

**CERTIFICATE OF ANALYSIS FOR**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 240b**

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

**Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 240b.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	5.65	5.60	5.70	5.62*	5.67*

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 240b.**

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion (sample weights 10-50g)</b>					
Au, Gold (ppm)	5.53	5.45	5.60	5.50*	5.55*
<b>Cyanide Leach</b>					
Au, Gold (ppm)	5.44	5.37	5.50	5.43*	5.44*
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	1.40	1.34	1.46	1.34	1.46
Al, Aluminium (wt.%)	6.49	6.31	6.68	6.34	6.65
As, Arsenic (ppm)	98	94	101	94	102
Ba, Barium (ppm)	324	315	333	313	335
Be, Beryllium (ppm)	0.47	0.43	0.51	0.45	0.49
Bi, Bismuth (ppm)	0.073	0.060	0.086	IND	IND
Ca, Calcium (wt.%)	5.66	5.50	5.83	5.57	5.76
Cd, Cadmium (ppm)	0.89	0.82	0.95	0.85	0.93
Ce, Cerium (ppm)	14.9	14.2	15.6	14.4	15.4
Co, Cobalt (ppm)	40.3	39.1	41.5	39.0	41.6
Cr, Chromium (ppm)	146	138	154	141	150
Cs, Caesium (ppm)	1.38	1.31	1.45	1.34	1.42
Cu, Copper (ppm)	164	159	170	160	168
Dy, Dysprosium (ppm)	3.35	3.05	3.66	3.24	3.47
Er, Erbium (ppm)	2.03	1.83	2.23	1.89	2.17
Eu, Europium (ppm)	0.87	0.80	0.94	0.82	0.92
Fe, Iron (wt.%)	7.31	7.12	7.50	7.17	7.46
Ga, Gallium (ppm)	14.8	14.1	15.5	14.3	15.4
Gd, Gadolinium (ppm)	2.89	2.60	3.17	2.73	3.04
Ge, Germanium (ppm)	0.095	0.069	0.121	IND	IND
Hf, Hafnium (ppm)	1.66	1.53	1.79	1.58	1.75
Ho, Holmium (ppm)	0.67	0.59	0.75	0.64	0.70
In, Indium (ppm)	0.075	0.068	0.083	0.067	0.084
K, Potassium (wt.%)	0.730	0.707	0.752	0.706	0.753
La, Lanthanum (ppm)	6.60	6.29	6.92	6.40	6.81
Li, Lithium (ppm)	13.5	12.9	14.0	13.1	13.8
Lu, Lutetium (ppm)	0.29	0.26	0.33	0.28	0.31
Mg, Magnesium (wt.%)	3.44	3.36	3.52	3.36	3.51
Mn, Manganese (wt.%)	0.140	0.136	0.144	0.137	0.142
Mo, Molybdenum (ppm)	2.90	2.75	3.05	2.77	3.03

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>					
Na, Sodium (wt.%)	1.87	1.83	1.91	1.81	1.93
Nb, Niobium (ppm)	3.54	3.32	3.76	3.36	3.72
Nd, Neodymium (ppm)	8.74	8.15	9.33	8.40	9.08
Ni, Nickel (ppm)	94	91	98	92	96
P, Phosphorus (wt.%)	0.043	0.042	0.044	0.042	0.044
Pb, Lead (ppm)	40.5	38.5	42.6	39.1	42.0
Pr, Praseodymium (ppm)	1.91	1.77	2.06	1.83	1.99
Rb, Rubidium (ppm)	21.1	20.1	22.0	20.3	21.8
Re, Rhenium (ppm)	0.002	0.002	0.003	IND	IND
S, Sulphur (wt.%)	0.549	0.535	0.563	0.537	0.562
Sb, Antimony (ppm)	2.14	2.01	2.27	2.00	2.27
Sc, Scandium (ppm)	33.3	32.1	34.5	32.3	34.4
Sm, Samarium (ppm)	2.42	2.19	2.66	2.28	2.57
Sn, Tin (ppm)	1.32	1.20	1.43	1.11	1.52
Sr, Strontium (ppm)	196	190	202	192	201
Ta, Tantalum (ppm)	0.24	0.22	0.27	0.22	0.26
Tb, Terbium (ppm)	0.50	0.46	0.55	0.47	0.53
Te, Tellurium (ppm)	0.15	0.12	0.18	IND	IND
Th, Thorium (ppm)	1.45	1.34	1.56	1.39	1.51
Ti, Titanium (wt.%)	0.543	0.529	0.558	0.528	0.559
Tl, Thallium (ppm)	0.34	0.32	0.36	0.32	0.36
Tm, Thulium (ppm)	0.29	0.26	0.32	0.27	0.32
U, Uranium (ppm)	0.42	0.39	0.46	0.40	0.45
V, Vanadium (ppm)	232	225	238	227	236
W, Tungsten (ppm)	47.1	45.0	49.3	45.1	49.1
Y, Yttrium (ppm)	18.3	17.5	19.0	17.7	18.8
Yb, Ytterbium (ppm)	1.87	1.70	2.03	1.76	1.98
Zn, Zinc (ppm)	173	168	179	169	178
Zr, Zirconium (ppm)	53	50	56	51	56
<b>Aqua Regia Digestion</b>					
Ag, Silver (ppm)	1.39	1.34	1.44	1.35	1.43
Al, Aluminium (wt.%)	2.99	2.87	3.10	2.92	3.06
As, Arsenic (ppm)	96	93	99	94	98
B, Boron (ppm)	22.6	17.3	27.9	20.7	24.4
Ba, Barium (ppm)	51	48	53	50	52
Be, Beryllium (ppm)	0.25	0.22	0.28	0.23	0.27
Bi, Bismuth (ppm)	0.067	0.059	0.076	IND	IND

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>					
Ca, Calcium (wt.%)	2.24	2.12	2.36	2.19	2.29
Cd, Cadmium (ppm)	0.86	0.81	0.91	0.83	0.89
Ce, Cerium (ppm)	11.7	11.2	12.2	11.4	12.0
Co, Cobalt (ppm)	29.8	28.7	30.9	29.1	30.5
Cr, Chromium (ppm)	34.7	33.1	36.3	33.6	35.7
Cs, Caesium (ppm)	1.00	0.96	1.03	0.96	1.03
Cu, Copper (ppm)	162	158	167	159	166
Dy, Dysprosium (ppm)	1.93	1.60	2.26	1.85	2.02
Er, Erbium (ppm)	1.11	0.93	1.30	1.06	1.17
Eu, Europium (ppm)	0.45	0.37	0.53	0.43	0.47
Fe, Iron (wt.%)	5.40	5.29	5.51	5.29	5.51
Ga, Gallium (ppm)	9.33	8.84	9.81	9.12	9.53
Gd, Gadolinium (ppm)	1.81	1.56	2.05	1.74	1.88
Ge, Germanium (ppm)	0.094	0.067	0.121	IND	IND
Hf, Hafnium (ppm)	0.43	0.39	0.48	0.41	0.46
Ho, Holmium (ppm)	0.37	0.33	0.41	0.36	0.38
In, Indium (ppm)	0.038	0.034	0.042	0.035	0.041
K, Potassium (wt.%)	0.209	0.201	0.218	0.203	0.216
La, Lanthanum (ppm)	5.49	5.29	5.68	5.36	5.61
Li, Lithium (ppm)	10.6	10.1	11.0	10.3	10.8
Lu, Lutetium (ppm)	0.13	0.10	0.15	IND	IND
Mg, Magnesium (wt.%)	1.70	1.65	1.75	1.66	1.73
Mn, Manganese (wt.%)	0.079	0.076	0.082	0.077	0.080
Mo, Molybdenum (ppm)	2.79	2.67	2.90	2.67	2.90
Na, Sodium (wt.%)	0.328	0.315	0.342	0.316	0.340
Nd, Neodymium (ppm)	6.26	5.55	6.96	6.09	6.43
Ni, Nickel (ppm)	68	66	70	67	69
P, Phosphorus (wt.%)	0.042	0.040	0.043	0.041	0.042
Pb, Lead (ppm)	40.7	39.2	42.1	39.6	41.8
Pr, Praseodymium (ppm)	1.39	1.23	1.56	1.35	1.43
Rb, Rubidium (ppm)	8.91	8.52	9.30	8.67	9.15
Re, Rhenium (ppm)	0.002	0.001	0.003	IND	IND
S, Sulphur (wt.%)	0.545	0.533	0.558	0.534	0.557
Sb, Antimony (ppm)	1.21	1.01	1.41	1.13	1.29
Sc, Scandium (ppm)	5.21	4.83	5.59	5.06	5.36
Sm, Samarium (ppm)	1.60	1.41	1.78	1.54	1.65
Sn, Tin (ppm)	0.75	0.68	0.81	0.68	0.82

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>					
Sr, Strontium (ppm)	43.3	40.2	46.3	41.5	45.0
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.29	0.27	0.31	0.28	0.30
Te, Tellurium (ppm)	0.14	0.12	0.15	IND	IND
Th, Thorium (ppm)	1.14	1.06	1.22	1.09	1.19
Ti, Titanium (wt.%)	0.282	0.258	0.307	0.272	0.292
Tl, Thallium (ppm)	0.18	0.17	0.19	0.13	0.23
Tm, Thulium (ppm)	0.14	0.11	0.17	IND	IND
U, Uranium (ppm)	0.28	0.27	0.30	0.27	0.30
V, Vanadium (ppm)	116	109	123	113	119
W, Tungsten (ppm)	33.7	31.2	36.3	32.8	34.6
Y, Yttrium (ppm)	10.0	9.6	10.4	9.8	10.2
Yb, Ytterbium (ppm)	0.91	0.79	1.02	0.80	1.01
Zn, Zinc (ppm)	158	153	163	156	161

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 240b.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppb	14.1	Pt	ppb	8.09			
<b>4-Acid Digestion</b>								
B	ppm	33.8	Hg	ppm	0.098	Se	ppm	1.05
<b>Aqua Regia Digestion</b>								
Hg	ppm	0.062	Pd	ppb	12.5	Si	wt.%	0.089
Ir	ppm	< 0.003	Pt	ppb	6.80			
Nb	ppm	0.11	Se	ppm	0.73			
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt.%	12.32	MgO	wt.%	5.86	S	wt.%	0.457
CaO	wt.%	8.16	MnO	wt.%	0.169	SiO <sub>2</sub>	wt.%	54.61
Fe <sub>2</sub> O <sub>3</sub>	wt.%	10.83	Na <sub>2</sub> O	wt.%	2.50	TiO <sub>2</sub>	wt.%	0.957
K <sub>2</sub> O	wt.%	0.853	P <sub>2</sub> O <sub>5</sub>	wt.%	0.095			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt.%	3.36						
<b>Infrared Combustion</b>								
C	wt.%	0.190	S	wt.%	0.515			

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	1.30	Hf	ppm	2.02	Sm	ppm	2.54
As	ppm	94	Ho	ppm	0.78	Sn	ppm	1.20
Ba	ppm	314	In	ppm	0.075	Sr	ppm	188
Be	ppm	0.70	La	ppm	6.69	Ta	ppm	0.23
Bi	ppm	0.070	Lu	ppm	0.32	Tb	ppm	0.54
Cd	ppm	0.95	Mn	wt.%	0.148	Te	ppm	< 0.2
Ce	ppm	14.4	Mo	ppm	2.90	Th	ppm	1.46
Co	ppm	41.2	Nb	ppm	3.54	Ti	wt.%	0.566
Cr	ppm	165	Nd	ppm	9.03	Tl	ppm	0.15
Cs	ppm	1.32	Ni	ppm	98	Tm	ppm	0.33
Cu	ppm	169	Pb	ppm	40.5	U	ppm	0.39
Dy	ppm	3.45	Pr	ppm	1.99	V	ppm	241
Er	ppm	2.20	Rb	ppm	20.0	W	ppm	46.3
Eu	ppm	0.88	Re	ppm	0.020	Y	ppm	19.3
Ga	ppm	14.3	Sb	ppm	2.20	Yb	ppm	2.08
Gd	ppm	3.11	Sc	ppm	34.1	Zn	ppm	173

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 240b-DataPack.1.0.230823\_205935.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  $\pm 3SD$  (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 240b was prepared from a blend of gold-bearing 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 volcanoclastics 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.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 240b 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 microns;
- Crushing and multi stage milling of the greenstone to >98% minus 75 microns;
- Blending in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 60g units sealed in laminated foil pouches and 500g units in plastic jars.



## PHYSICAL PROPERTIES

OREAS 240b 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 240b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
775	0.56	N7	Light 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 240b based on semi-quantitative XRD analysis.**

Mineral / Mineral Group	% (mass ratio)
Stilpnomelane	< 1
Chlorite	13
Biotite	5
Muscovite	4
Calcic amphibole	3
Clinopyroxene	9
Epidote	4
Prehnite	1
Plagioclase	33
K-feldspar	3
Quartz	25
Gypsum	< 1

## 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 (15-50g charge weight) with AAS (24 laboratories), ICP-OES (6 laboratories) finish and ICP-MS (1 laboratory) finish;
- 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: 5g (2 laboratories by AAS finish), 20g (1 laboratory by AAS finish), 30g (4 laboratories by AAS finish and 1 laboratory by ICP-OES finish), 50g (1 laboratory by AAS, 1 laboratory by ICP-OES finish and 2 laboratories by ICP-

- MS finish), 60g (1 laboratory by AAS finish) and 200g (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 85mg 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 3kg 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 3kg 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 240b.**

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>Pb Fire Assay</b>											
Au, ppm	5.65	0.143	5.36	5.93	5.22	6.08	2.53%	5.05%	7.58%	5.37	5.93
<b>Aqua Regia Digestion (sample weights 10-50g)</b>											
Au, ppm	5.53	0.171	5.18	5.87	5.01	6.04	3.10%	6.19%	9.29%	5.25	5.80
<b>Cyanide Leach</b>											
Au, ppm	5.44	0.143	5.15	5.72	5.01	5.86	2.63%	5.27%	7.90%	5.16	5.71
<b>4-Acid Digestion</b>											
Ag, ppm	1.40	0.049	1.30	1.50	1.25	1.55	3.47%	6.94%	10.40%	1.33	1.47
Al, wt. %	6.49	0.273	5.95	7.04	5.68	7.31	4.20%	8.40%	12.60%	6.17	6.82
As, ppm	98	4.5	89	107	84	112	4.63%	9.26%	13.89%	93	103
Ba, ppm	324	10	304	344	294	354	3.08%	6.16%	9.24%	308	340
Be, ppm	0.47	0.05	0.37	0.57	0.33	0.61	10.20%	20.41%	30.61%	0.45	0.49
Bi, ppm	0.073	0.010	0.053	0.093	0.042	0.103	13.93%	27.86%	41.79%	0.069	0.077
Ca, wt. %	5.66	0.220	5.22	6.10	5.00	6.32	3.88%	7.76%	11.64%	5.38	5.95
Cd, ppm	0.89	0.050	0.79	0.99	0.73	1.04	5.69%	11.38%	17.07%	0.84	0.93
Ce, ppm	14.9	0.84	13.2	16.6	12.4	17.4	5.66%	11.32%	16.99%	14.1	15.6
Co, ppm	40.3	1.68	36.9	43.7	35.2	45.3	4.17%	8.35%	12.52%	38.3	42.3
Cr, ppm	146	15	116	176	101	191	10.30%	20.61%	30.91%	138	153
Cs, ppm	1.38	0.083	1.21	1.54	1.13	1.63	6.03%	12.05%	18.08%	1.31	1.45
Cu, ppm	164	6	153	176	147	181	3.42%	6.85%	10.27%	156	173
Dy, ppm	3.35	0.109	3.14	3.57	3.03	3.68	3.25%	6.51%	9.76%	3.19	3.52
Er, ppm	2.03	0.128	1.77	2.29	1.65	2.41	6.29%	12.58%	18.87%	1.93	2.13
Eu, ppm	0.87	0.054	0.76	0.98	0.71	1.03	6.18%	12.35%	18.53%	0.83	0.91
Fe, wt. %	7.31	0.335	6.64	7.98	6.31	8.32	4.58%	9.16%	13.74%	6.95	7.68
Ga, ppm	14.8	0.84	13.2	16.5	12.3	17.3	5.63%	11.26%	16.90%	14.1	15.6
Gd, ppm	2.89	0.255	2.38	3.40	2.12	3.65	8.83%	17.66%	26.49%	2.74	3.03
Ge, ppm	0.095	0.031	0.033	0.158	0.002	0.189	32.78%	65.55%	98.33%	0.091	0.100
Hf, ppm	1.66	0.153	1.36	1.97	1.20	2.12	9.20%	18.39%	27.59%	1.58	1.75
Ho, ppm	0.67	0.038	0.59	0.75	0.56	0.78	5.64%	11.27%	16.91%	0.64	0.70
In, ppm	0.075	0.006	0.063	0.088	0.057	0.094	8.11%	16.22%	24.33%	0.072	0.079
K, wt. %	0.730	0.030	0.669	0.790	0.639	0.820	4.15%	8.29%	12.44%	0.693	0.766
La, ppm	6.60	0.387	5.83	7.38	5.44	7.77	5.86%	11.73%	17.59%	6.27	6.94
Li, ppm	13.5	0.92	11.6	15.3	10.7	16.2	6.81%	13.62%	20.43%	12.8	14.1
Lu, ppm	0.29	0.025	0.24	0.34	0.22	0.37	8.46%	16.92%	25.38%	0.28	0.31
Mg, wt. %	3.44	0.152	3.13	3.74	2.98	3.89	4.41%	8.83%	13.24%	3.27	3.61
Mn, wt. %	0.140	0.004	0.132	0.147	0.128	0.151	2.82%	5.64%	8.45%	0.133	0.147
Mo, ppm	2.90	0.252	2.40	3.41	2.15	3.66	8.69%	17.38%	26.07%	2.76	3.05
Na, wt. %	1.87	0.061	1.75	1.99	1.69	2.05	3.27%	6.54%	9.81%	1.78	1.96
Nb, ppm	3.54	0.226	3.09	3.99	2.86	4.22	6.38%	12.76%	19.14%	3.36	3.72
Nd, ppm	8.74	0.361	8.02	9.46	7.66	9.82	4.13%	8.26%	12.38%	8.30	9.18
Ni, ppm	94	3.8	87	102	83	106	4.08%	8.16%	12.23%	90	99

SI unit equivalents: ppm (parts per million;  $1 \times 10^6$ )  $\equiv$  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>											
P, wt.%	0.043	0.001	0.041	0.045	0.040	0.046	2.34%	4.68%	7.03%	0.041	0.045
Pb, ppm	40.5	2.85	34.8	46.2	32.0	49.1	7.03%	14.06%	21.08%	38.5	42.6
Pr, ppm	1.91	0.093	1.73	2.10	1.63	2.19	4.85%	9.71%	14.56%	1.82	2.01
Rb, ppm	21.1	1.10	18.9	23.3	17.8	24.4	5.21%	10.43%	15.64%	20.0	22.1
Re, ppm	0.002	0.001	0.001	0.004	0.001	0.004	26.31%	52.62%	78.93%	0.002	0.003
S, wt.%	0.549	0.015	0.519	0.580	0.504	0.595	2.76%	5.52%	8.28%	0.522	0.577
Sb, ppm	2.14	0.169	1.80	2.48	1.63	2.65	7.88%	15.76%	23.64%	2.03	2.25
Sc, ppm	33.3	1.43	30.5	36.2	29.0	37.6	4.30%	8.60%	12.89%	31.7	35.0
Sm, ppm	2.42	0.133	2.16	2.69	2.02	2.82	5.49%	10.98%	16.47%	2.30	2.55
Sn, ppm	1.32	0.15	1.01	1.62	0.86	1.77	11.59%	23.19%	34.78%	1.25	1.38
Sr, ppm	196	9	179	214	170	223	4.50%	9.00%	13.50%	186	206
Ta, ppm	0.24	0.04	0.17	0.31	0.14	0.35	14.47%	28.94%	43.41%	0.23	0.25
Tb, ppm	0.50	0.039	0.43	0.58	0.39	0.62	7.67%	15.34%	23.01%	0.48	0.53
Te, ppm	0.15	0.03	0.09	0.21	0.06	0.24	19.23%	38.45%	57.68%	0.15	0.16
Th, ppm	1.45	0.129	1.19	1.71	1.06	1.84	8.86%	17.73%	26.59%	1.38	1.52
Ti, wt.%	0.543	0.015	0.514	0.573	0.499	0.587	2.70%	5.40%	8.09%	0.516	0.571
Tl, ppm	0.34	0.022	0.30	0.39	0.28	0.41	6.33%	12.66%	18.99%	0.33	0.36
Tm, ppm	0.29	0.019	0.25	0.33	0.23	0.35	6.62%	13.23%	19.85%	0.28	0.31
U, ppm	0.42	0.041	0.34	0.51	0.30	0.55	9.75%	19.49%	29.24%	0.40	0.44
V, ppm	232	7	217	246	210	253	3.05%	6.11%	9.16%	220	243
W, ppm	47.1	3.27	40.6	53.7	37.3	56.9	6.95%	13.89%	20.84%	44.8	49.5
Y, ppm	18.3	1.00	16.3	20.3	15.3	21.3	5.50%	11.00%	16.50%	17.4	19.2
Yb, ppm	1.87	0.139	1.59	2.14	1.45	2.28	7.46%	14.92%	22.38%	1.77	1.96
Zn, ppm	173	8	158	189	150	197	4.54%	9.08%	13.62%	165	182
Zr, ppm	53	4.6	44	62	39	67	8.74%	17.47%	26.21%	50	56
<b>Aqua Regia Digestion</b>											
Ag, ppm	1.39	0.056	1.28	1.50	1.22	1.56	4.04%	8.09%	12.13%	1.32	1.46
Al, wt.%	2.99	0.222	2.55	3.43	2.32	3.66	7.43%	14.86%	22.29%	2.84	3.14
As, ppm	96	4.5	87	105	82	109	4.68%	9.37%	14.05%	91	100
B, ppm	22.6	7.2	8.1	37.0	0.9	44.2	32.01%	64.02%	96.03%	21.4	23.7
Ba, ppm	51	4.5	42	60	37	64	8.87%	17.74%	26.61%	48	53
Be, ppm	0.25	0.04	0.16	0.33	0.12	0.37	16.81%	33.62%	50.44%	0.24	0.26
Bi, ppm	0.067	0.010	0.047	0.087	0.037	0.097	14.82%	29.64%	44.45%	0.064	0.071
Ca, wt.%	2.24	0.26	1.72	2.76	1.46	3.02	11.62%	23.25%	34.87%	2.13	2.35
Cd, ppm	0.86	0.056	0.75	0.97	0.69	1.03	6.55%	13.11%	19.66%	0.82	0.90
Ce, ppm	11.7	0.54	10.6	12.8	10.1	13.3	4.61%	9.21%	13.82%	11.1	12.3
Co, ppm	29.8	1.54	26.7	32.9	25.2	34.4	5.15%	10.30%	15.45%	28.3	31.3
Cr, ppm	34.7	2.87	28.9	40.4	26.1	43.3	8.27%	16.54%	24.82%	32.9	36.4
Cs, ppm	1.00	0.033	0.93	1.06	0.90	1.10	3.35%	6.70%	10.05%	0.95	1.05
Cu, ppm	162	5	152	173	147	178	3.09%	6.19%	9.28%	154	171
Dy, ppm	1.93	0.27	1.39	2.47	1.13	2.74	13.92%	27.84%	41.76%	1.84	2.03
Er, ppm	1.11	0.16	0.79	1.44	0.63	1.60	14.41%	28.82%	43.24%	1.06	1.17
Eu, ppm	0.45	0.07	0.31	0.59	0.23	0.67	16.02%	32.05%	48.07%	0.43	0.47

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>											
Fe, wt.%	5.40	0.151	5.10	5.70	4.94	5.85	2.81%	5.61%	8.42%	5.13	5.67
Ga, ppm	9.33	0.821	7.69	10.97	6.87	11.79	8.80%	17.60%	26.41%	8.86	9.80
Gd, ppm	1.81	0.19	1.42	2.20	1.22	2.39	10.76%	21.51%	32.27%	1.72	1.90
Ge, ppm	0.094	0.029	0.036	0.152	0.007	0.181	30.68%	61.37%	92.05%	0.089	0.099
Hf, ppm	0.43	0.06	0.32	0.55	0.26	0.61	13.64%	27.27%	40.91%	0.41	0.46
Ho, ppm	0.37	0.031	0.31	0.43	0.28	0.46	8.21%	16.42%	24.63%	0.35	0.39
In, ppm	0.038	0.003	0.031	0.045	0.028	0.049	8.98%	17.96%	26.94%	0.036	0.040
K, wt.%	0.209	0.013	0.184	0.235	0.171	0.248	6.13%	12.25%	18.38%	0.199	0.220
La, ppm	5.49	0.271	4.94	6.03	4.67	6.30	4.93%	9.86%	14.79%	5.21	5.76
Li, ppm	10.6	0.61	9.3	11.8	8.7	12.4	5.80%	11.59%	17.39%	10.0	11.1
Lu, ppm	0.13	0.03	0.07	0.19	0.04	0.22	22.02%	44.04%	66.06%	0.12	0.14
Mg, wt.%	1.70	0.085	1.53	1.87	1.44	1.95	5.01%	10.02%	15.03%	1.61	1.78
Mn, wt.%	0.079	0.005	0.068	0.090	0.063	0.095	6.78%	13.55%	20.33%	0.075	0.083
Mo, ppm	2.79	0.126	2.53	3.04	2.41	3.16	4.52%	9.04%	13.56%	2.65	2.92
Na, wt.%	0.328	0.023	0.282	0.375	0.259	0.398	7.04%	14.09%	21.13%	0.312	0.345
Nd, ppm	6.26	0.572	5.11	7.40	4.54	7.97	9.14%	18.29%	27.43%	5.94	6.57
Ni, ppm	68	3.2	61	74	58	78	4.73%	9.46%	14.19%	65	71
P, wt.%	0.042	0.002	0.038	0.045	0.036	0.047	4.40%	8.81%	13.21%	0.040	0.044
Pb, ppm	40.7	2.00	36.7	44.7	34.7	46.7	4.93%	9.85%	14.78%	38.7	42.7
Pr, ppm	1.39	0.108	1.18	1.61	1.07	1.72	7.75%	15.51%	23.26%	1.32	1.46
Rb, ppm	8.91	0.608	7.69	10.12	7.09	10.73	6.82%	13.64%	20.46%	8.46	9.35
Re, ppm	0.002	0.000	0.001	0.003	0.001	0.003	19.86%	39.71%	59.57%	0.002	0.002
S, wt.%	0.545	0.017	0.511	0.579	0.494	0.596	3.11%	6.21%	9.32%	0.518	0.573
Sb, ppm	1.21	0.42	0.38	2.04	0.00	2.46	34.32%	68.64%	102.97%	1.15	1.27
Sc, ppm	5.21	0.65	3.91	6.52	3.25	7.17	12.51%	25.03%	37.54%	4.95	5.47
Sm, ppm	1.60	0.17	1.25	1.94	1.08	2.11	10.78%	21.56%	32.34%	1.52	1.67
Sn, ppm	0.75	0.069	0.61	0.89	0.54	0.96	9.18%	18.37%	27.55%	0.71	0.79
Sr, ppm	43.3	6.0	31.2	55.4	25.1	61.4	13.98%	27.95%	41.93%	41.1	45.4
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.29	0.010	0.27	0.31	0.26	0.32	3.49%	6.97%	10.46%	0.28	0.30
Te, ppm	0.14	0.02	0.10	0.17	0.09	0.19	12.77%	25.54%	38.31%	0.13	0.15
Th, ppm	1.14	0.092	0.95	1.32	0.86	1.41	8.05%	16.09%	24.14%	1.08	1.19
Ti, wt.%	0.282	0.054	0.174	0.390	0.120	0.444	19.14%	38.28%	57.42%	0.268	0.296
Tl, ppm	0.18	0.011	0.16	0.20	0.15	0.21	5.84%	11.68%	17.52%	0.17	0.19
Tm, ppm	0.14	0.03	0.08	0.21	0.04	0.24	23.00%	46.00%	68.99%	0.13	0.15
U, ppm	0.28	0.017	0.25	0.32	0.23	0.34	6.15%	12.31%	18.46%	0.27	0.30
V, ppm	116	13	90	141	77	154	11.07%	22.15%	33.22%	110	121
W, ppm	33.7	5.5	22.8	44.6	17.4	50.1	16.16%	32.32%	48.49%	32.0	35.4
Y, ppm	10.0	0.55	8.9	11.1	8.3	11.7	5.54%	11.07%	16.61%	9.5	10.5
Yb, ppm	0.91	0.12	0.67	1.14	0.55	1.26	13.02%	26.05%	39.07%	0.86	0.95
Zn, ppm	158	8	142	174	134	183	5.15%	10.31%	15.46%	150	166
Zr, ppm	14.5	2.0	10.5	18.4	8.6	20.3	13.54%	27.09%	40.63%	13.7	15.2

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 160 and 168 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 85mg subsamples of OREAS 240b. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.14% calculated for a 30g fire assay sample (2.68% at 85mg weights) confirms the high level of gold homogeneity in OREAS 240b.

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 240b 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 240b. 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 0.788 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 240b 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 240b 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 85mg subsamples and showing the equivalent results scaled to a 30g sample mass typical of fire assay determination.**

Replicate No	Au 85mg actual	Au 30g equivalent*
1	5.905	5.841
2	5.533	5.821
3	5.898	5.840
4	5.675	5.828
5	5.826	5.836
6	5.736	5.831
7	5.648	5.827
8	5.723	5.831
9	5.906	5.841
10	5.686	5.829
11	5.842	5.837
12	5.897	5.840
13	6.195	5.856
14	5.727	5.831
15	5.943	5.843
16	5.905	5.841
17	5.994	5.845
18	5.832	5.837
19	5.777	5.834
20	6.089	5.850
Mean	5.837	5.837
Median	5.837	5.837
Std Dev.	0.157	0.008
<b>Rel.Std.Dev.</b>	<b>2.68%</b>	<b>0.14%</b>

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

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



## 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, Lima, Peru
6. ALS, Loughrea, Galway, Ireland
7. ALS, Malaga, WA, Australia
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ANSTO, Lucas Heights, NSW, Australia
11. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
12. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. CRS Laboratories Oy, Kempele, Northern Ostrobothnia, Finland
15. Gekko Assay Labs, Ballarat, VIC, Australia
16. Inspectorate (BV), Lima, Peru
17. Inspectorate Griffith India, Gandhidham, Gujarat, India
18. Intertek Genalysis, Adelaide, SA, Australia
19. Intertek Genalysis, Perth, WA, Australia
20. Intertek Tarkwa, Tarkwa, Ghana
21. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
22. Koza Gold (Ovacik Gold Mine), Bergama, Izmir, Turkey
23. MSALABS, Vancouver, BC, Canada
24. Nagrom, Perth, WA, 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 Canada Inc., Vancouver, BC, Canada
31. SGS del Peru, Lima, Peru
32. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
33. SGS Tarkwa, Tarkwa, Western Region, Ghana
34. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
35. Skyline Assayers & Laboratories, Tucson, Arizona, USA
36. 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 240b

SPC.1710.RR1.OREAS 240b.2.Fire Assay.Au.Lab.230713.110730.SS

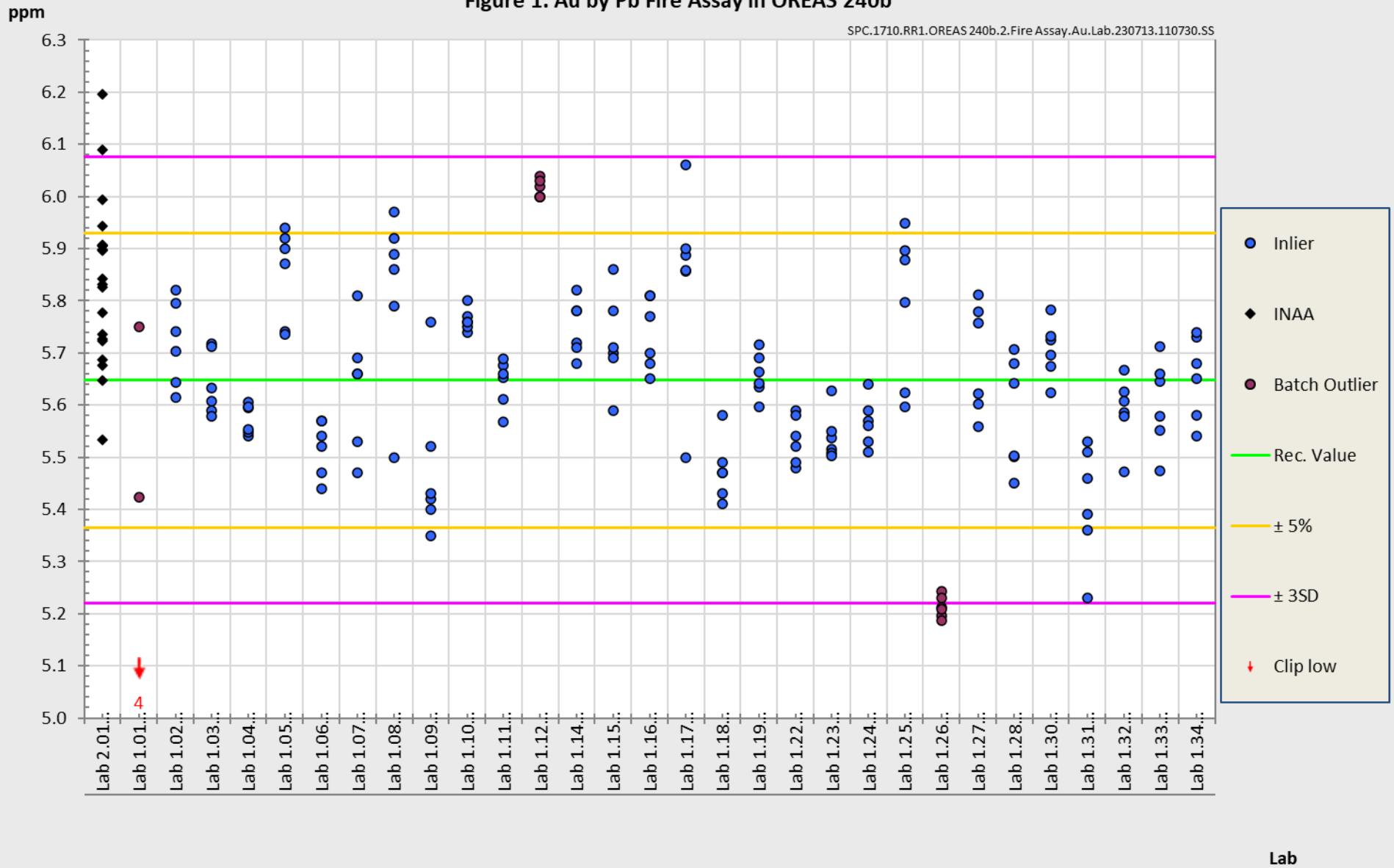


Figure 2. Au by Aqua Regia Digestion (sample weights 10-50g) in OREAS 240b

SPC.1710.RR1.OREAS 240b.2.AR Digest 10-50g.Au.Lab.230713.110830.SN

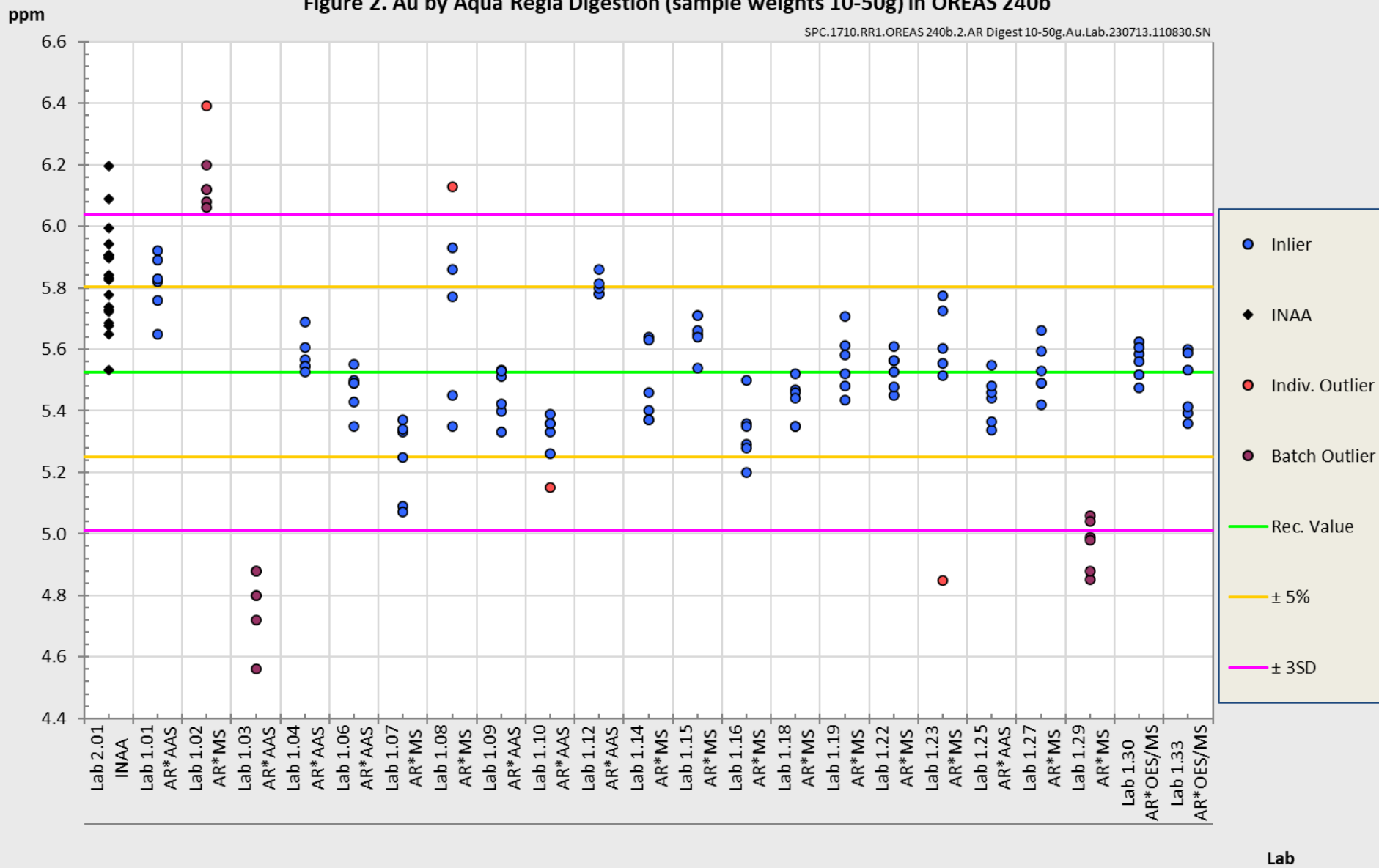
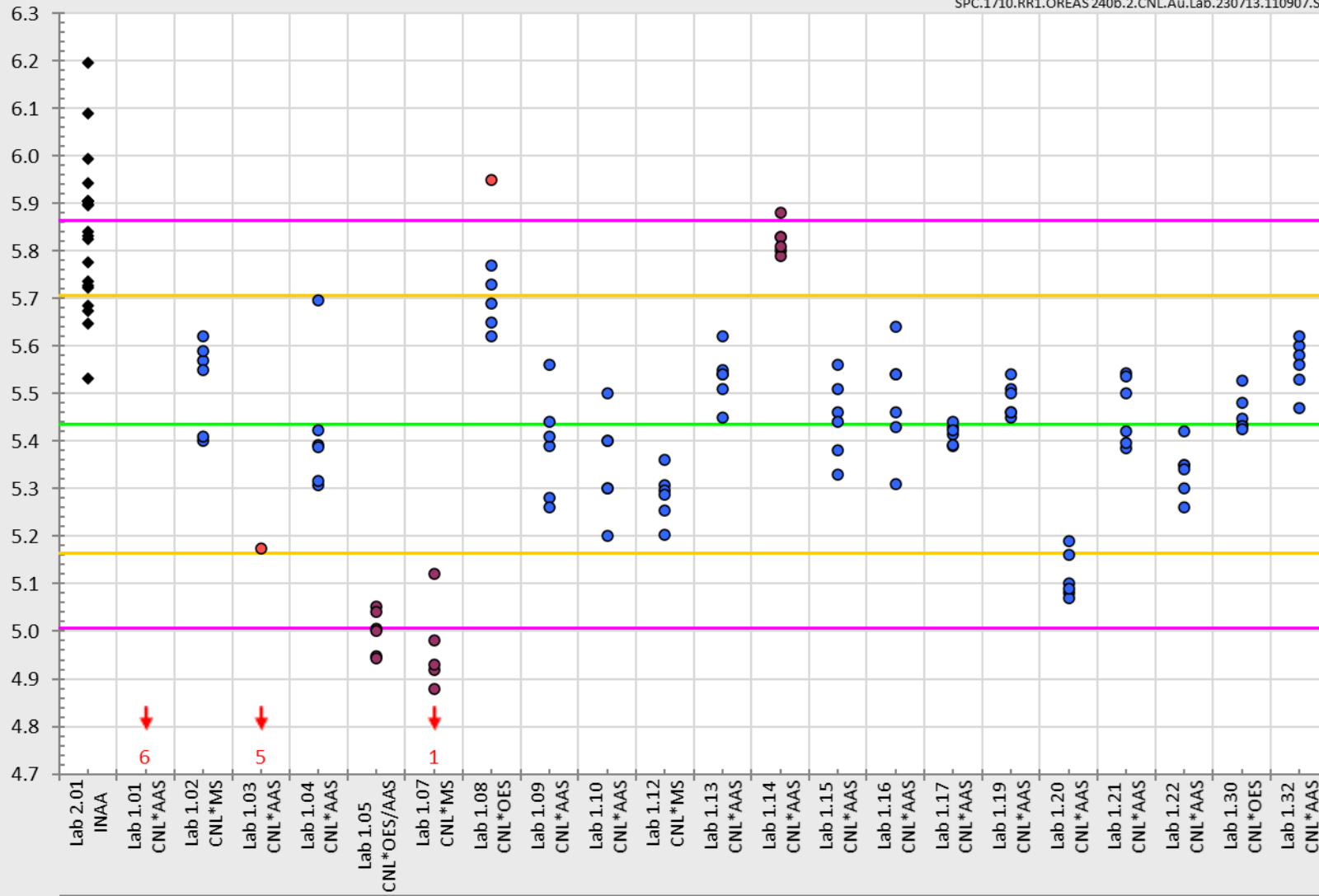


Figure 3. Au by Cyanide Leach in OREAS 240b

SPC.1710.RR1.OREAS 240b.2.CNL.Au.Lab.230713.110907.SS

ppm



- Inlier
- ◆ INAA
- Individ. Outlier
- Batch Outlier
- Rec. Value
- ± 5%
- ± 3SD
- ↓ Clip low

Lab

## PREPARER AND SUPPLIER

Certified reference material OREAS 240b 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)). 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 '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. 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, 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 35:2017, 9.2.4c).**"* 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 240b 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 240b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 240b 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:  $\geq 15\text{g}$ ;
- Au by aqua regia digestion:  $\geq 10\text{g}$ ;
- Au by cyanide leach:  $\geq 5\text{g}$ ;
- 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25\text{g}$ ;
- Aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5\text{g}$ .

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 240b 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., 500g unit)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 240b 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 (0.52 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>
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.

22<sup>nd</sup> August, 2023

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

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