

#### **CERTIFICATE OF ANALYSIS FOR**

# Gold Ore (Fosterville Gold Mine, Victoria, Australia) CERTIFIED REFERENCE MATERIAL OREAS 239

Table 1. Certified Values and Performance Gates for OREAS 239.

Constituent 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
Pb Fire Assay	1										
Au, ppm	3.55	0.086	3.38	3.72	3.29	3.81	2.42%	4.84%	7.26%	3.37	3.72
Aqua Regia D	Aqua Regia Digestion (sample weights 10-50g)										
Au, ppm	3.41	0.162	3.09	3.74	2.93	3.90	4.73%	9.46%	14.20%	3.24	3.58
Cyanide Leac	Cyanide Leach										
Au, ppm	3.09	0.138	2.82	3.37	2.68	3.51	4.46%	8.92%	13.38%	2.94	3.25
Aqua Regia D	Aqua Regia Digestion										
Ag, ppm	0.244	0.013	0.219	0.270	0.206	0.282	5.18%	10.36%	15.54%	0.232	0.257
Al, wt.%	2.39	0.153	2.08	2.69	1.93	2.84	6.42%	12.84%	19.25%	2.27	2.50
As, ppm	704	33	638	771	605	804	4.72%	9.44%	14.16%	669	739
Ba, ppm	96	13	70	122	57	135	13.54%	27.08%	40.61%	91	101
Be, ppm	1.15	0.064	1.02	1.28	0.96	1.34	5.56%	11.13%	16.69%	1.09	1.21
Bi, ppm	0.38	0.027	0.32	0.43	0.29	0.46	7.29%	14.59%	21.88%	0.36	0.39
Ca, wt.%	0.245	0.018	0.208	0.282	0.189	0.300	7.54%	15.09%	22.63%	0.232	0.257
Cd, ppm	0.039	0.010	0.019	0.058	0.009	0.068	25.37%	50.73%	76.10%	0.037	0.041
Ce, ppm	54	9	37	72	28	81	16.22%	32.43%	48.65%	51	57
Co, ppm	12.1	0.66	10.8	13.5	10.2	14.1	5.47%	10.93%	16.40%	11.5	12.8
Cr, ppm	95	3.3	88	101	85	105	3.46%	6.92%	10.38%	90	100

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

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.



Document: COA-1370-OREAS239-R0 30-May-2019 (Template:BUP-70-10-01 Rev:2.0)

#### Table 1 continued.

Table I continued.											
Constituent	Certified		Absolute	Standard	Deviations	3	Relative Standard Deviations			5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Aqua Regia D	igestion co	ntinued									
Cs, ppm	7.00	0.556	5.89	8.11	5.33	8.67	7.94%	15.88%	23.82%	6.65	7.35
Cu, ppm	26.8	1.99	22.8	30.8	20.8	32.8	7.42%	14.84%	22.26%	25.5	28.1
Dy, ppm	2.18	0.170	1.84	2.52	1.67	2.69	7.82%	15.64%	23.46%	2.07	2.29
Er, ppm	0.97	0.057	0.86	1.09	0.80	1.14	5.81%	11.63%	17.44%	0.92	1.02
Eu, ppm	0.76	0.18	0.40	1.12	0.22	1.30	23.56%	47.12%	70.68%	0.72	0.80
Fe, wt.%	3.36	0.174	3.01	3.70	2.84	3.88	5.17%	10.34%	15.52%	3.19	3.52
Ga, ppm	7.48	0.673	6.13	8.82	5.46	9.49	8.99%	17.99%	26.98%	7.10	7.85
Gd, ppm	3.71	0.38	2.94	4.47	2.56	4.85	10.27%	20.53%	30.80%	3.52	3.89
Ge, ppm	0.090	0.012	0.067	0.113	0.055	0.125	12.86%	25.71%	38.57%	0.086	0.095
Hf, ppm	0.75	0.055	0.64	0.86	0.58	0.91	7.29%	14.58%	21.87%	0.71	0.79
Ho, ppm	0.35	0.05	0.24	0.46	0.19	0.51	15.50%	30.99%	46.49%	0.33	0.37
In, ppm	0.023	0.003	0.017	0.029	0.014	0.032	13.38%	26.75%	40.13%	0.022	0.024
K, wt.%	0.931	0.044	0.842	1.020	0.798	1.064	4.77%	9.54%	14.30%	0.884	0.978
La, ppm	27.7	4.2	19.2	36.1	15.0	40.4	15.32%	30.64%	45.96%	26.3	29.1
Li, ppm	31.5	3.02	25.4	37.5	22.4	40.6	9.60%	19.21%	28.81%	29.9	33.1
Lu, ppm	0.12	0.01	0.09	0.14	0.08	0.15	10.75%	21.50%	32.25%	0.11	0.12
Mg, wt.%	1.23	0.044	1.14	1.32	1.10	1.36	3.60%	7.21%	10.81%	1.17	1.29
Mn, wt.%	0.022	0.001	0.020	0.024	0.019	0.025	4.28%	8.56%	12.85%	0.021	0.023
Mo, ppm	0.59	0.055	0.48	0.70	0.43	0.76	9.25%	18.50%	27.76%	0.56	0.62
Na, wt.%	0.070	0.011	0.048	0.092	0.037	0.103	15.64%	31.28%	46.92%	0.066	0.073
Ni, ppm	55	2.7	50	61	47	63	4.96%	9.93%	14.89%	52	58
P, wt.%	0.048	0.003	0.042	0.054	0.040	0.057	5.97%	11.94%	17.91%	0.046	0.051
Pb, ppm	10.9	0.86	9.1	12.6	8.3	13.4	7.91%	15.83%	23.74%	10.3	11.4
Pr, ppm	6.96	1.05	4.86	9.07	3.81	10.12	15.11%	30.22%	45.33%	6.61	7.31
Rb, ppm	88	6.4	75	101	69	107	7.30%	14.60%	21.89%	84	93
S, wt.%	0.155	0.011	0.133	0.177	0.122	0.188	7.05%	14.10%	21.16%	0.147	0.163
Sb, ppm	518	100	318	719	217	819	19.36%	38.71%	58.07%	492	544
Sc, ppm	5.67	0.379	4.91	6.43	4.53	6.81	6.69%	13.37%	20.06%	5.39	5.95
Sm, ppm	4.65	0.68	3.29	6.01	2.61	6.69	14.61%	29.22%	43.84%	4.42	4.89
Sn, ppm	1.29	0.089	1.11	1.47	1.02	1.56	6.92%	13.83%	20.75%	1.23	1.35
Sr, ppm	20.2	1.75	16.7	23.7	14.9	25.4	8.68%	17.37%	26.05%	19.2	21.2
Tb, ppm	0.40	0.08	0.24	0.55	0.16	0.63	19.73%	39.46%	59.19%	0.38	0.41
Th, ppm	12.6	0.97	10.7	14.5	9.7	15.5	7.69%	15.38%	23.07%	12.0	13.2
Ti, wt.%	0.128	0.015	0.098	0.159	0.082	0.174	11.95%	23.89%	35.84%	0.122	0.135
TI, ppm	0.57	0.037	0.50	0.65	0.46	0.68	6.39%	12.77%	19.16%	0.54	0.60
Tm, ppm	0.12	0.02	0.08	0.16	0.06	0.18	15.96%	31.92%	47.88%	0.12	0.13
U, ppm	1.51	0.149	1.22	1.81	1.07	1.96	9.82%	19.63%	29.45%	1.44	1.59
V, ppm	63	1.9	59	67	57	69	3.00%	6.00%	9.01%	60	66
W, ppm	0.54	0.033	0.47	0.61	0.44	0.64	6.09%	12.18%	18.27%	0.51	0.57
Y, ppm	8.95	0.667	7.62	10.29	6.95	10.95	7.45%	14.89%	22.34%	8.51	9.40
Yb, ppm	0.80	0.09	0.61	0.99	0.52	1.09	11.81%	23.62%	35.43%	0.76	0.84
Zn, ppm	75	4.5	66	84	62	89	5.95%	11.90%	17.85%	72	79
Zr, ppm	27.0	5.2	16.6	37.4	11.4	42.7	19.30%	38.60%	57.90%	25.7	28.4
SI unit equival				l						l	

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

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#### 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. OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself. In evaluating laboratory performance with this CRM, the section headed 'Intended Use' (page 19) should be read carefully.

## **SOURCE MATERIAL**

OREAS 239 was prepared from a blend of high grade gold-bearing ore and barren metasediments. The ore was sourced from the Fosterville Mine, located 20km from the city of Bendigo in the state of Victoria, Australia. The deposit is hosted by a metamorphosed interbedded turbidite sequence of sandstones, siltstones and shales. Primary gold mineralization occurs as disseminated arsenopyrite and pyrite in a quartz–carbonate veinlet stockwork. Primary gold also occurs as visible gold where it variably overprints sulphide mineralization and is found as disseminated fine specks (>1 mm) of gold within host quartz veins. The visible gold is spatially associated with antimony mineralization, in the form of stibnite that occurs with quartz and varies from replacement and infill of earlier quartz-carbonate stockwork veins, to massive stibnite-only veins of up to 0.5m in width (Hitchman, Philips, & Greenberger, 2017). The approximate major and trace element composition of OREAS 239 is provided in Table 2 below.

# **PERFORMANCE GATES**

Table 1 above shows intervals 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 (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit www.westgard.com/mltirule.htm). A second method utilises a 5% window calculated directly from the certified value.

Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL)  $\pm$  10%.

i.e. Certified Value ± 10% ± 2DL (adapted from Govett, 1983).



Table 2. Indicative Values for OREAS 239.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
		value	Constituent	Offic	Value	Constituent	Offic	value
Pb Fire Assa		_	T			T		
Pd	ppb	< 5	Pt	ppb	3.75			
X-ray Photor			T			1		
Au	ppm	3.68						
Aqua Regia	Digestior		T					
В	ppm	< 10	Pd	ppb	< 10	Si	wt.%	0.041
Hg	ppm	0.015	Pt	ppb	< 5	Та	ppm	< 0.01
Nb	ppm	0.31	Re	ppm	< 0.001	Te	ppm	0.026
Nd	ppm	25.5	Se	ppm	0.24			
Borate Fusion	on XRF		l			1		
$Al_2O_3$	wt.%	14.73	MgO	wt.%	2.64	SiO <sub>2</sub>	wt.%	67.82
CaO	wt.%	1.08	MnO	wt.%	0.050	SO <sub>3</sub>	wt.%	0.379
$Fe_2O_3$	wt.%	5.74	Na₂O	wt.%	0.935	TiO <sub>2</sub>	wt.%	0.820
K₂O	wt.%	3.37	P <sub>2</sub> O <sub>5</sub>	wt.%	0.134			
4-Acid Diges	tion							
As	ppm	700	Cr	ppm	228	Sb	ppm	605
В	ppm	80	Cu	ppm	121	Se	ppm	< 40
Ва	ppm	720	Ge	ppm	< 10	Sn	ppm	51
Be	ppm	< 1	Li	ppm	49.3	Sr	ppm	114
Bi	ppm	34.8	Мо	ppm	6.67	Y	ppm	27.5
Cd	ppm	8.83	Nb	ppm	5.83	Zn	ppm	121
Co	ppm	36.8	Ni	ppm	92			
Thermograv	imetry							
LOI <sup>1000</sup>	wt.%	2.61						
Infrared Con	nbustion							
С	wt.%	0.100	S	wt.%	0.150			
Laser Ablatic	on ICP-M	S	1					
Ag	ppm	0.250	Hf	ppm	6.10	Sm	ppm	6.97
As	ppm	734	Но	ppm	1.13	Sn	ppm	4.10
Ва	ppm	696	In	ppm	0.050	Sr	ppm	98
Be	ppm	2.00	La	ppm	40.4	Та	ppm	1.17
Bi	ppm	0.41	Lu	ppm	0.46	Tb	ppm	0.95
Cd	ppm	0.10	Mn	wt.%	0.035	Te	ppm	< 0.2
Ce	ppm	79	Мо	ppm	0.70	Th	ppm	15.3
Co	ppm	14.3	Nb	ppm	15.4	Ti	wt.%	0.490
Cr	ppm	145	Nd	ppm	35.2	TI	ppm	1.20
Cs	ppm	9.40	Ni	ppm	64	Tm	ppm	0.51
Cu	ppm	31.0	Pb	ppm	21.5	U	ppm	2.97
Dy	ppm	<i>5.4</i> 9	Pr	ppm	9.26	V	ppm	106
Ér	ppm	3.34	Rb	ppm	163	W	ppm	3.25
Eu	ppm	1.34	Re	ppm	< 0.01	Y	ppm	30.9
Ga	ppm	19.5	Sb	ppm	724	Yb	ppm	3.21
Gd	ppm	6.01	Sc	ppm	14.7	Zn	ppm	98
Ge	ppm	1.80	Se	ppm	< 5	Zr	ppm	215
			) ≡ ma/ka ≡ µa/a					

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

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.



#### COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 239 was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of the barren metasediments to 98% 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.

#### PHYSICAL PROPERTIES

OREAS 239 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

Table 3. Physical properties of OREAS 239.

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
829.6	0.61	N8	Very Light Gray

<sup>&</sup>lt;sup>‡</sup>The Munsell Rock Color Chart helps geologists and archeologists communicate with colour more effectively by cross-referencing ISCC-NBS colour names with unique Munsell alpha-numeric colour notations for rock colour samples.

## ANALYTICAL PROGRAM

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

- Gold via 15-50g fire assay with AAS finish (27 laboratories) and ICP-OES (7 laboratories) finish;
- Gold via 15-50g aqua regia digestion with ICP-MS finish (13 laboratories), AAS (11 laboratories) and ICP-OES (1 laboratory) finish;
- Gold by cyanide leach A variety of cyanide leach methods were undertaken by the participating laboratories including the use of LeachWELL tablets, alkaline added sodium cyanide solution as well as sodium cyanide liquor with LeachWELL powder. The sample weights included: 10g (1 laboratory by ICP-OES finish), 30g (9 laboratories by AAS finish and 1 laboratory by ICP-MS finish), 50g (1 laboratory by AAS finish and 1 laboratory by ICP-MS finish);
- Aqua regia digestion for full elemental suite ICP-OES and ICP-MS finish (up to 23 laboratories depending on the element);

To confirm homogeneity, gold by instrumental neutron activation analysis (INAA) was undertaken on 20 x 85mg subsamples (ANSTO, Lucas Heights).

Gold was also determined by Chrysos Corporation's new Photon Assay technique at Minanalytical Services, Perth. This value is included in Table 2 as an indicative value since

it is reported by one laboratory only. Table 2 also includes major and trace element characterisation by BV Perth Geoanalytical laboratory using the following methodologies:

- Major oxides by lithium borate fusion with X-ray fluorescence;
- LOI at 1000°C by thermogravimetric analyser;
- Infrared combustion furnace for C and S;
- Trace element characterisation by laser ablation with ICP-MS finish.

Table 2 also presents twenty additional indicative values (As to Zn) by 4-acid digestion with ICP-OES/MS finish performed by one of the participating laboratories.

For the round robin program twenty 1.5kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 100-350g pulp samples (the weight provided depended on whether the laboratory was anticipated to undertake assays by gold cyanide leach) were submitted to each laboratory for analysis. The samples received by each laboratory were obtained by taking two samples from each of three separate 1.5kg test units. This format enabled a 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 provides performance gate intervals for the 57 certified values based on their pooled 1SD's and Table 2 shows 156 indicative values for major and trace element composition. Table 3 provides some indicative physical properties and Table 4 presents 95% confidence and tolerance limits. Gold homogeneity has been evaluated and confirmed by instrumental neutron activation analysis (INAA) on twenty ~85mg sample portions (Table 5) and by a nested ANOVA program for both fire assay and aqua regia digestion (see 'nested ANOVA' section).

Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM³) are presented in the detailed certification data for this CRM (OREAS 239 DataPack-1.0.190527\_153119.xlsx).

Results are also presented in scatter plots for gold by fire assay, aqua regia digestion and cyanide leach (Figures 1 to 3, respectively) together with ±3SD (magenta) and ±5% (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

#### STATISTICAL ANALYSIS

**Standard Deviation** intervals (see Table 1) 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. They 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 Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

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 (see Intended Use section for more detail).

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 all 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.

Certified Values, Standard Deviations, Confidence Limits and Tolerance Limits (Table 4) 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 5) 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 239 (see 'Homogeneity Evaluation' section below).

**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  $TiO_2$ ), laser ablation with ICP-MS (Ag to Zr), LOI at 1000°C and C by infrared combustion furnace 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.

Table 4. 95% Confidence & Tolerance Limits for OREAS 239.

	5% Confidence 8  Certified	95% Confid					
Constituent	Value	Low	High	Low	High		
Pb Fire Assay			J		J		
Au, Gold (ppm)	3.55	3.52	3.58	3.53	3.56		
Aqua Regia Digestion (sample	weights 10-50g)						
Au, Gold (ppm)	3.41	3.35	3.48	3.40	3.43		
Cyanide Leach							
Au, Gold (ppm)	3.09	3.03	3.16	3.08	3.11		
Aqua Regia Digestion							
Ag, Silver (ppm)	0.244	0.239	0.249	0.229	0.260		
Al, Aluminium (wt.%)	2.39	2.31	2.46	2.33	2.44		
As, Arsenic (ppm)	704	689	720	692	716		
Ba, Barium (ppm)	96	90	102	94	98		
Be, Beryllium (ppm)	1.15	1.10	1.20	1.08	1.22		
Bi, Bismuth (ppm)	0.38	0.36	0.39	0.36	0.40		
Ca, Calcium (wt.%)	0.245	0.236	0.253	0.237	0.252		
Cd, Cadmium (ppm)	0.039	0.034	0.044	IND	IND		
Ce, Cerium (ppm)	54	49	59	53	56		
Co, Cobalt (ppm)	12.1	11.8	12.5	11.8	12.5		
Cr, Chromium (ppm)	95	93	96	93	97		
Cs, Caesium (ppm)	7.00	6.69	7.31	6.79	7.21		
Cu, Copper (ppm)	26.8	25.9	27.7	25.8	27.8		
Dy, Dysprosium (ppm)	2.18	1.96	2.40	2.06	2.29		
Er, Erbium (ppm)	0.97	0.92	1.03	0.92	1.02		
Eu, Europium (ppm)	0.76	0.52	1.00	0.70	0.82		
Fe, Iron (wt.%)	3.36	3.28	3.43	3.27	3.44		
Ga, Gallium (ppm)	7.48	7.13	7.82	7.18	7.77		
Gd, Gadolinium (ppm)	3.71	3.29	4.12	3.53	3.88		
Ge, Germanium (ppm)	0.090	0.079	0.101	IND	IND		
Hf, Hafnium (ppm)	0.75	0.71	0.78	0.72	0.78		
Ho, Holmium (ppm)	0.35	0.29	0.41	0.33	0.37		
In, Indium (ppm)	0.023	0.022	0.024	0.019	0.026		
K, Potassium (wt.%)	0.931	0.909	0.953	0.912	0.950		
La, Lanthanum (ppm)	27.7	25.6	29.7	26.7	28.6		
Li, Lithium (ppm)	31.5	29.9	33.1	30.8	32.1		
Lu, Lutetium (ppm)	0.12	0.10	0.13	0.10	0.13		
Mg, Magnesium (wt.%)	1.23	1.21	1.25	1.20	1.26		
Mn, Manganese (wt.%)	0.022	0.022	0.022	0.022	0.022		
Mo, Molybdenum (ppm)	0.59	0.56	0.62	0.54	0.65		
Na, Sodium (wt.%)	0.070	0.065	0.075	0.067	0.072		

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

Note: intervals may appear asymmetric due to rounding.



<sup>\*</sup>Gold Tolerance Limits for typical 30g fire assay, 25g aqua regia digestion and 30g cyanide leach methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Table 4 continued.

	Certified	95% Confid	ence Limits	95% Tolera	rance Limits	
Constituent	Value	Low	High	Low	High	
Aqua Regia Digestion continu	ed					
Ni, Nickel (ppm)	55	54	56	53	57	
P, Phosphorus (wt.%)	0.048	0.047	0.049	0.047	0.050	
Pb, Lead (ppm)	10.9	10.4	11.3	10.5	11.3	
Pr, Praseodymium (ppm)	6.96	5.79	8.13	6.59	7.34	
Rb, Rubidium (ppm)	88	85	92	86	90	
S, Sulphur (wt.%)	0.155	0.150	0.160	0.151	0.159	
Sb, Antimony (ppm)	518	471	565	503	533	
Sc, Scandium (ppm)	5.67	5.49	5.85	5.42	5.92	
Sm, Samarium (ppm)	4.65	3.74	5.56	4.35	4.95	
Sn, Tin (ppm)	1.29	1.24	1.34	1.25	1.33	
Sr, Strontium (ppm)	20.2	19.3	21.1	19.4	21.0	
Tb, Terbium (ppm)	0.40	0.32	0.47	0.37	0.42	
Th, Thorium (ppm)	12.6	12.1	13.1	12.2	13.1	
Ti, Titanium (wt.%)	0.128	0.121	0.135	0.124	0.132	
TI, Thallium (ppm)	0.57	0.55	0.59	0.55	0.60	
Tm, Thulium (ppm)	0.12	0.10	0.15	IND	IND	
U, Uranium (ppm)	1.51	1.44	1.59	1.44	1.59	
V, Vanadium (ppm)	63	62	64	61	65	
W, Tungsten (ppm)	0.54	0.52	0.56	0.50	0.58	
Y, Yttrium (ppm)	8.95	8.62	9.29	8.65	9.26	
Yb, Ytterbium (ppm)	0.80	0.72	0.89	0.78	0.83	
Zn, Zinc (ppm)	75	73	77	73	77	
Zr, Zirconium (ppm)	27.0	24.5	29.6	25.9	28.1	

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

Note: intervals may appear asymmetric due to rounding.

### **Homogeneity Evaluation**

For analytes other than gold the tolerance limits (ISO 16269:2014) shown in Table 4 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 antimony by aqua-regia digestion, where 99% of the time  $(1-\alpha=0.99)$  at least 95% of subsamples (p=0.95) will have concentrations lying between 503 and 533 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35). **Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.** 

Table 5 shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 239. An equivalent scaled version of the results is also provided to demonstrate the level of repeatability that would be achieved if 30g fire assay determinations were undertaken without the normal measurement error associated with this methodology. The homogeneity of gold 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 (i.e. sampling error) and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.135% was calculated for a 30g fire assay or aqua regia sample (2.54% at 85mg weights) and confirms the high level of gold homogeneity in OREAS 239.

Table 5. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples and showing the equivalent results scaled to a 30g sample mass typical of fire assay determination.

Replicate	Au	Au
No	85mg actual	30g equivalent*
1	3.44	3.54
2	3.45	3.54
3	3.56	3.54
4	3.50	3.54
5	3.49	3.54
6	3.53	3.54
7	3.59	3.54
8	3.64	3.55
9	3.71	3.55
10	3.59	3.54
11	3.44	3.54
12	3.50	3.54
13	3.49	3.54
14	3.49	3.54
15	3.52	3.54
16	3.52	3.54
17	3.47	3.54
18	3.77	3.55
19	3.63	3.55
20	3.52	3.54
Mean	3.54	3.54
Median	3.52	3.54
Std Dev.	0.090	0.005
Rel.Std.Dev.	2.54%	0.135%

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

The homogeneity of OREAS 239 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty-five 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 239. The test was performed using the following parameters:

- Gold fire assay 204 samples (34 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion 150 samples (25 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 datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of *p*-values. This process derived *p*-values of 0.9871 for Au by fire assay and 0.8959 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. Please note that only results for constituents present in concentrations well above the detection levels (i.e. >20 x Lower Limit of Detection) for the various methods undertaken were considered for the objective of evaluating homogeneity.

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 239 and whether the variance between two subsamples from the same unit is statistically distinguishable from the variance of two subsamples taken from any two separate units. A reference material therefore can possess poor absolute homogeneity yet still pass a relative homogeneity (ANOVA) 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 239 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

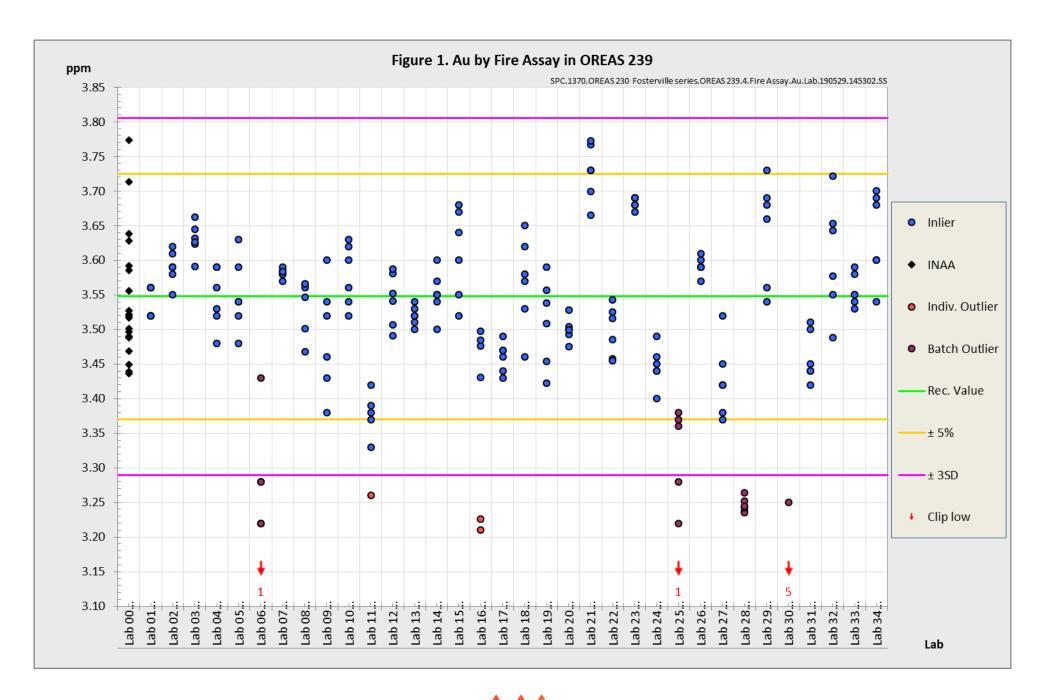
#### PARTICIPATING LABORATORIES

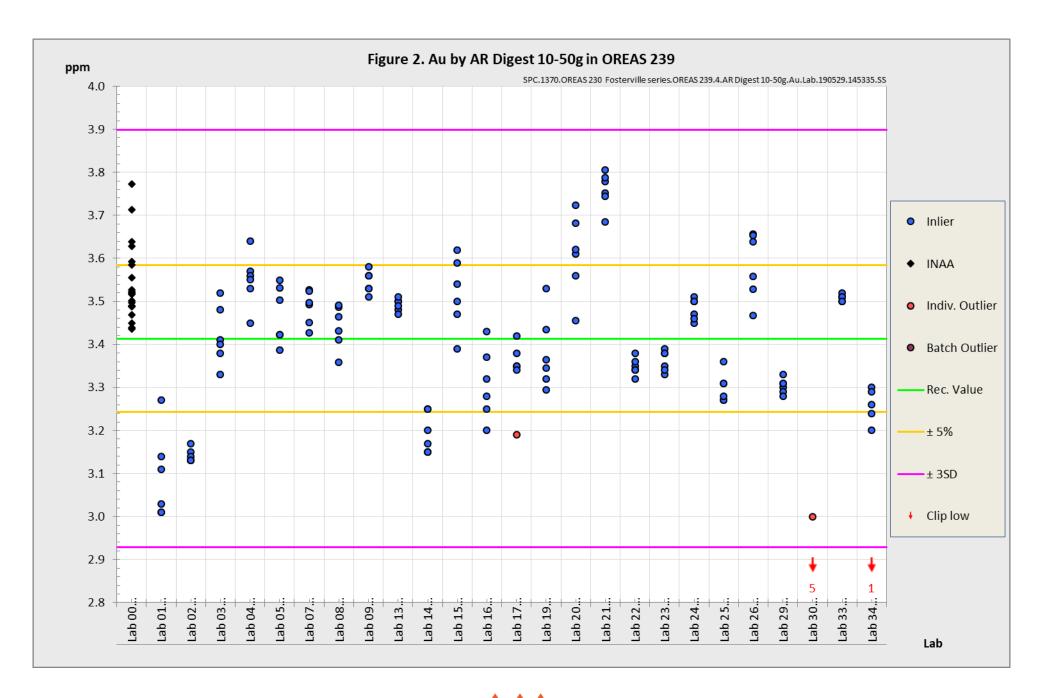
- 1. Actlabs, Ancaster, Ontario, Canada
- 2. Alex Stewart International, Mendoza, Argentina
- 3. ALS, Lima, Peru
- 4. ALS, Loughrea, Galway, Ireland
- 5. ALS, Perth, WA, Australia
- 6. ALS, Reno, Nevada, USA
- 7. ALS, Vancouver, BC, Canada
- 8. American Assay Laboratories, Sparks, Nevada, USA
- 9. ANSTO, Lucas Heights, NSW, Australia
- 10. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
- 11. Aurum Laboratories Pty Ltd., Perth, Western Australia, Australia
- 12. BGRIMM MTC Technology Co., Ltd., Beijing, Daxing District, China
- 13. Bureau Veritas, Abidjan, Cote D'ivoire
- 14. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada

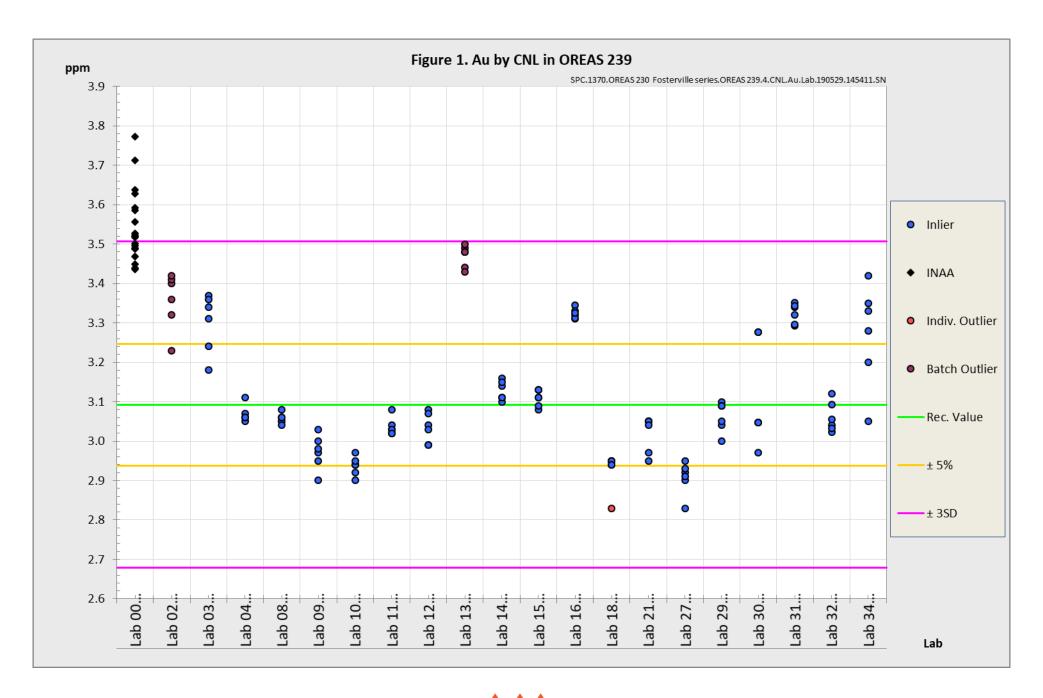
- 15. Bureau Veritas Geoanalytical, Perth, WA, Australia
- 16. Gekko Assay Labs, Ballarat, VIC, Australia
- 17. Inspectorate (BV), Lima, Peru
- 18. Inspectorate America Corporation (BV), Sparks, Nevada, USA
- 19. Intertek Genalysis, Perth, WA, Australia
- 20. Intertek Tarkwa, Tarkwa, Ghana
- 21. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 22. MinAnalytical Services, Perth, WA, Australia
- 23. Nagrom, Perth, WA, Australia
- 24. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 25. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 26. Quality Laboratory Services, Dar es Salaam, Chunya, United Republic Of Tanzania
- 27. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
- 28. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
- 29. SGS, Randfontein, Gauteng, South Africa
- 30. SGS Australia Mineral Services, Kalgoorlie, WA, Australia
- 31. SGS Australia Mineral Services, Perth, WA, Australia
- 32. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 33. SGS Tarkwa, Tarkwa, Western Region, Ghana
- 34. Shandong Humon Smelting Co., Ltd., Yantai City, Shandong Province, China
- 35. Shiva Analyticals Ltd, Bangalore North, Karnataka, India

Please note: To preserve anonymity, the above numbered alphabetical list of participating laboratories <u>does not</u> correspond with the Lab ID numbering on the scatter plots below.









# PREPARER AND SUPPLIER

Certified reference material OREAS 239 was prepared, certified and supplied by:



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#### **METROLOGICAL TRACEABILITY**

The analytical samples were selected in a manner representative of the entire batch of the prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment, as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, "Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results (see ISO Guide 35:2006, Clause 10)."

#### COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their field samples.

#### INTENDED USE

OREAS 239 is intended to cover all activities needed to produce a measurement result. This includes extraction, possible separation steps and the actual measurement process (the signal producing step). OREAS 239 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 239 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.

#### For use with the agua regia digestion method

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 from 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.

# QC monitoring using multiples of the Standard Deviation (SD)

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-laboratory 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.

#### STABILITY AND STORAGE INSTRUCTIONS

OREAS 239 has been prepared from primary gold ore blended with barren metasediments. It is low in reactive sulphide (~0.16 wt.% S) and in its unopened state and

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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 239 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 including the use of safety glasses and dust masks are advised.

### LEGAL NOTICE

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

## **DOCUMENT HISTORY**

Revision No.	Date	Changes applied
0	30 <sup>th</sup> May 2019	First publication.

#### QMS CERTIFICATION

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





#### **CERTIFYING OFFICER**

30<sup>th</sup> May, 2019

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



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