

# CERTIFICATE OF ANALYSIS FOR

# Gold Ore (Frogs Leg Gold Mine, Western Australia)

# CERTIFIED REFERENCE MATERIAL OREAS 231

Ormatiturent	Certified		Absolute Standard Deviations Relative Standard Deviations 59						5% w	indow	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Pb Fire Assay	,										
Au, ppm	0.542	0.015	0.512	0.573	0.497	0.588	2.78%	5.55%	8.33%	0.515	0.570
Aqua Regia D	igestion (sa	mple wei	ghts 10-5	0g)							
Au, ppm	0.521	0.018	0.485	0.556	0.468	0.573	3.38%	6.76%	10.15%	0.495	0.547
Cyanide Leac	h										
Au, ppm	0.516	0.027	0.461	0.570	0.434	0.597	5.25%	10.49%	15.74%	0.490	0.541
4-Acid Digest	ion										
Ag, ppm	0.177	0.024	0.130	0.224	0.106	0.247	13.28%	26.57%	39.85%	0.168	0.186
AI, wt.%	7.14	0.256	6.62	7.65	6.37	7.90	3.59%	7.19%	10.78%	6.78	7.49
As, ppm	27.2	2.08	23.1	31.4	21.0	33.4	7.64%	15.28%	22.92%	25.8	28.6
Ba, ppm	112	4	104	120	100	124	3.57%	7.13%	10.70%	106	118
Be, ppm	0.36	0.05	0.27	0.46	0.22	0.50	12.83%	25.66%	38.49%	0.34	0.38
Bi, ppm	0.037	0.010	0.018	0.056	0.008	0.066	26.13%	52.27%	78.40%	0.035	0.039
Ca, wt.%	7.47	0.256	6.96	7.98	6.70	8.24	3.43%	6.86%	10.29%	7.10	7.84
Cd, ppm	0.35	0.05	0.26	0.44	0.22	0.49	12.98%	25.95%	38.93%	0.33	0.37
Ce, ppm	11.0	0.72	9.5	12.4	8.8	13.1	6.61%	13.21%	19.82%	10.4	11.5
Co, ppm	44.7	2.92	38.8	50.5	35.9	53.4	6.54%	13.08%	19.62%	42.4	46.9

### Table 1. Certified Values and Performance Gates for OREAS 231.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/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.



	Certified		Absolute	Standard	Deviations	6	Relative Standard Deviations 5% win			indow	
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion continued						I					
Cr, ppm	133	18	97	169	79	186	13.41%	26.81%	40.22%	126	140
Cs, ppm	0.56	0.019	0.52	0.60	0.50	0.62	3.45%	6.91%	10.36%	0.53	0.59
Cu, ppm	161	9	143	178	134	187	5.47%	10.94%	16.41%	153	169
Dy, ppm	3.71	0.138	3.44	3.99	3.30	4.13	3.72%	7.44%	11.15%	3.53	3.90
Er, ppm	2.35	0.129	2.09	2.61	1.96	2.74	5.49%	10.98%	16.47%	2.23	2.47
Eu, ppm	0.93	0.062	0.81	1.06	0.75	1.12	6.64%	13.28%	19.92%	0.89	0.98
Fe, wt.%	8.05	0.281	7.49	8.61	7.21	8.90	3.48%	6.97%	10.45%	7.65	8.46
Ga, ppm	15.9	0.55	14.8	17.0	14.3	17.6	3.44%	6.89%	10.33%	15.1	16.7
Gd, ppm	3.23	0.188	2.86	3.61	2.67	3.80	5.82%	11.64%	17.45%	3.07	3.39
Hf, ppm	1.63	0.125	1.38	1.88	1.25	2.00	7.71%	15.41%	23.12%	1.55	1.71
Ho, ppm	0.82	0.045	0.73	0.91	0.69	0.96	5.49%	10.97%	16.46%	0.78	0.86
In, ppm	0.070	0.006	0.058	0.083	0.051	0.089	8.91%	17.82%	26.72%	0.067	0.074
K, wt.%	0.288	0.012	0.264	0.313	0.251	0.325	4.26%	8.53%	12.79%	0.274	0.303
La, ppm	4.47	0.257	3.95	4.98	3.70	5.24	5.75%	11.51%	17.26%	4.24	4.69
Li, ppm	11.1	0.94	9.2	13.0	8.3	13.9	8.44%	16.89%	25.33%	10.5	11.6
Lu, ppm	0.33	0.017	0.29	0.36	0.27	0.38	5.27%	10.53%	15.80%	0.31	0.34
Mg, wt.%	3.90	0.149	3.61	4.20	3.46	4.35	3.81%	7.62%	11.44%	3.71	4.10
Mn, wt.%	0.139	0.007	0.125	0.152	0.119	0.159	4.84%	9.67%	14.51%	0.132	0.146
Mo, ppm	0.86	0.09	0.69	1.04	0.60	1.12	10.09%	20.17%	30.26%	0.82	0.91
Na, wt.%	1.75	0.052	1.64	1.85	1.59	1.91	2.98%	5.96%	8.94%	1.66	1.84
Nb, ppm	3.50	0.282	2.93	4.06	2.65	4.34	8.07%	16.13%	24.20%	3.32	3.67
Nd, ppm	7.98	0.303	7.37	8.59	7.07	8.89	3.80%	7.60%	11.40%	7.58	8.38
Ni, ppm	88	5.4	78	99	72	104	6.09%	12.17%	18.26%	84	93
P, wt.%	0.039	0.002	0.035	0.043	0.033	0.045	4.92%	9.85%	14.77%	0.037	0.041
Pb, ppm	12.8	1.20	10.4	15.2	9.2	16.4	9.35%	18.70%	28.05%	12.1	13.4
Pr, ppm	1.66	0.077	1.50	1.81	1.43	1.89	4.64%	9.28%	13.92%	1.57	1.74
Rb, ppm	8.08	0.437	7.20	8.95	6.76	9.39	5.42%	10.83%	16.25%	7.67	8.48
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.220	0.015	0.189	0.251	0.174	0.266	7.02%	14.04%	21.07%	0.209	0.231
Sb, ppm	0.71	0.061	0.58	0.83	0.52	0.89	8.68%	17.36%	26.04%	0.67	0.74
Sc, ppm	42.2	2.36	37.5	46.9	35.1	49.3	5.60%	11.19%	16.79%	40.1	44.3
Sm, ppm	2.49	0.143	2.20	2.78	2.06	2.92	5.74%	11.49%	17.23%	2.37	2.62
Sn, ppm	0.90	0.13	0.65	1.15	0.52	1.28	14.07%	28.13%	42.20%	0.86	0.95
Sr, ppm	179	7	165	194	158	201	4.03%	8.07%	12.10%	171	188
Ta, ppm	0.22	0.015	0.19	0.25	0.18	0.26	6.64%	13.27%	19.91%	0.21	0.23
Tb, ppm	0.58	0.038	0.50	0.65	0.46	0.69	6.54%	13.09%	19.63%	0.55	0.61
Te, ppm	0.068	0.010	0.048	0.088	0.039	0.098	14.48%	28.96%	43.44%	0.065	0.072
Th, ppm	0.72	0.08	0.56	0.88	0.47	0.96	11.30%	22.60%	33.90%	0.68	0.75
Ti, wt.%	0.617	0.027	0.564	0.670	0.537	0.697	4.31%	8.61%	12.92%	0.586	0.648
TI, ppm	0.12	0.008	0.10	0.13	0.09	0.14	6.59%	13.19%	19.78%	0.11	0.12

#### Table 1 continued.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/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.



Constituent	Certified		Absolute Standard Deviations Relative Standard Deviations					5% w	indow		
Constituent	Value 1	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digest	ion continue	əd									
Tm, ppm	0.33	0.028	0.27	0.39	0.25	0.42	8.51%	17.03%	25.54%	0.31	0.35
U, ppm	0.20	0.006	0.19	0.21	0.18	0.22	2.96%	5.93%	8.89%	0.19	0.21
V, ppm	285	17	252	318	236	335	5.79%	11.58%	17.38%	271	299
W, ppm	15.2	1.07	13.0	17.3	12.0	18.4	7.04%	14.08%	21.11%	14.4	15.9
Y, ppm	20.7	0.89	18.9	22.5	18.0	23.4	4.30%	8.60%	12.90%	19.7	21.8
Yb, ppm	2.20	0.124	1.96	2.45	1.83	2.58	5.61%	11.22%	16.83%	2.09	2.32
Zn, ppm	113	5	102	123	96	129	4.77%	9.54%	14.30%	107	118
Zr, ppm	50	4.3	42	59	38	63	8.45%	16.90%	25.36%	48	53

#### Table 1 continued.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/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.



# TABLE OF CONTENTS

INTRODUCTION	5
SOURCE MATERIAL	5
PERFORMANCE GATES	5
COMMINUTION AND HOMOGENISATION PROCEDURES	7
PHYSICAL PROPERTIES	7
ANALYTICAL PROGRAM	7
STATISTICAL ANALYSIS	8
Homogeneity Evaluation	11
PARTICIPATING LABORATORIES	14
PREPARER AND SUPPLIER	18
METROLOGICAL TRACEABILITY	18
COMMUTABILITY	18
INTENDED USE	19
STABILITY AND STORAGE INSTRUCTIONS	19
INSTRUCTIONS FOR CORRECT USE	19
HANDLING INSTRUCTIONS	19
LEGAL NOTICE	20
DOCUMENT HISTORY	20
QMS CERTIFICATION	20
CERTIFYING OFFICER	20
REFERENCES	20

# LIST OF TABLES

Table 1. Certified Values and Performance Gates for OREAS 231	1
Table 2. Indicative Values for OREAS 231	6
Table 3. Physical properties of OREAS 231	7
Table 4. 95% Confidence & Tolerance Limits for OREAS 231	.10
Table 5. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples	.12

## LIST OF FIGURES

Figure 1. Au by Fire Assay in OREAS 231	15
Figure 2. Au by aqua regia digestion in OREAS 231	16
Figure 3. Au by cyanide leach in OREAS 231	17



# 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' should be read carefully.

# SOURCE MATERIAL

Certified Reference Material (CRM) OREAS 231 was prepared from a blend of goldbearing ore and barren greenstone. The ore was sourced from the Frogs Leg Gold Mine located 19km west of Kalgoorlie in Western Australia. The ore lodes lie within sheared contacts between volcaniclastics and basalt and are hosted in laminated quartz veins, breccia and wall rock alteration. The Cambrian greenstone was sourced from a quarry 145km north of Melbourne, Australia. The approximate major and trace element composition of OREAS 231 is provided in Table 2.

# 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  $\pm$  10%  $\pm$  2DL (adapted from Govett, 1983).



Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assa	ay							
Pd	ppb	19.2	Pt	ppb	15.3			
X-ray Photo	n Assay							
Au	ppm	0.525						
Aqua Regia	Digestio	n						
Ag	ppm	< 0.2	In	ppm	0.029	Rh	ppm	< 0.003
As	ppm	27.8	lr	ppm	< 0.003	Sb	ppm	0.52
Bi	ppm	0.027	Мо	ppm	0.77	Se	ppm	0.30
Cd	ppm	0.31	Ni	ppm	57	Sn	ppm	0.54
Со	ppm	30.0	Pb	ppm	12.3	Те	ppm	0.060
Cu	ppm	150	Pd	ppb	18.4	TI	ppm	0.064
Hg	ppm	< 0.08	Pt	ppb	19.2	Zn	ppm	95
Borate Fusio	on XRF							
Al <sub>2</sub> O <sub>3</sub>	wt.%	13.68	K <sub>2</sub> O	wt.%	0.336	$P_2O_5$	wt.%	0.091
CaO	wt.%	10.85	MgO	wt.%	6.76	SiO <sub>2</sub>	wt.%	50.10
Cl	ppm	1165	MnO	wt.%	0.190	SO <sub>3</sub>	wt.%	0.532
Fe <sub>2</sub> O <sub>3</sub>	wt.%	11.85	Na <sub>2</sub> O	wt.%	2.34	TiO <sub>2</sub>	wt.%	1.07
4-Acid Diges	stion							
Ge	ppm	0.17	Hg	ppm	0.095	Se	ppm	1.05
Thermograv	rimetry							
LOI <sup>1000</sup>	wt.%	3.18						
Infrared Con	nbustion	I						
С	wt.%	0.170	S	wt.%	0.185			
Laser Ablati	on ICP-N	IS						
Ag	ppm	0.250	Hf	ppm	1.80	Sm	ppm	2.50
As	ppm	27.2	Но	ppm	0.82	Sn	ppm	0.80
Ва	ppm	113	In	ppm	0.050	Sr	ppm	174
Be	ppm	0.40	La	ppm	4.53	Та	ppm	0.21
Bi	ppm	0.020	Lu	ppm	0.33	Tb	ppm	0.61
Cd	ppm	0.40	Mn	wt.%	0.144	Те	ppm	< 0.2
Ce	ppm	10.8	Мо	ppm	0.70	Th	ppm	0.71
Co	ppm	45.1	Nb	ppm	3.45	Ti	wt.%	0.653
Cr	ppm	157	Nd	ppm	7.84	TI	ppm	< 0.2
Cs	ppm	0.51	Ni	ppm	95	Tm	ppm	0.34
Cu	ppm	160	Pb	ppm	13.0	U	ppm	0.20
Dy	ppm	3.83	Pr	ppm	1.69	V	ppm	296
Er	ppm	2.37	Rb	ppm	7.78	W	ppm	15.1
Eu	ppm	0.91	Re	ppm	< 0.01	Y	ppm	21.3
Ga	ppm	15.7	Sb	ppm	0.75	Yb	ppm	2.30
Gd	ppm	3.28	Sc	ppm	42.8	Zn	ppm	115
Ge	ppm	1.33	Se	ppm	< 5	Zr	ppm	63

### Table 2. Indicative Values for OREAS 231.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/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 231 was prepared in the following manner:

- Drying the gold ore and greenstone to constant mass at 105°C;
- Crushing and multi stage milling of the gold ore to 100% minus 30 microns;
- Crushing and multi stage milling of the greenstone to >98% minus 75 microns;
- Final homogenisation;
- Packaging in 60g units sealed in laminated foil pouches and 500g units in plastic jars.

# PHYSICAL PROPERTIES

OREAS 231 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.

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
740	0.58	N7	Light Gray

Table 3. Physical properties of OREAS 231.

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

- Gold by fire assay (25-50g charge weight) with AAS (25 laboratories), ICP-OES (5 laboratories) and ICP-MS (2 laboratory) finish;
- Full ICP-OES and ICP-MS elemental suites by 4-acid (HNO<sub>3</sub>-HF-HCIO<sub>4</sub>-HCI) digestion (up to 26 laboratories depending on the element);
- Gold by 15-50g aqua regia digestion with ICP-MS (12 laboratories) and AAS (8 laboratories) 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: 5g (1 laboratory by AAS finish), 10g (1 laboratory by ICP-OES finish), 15g (1 laboratory by AAS finish), 30g (6 laboratories by AAS finish), 50g (2 laboratories by ICP-MS and 1 laboratory by AAS finish), 60g (1 laboratory by AAS finish), and 200g (5 laboratories by AAS and 1 laboratory by ICP-MS finish).

To confirm homogeneity, gold by instrumental neutron activation analysis (INAA) was undertaken on 20 x 85mg subsamples by the Australian Nuclear Science and Technology Organisation (ANSTO) located in Lucas Heights, NSW, Australia (see Table 5 in the 'Homogeneity Evaluation' section below).



For the round robin program twenty 2kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six pulp samples were submitted to each laboratory for analysis (the weight provided depended on whether the laboratory was anticipated to undertake assays by gold cyanide leach). The samples received by each laboratory were obtained by taking two samples from each of three separate 2kg 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 61 certified values based on their pooled 1SD's. Table 2 shows 93 indicative values including gold by Photon Assay (undertaken at 4 laboratories) and major and trace element characterisation by Bureau Veritas in Perth, Western Australia which includes:

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

Table 3 provides some indicative physical properties and Table 4 presents the 95% confidence and tolerance limits for all certified values. Gold homogeneity (via INAA) is shown in Table 5 and is also demonstrated by a nested ANOVA program using fire assay (see '**nested ANOVA**' section).

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 231-DataPack.1.0.200311\_144109.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  $\pm 3$ SD (magenta) and  $\pm 5$ % (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 231 (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 present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor.



Constituent	Certified	95% Confid	ence Limits	95% Tolera	ance Limits	
Constituent	Value	Low	High	Value	Low	
Pb Fire Assay	Pb Fire Assay					
Au, Gold (ppm)	0.542	0.538	0.547	0.540*	0.545*	
Aqua Regia Digestion (sample weights 10-50g)						
Au, Gold (ppm)	0.521	0.513	0.528	0.518*	0.523*	
Cyanide Leach						
Au, Gold (ppm)	0.516	0.504	0.527	0.515*	0.516*	
4-Acid Digestion						
Ag, Silver (ppm)	0.177	0.166	0.188	0.158	0.196	
Al, Aluminium (wt.%)	7.14	7.03	7.24	7.02	7.25	
As, Arsenic (ppm)	27.2	26.3	28.2	26.2	28.2	
Ba, Barium (ppm)	112	110	114	109	115	
Be, Beryllium (ppm)	0.36	0.34	0.38	0.33	0.40	
Bi, Bismuth (ppm)	0.037	0.033	0.041	IND	IND	
Ca, Calcium (wt.%)	7.47	7.36	7.58	7.38	7.56	
Cd, Cadmium (ppm)	0.35	0.33	0.38	0.33	0.38	
Ce, Cerium (ppm)	11.0	10.6	11.3	10.7	11.2	
Co, Cobalt (ppm)	44.7	43.5	45.8	43.4	45.9	
Cr, Chromium (ppm)	133	125	141	129	137	
Cs, Caesium (ppm)	0.56	0.55	0.57	0.54	0.58	
Cu, Copper (ppm)	161	157	164	156	165	
Dy, Dysprosium (ppm)	3.71	3.63	3.79	3.60	3.82	
Er, Erbium (ppm)	2.35	2.27	2.43	2.28	2.42	
Eu, Europium (ppm)	0.93	0.89	0.98	0.91	0.96	
Fe, Iron (wt.%)	8.05	7.94	8.17	7.95	8.16	
Ga, Gallium (ppm)	15.9	15.7	16.2	15.5	16.4	
Gd, Gadolinium (ppm)	3.23	3.11	3.35	3.11	3.35	
Hf, Hafnium (ppm)	1.63	1.57	1.68	1.52	1.73	
Ho, Holmium (ppm)	0.82	0.79	0.86	0.79	0.85	
In, Indium (ppm)	0.070	0.068	0.073	0.066	0.074	
K, Potassium (wt.%)	0.288	0.283	0.294	0.280	0.297	
La, Lanthanum (ppm)	4.47	4.36	4.58	4.27	4.66	
Li, Lithium (ppm)	11.1	10.6	11.5	10.6	11.6	
Lu, Lutetium (ppm)	0.33	0.31	0.34	0.30	0.35	
Mg, Magnesium (wt.%)	3.90	3.84	3.97	3.84	3.97	
Mn, Manganese (wt.%)	0.139	0.136	0.142	0.136	0.142	
Mo, Molybdenum (ppm)	0.86	0.82	0.90	0.79	0.93	
Na, Sodium (wt. <del>%</del> )	1.75	1.73	1.77	1.72	1.78	

Table 4. 95% Confidence & Tolerance Limits for OREAS 231.

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

\*Gold Tolerance Limits for typical 30g fire assay, 25g aqua regia digestion and 200g cyanide leach methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973). Note: intervals may appear asymmetric due to rounding.

Constituent	Certified	95% Confid	ence Limits	95% Tolera	ance Limits
	Value	Low	High	Value	Low
4-Acid Digestion continue	ed	ſ	Γ	ſ	Γ
Nb, Niobium (ppm)	3.50	3.37	3.62	3.37	3.63
Nd, Neodymium (ppm)	7.98	7.81	8.15	7.73	8.23
Ni, Nickel (ppm)	88	86	90	86	91
P, Phosphorus (wt.%)	0.039	0.038	0.040	0.038	0.041
Pb, Lead (ppm)	12.8	12.2	13.4	12.2	13.4
Pr, Praseodymium (ppm)	1.66	1.61	1.70	1.60	1.71
Rb, Rubidium (ppm)	8.08	7.89	8.26	7.83	8.32
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.220	0.213	0.227	0.211	0.229
Sb, Antimony (ppm)	0.71	0.68	0.74	0.64	0.77
Sc, Scandium (ppm)	42.2	41.2	43.2	41.2	43.2
Sm, Samarium (ppm)	2.49	2.41	2.57	2.39	2.60
Sn, Tin (ppm)	0.90	0.84	0.96	IND	IND
Sr, Strontium (ppm)	179	177	182	175	184
Ta, Tantalum (ppm)	0.22	0.21	0.23	0.20	0.24
Tb, Terbium (ppm)	0.58	0.55	0.60	0.55	0.60
Te, Tellurium (ppm)	0.068	0.063	0.073	IND	IND
Th, Thorium (ppm)	0.72	0.69	0.75	0.69	0.75
Ti, Titanium (wt.%)	0.617	0.606	0.629	0.602	0.633
TI, Thallium (ppm)	0.12	0.11	0.12	IND	IND
Tm, Thulium (ppm)	0.33	0.32	0.35	0.31	0.35
U, Uranium (ppm)	0.20	0.20	0.20	0.19	0.21
V, Vanadium (ppm)	285	278	292	279	291
W, Tungsten (ppm)	15.2	14.7	15.7	14.8	15.6
Y, Yttrium (ppm)	20.7	20.4	21.1	20.2	21.2
Yb, Ytterbium (ppm)	2.20	2.14	2.27	2.12	2.29
Zn, Zinc (ppm)	113	111	115	110	116
Zr, Zirconium (ppm)	50	49	52	48	52

Table 4 continued.

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 copper 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 156 and 165 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 below shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 231. 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 0.15% was calculated for a 30g fire assay sample (2.78% at 85mg weights) and confirms the high level of gold homogeneity in OREAS 231.

Replicate	Au	Au
No	85mg actual	30g equivalent*
1	0.543	0.537
2	0.515	0.536
3	0.555	0.538
4	0.564	0.538
5	0.534	0.537
6	0.540	0.537
7	0.514	0.536
8	0.525	0.536
9	0.543	0.537
10	0.538	0.537
11	0.553	0.538
12	0.527	0.536
13	0.530	0.537
14	0.556	0.538
15	0.531	0.537
16	0.554	0.538
17	0.531	0.537
18	0.548	0.538
19	0.520	0.536
20	0.517	0.536
Mean	0.537	0.537
Median	0.536	0.537
Std Dev.	0.015	0.001
Rel.Std.Dev.	2.78%	0.15%

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.

\*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

 $\overline{X}$  = mean of 85mg INAA results



The homogeneity of OREAS 231 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the forty-two 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 231. The test was performed using the following parameters:

- Gold fire assay 192 samples (32 laboratories each providing analyses on 3 pairs of samples);
- Gold aqua regia digestion 120 samples (20 laboratories each providing analyses on 3 pairs of samples);
- Gold cyanide leach 114 samples (19 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,  $H_1$ : Between-unit variance is greater than within-unit variance.

*P*-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the *p*-value. This process derived *p*-values of 1.00 for Au by fire assay, 0.70 for Au by aqua regia digestion and 0.94 for Au by cyanide leach. All three *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 231 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 231 is fit-for-purpose as a certified reference material (see 'Intended Use' below).



# PARTICIPATING LABORATORIES

- 1. Actlabs, Ancaster, Ontario, Canada
- 2. AGAT Laboratories, Mississauga, Ontario, Canada
- 3. ALS, Lima, Peru
- 4. ALS, Loughrea, Galway, Ireland
- 5. ALS, Perth, WA, Australia
- 6. ALS, Vancouver, BC, Canada
- 7. American Assay Laboratories, Sparks, Nevada, USA
- 8. ANSTO, Lucas Heights, NSW, Australia
- 9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
- 10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 11. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 12. Bureau Veritas Geoanalytical, Perth, WA, Australia
- 13. Chrysos Corporation, Perth, WA, Australia
- 14. Gekko Assay Labs, Ballarat, VIC, Australia
- 15. Inspectorate (BV), Lima, Peru
- 16. Inspectorate America Corporation (BV), Sparks, Nevada, USA
- 17. Intertek Genalysis, Perth, WA, Australia
- 18. Intertek Tarkwa, Tarkwa, Ghana
- 19. Intertek Testing Services, Townsville, QLD, Australia
- 20. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 21. MinAnalytical Services, Kalgoorlie, WA, Australia
- 22. MinAnalytical Services, Perth, WA, Australia
- 23. Nagrom, Perth, WA, Australia
- 24. On Site Laboratory Services, Bendigo, VIC, Australia
- 25. Ontario Geological Survey, Sudbury, Ontario, Canada
- 26. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 27. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
- 28. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
- 29. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
- 30. SGS, Randfontein, Gauteng, South Africa
- 31. SGS Canada Inc., Vancouver, BC, Canada
- 32. SGS del Peru, Lima, Peru
- 33. SGS Tarkwa, Tarkwa, Western Region, Ghana
- 34. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 35. Skyline Assayers & Laboratories, Tucson, Arizona, USA

# 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 231 was prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd 37A Hosie Street Bayswater North VIC 3153 AUSTRALIA 
 Tel:
 +613-9729 0333

 Fax:
 +613-9729 8338

 Web:
 www.ore.com.au

 Email:
 info@ore.com.au

# 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 231 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 231 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 231 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.

### 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 231 has been prepared from gold ore blended with greenstone. It is low in reactive sulphide (0.22 wt.% S) and in its unopened state and under normal conditions of storage has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

# INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 231 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	12 <sup>th</sup> March, 2020	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**

12<sup>th</sup> March, 2020

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

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