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**CERTIFICATE OF ANALYSIS FOR**  
**CERTIFIED REFERENCE MATERIAL**  
**OREAS 231b**  
**Gold Ore (Frogs Leg Gold Mine, Western Australia)**

**Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 231b.**

Constituent	Certified Value <sup>†</sup>	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	0.556	0.550	0.563	0.552*	0.561*

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg.

<sup>†</sup>This operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

\*Gold Tolerance Limits for typical 30g fire assay are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.



Accredited for compliance with ISO 17034



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**Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 231b.**

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
<b>PhotonAssay</b>					
Au, Gold (ppm)	0.556	0.549	0.564	0.555	0.557
<b>Aqua Regia Digestion (sample weights 10-50 g)</b>					
Au, Gold (ppm)	0.526	0.517	0.535	0.521*	0.531*
<b>Cyanide Leach</b>					
Au, Gold (ppm)	0.524	0.515	0.532	0.522*	0.525*
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	0.182	0.170	0.194	0.166	0.198
Al, Aluminium (wt.%)	6.89	6.70	7.07	6.77	7.00
As, Arsenic (ppm)	34.0	31.9	36.1	32.2	35.8
Ba, Barium (ppm)	299	287	311	289	310
Be, Beryllium (ppm)	0.37	0.34	0.40	0.35	0.39
Bi, Bismuth (ppm)	0.029	0.021	0.036	IND	IND
Ca, Calcium (wt.%)	6.43	6.26	6.60	6.33	6.54
Cd, Cadmium (ppm)	0.39	0.36	0.43	0.37	0.42
Ce, Cerium (ppm)	11.3	10.7	12.0	11.0	11.7
Co, Cobalt (ppm)	46.6	44.8	48.3	45.5	47.6
Cr, Chromium (ppm)	154	146	162	149	159
Cs, Caesium (ppm)	0.58	0.55	0.62	0.56	0.61
Cu, Copper (ppm)	156	150	161	153	159
Dy, Dysprosium (ppm)	3.77	3.55	3.99	3.63	3.91
Er, Erbium (ppm)	2.32	2.16	2.47	2.21	2.42
Eu, Europium (ppm)	0.92	0.86	0.98	0.89	0.95
Fe, Iron (wt.%)	8.31	8.07	8.54	8.17	8.44
Ga, Gallium (ppm)	15.4	14.6	16.1	14.8	16.0
Gd, Gadolinium (ppm)	3.04	2.77	3.32	2.97	3.12
Hf, Hafnium (ppm)	1.54	1.40	1.69	1.46	1.63
Ho, Holmium (ppm)	0.77	0.73	0.82	0.73	0.82
In, Indium (ppm)	0.071	0.062	0.080	0.063	0.079
K, Potassium (wt.%)	0.477	0.459	0.495	0.467	0.487
La, Lanthanum (ppm)	4.52	4.30	4.75	4.36	4.69
Li, Lithium (ppm)	12.0	11.1	12.8	11.6	12.4
Lu, Lutetium (ppm)	0.33	0.29	0.37	0.32	0.34
Mg, Magnesium (wt.%)	4.14	4.04	4.24	4.06	4.23
Mn, Manganese (wt.%)	0.158	0.153	0.162	0.155	0.160
Mo, Molybdenum (ppm)	1.46	1.38	1.54	1.37	1.55
Na, Sodium (wt.%)	2.04	1.99	2.09	2.00	2.08

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

\*Gold Tolerance Limits for typical 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; IND = indeterminate (due to limited reading resolution of the methods employed).

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
Nb, Niobium (ppm)	3.66	3.39	3.94	3.49	3.84
Nd, Neodymium (ppm)	7.98	7.64	8.32	7.83	8.13
Ni, Nickel (ppm)	105	101	109	103	107
P, Phosphorus (wt.%)	0.044	0.042	0.045	0.043	0.045
Pb, Lead (ppm)	13.6	12.8	14.3	13.0	14.2
Pr, Praseodymium (ppm)	1.64	1.52	1.76	1.58	1.71
Rb, Rubidium (ppm)	10.4	9.7	11.0	10.0	10.7
Re, Rhenium (ppm)	0.003	0.002	0.004	IND	IND
S, Sulphur (wt.%)	0.264	0.250	0.278	0.257	0.272
Sb, Antimony (ppm)	0.82	0.75	0.89	0.77	0.87
Sc, Scandium (ppm)	39.4	37.8	41.1	38.2	40.6
Sm, Samarium (ppm)	2.44	2.21	2.67	2.32	2.56
Sn, Tin (ppm)	0.94	0.82	1.05	0.82	1.05
Sr, Strontium (ppm)	203	197	210	200	207
Ta, Tantalum (ppm)	0.24	0.21	0.27	0.21	0.27
Tb, Terbium (ppm)	0.56	0.52	0.60	0.54	0.58
Te, Tellurium (ppm)	0.074	0.054	0.093	IND	IND
Th, Thorium (ppm)	0.65	0.61	0.70	0.62	0.68
Ti, Titanium (wt.%)	0.638	0.623	0.653	0.621	0.655
Tl, Thallium (ppm)	0.12	0.11	0.14	IND	IND
Tm, Thulium (ppm)	0.33	0.31	0.36	0.31	0.35
U, Uranium (ppm)	0.20	0.20	0.21	0.19	0.21
V, Vanadium (ppm)	283	275	291	278	288
W, Tungsten (ppm)	17.0	16.2	17.7	16.5	17.4
Y, Yttrium (ppm)	20.5	19.4	21.6	19.9	21.1
Yb, Ytterbium (ppm)	2.13	1.96	2.30	2.02	2.24
Zn, Zinc (ppm)	118	113	122	115	121
Zr, Zirconium (ppm)	45.4	41.7	49.1	42.9	47.9
<b>Aqua Regia Digestion</b>					
Ag, Silver (ppm)	0.182	0.169	0.195	0.167	0.197
Al, Aluminium (wt.%)	3.13	3.00	3.27	3.07	3.20
As, Arsenic (ppm)	33.3	32.1	34.6	32.4	34.3
B, Boron (ppm)	28.6	22.8	34.4	26.9	30.3
Ba, Barium (ppm)	35.8	33.4	38.3	34.5	37.1
Be, Beryllium (ppm)	0.17	0.15	0.19	IND	IND
Bi, Bismuth (ppm)	0.027	0.019	0.035	IND	IND
Ca, Calcium (wt.%)	2.11	1.97	2.24	2.05	2.16

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed).

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Cd, Cadmium (ppm)	0.34	0.32	0.37	0.32	0.37
Ce, Cerium (ppm)	7.94	7.65	8.22	7.76	8.11
Co, Cobalt (ppm)	33.4	32.2	34.6	32.5	34.2
Cr, Chromium (ppm)	23.9	22.7	25.1	23.1	24.7
Cs, Caesium (ppm)	0.44	0.42	0.46	0.42	0.46
Cu, Copper (ppm)	155	151	159	152	158
Dy, Dysprosium (ppm)	2.12	1.77	2.46	1.99	2.24
Er, Erbium (ppm)	1.22	1.01	1.42	1.17	1.27
Eu, Europium (ppm)	0.44	0.34	0.55	0.43	0.46
Fe, Iron (wt.%)	5.91	5.78	6.03	5.78	6.03
Ga, Gallium (ppm)	10.2	9.6	10.8	10.0	10.4
Gd, Gadolinium (ppm)	1.82	1.57	2.06	1.66	1.97
Ge, Germanium (ppm)	0.13	0.10	0.16	IND	IND
Hf, Hafnium (ppm)	0.40	0.36	0.43	0.37	0.42
Ho, Holmium (ppm)	0.42	0.36	0.48	0.38	0.47
In, Indium (ppm)	0.024	0.022	0.027	0.021	0.027
K, Potassium (wt.%)	0.104	0.099	0.108	0.100	0.107
La, Lanthanum (ppm)	3.34	3.23	3.46	3.23	3.45
Li, Lithium (ppm)	10.6	10.1	11.1	10.4	10.8
Lu, Lutetium (ppm)	0.13	0.11	0.15	IND	IND
Mg, Magnesium (wt.%)	1.96	1.91	2.01	1.92	2.00
Mn, Manganese (wt.%)	0.084	0.081	0.087	0.083	0.086
Mo, Molybdenum (ppm)	1.39	1.32	1.47	1.32	1.47
Na, Sodium (wt.%)	0.147	0.140	0.154	0.137	0.156
Nd, Neodymium (ppm)	5.31	4.84	5.78	5.15	5.47
Ni, Nickel (ppm)	73	71	75	71	74
P, Phosphorus (wt.%)	0.043	0.042	0.045	0.042	0.044
Pb, Lead (ppm)	13.3	12.8	13.9	12.8	13.9
Pr, Praseodymium (ppm)	1.07	0.96	1.18	1.02	1.12
Rb, Rubidium (ppm)	3.99	3.76	4.22	3.86	4.12
Re, Rhenium (ppm)	0.002	0.001	0.003	IND	IND
S, Sulphur (wt.%)	0.262	0.251	0.272	0.253	0.270
Sb, Antimony (ppm)	0.50	0.44	0.56	0.47	0.54
Sc, Scandium (ppm)	4.46	4.22	4.70	4.28	4.65
Sm, Samarium (ppm)	1.53	1.37	1.70	1.48	1.58
Sn, Tin (ppm)	0.52	0.46	0.59	0.48	0.57
Sr, Strontium (ppm)	32.0	29.0	35.1	30.2	33.9

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

**Table 2 continued.**

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.31	0.29	0.33	0.29	0.32
Te, Tellurium (ppm)	0.060	0.047	0.072	IND	IND
Th, Thorium (ppm)	0.46	0.40	0.52	0.43	0.49
Ti, Titanium (wt.%)	0.329	0.301	0.357	0.319	0.339
Tl, Thallium (ppm)	0.064	0.057	0.072	IND	IND
Tm, Thulium (ppm)	0.17	0.14	0.19	IND	IND
U, Uranium (ppm)	0.13	0.12	0.14	0.12	0.14
V, Vanadium (ppm)	141	131	150	137	144
W, Tungsten (ppm)	11.9	10.8	12.9	11.4	12.3
Y, Yttrium (ppm)	10.8	10.3	11.2	10.5	11.0
Yb, Ytterbium (ppm)	0.98	0.84	1.12	0.93	1.03
Zn, Zinc (ppm)	102	99	105	100	104
Zr, Zirconium (ppm)	12.1	11.0	13.3	11.5	12.8

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

**Table 3. Indicative Values for OREAS 231b.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppb	20.8	Pt	ppb	10.3			
<b>4-Acid Digestion</b>								
B	ppm	32.6	Hg	ppm	0.19			
Ge	ppm	0.16	Se	ppm	1.00			
<b>Aqua Regia Digestion</b>								
Hg	ppm	0.036	Pd	ppb	16.4	Si	wt.%	0.081
Ir	ppm	< 0.003	Pt	ppb	8.77			
Nb	ppm	0.11	Se	ppm	0.52			
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt.%	13.15	MgO	wt.%	7.05	S	wt.%	0.219
CaO	wt.%	9.38	MnO	wt.%	0.192	SiO <sub>2</sub>	wt.%	50.17
Fe <sub>2</sub> O <sub>3</sub>	wt.%	12.31	Na <sub>2</sub> O	wt.%	2.74	TiO <sub>2</sub>	wt.%	1.11
K <sub>2</sub> O	wt.%	0.550	P <sub>2</sub> O <sub>5</sub>	wt.%	0.097			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt.%	2.98						
<b>Infrared Combustion</b>								
C	wt.%	0.100	S	wt.%	0.245			

SI unit equivalents: ppb (parts per billion;  $1 \times 10^{-9}$ )  $\equiv$   $\mu$ g/kg; ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

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.

**Table 3 continued.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	0.200	Hf	ppm	1.88	Sm	ppm	2.61
As	ppm	33.2	Ho	ppm	0.84	Sn	ppm	0.80
Ba	ppm	295	In	ppm	0.050	Sr	ppm	197
Be	ppm	0.60	La	ppm	4.87	Ta	ppm	0.21
Bi	ppm	0.040	Lu	ppm	0.35	Tb	ppm	0.58
Cd	ppm	0.40	Mn	wt.%	0.168	Te	ppm	< 0.2
Ce	ppm	11.0	Mo	ppm	1.30	Th	ppm	0.65
Co	ppm	47.8	Nb	ppm	3.59	Ti	wt.%	0.667
Cr	ppm	190	Nd	ppm	8.24	Tl	ppm	< 0.2
Cs	ppm	0.55	Ni	ppm	115	Tm	ppm	0.35
Cu	ppm	160	Pb	ppm	13.5	U	ppm	0.17
Dy	ppm	3.86	Pr	ppm	1.74	V	ppm	298
Er	ppm	2.46	Rb	ppm	9.90	W	ppm	16.5
Eu	ppm	0.89	Re	ppm	0.018	Y	ppm	21.5
Ga	ppm	15.1	Sb	ppm	0.80	Yb	ppm	2.44
Gd	ppm	3.21	Sc	ppm	41.3	Zn	ppm	120
Ge	ppm	1.53	Se	ppm	< 5	Zr	ppm	65

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

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.

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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 'Instructions for correct use' should be read carefully.

Tables 1 and 2 provide the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties, Table 5 provides indicative mineralogy based on semi-quantitative XRD analysis and Table 6 presents the performance gate intervals for all certified values. Gold homogeneity (via INAA) is shown in Table 7 and is also demonstrated by a nested ANOVA program using fire assay (see '**nested ANOVA**' section).

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent 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 231b-DataPack.1.2.241202\_115012.xlsx**).

Results are also presented in scatter plots for gold by fire assay, aqua regia digestion, cyanide leach and photon assay (Figures 1 to 4, respectively) together with  $\pm 3SD$  (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.

## SOURCE MATERIAL

OREAS 231b was prepared from a blend of gold-bearing ore and barren greenstone. The ore was sourced from the Frogs Leg Gold Mine located 19 km west of Kalgoorlie in Western Australia. The ore lodes lie within sheared contacts between volcanoclastics and basalt and are hosted in laminated quartz veins, breccia and wall rock alteration. The Cambrian greenstone was sourced from a quarry 145 km north of Melbourne, Australia.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 231b was prepared in the following manner:

- Drying to constant mass at 105 °C;
- Crushing and multi stage milling of the gold ore to 100 % minus 30  $\mu\text{m}$ ;
- Crushing and multi stage milling of the greenstone to > 98 % minus 75  $\mu\text{m}$ ;
- Blending in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 60 g units sealed in laminated foil pouches and 500 g units in plastic jars.



## PHYSICAL PROPERTIES

OREAS 231b was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

**Table 4. Physical properties of OREAS 231b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
827	0.65	5GY 6/1	Greenish Gray

<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.

## MINERALOGY

The semi-quantitative XRD results shown in Table 5 below have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. Some amorphous material is likely present.

**Table 5. Indicative mineralogy of OREAS 231b based on semi-quantitative XRD analysis.**

Mineral / Mineral Group	% (mass ratio)
Stilpnomelane	< 1
Chlorite	18
Biotite	3
Muscovite	2
Calcic amphibole	2
Clinopyroxene	15
Epidote	4
Prehnite	1
Plagioclase	39
K-feldspar	3
Quartz	12
Gypsum	0

## ANALYTICAL PROGRAM

Thirty-six commercial analytical laboratories participated in the program to certify the elements reported in Tables 1 and 2. The following methods were employed:

- Gold by fire assay (15-50 g charge weight) with AAS (24 laboratories), ICP-OES (6 laboratories) finish and ICP-MS (1 laboratory) finish;
- Gold by X-ray PhotonAssay on ~350 g sample weights (11 laboratories with data generated by both Chrysos and local laboratory staff and on multiple PhotonAssay machines where available);
- Gold by aqua regia digestion (10-50g sample weight) with ICP-OES and/or ICP-MS (23 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: 5 g (2 laboratories by AAS finish), 20 g (1 laboratory by AAS finish), 30 g (4 laboratories by AAS finish and 1 laboratory by ICP-OES finish), 50 g (1 laboratory by AAS, 1 laboratory by ICP-OES finish and 2 laboratories by ICP-MS finish), 60 g (1 laboratory by AAS finish) and 200 g (7 laboratories by AAS and 1 laboratory by ICP-MS finish).

- Full ICP-OES and ICP-MS elemental suites by 4-acid ( $\text{HNO}_3\text{-HF-HClO}_4\text{-HCl}$ ) digestion (up to 29 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by aqua regia digestion (up to 31 laboratories depending on the element).

Instrumental neutron activation analysis for Au on 20 x 85 mg subsamples was also undertaken at ANSTO, Lucas Heights to confirm homogeneity (see Table 7 below).

Table 3 shows indicative values including 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 elements by laser ablation (on the fused bead) with ICP-MS finish.

For the round robin program twenty 3 kg 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 3 kg 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 (see 'Homogeneity Evaluation' section below).

## STATISTICAL ANALYSIS

**Certified Values and their uncertainty intervals** (Tables 1 and 2) 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. 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. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

**Certified Values** are the means of accepted laboratory means after outlier filtering and are the present best estimate of the true value. The INAA data (see Table 7) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation (see 'Homogeneity Evaluation' section below).

**95 % Expanded Uncertainty** provides a 95 % probability that the true value of the analyte under consideration lies between the upper and lower limits and is calculated according to the method outlined in ISO 98-3 [6]. All known or suspected sources of bias have been investigated or taken into account.

**Indicative (uncertified) values** (Table 3) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor.

**Standard Deviation** intervals (see Table 6) 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.***

## PERFORMANCE GATES

Table 6 below 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](http://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  $\pm 10$  %  $\pm 2DL$  [1].*

**Table 6. Performance Gates for OREAS 231b.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>PhotonAssay</b>											
Au, ppm	0.556	0.030	0.495	0.617	0.465	0.647	5.47%	10.95%	16.42%	0.528	0.584
<b>Pb Fire Assay</b>											
Au, ppm	0.556	0.017	0.522	0.591	0.505	0.608	3.11%	6.21%	9.32%	0.529	0.584
<b>Aqua Regia Digestion (sample weights 10-50g)</b>											
Au, ppm	0.526	0.024	0.478	0.573	0.455	0.597	4.52%	9.04%	13.57%	0.500	0.552
<b>Cyanide Leach</b>											
Au, ppm	0.524	0.024	0.476	0.571	0.452	0.595	4.56%	9.12%	13.68%	0.498	0.550
<b>4-Acid Digestion</b>											
Ag, ppm	0.182	0.011	0.159	0.205	0.148	0.217	6.31%	12.61%	18.92%	0.173	0.191
Al, wt.%	6.89	0.234	6.42	7.35	6.18	7.59	3.40%	6.81%	10.21%	6.54	7.23
As, ppm	34.0	2.64	28.7	39.3	26.1	41.9	7.76%	15.52%	23.29%	32.3	35.7
Ba, ppm	299	14	271	328	256	342	4.76%	9.53%	14.29%	284	314
Be, ppm	0.37	0.05	0.28	0.46	0.23	0.51	12.58%	25.16%	37.74%	0.35	0.39
Bi, ppm	0.029	0.005	0.019	0.038	0.015	0.043	16.32%	32.64%	48.96%	0.027	0.030
Ca, wt.%	6.43	0.267	5.90	6.97	5.63	7.23	4.15%	8.31%	12.46%	6.11	6.76
Cd, ppm	0.39	0.037	0.32	0.47	0.28	0.50	9.56%	19.11%	28.67%	0.37	0.41
Ce, ppm	11.3	0.56	10.2	12.5	9.7	13.0	4.96%	9.91%	14.87%	10.8	11.9
Co, ppm	46.6	1.66	43.2	49.9	41.6	51.6	3.57%	7.14%	10.71%	44.2	48.9
Cr, ppm	154	13	127	180	114	194	8.69%	17.38%	26.07%	146	161
Cs, ppm	0.58	0.043	0.50	0.67	0.46	0.71	7.33%	14.66%	22.00%	0.56	0.61
Cu, ppm	156	6	143	168	137	175	4.04%	8.08%	12.11%	148	163
Dy, ppm	3.77	0.129	3.51	4.03	3.38	4.16	3.43%	6.85%	10.28%	3.58	3.96
Er, ppm	2.32	0.093	2.13	2.50	2.04	2.59	4.02%	8.05%	12.07%	2.20	2.43
Eu, ppm	0.92	0.049	0.82	1.02	0.78	1.07	5.33%	10.67%	16.00%	0.88	0.97
Fe, wt.%	8.31	0.371	7.56	9.05	7.19	9.42	4.47%	8.94%	13.41%	7.89	8.72
Ga, ppm	15.4	1.05	13.3	17.5	12.2	18.5	6.84%	13.68%	20.53%	14.6	16.1
Gd, ppm	3.04	0.234	2.57	3.51	2.34	3.74	7.69%	15.39%	23.08%	2.89	3.19
Hf, ppm	1.54	0.21	1.12	1.96	0.91	2.17	13.59%	27.19%	40.78%	1.47	1.62
Ho, ppm	0.77	0.043	0.69	0.86	0.65	0.90	5.55%	11.09%	16.64%	0.74	0.81
In, ppm	0.071	0.009	0.054	0.089	0.045	0.097	12.32%	24.64%	36.96%	0.068	0.075
K, wt.%	0.477	0.024	0.429	0.524	0.406	0.548	4.99%	9.97%	14.96%	0.453	0.501
La, ppm	4.52	0.189	4.15	4.90	3.96	5.09	4.17%	8.34%	12.51%	4.30	4.75
Li, ppm	12.0	1.4	9.2	14.7	7.9	16.0	11.35%	22.69%	34.04%	11.4	12.6
Lu, ppm	0.33	0.028	0.27	0.39	0.25	0.41	8.42%	16.84%	25.27%	0.31	0.35
Mg, wt.%	4.14	0.121	3.90	4.38	3.78	4.50	2.92%	5.84%	8.76%	3.93	4.35
Mn, wt.%	0.158	0.006	0.146	0.169	0.141	0.175	3.65%	7.30%	10.95%	0.150	0.166
Mo, ppm	1.46	0.089	1.28	1.64	1.19	1.73	6.11%	12.21%	18.32%	1.39	1.53
Na, wt.%	2.04	0.078	1.88	2.20	1.80	2.27	3.84%	7.69%	11.53%	1.94	2.14
Nb, ppm	3.66	0.344	2.98	4.35	2.63	4.69	9.39%	18.77%	28.16%	3.48	3.85
Nd, ppm	7.98	0.282	7.42	8.54	7.13	8.83	3.53%	7.07%	10.60%	7.58	8.38
Ni, ppm	105	5	95	115	90	120	4.69%	9.38%	14.07%	100	110
P, wt.%	0.044	0.002	0.040	0.047	0.039	0.049	3.88%	7.76%	11.64%	0.042	0.046

SI unit equivalents: ppm (parts per million;  $1 \times 10^6 \equiv \text{mg/kg}$ ; wt.% (weight per cent)  $\equiv \%$  (mass fraction).

TBA: To be advised (certified values & performance gates data coming soon).

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 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>4-Acid Digestion continued</b>											
Pb, ppm	13.6	1.16	11.2	15.9	10.1	17.1	8.56%	17.13%	25.69%	12.9	14.2
Pr, ppm	1.64	0.108	1.43	1.86	1.32	1.97	6.57%	13.14%	19.71%	1.56	1.72
Rb, ppm	10.4	0.57	9.2	11.5	8.6	12.1	5.51%	11.01%	16.52%	9.8	10.9
Re, ppm	0.003	0.001	0.001	0.004	0.001	0.004	23.24%	46.49%	69.73%	0.002	0.003
S, wt. %	0.264	0.014	0.236	0.292	0.222	0.306	5.29%	10.58%	15.86%	0.251	0.277
Sb, ppm	0.82	0.062	0.70	0.95	0.63	1.01	7.56%	15.13%	22.69%	0.78	0.86
Sc, ppm	39.4	2.25	34.9	43.9	32.7	46.2	5.71%	11.43%	17.14%	37.5	41.4
Sm, ppm	2.44	0.188	2.06	2.82	1.88	3.00	7.69%	15.37%	23.06%	2.32	2.56
Sn, ppm	0.94	0.14	0.66	1.21	0.53	1.34	14.44%	28.88%	43.32%	0.89	0.98
Sr, ppm	203	9	186	220	177	229	4.23%	8.47%	12.70%	193	213
Ta, ppm	0.24	0.05	0.14	0.34	0.09	0.39	20.55%	41.09%	61.64%	0.23	0.25
Tb, ppm	0.56	0.041	0.48	0.64	0.44	0.68	7.25%	14.49%	21.74%	0.53	0.59
Te, ppm	0.074	0.018	0.037	0.110	0.019	0.128	24.91%	49.83%	74.74%	0.070	0.077
Th, ppm	0.65	0.046	0.56	0.74	0.52	0.79	7.01%	14.01%	21.02%	0.62	0.69
Ti, wt. %	0.638	0.018	0.602	0.674	0.584	0.692	2.83%	5.66%	8.48%	0.606	0.670
Tl, ppm	0.12	0.01	0.10	0.15	0.09	0.16	10.08%	20.17%	30.25%	0.12	0.13
Tm, ppm	0.33	0.024	0.29	0.38	0.26	0.40	7.09%	14.18%	21.27%	0.32	0.35
U, ppm	0.20	0.007	0.19	0.22	0.18	0.22	3.63%	7.26%	10.89%	0.19	0.21
V, ppm	283	10	263	303	253	312	3.49%	6.97%	10.46%	269	297
W, ppm	17.0	1.29	14.4	19.5	13.1	20.8	7.59%	15.17%	22.76%	16.1	17.8
Y, ppm	20.5	1.34	17.8	23.2	16.5	24.5	6.54%	13.09%	19.63%	19.4	21.5
Yb, ppm	2.13	0.171	1.79	2.47	1.62	2.64	8.04%	16.09%	24.13%	2.02	2.24
Zn, ppm	118	7	104	131	98	138	5.70%	11.40%	17.10%	112	124
Zr, ppm	45.4	6.5	32.5	58.4	26.0	64.9	14.26%	28.52%	42.78%	43.2	47.7
<b>Aqua Regia Digestion</b>											
Ag, ppm	0.182	0.015	0.153	0.212	0.138	0.226	8.07%	16.14%	24.21%	0.173	0.191
Al, wt. %	3.13	0.248	2.64	3.63	2.39	3.87	7.91%	15.82%	23.72%	2.98	3.29
As, ppm	33.3	1.64	30.0	36.6	28.4	38.2	4.91%	9.82%	14.74%	31.7	35.0
B, ppm	28.6	8.1	12.5	44.7	4.4	52.8	28.19%	56.37%	84.56%	27.2	30.0
Ba, ppm	35.8	4.4	27.1	44.6	22.7	49.0	12.25%	24.51%	36.76%	34.0	37.6
Be, ppm	0.17	0.02	0.12	0.22	0.10	0.24	13.99%	27.99%	41.98%	0.16	0.18
Bi, ppm	0.027	0.005	0.018	0.037	0.013	0.042	17.93%	35.86%	53.79%	0.026	0.029
Ca, wt. %	2.11	0.32	1.46	2.75	1.14	3.07	15.28%	30.56%	45.85%	2.00	2.21
Cd, ppm	0.34	0.028	0.29	0.40	0.26	0.43	8.11%	16.22%	24.32%	0.33	0.36
Ce, ppm	7.94	0.421	7.09	8.78	6.67	9.20	5.30%	10.60%	15.90%	7.54	8.33
Co, ppm	33.4	2.15	29.1	37.7	26.9	39.8	6.45%	12.89%	19.34%	31.7	35.0
Cr, ppm	23.9	2.29	19.3	28.5	17.0	30.8	9.57%	19.14%	28.72%	22.7	25.1
Cs, ppm	0.44	0.030	0.38	0.50	0.35	0.53	6.79%	13.58%	20.37%	0.42	0.46
Cu, ppm	155	5	145	165	139	170	3.34%	6.68%	10.02%	147	163
Dy, ppm	2.12	0.29	1.53	2.70	1.24	2.99	13.75%	27.50%	41.26%	2.01	2.22
Er, ppm	1.22	0.18	0.87	1.57	0.69	1.75	14.48%	28.95%	43.43%	1.16	1.28
Eu, ppm	0.44	0.09	0.26	0.63	0.17	0.72	20.50%	41.00%	61.50%	0.42	0.47
Fe, wt. %	5.91	0.172	5.56	6.25	5.39	6.42	2.91%	5.81%	8.72%	5.61	6.20

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt. % (weight per cent)  $\equiv$  % (mass fraction).

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 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Aqua Regia Digestion continued</b>											
Ga, ppm	10.2	0.91	8.4	12.0	7.5	12.9	8.95%	17.90%	26.85%	9.7	10.7
Gd, ppm	1.82	0.20	1.42	2.21	1.22	2.41	10.87%	21.75%	32.62%	1.73	1.91
Ge, ppm	0.13	0.03	0.07	0.19	0.05	0.21	21.34%	42.68%	64.01%	0.12	0.14
Hf, ppm	0.40	0.038	0.32	0.47	0.28	0.51	9.49%	18.99%	28.48%	0.38	0.42
Ho, ppm	0.42	0.042	0.34	0.51	0.30	0.55	9.94%	19.88%	29.82%	0.40	0.45
In, ppm	0.024	0.004	0.017	0.032	0.013	0.036	15.74%	31.48%	47.23%	0.023	0.026
K, wt. %	0.104	0.006	0.092	0.116	0.086	0.122	5.82%	11.64%	17.45%	0.099	0.109
La, ppm	3.34	0.170	3.00	3.68	2.83	3.85	5.09%	10.17%	15.26%	3.18	3.51
Li, ppm	10.6	0.65	9.3	11.9	8.6	12.5	6.15%	12.29%	18.44%	10.1	11.1
Lu, ppm	0.13	0.03	0.08	0.18	0.05	0.21	20.93%	41.86%	62.78%	0.12	0.14
Mg, wt. %	1.96	0.089	1.78	2.14	1.69	2.22	4.57%	9.13%	13.70%	1.86	2.05
Mn, wt. %	0.084	0.006	0.072	0.096	0.066	0.102	7.11%	14.22%	21.32%	0.080	0.088
Mo, ppm	1.39	0.095	1.21	1.58	1.11	1.68	6.78%	13.56%	20.34%	1.33	1.46
Na, wt. %	0.147	0.011	0.124	0.169	0.113	0.180	7.62%	15.24%	22.86%	0.139	0.154
Nd, ppm	5.31	0.366	4.58	6.05	4.22	6.41	6.88%	13.76%	20.64%	5.05	5.58
Ni, ppm	73	3.3	66	80	63	83	4.57%	9.14%	13.72%	69	77
P, wt. %	0.043	0.002	0.038	0.048	0.036	0.050	5.50%	11.01%	16.51%	0.041	0.045
Pb, ppm	13.3	0.58	12.2	14.5	11.6	15.1	4.36%	8.72%	13.07%	12.7	14.0
Pr, ppm	1.07	0.073	0.93	1.22	0.85	1.29	6.79%	13.58%	20.38%	1.02	1.13
Rb, ppm	3.99	0.40	3.19	4.79	2.79	5.19	10.04%	20.07%	30.11%	3.79	4.19
Re, ppm	0.002	0.000	0.001	0.003	0.000	0.003	25.55%	51.09%	76.64%	0.002	0.002
S, wt. %	0.262	0.013	0.236	0.287	0.223	0.300	4.93%	9.87%	14.80%	0.248	0.275
Sb, ppm	0.50	0.11	0.28	0.72	0.17	0.83	21.78%	43.56%	65.34%	0.48	0.53
Sc, ppm	4.46	0.293	3.88	5.05	3.58	5.34	6.57%	13.14%	19.72%	4.24	4.68
Sm, ppm	1.53	0.124	1.28	1.78	1.16	1.90	8.12%	16.24%	24.35%	1.45	1.61
Sn, ppm	0.52	0.05	0.41	0.63	0.36	0.68	10.23%	20.46%	30.69%	0.49	0.55
Sr, ppm	32.0	6.5	19.0	45.1	12.5	51.6	20.38%	40.76%	61.14%	30.4	33.6
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.31	0.011	0.28	0.33	0.27	0.34	3.58%	7.16%	10.74%	0.29	0.32
Te, ppm	0.060	0.008	0.044	0.075	0.036	0.083	12.96%	25.92%	38.89%	0.057	0.063
Th, ppm	0.46	0.05	0.35	0.56	0.30	0.62	11.38%	22.77%	34.15%	0.44	0.48
Ti, wt. %	0.329	0.059	0.211	0.447	0.151	0.506	17.99%	35.97%	53.96%	0.312	0.345
Tl, ppm	0.064	0.006	0.052	0.077	0.046	0.083	9.73%	19.46%	29.18%	0.061	0.068
Tm, ppm	0.17	0.02	0.13	0.20	0.11	0.22	11.67%	23.34%	35.01%	0.16	0.17
U, ppm	0.13	0.011	0.11	0.15	0.10	0.16	8.39%	16.78%	25.17%	0.12	0.14
V, ppm	141	20	100	181	80	201	14.40%	28.80%	43.20%	134	148
W, ppm	11.9	2.0	8.0	15.8	6.0	17.7	16.44%	32.87%	49.31%	11.3	12.5
Y, ppm	10.8	0.64	9.5	12.1	8.8	12.7	5.97%	11.93%	17.90%	10.2	11.3
Yb, ppm	0.98	0.14	0.71	1.26	0.57	1.40	14.07%	28.14%	42.20%	0.93	1.03
Zn, ppm	102	6	91	114	85	119	5.62%	11.24%	16.86%	97	107
Zr, ppm	12.1	1.8	8.5	15.8	6.6	17.6	15.10%	30.20%	45.30%	11.5	12.7

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt. % (weight per cent)  $\equiv$  % (mass fraction).

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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.

## Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Tables 1 and 2 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 153 and 159 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 7 below shows the gold INAA data determined on 20 x 85 mg subsamples of OREAS 231b. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30 g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.27 % calculated for a 30 g fire assay sample (4.98 % at 85 mg weights) confirms the high level of gold homogeneity in OREAS 231b.

The homogeneity of gold has been determined by INAA at ANSTO using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973 [2]). 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.

The homogeneity of gold in OREAS 231b has also been evaluated in a nested Analysis of Variance (**ANOVA**) of the round robin program. Each participating laboratory received six samples 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 231b. The test was performed using the following parameters:

- Gold fire assay – 186 samples (31 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis,  $H_0$ : Between-unit variance is no greater than within-unit variance (reject  $H_0$  if  $p$ -value < 0.05);
- Alternative Hypothesis,  $H_1$ : Between-unit variance is greater than within-unit variance.

$P$ -values are a measure of probability where values less than 0.05 indicate a greater than 95 % probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the  $p$ -value.

This process derived a  $p$ -value of 0.898 for Au by fire assay, an insignificant result 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 231b 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 ANOVA and the results of the interlaboratory certification program, it can be concluded that OREAS 231b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

**Table 7. Neutron Activation Analysis of Au (in ppm) on 20 x 85 mg subsamples and showing the equivalent results scaled to a 30 g sample mass typical of fire assay determination.**

Replicate No	Au 85 mg actual	Au 30 g equivalent*
1	0.582	0.578
2	0.598	0.579
3	0.532	0.575
4	0.580	0.578
5	0.558	0.577
6	0.550	0.576
7	0.537	0.575
8	0.569	0.577
9	0.528	0.575
10	0.581	0.578
11	0.589	0.578
12	0.572	0.577
13	0.619	0.580
14	0.593	0.578
15	0.555	0.576
16	0.621	0.580
17	0.570	0.577
18	0.593	0.578
19	0.632	0.581
20	0.592	0.578
Mean	0.578	0.578
Median	0.580	0.578
Std Dev.	0.029	0.002
<b>Rel.Std.Dev.</b>	<b>4.98%</b>	<b>0.27%</b>

\*Results calculated for a 30 g 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 30 g sample mass  
 $(x^{INAA})$  = raw INAA result at 85mg  
 $\bar{X}$  = mean of 85 mg INAA results



## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. AGAT Laboratories, Mississauga, Ontario, Canada
4. ALS, Brisbane, QLD, Australia
5. ALS, Canning Vale, WA, Australia
6. ALS, Kalgoorlie, WA, Australia
7. ALS, Lima, Peru
8. ALS, Loughrea, Galway, Ireland
9. ALS, Perth, WA, Australia
10. ALS, Vancouver, BC, Canada
11. American Assay Laboratories, Sparks, Nevada, USA
12. ANSTO, Lucas Heights, NSW, Australia
13. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
14. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
15. Bureau Veritas Geoanalytical, Perth, WA, Australia
16. CRS Laboratories Oy, Kempele, Northern Ostrobothnia, Finland
17. Gekko Assay Labs, Ballarat, VIC, Australia
18. Inspectorate (BV), Lima, Peru
19. Inspectorate Griffith India, Gandhidham, Gujarat, India
20. Intertek Genalysis, Adelaide, SA, Australia
21. Intertek Genalysis, Perth, WA, Australia
22. Intertek Tarkwa, Tarkwa, Ghana
23. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
24. Koza Gold (Ovacik Gold Mine), Bergama, Izmir, Turkey
25. MSA ENVAL Laboratories, Yamoussoukro, Côte d'Ivoire
26. MSALABS, Prince George, BC, Canada
27. MSALABS, Val-d'Or, Quebec, Canada
28. MSALABS, Vancouver, BC, Canada
29. MSALABS Bulyanhulu Gold Mine, Bubada, Shinyanga, United Republic of Tanzania
30. MSALABS Kibali Gold Mines, Doko, Haut-Uélé, Congo, Democratic Republic of the (Zaire)
31. Nagrom, Perth, WA, Australia
32. On Site Laboratory Services, Bendigo, VIC, Australia
33. Ontario Geological Survey, Sudbury, Ontario, Canada
34. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
35. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
36. Ravenswood Gold, Ravenswood, QLD, Australia
37. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
38. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
39. SGS Australia Mineral Services, Perth, WA, Australia
40. SGS Canada Inc., Vancouver, BC, Canada
41. SGS del Peru, Lima, Peru
42. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
43. SGS Tarkwa, Tarkwa, Western Region, Ghana
44. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
45. Skyline Assayers & Laboratories, Tucson, Arizona, USA
46. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

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

Figure 1. Au by Pb Fire Assay in OREAS 231b

SPC.1710.RR1.OREAS 231b.2.Fire Assay.Au.Lab.230705.222410.SN

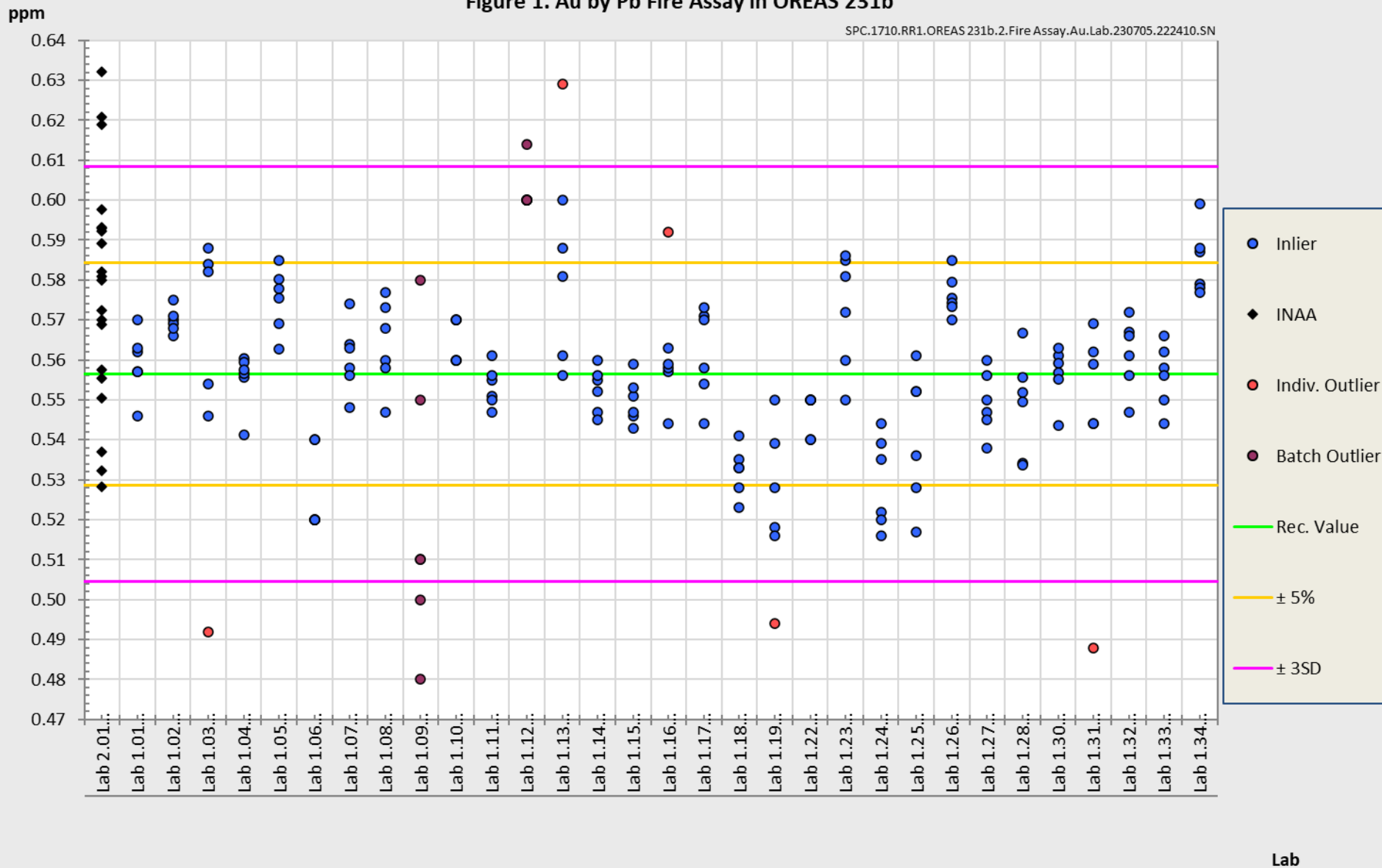


Figure 2. Au by Aqua Regia Digestion (sample weights 10-50g) in OREAS 231b

SPC.1710.RR1.OREAS 231b.2.AR Digest 10-50g.Au.Lab.230705.222753.SN

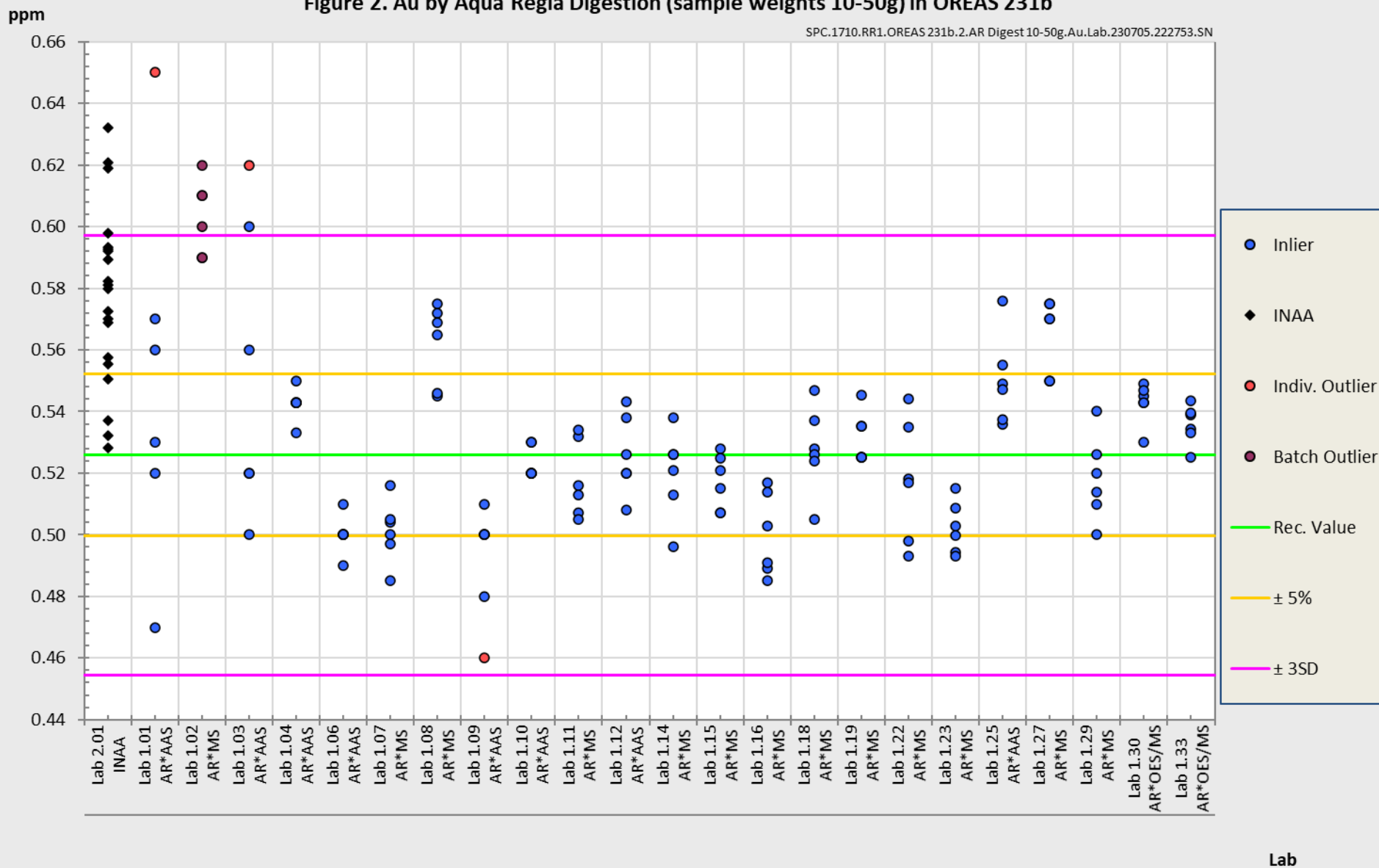


Figure 3. Au by Cyanide Leach in OREAS 231b

SPC.1710.RR1.OREAS 231b.2.CNL.Au.Lab.230705.222837.SN

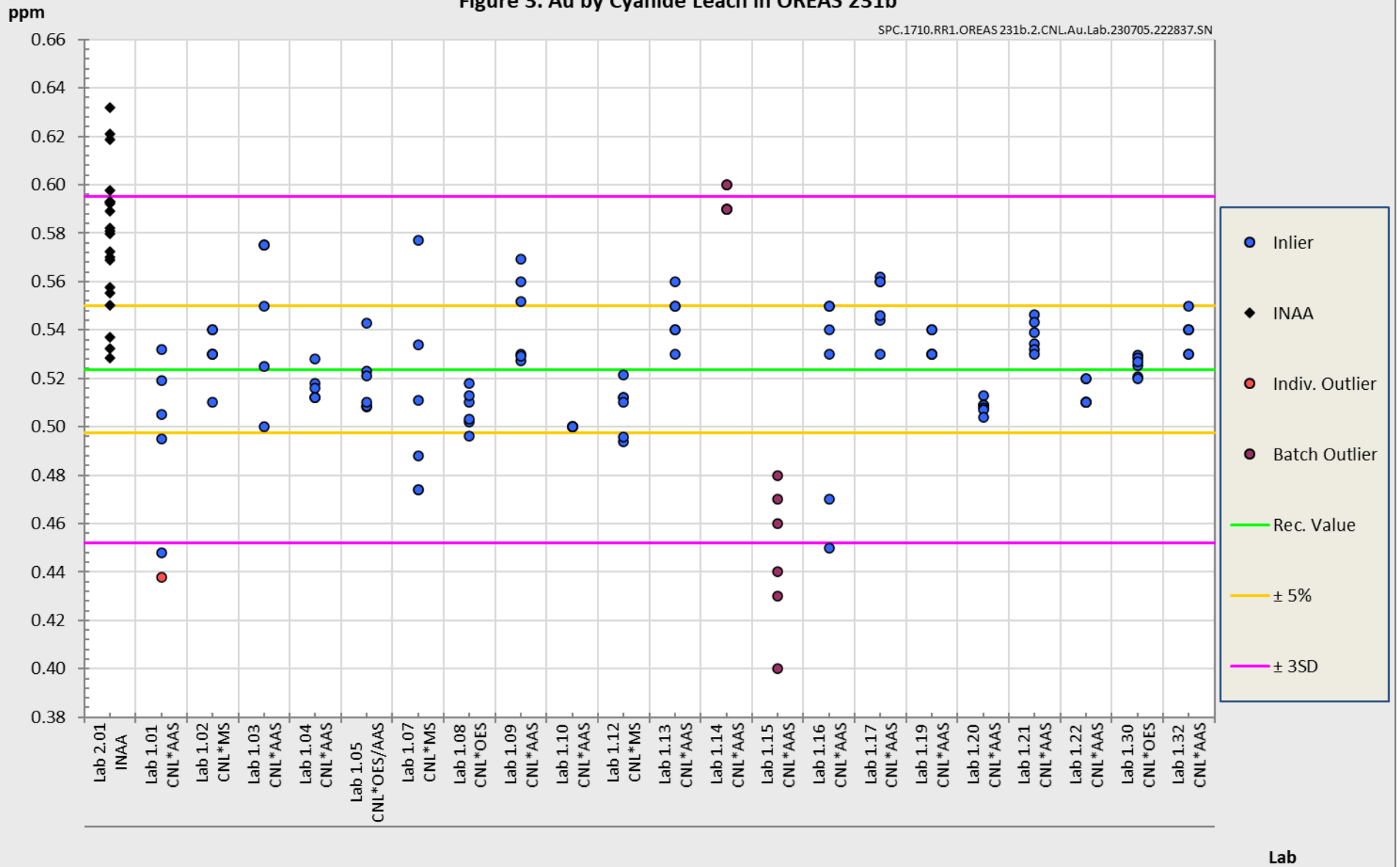
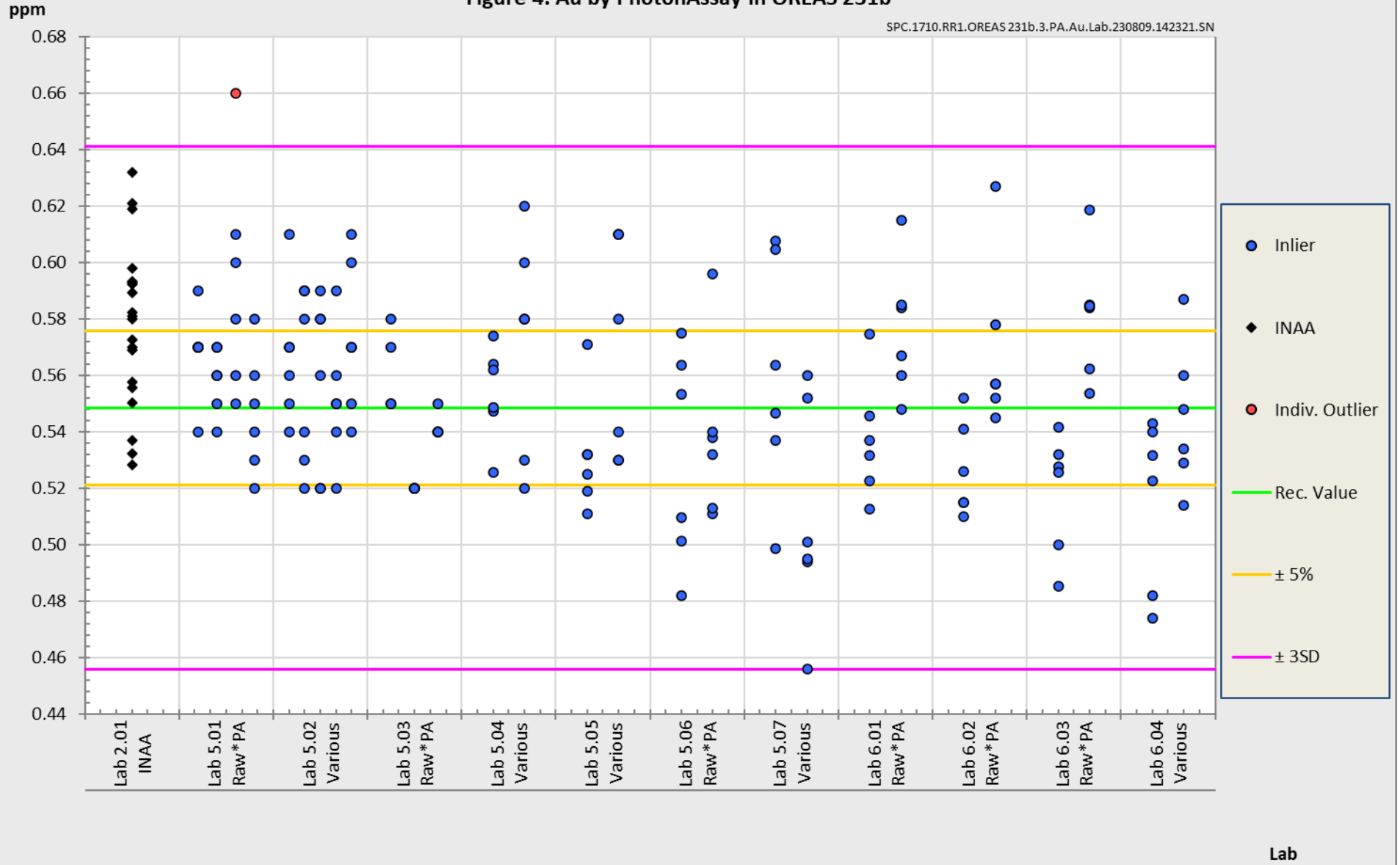


Figure 4. Au by PhotonAssay in OREAS 231b

SPC.1710.RR1.OREAS 231b.3.PA.Au.Lab.230809.142321.SN



## PREPARER AND SUPPLIER

Certified reference material OREAS 231b is prepared, certified and supplied by:



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## METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified values are metrologically traceable to the international measurement scale (SI) of mass (either as a % mass fraction or as milligrams per kilogram (mg/kg)) [15]. In line with popular use, all data within tables in this certificate are expressed as the mass fraction in either weight percent (wt.%) or parts per million (ppm).

The analytical samples sent to participating laboratories were selected in a manner to be representative of the entire prepared batch of CRM. This representativeness 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. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [10], each analytical data set received from the participating laboratories has been validated by its assayer through the inclusion of internal reference materials and QC checks during and post analysis.

The participating laboratories were chosen on the basis of their competence (from past performance in interlaboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. The operationally defined measurands characterised in this certificate are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016 [8], 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 procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 33405:2024, 9.2.4c) [5].**”* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

## COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (fusion/digestion) 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 the field samples being analysed.

## INTENDED USE

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

OREAS 231b is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 and 2 in geological samples;
- For the verification of analytical methods for analytes reported in Tables 1 and 2;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 and 2. When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. Users can determine an approximation of the standard uncertainty by calculating one fourth of the width of the Expanded Uncertainty interval given in this certificate (Expanded Uncertainty intervals are provided in Tables 1 and 2).

## MINIMUM SAMPLE SIZE

To relate analytical determinations to the values in this certificate, the minimum mass of sample used should match the typical mass that the laboratories used in the interlaboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Au by fire assay:  $\geq 15$  g;
- Au by PhotonAssay:  $\sim 350$  g;
- Au by aqua regia digestion:  $\geq 10$  g;
- Au by cyanide leach:  $\geq 5$  g;
- 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25$  g;
- Aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5$  g.

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 231b remains valid, within the specified measurement uncertainties, until July 2033, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

Store in a clean and cool dry place away from direct sunlight.

Long-term stability will be monitored at appropriate intervals and purchasers notified if any changes are observed. The period of validity may well be indefinite and will be reassessed prior to expiry with the aim of extending the validity if possible.

### Single-use sachets

Following analysis, it is the manufacturer's expectation that any remaining material is discarded unless the sachet is promptly resealed. It is the user's responsibility to prevent contamination and minimise exposure to the atmosphere.

### Repeat-use packaging (e.g., 500 g unit)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 231b contains a non-hygroscopic\* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (0.26 wt.% S).

\*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorbed moisture (weakly held H<sub>2</sub>O- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2mm thick) layer and left exposed for a period of 2 hours.

## INSTRUCTIONS FOR HANDLING & CORRECT USE

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [13].

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised.

### 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 interlaboratory 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.

The performance gates shown in Table 6 are intended only to be used as a preliminary guide as to what a laboratory may be able to achieve. Over a period of time monitoring your own laboratory's data for this CRM, SD's should be calculated directly from your own laboratory's process. This will enable you to establish more specific performance gates that are fit for purpose for your application as well as the ability to monitor bias. If your long-term trend analysis shows an average value that is within the 95 % expanded uncertainty then generally there is no cause for concern in regard to bias.

### **For use with the aqua regia digestion method**

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. This method is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions and 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.

## **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.

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## **DOCUMENT HISTORY**

<b>Revision No.</b>	<b>Date</b>	<b>Changes applied</b>
1	2 <sup>nd</sup> December, 2024	Added Au by PhotonAssay certification (Table 2).
0	22 <sup>nd</sup> August, 2023	First publication.

## QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034.



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

A handwritten signature in blue ink, appearing to read 'S.H.', is positioned above the name of the certifying officer.

2<sup>nd</sup> December, 2024

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

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