

**CERTIFICATE OF ANALYSIS FOR**

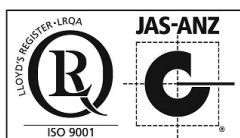
**CERTIFIED REFERENCE MATERIAL**

**OREAS 608b**

**High Sulphidation Epithermal Au-Cu-Ag Ore**  
**(Mt Carlton, Queensland, Australia)**



Accredited for compliance with ISO 17034



COA-1656-OREA608b-R0 BUP-70-10-01 Ver:2.0	19-Jun-2024
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**Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 608b.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	1.29	1.28	1.31	1.29*	1.30*

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg. Note: intervals may appear asymmetric due to rounding.

<sup>†</sup>The 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).

**Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 608b.**

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)	1.29	1.27	1.31	1.29*	1.30*
<b>Infrared Combustion</b>					
S, Sulphur (wt.%)	1.33	1.30	1.36	1.31	1.35
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	15.2	14.7	15.6	14.8	15.5
Al, Aluminium (wt.%)	7.26	7.05	7.47	7.09	7.43
As, Arsenic (ppm)	268	259	278	261	276
Be, Beryllium (ppm)	2.58	2.45	2.72	2.51	2.66
Bi, Bismuth (ppm)	15.2	14.4	16.0	14.8	15.6
Ca, Calcium (wt.%)	0.943	0.914	0.973	0.921	0.966
Cd, Cadmium (ppm)	2.91	2.76	3.06	2.79	3.03
Ce, Cerium (ppm)	79	75	84	77	81
Co, Cobalt (ppm)	2.99	2.82	3.16	2.83	3.14
Cr, Chromium (ppm)	15.9	14.0	17.8	14.6	17.2
Cs, Caesium (ppm)	5.30	5.03	5.58	5.16	5.45
Cu, Copper (wt.%)	0.121	0.118	0.124	0.119	0.123
Dy, Dysprosium (ppm)	3.14	2.96	3.33	2.99	3.30
Er, Erbium (ppm)	0.94	0.85	1.04	0.90	0.99
Eu, Europium (ppm)	1.14	1.06	1.21	1.10	1.18
Fe, Iron (wt.%)	2.33	2.25	2.41	2.28	2.39
Ga, Gallium (ppm)	23.4	22.3	24.4	22.6	24.2
Gd, Gadolinium (ppm)	4.96	4.54	5.38	4.79	5.14
Ge, Germanium (ppm)	0.18	0.11	0.25	0.16	0.20
Hf, Hafnium (ppm)	5.68	5.42	5.95	5.49	5.88
Ho, Holmium (ppm)	0.43	0.38	0.48	0.40	0.46
In, Indium (ppm)	0.29	0.28	0.31	0.28	0.31
K, Potassium (wt.%)	2.82	2.72	2.92	2.75	2.89

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 15-50g aqua regia digestion methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
La, Lanthanum (ppm)	39.4	37.0	41.9	38.3	40.5
Li, Lithium (ppm)	29.1	28.1	30.2	28.3	29.9
Lu, Lutetium (ppm)	0.078	0.064	0.091	IND	IND
Mg, Magnesium (wt.%)	0.115	0.109	0.122	0.112	0.119
Mn, Manganese (wt.%)	0.025	0.024	0.026	0.024	0.025
Mo, Molybdenum (ppm)	5.63	5.34	5.93	5.40	5.87
Na, Sodium (wt.%)	2.30	2.23	2.38	2.24	2.36
Nb, Niobium (ppm)	16.6	15.8	17.5	16.1	17.2
Nd, Neodymium (ppm)	33.9	31.4	36.5	32.9	35.0
Ni, Nickel (ppm)	4.05	3.65	4.46	3.77	4.33
P, Phosphorus (wt.%)	0.031	0.030	0.032	0.030	0.032
Pb, Lead (ppm)	377	363	391	368	386
Pr, Praseodymium (ppm)	9.19	8.41	9.97	8.83	9.55
Rb, Rubidium (ppm)	120	114	125	117	123
Re, Rhenium (ppm)	< 0.005	IND	IND	IND	IND
S, Sulphur (wt.%)	1.29	1.25	1.33	1.27	1.32
Sb, Antimony (ppm)	46.8	44.8	48.8	45.4	48.2
Sc, Scandium (ppm)	4.10	3.82	4.38	3.97	4.23
Se, Selenium (ppm)	3.75	3.02	4.49	3.49	4.02
Sm, Samarium (ppm)	6.30	5.80	6.81	6.11	6.50
Sn, Tin (ppm)	3.50	3.33	3.67	3.35	3.65
Sr, Strontium (ppm)	218	209	227	213	223
Ta, Tantalum (ppm)	1.21	1.14	1.28	1.18	1.25
Tb, Terbium (ppm)	0.67	0.60	0.74	0.64	0.70
Te, Tellurium (ppm)	2.33	2.15	2.52	2.19	2.48
Th, Thorium (ppm)	13.2	12.4	13.9	12.7	13.6
Ti, Titanium (wt.%)	0.118	0.114	0.122	0.115	0.121
Tl, Thallium (ppm)	1.05	0.99	1.10	1.01	1.08
Tm, Thulium (ppm)	0.10	0.09	0.11	IND	IND
U, Uranium (ppm)	5.01	4.83	5.19	4.83	5.19
V, Vanadium (ppm)	8.60	8.00	9.21	8.07	9.14
W, Tungsten (ppm)	2.41	2.27	2.54	2.30	2.51
Y, Yttrium (ppm)	13.1	12.6	13.6	12.8	13.4
Yb, Ytterbium (ppm)	0.59	0.53	0.66	0.57	0.62
Zn, Zinc (ppm)	651	628	675	637	666
Zr, Zirconium (ppm)	214	206	222	208	220

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</b>					
Ag, Silver (ppm)	14.8	14.4	15.3	14.6	15.1
Al, Aluminium (wt.%)	0.836	0.798	0.875	0.814	0.858
As, Arsenic (ppm)	255	246	264	251	259
B, Boron (ppm)	< 10	IND	IND	IND	IND
Ba, Barium (ppm)	341	308	373	330	352
Be, Beryllium (ppm)	0.45	0.41	0.48	0.43	0.47
Bi, Bismuth (ppm)	15.7	15.0	16.3	15.2	16.2
Ca, Calcium (wt.%)	0.551	0.536	0.566	0.539	0.562
Cd, Cadmium (ppm)	2.87	2.75	2.99	2.77	2.96
Ce, Cerium (ppm)	46.2	43.8	48.6	44.8	47.7
Co, Cobalt (ppm)	2.77	2.66	2.88	2.65	2.89
Cr, Chromium (ppm)	16.9	15.8	17.9	16.1	17.6
Cs, Caesium (ppm)	1.29	1.20	1.38	1.25	1.32
Cu, Copper (wt.%)	0.122	0.118	0.125	0.120	0.124
Dy, Dysprosium (ppm)	1.76	1.60	1.91	1.66	1.85
Er, Erbium (ppm)	0.43	0.38	0.48	0.40	0.46
Eu, Europium (ppm)	0.59	0.51	0.67	0.57	0.61
Fe, Iron (wt.%)	2.02	1.96	2.07	1.98	2.05
Ga, Gallium (ppm)	5.78	5.53	6.02	5.59	5.97
Gd, Gadolinium (ppm)	3.16	2.82	3.51	3.00	3.33
Ge, Germanium (ppm)	0.10	0.07	0.14	IND	IND
Hf, Hafnium (ppm)	1.12	1.05	1.18	1.08	1.15
Hg, Mercury (ppm)	0.15	0.13	0.16	IND	IND
Ho, Holmium (ppm)	0.20	0.18	0.23	0.19	0.21
In, Indium (ppm)	0.26	0.25	0.27	0.25	0.27
K, Potassium (wt.%)	0.240	0.230	0.251	0.231	0.250
La, Lanthanum (ppm)	23.0	21.9	24.1	22.3	23.8
Li, Lithium (ppm)	17.7	16.6	18.7	17.3	18.1
Lu, Lutetium (ppm)	0.027	0.022	0.031	IND	IND
Mg, Magnesium (wt.%)	0.078	0.076	0.081	0.076	0.081
Mn, Manganese (wt.%)	0.022	0.022	0.023	0.022	0.023
Mo, Molybdenum (ppm)	5.20	4.96	5.44	5.01	5.39
Na, Sodium (wt.%)	0.069	0.066	0.073	0.066	0.072
Nb, Niobium (ppm)	0.78	0.65	0.90	0.68	0.87
Nd, Neodymium (ppm)	20.5	18.3	22.6	19.7	21.2
Ni, Nickel (ppm)	3.96	3.66	4.26	3.77	4.15
P, Phosphorus (wt.%)	0.020	0.019	0.021	0.019	0.021

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>					
Pb, Lead (ppm)	323	314	331	318	328
Pr, Praseodymium (ppm)	5.44	4.92	5.96	5.18	5.70
Rb, Rubidium (ppm)	11.6	10.8	12.5	11.3	11.9
Re, Rhenium (ppm)	< 0.05	IND	IND	IND	IND
S, Sulphur (wt.%)	0.682	0.660	0.704	0.667	0.697
Sb, Antimony (ppm)	40.4	38.4	42.4	39.0	41.8
Sc, Scandium (ppm)	1.45	1.29	1.61	1.35	1.55
Se, Selenium (ppm)	3.43	3.13	3.72	3.20	3.66
Sm, Samarium (ppm)	3.89	3.49	4.30	3.72	4.06
Sn, Tin (ppm)	1.23	1.10	1.35	1.16	1.30
Sr, Strontium (ppm)	29.7	28.4	31.0	28.9	30.5
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.37	0.32	0.42	0.35	0.39
Te, Tellurium (ppm)	2.29	2.10	2.47	2.15	2.43
Th, Thorium (ppm)	9.23	8.83	9.64	8.88	9.58
Ti, Titanium (wt.%)	0.017	0.015	0.019	0.016	0.018
Tl, Thallium (ppm)	0.50	0.47	0.52	0.48	0.51
U, Uranium (ppm)	2.31	2.21	2.40	2.23	2.39
V, Vanadium (ppm)	2.94	2.67	3.20	2.74	3.13
W, Tungsten (ppm)	0.79	0.73	0.84	0.76	0.81
Y, Yttrium (ppm)	6.58	6.29	6.88	6.41	6.75
Yb, Ytterbium (ppm)	0.22	0.20	0.24	0.21	0.23
Zn, Zinc (ppm)	620	598	641	612	628
Zr, Zirconium (ppm)	37.7	35.4	40.1	36.8	38.7
<b>Alkaline Leach</b>					
S-(Sulphide), Sulphur as S <sup>2-</sup> (wt.%)	0.543	0.433	0.653	0.522	0.564

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 608b.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Infrared Combustion</b>								
C	wt.%	0.109	C-(Inorganic)	wt.%	0.095	C-(Organic)	wt.%	0.042
<b>Alkaline Leach</b>								
S-(Sulphate)	wt.%	0.607						
<b>Acid Leach</b>								
S-(Sulphate)	wt.%	0.393						
<b>4-Acid Digestion</b>								
B	ppm	14.0	Ba	ppm	1656	Hg	ppm	0.052
<b>Aqua Regia Digestion</b>								
Pd	ppb	15.3	Pt	ppb	6.33	Tm	ppm	0.043
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt.%	14.42	Fe <sub>2</sub> O <sub>3</sub>	wt.%	3.33	S	wt.%	1.32
As	ppm	255	K <sub>2</sub> O	wt.%	3.44	SiO <sub>2</sub>	wt.%	68.70
BaO	ppm	3176	MgO	wt.%	0.230	Sn	ppm	35.0
CaO	wt.%	1.31	MnO	wt.%	0.028	Sr	ppm	230
Cl	ppm	20.0	Na <sub>2</sub> O	wt.%	3.16	TiO <sub>2</sub>	wt.%	0.210
Co	ppm	< 10	Ni	ppm	10.0	V <sub>2</sub> O <sub>5</sub>	ppm	17.9
Cr <sub>2</sub> O <sub>3</sub>	ppm	29.2	P <sub>2</sub> O <sub>5</sub>	wt.%	0.066	Zn	ppm	635
Cu	wt.%	0.124	Pb	ppm	400	Zr	ppm	240
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt.%	3.95						
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	20.5	Hf	ppm	6.98	Sm	ppm	6.47
As	ppm	301	Ho	ppm	0.45	Sn	ppm	3.80
Ba	ppm	2675	In	ppm	0.30	Sr	ppm	224
Be	ppm	3.10	La	ppm	42.0	Ta	ppm	1.23
Bi	ppm	18.5	Lu	ppm	0.065	Tb	ppm	0.65
Cd	ppm	3.65	Mn	wt.%	0.026	Te	ppm	3.60
Ce	ppm	79	Mo	ppm	5.90	Th	ppm	13.5
Co	ppm	3.75	Nb	ppm	16.6	Ti	wt.%	0.126
Cr	ppm	18.5	Nd	ppm	34.7	Tl	ppm	1.20
Cs	ppm	5.49	Ni	ppm	6.00	Tm	ppm	0.11
Cu	wt.%	0.130	Pb	ppm	452	U	ppm	5.22
Dy	ppm	3.07	Pr	ppm	9.83	V	ppm	8.95
Er	ppm	0.96	Rb	ppm	120	W	ppm	2.25
Eu	ppm	1.08	Re	ppm	0.008	Y	ppm	13.8
Ga	ppm	23.4	Sb	ppm	56	Yb	ppm	0.58
Gd	ppm	4.88	Sc	ppm	3.65	Zn	ppm	773
Ge	ppm	1.63	Se	ppm	< 5	Zr	ppm	253
<b>3-Acid Digestion (no HF)</b>								
Ag	ppm	15.6	Gd	ppm	4.87	S	wt.%	1.33
Al <sub>2</sub> O <sub>3</sub>	wt.%	13.78	Hf	ppm	5.68	Sc	ppm	3.88
Ba	ppm	2553	Ho	ppm	0.38	Sm	ppm	6.08
Be	ppm	2.67	K <sub>2</sub> O	wt.%	3.28	Sn	ppm	3.41

SI unit equivalents: ppb (parts per billion;  $1 \times 10^{-9}$ )  $\equiv$   $\mu\text{g}/\text{kg}$ ; ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$   $\text{mg}/\text{kg}$ ; 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>3-Acid Digestion (no HF) continued</b>								
Bi	ppm	15.1	La	ppm	41.3	Sr	ppm	224
CaO	wt. %	1.31	Li	ppm	28.3	Ta	ppm	1.17
Cd	ppm	2.87	MgO	wt. %	0.190	Tb	ppm	0.59
Ce	ppm	75	MnO	wt. %	0.031	Th	ppm	12.2
Co	ppm	2.88	Mo	ppm	5.75	TiO <sub>2</sub>	wt. %	0.191
Cr	ppm	16.5	Na <sub>2</sub> O	wt. %	3.03	U	ppm	4.87
Cs	ppm	5.17	Nb	ppm	15.7	V	ppm	8.78
Cu	wt. %	0.120	Nd	ppm	32.1	W	ppm	2.58
Dy	ppm	3.04	Ni	ppm	4.80	Y	ppm	12.5
Er	ppm	0.89	P <sub>2</sub> O <sub>5</sub>	wt. %	0.069	Yb	ppm	0.57
Eu	ppm	1.64	Pb	ppm	386	Zn	ppm	653
Fe <sub>2</sub> O <sub>3</sub>	wt. %	3.11	Pr	ppm	9.02	Zr	ppm	250
Ga	ppm	21.9	Rb	ppm	117			

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.

Table 1 (all laboratories accredited to ISO 17025) and Table 2 (most laboratories accredited to ISO 17025) 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 and Table 5 provides indicative mineralogy based on semi-quantitative XRD analysis. Table 6 shows gold homogeneity via INAA with a nested ANOVA (see 'Homogeneity Evaluation' section), while Table 7 presents the performance gate intervals for all certified values.

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 608b-DataPack.1.0.240517\_171549.xlsx**). Results are also presented in scatter plots for gold by fire assay, copper by 4-acid digestion and silver by 4-acid digestion (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 608b was prepared from a blend of gold-copper-silver ores from Evolution Mining's Mount Carlton Operation in Queensland, Australia and argillic rhyodacite sourced from a quarry east of Melbourne, Australia. The mineralisation assemblage at Mount Carlton consists of pyrite, enargite/tennantite, tetrahedrite, digenite, covellite, sphalerite, galena, alunite, dickite, kaolinite and vuggy silica, hosted in advanced argillic altered rhyodacite containing sulphur-salts.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 608b was prepared in the following manner:

- Drying of ore materials to constant mass at 85° C;
- Drying of barren rhyodacite to constant mass at 105° C;
- Crushing and milling of ore materials to 100% minus 30 microns;
- Crushing and milling of barren rhyodacite to 98% minus 75 microns;
- Blending in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10g and 60g units in laminated foil pouches and 500g units in plastic jars.

## PHYSICAL PROPERTIES

OREAS 608b 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 608b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
710	0.55	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 were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results have been normalised to 100 per cent and represent the relative proportion of crystalline material. Totals greater or less than 100 per cent are due to rounding errors. The Kandite group appears to be mainly kaolinite and dickite. A trace amount of kandite group mineral might be present. Chalcopyrite and calcite are reported together due to their overlapping patterns. Samples might contain some illite which is reported under muscovite. Some amorphous material might be present in the samples.

**Table 5. Indicative mineralogy of OREAS 608b based on semi-quantitative XRD analysis.**

Mineral / Mineral Group	% (mass ratio)
Kandite group	4
Chlorite	4
Annite - biotite - phlogopite	< 1
Muscovite	3
Plagioclase	15
K-feldspar	7
Quartz	56
Magnetite	2
Pyrite	3
Chalcopyrite and/or calcite	1
Alunite	6

## ANALYTICAL PROGRAM

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

- Gold by fire assay using a 25-50g charge weight with AAS finish (22 laboratories) and ICP-OES (6 laboratories);
- Gold by aqua regia digestion using a 10-50g sample mass with ICP-MS finish (12 laboratories), AAS (10 laboratories) finish and ICP-OES (1 laboratory);
- Sulphur by infrared combustion furnace (25 laboratories);

- Full ICP-OES and MS elemental suites by 4-acid digestion (up to 26 laboratories depending on the element);
- Full ICP-OES and MS elemental suites by aqua regia digestion (up to 26 laboratories depending on the element).

The following Sulphur “species” were also requested from the laboratories offering this methodology:

- Sulphate S by Na<sub>2</sub>CO<sub>3</sub> leach of sulphates, precipitation as barium sulphate with gravimetric finish (or by difference using the Total S value minus the Sulphide S);
- Sulphide S by Na<sub>2</sub>CO<sub>3</sub> leach of sulphates followed by infrared combustion furnace (or by difference using the Total S value minus the Sulphate S).

In addition, instrumental neutron activation analysis (INAA) of Au on 20 x 85mg subsamples was undertaken at ANSTO, Lucas Heights to confirm homogeneity (see Table 6).

Table 3 above shows indicative values including major and trace element characterisation based on two samples analysed at 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;
- Trace elements by laser ablation (on the fused bead) with ICP-MS finish.

Table 3 also includes indicative values for the Sulphur “species” and other elements where interlaboratory consensus or the quantity of data was insufficient for certification.

For the round robin program twelve 3kg test units (lots) were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 100g pulp samples were submitted to each laboratory for analysis and were selected systematically to maximise representation. For example, from the 12 sampling lot intervals, the six samples a laboratory may receive could be from the odd lots, or the even lots. I.e., 1, 3, 5, 7, 9 and 11 or 2, 4, 6, 8, 10 and 12). All samples lots within a laboratory sample set were randomised prior to assigning sample numbers.

The 20 individual INAA results upon which much of the homogeneity evaluation is based, included paired 10g samples taken from 10 different sampling lot intervals. This format enabled a nested ANOVA treatment of the INAA results to evaluate homogeneity (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 6) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation.

**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 98-3:2008 [6, 16]. 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. This data is intended for 'informational purposes' only.

### 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 0.119 and 0.123 wt.%. 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. **Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.**

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. Table 6 below shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 608b. 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 0.13% calculated for a 30g fire assay sample (2.46% at 85mg weights) confirms the high level of gold homogeneity in OREAS 608b.

**Table 6. 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	1.360	1.353
2	1.372	1.354
3	1.373	1.354
4	1.381	1.354
5	1.409	1.356
6	1.317	1.351
7	1.360	1.353
8	1.320	1.351
9	1.337	1.352
10	1.307	1.351
11	1.383	1.355
12	1.373	1.354
13	1.286	1.349
14	1.329	1.352
15	1.339	1.352
16	1.343	1.352
17	1.393	1.355
18	1.380	1.354
19	1.384	1.355
20	1.315	1.351
Mean	1.353	1.353
Median	1.360	1.353
Std Dev.	0.033	0.002
<b>Rel.Std.Dev.</b>	<b>2.46%</b>	<b>0.13%</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

The homogeneity of OREAS 608b has also been evaluated in an Analysis of Variance (**ANOVA**) of the INAA data. The 20 samples were comprised of paired samples from each of 10 different sampling lot intervals (representative of the prepared batch) and were randomised prior to assigning sample numbers. The duplicate samples enabled an ANOVA by comparison of within- and between-unit variances across the 10 pairs. The purpose of the ANOVA 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 608b. The test was performed using the following parameters:

- Gold INAA – 20 results (1 laboratory providing duplicate analyses on 10 samples where each sample can be viewed as a ‘unit’);
- 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.

The Au data by INAA was not filtered for outliers prior to the calculation of the  $p$ -value. This process derived a  $p$ -value of 0.098, a statistically insignificant result so the Null Hypothesis is accepted.

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 608b 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 608b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PERFORMANCE GATES

The standard deviations (SD's) intervals reported in Table 7 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.***

Table 7 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 7. Performance Gates for OREAS 608b.**

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	1.29	0.042	1.21	1.37	1.16	1.42	3.26%	6.51%	9.77%	1.23	1.36
<b>Aqua Regia Digestion (sample weights 10-50g)</b>											
Au, ppm	1.29	0.058	1.18	1.41	1.12	1.47	4.50%	8.99%	13.49%	1.23	1.36
<b>Infrared Combustion</b>											
S, wt. %	1.33	0.046	1.24	1.42	1.19	1.46	3.44%	6.87%	10.31%	1.26	1.39
<b>4-Acid Digestion</b>											
Ag, ppm	15.2	0.34	14.5	15.9	14.2	16.2	2.22%	4.44%	6.66%	14.4	15.9
Al, wt. %	7.26	0.252	6.76	7.77	6.51	8.02	3.47%	6.94%	10.41%	6.90	7.63
As, ppm	268	14	241	296	227	309	5.12%	10.24%	15.36%	255	282
Be, ppm	2.58	0.128	2.32	2.84	2.20	2.97	4.98%	9.95%	14.93%	2.45	2.71
Bi, ppm	15.2	0.93	13.3	17.1	12.4	18.0	6.10%	12.21%	18.31%	14.4	16.0
Ca, wt. %	0.943	0.029	0.886	1.001	0.857	1.030	3.06%	6.12%	9.18%	0.896	0.991
Cd, ppm	2.91	0.178	2.55	3.27	2.38	3.44	6.12%	12.24%	18.36%	2.76	3.06
Ce, ppm	79	4.3	71	88	66	92	5.44%	10.89%	16.33%	75	83
Co, ppm	2.99	0.207	2.57	3.40	2.37	3.61	6.92%	13.85%	20.77%	2.84	3.14
Cr, ppm	15.9	1.8	12.4	19.4	10.6	21.2	11.11%	22.23%	33.34%	15.1	16.7
Cs, ppm	5.30	0.355	4.59	6.01	4.24	6.37	6.69%	13.37%	20.06%	5.04	5.57
Cu, wt. %	0.121	0.004	0.113	0.128	0.110	0.132	3.12%	6.24%	9.36%	0.115	0.127
Dy, ppm	3.14	0.160	2.82	3.47	2.66	3.63	5.10%	10.20%	15.30%	2.99	3.30
Er, ppm	0.94	0.056	0.83	1.06	0.77	1.11	5.97%	11.95%	17.92%	0.90	0.99
Eu, ppm	1.14	0.040	1.06	1.22	1.02	1.26	3.49%	6.99%	10.48%	1.08	1.19
Fe, wt. %	2.33	0.078	2.18	2.49	2.10	2.57	3.35%	6.71%	10.06%	2.22	2.45
Ga, ppm	23.4	1.64	20.1	26.7	18.4	28.3	7.03%	14.05%	21.08%	22.2	24.5
Gd, ppm	4.96	0.334	4.30	5.63	3.96	5.96	6.72%	13.44%	20.16%	4.71	5.21
Ge, ppm	0.18	0.06	0.06	0.30	0.00	0.36	32.81%	65.62%	98.42%	0.17	0.19
Hf, ppm	5.68	0.268	5.15	6.22	4.88	6.49	4.71%	9.42%	14.13%	5.40	5.97
Ho, ppm	0.43	0.041	0.35	0.51	0.30	0.55	9.61%	19.23%	28.84%	0.41	0.45
In, ppm	0.29	0.020	0.25	0.33	0.23	0.35	6.75%	13.50%	20.25%	0.28	0.31
K, wt. %	2.82	0.151	2.52	3.12	2.37	3.27	5.36%	10.72%	16.08%	2.68	2.96
La, ppm	39.4	4.0	31.4	47.5	27.4	51.5	10.19%	20.38%	30.57%	37.4	41.4
Li, ppm	29.1	1.23	26.7	31.6	25.4	32.8	4.23%	8.46%	12.69%	27.7	30.6
Lu, ppm	0.078	0.009	0.061	0.095	0.052	0.103	11.02%	22.05%	33.07%	0.074	0.082
Mg, wt. %	0.115	0.007	0.101	0.129	0.094	0.137	6.17%	12.34%	18.52%	0.109	0.121
Mn, wt. %	0.025	0.001	0.023	0.027	0.022	0.028	4.39%	8.78%	13.17%	0.024	0.026
Mo, ppm	5.63	0.239	5.16	6.11	4.92	6.35	4.25%	8.49%	12.74%	5.35	5.92
Na, wt. %	2.30	0.095	2.11	2.49	2.01	2.59	4.14%	8.28%	12.42%	2.19	2.42
Nb, ppm	16.6	0.99	14.6	18.6	13.6	19.6	5.97%	11.94%	17.92%	15.8	17.4
Nd, ppm	33.9	2.50	28.9	38.9	26.5	41.4	7.35%	14.71%	22.06%	32.2	35.6
Ni, ppm	4.05	0.43	3.20	4.91	2.77	5.33	10.56%	21.12%	31.68%	3.85	4.25
P, wt. %	0.031	0.001	0.028	0.033	0.027	0.035	4.39%	8.78%	13.17%	0.029	0.032
Pb, ppm	377	25	327	428	301	453	6.69%	13.39%	20.08%	358	396
Pr, ppm	9.19	0.754	7.68	10.70	6.93	11.45	8.20%	16.41%	24.61%	8.73	9.65
Rb, ppm	120	7	106	134	99	141	5.85%	11.69%	17.54%	114	126

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 7 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>											
Re, ppm	< 0.005	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	1.29	0.053	1.19	1.40	1.13	1.45	4.11%	8.23%	12.34%	1.23	1.36
Sb, ppm	46.8	2.92	41.0	52.6	38.0	55.5	6.23%	12.46%	18.70%	44.5	49.1
Sc, ppm	4.10	0.403	3.29	4.91	2.89	5.31	9.82%	19.65%	29.47%	3.89	4.30
Se, ppm	3.75	0.49	2.78	4.73	2.29	5.22	13.02%	26.03%	39.05%	3.57	3.94
Sm, ppm	6.30	0.399	5.50	7.10	5.11	7.50	6.33%	12.67%	19.00%	5.99	6.62
Sn, ppm	3.50	0.171	3.16	3.84	2.99	4.01	4.89%	9.78%	14.67%	3.33	3.68
Sr, ppm	218	15	188	249	172	264	7.02%	14.03%	21.05%	207	229
Ta, ppm	1.21	0.087	1.04	1.39	0.95	1.47	7.17%	14.34%	21.51%	1.15	1.27
Tb, ppm	0.67	0.07	0.52	0.82	0.45	0.89	11.10%	22.20%	33.30%	0.64	0.70
Te, ppm	2.33	0.159	2.02	2.65	1.86	2.81	6.83%	13.66%	20.50%	2.22	2.45
Th, ppm	13.2	1.15	10.9	15.5	9.7	16.6	8.75%	17.50%	26.24%	12.5	13.8
Ti, wt. %	0.118	0.004	0.111	0.125	0.107	0.129	3.09%	6.19%	9.28%	0.112	0.124
Tl, ppm	1.05	0.058	0.93	1.16	0.87	1.22	5.56%	11.12%	16.68%	1.00	1.10
Tm, ppm	0.10	0.008	0.09	0.12	0.08	0.13	7.62%	15.25%	22.87%	0.10	0.11
U, ppm	5.01	0.163	4.68	5.33	4.52	5.50	3.25%	6.51%	9.76%	4.76	5.26
V, ppm	8.60	0.624	7.36	9.85	6.73	10.48	7.25%	14.51%	21.76%	8.17	9.04
W, ppm	2.41	0.145	2.12	2.70	1.97	2.84	6.02%	12.04%	18.05%	2.29	2.53
Y, ppm	13.1	0.53	12.0	14.2	11.5	14.7	4.08%	8.15%	12.23%	12.4	13.8
Yb, ppm	0.59	0.058	0.48	0.71	0.42	0.77	9.79%	19.58%	29.36%	0.57	0.62
Zn, ppm	651	41	570	733	529	773	6.25%	12.49%	18.74%	619	684
Zr, ppm	214	10	195	233	185	243	4.53%	9.06%	13.59%	203	225
<b>Aqua Regia Digestion</b>											
Ag, ppm	14.8	0.57	13.7	16.0	13.1	16.5	3.83%	7.65%	11.48%	14.1	15.6
Al, wt. %	0.836	0.054	0.728	0.944	0.674	0.999	6.48%	12.95%	19.43%	0.794	0.878
As, ppm	255	15	225	285	211	299	5.81%	11.61%	17.42%	242	268
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	341	60	221	461	161	521	17.63%	35.27%	52.90%	324	358
Be, ppm	0.45	0.05	0.35	0.55	0.30	0.60	10.98%	21.95%	32.93%	0.43	0.47
Bi, ppm	15.7	1.06	13.6	17.8	12.5	18.9	6.75%	13.50%	20.24%	14.9	16.5
Ca, wt. %	0.551	0.024	0.502	0.599	0.478	0.624	4.42%	8.84%	13.25%	0.523	0.578
Cd, ppm	2.87	0.132	2.60	3.13	2.47	3.26	4.61%	9.23%	13.84%	2.72	3.01
Ce, ppm	46.2	3.86	38.5	53.9	34.6	57.8	8.35%	16.69%	25.04%	43.9	48.5
Co, ppm	2.77	0.131	2.51	3.03	2.38	3.16	4.72%	9.45%	14.17%	2.63	2.91
Cr, ppm	16.9	0.73	15.4	18.3	14.7	19.1	4.35%	8.69%	13.04%	16.0	17.7
Cs, ppm	1.29	0.122	1.04	1.53	0.92	1.65	9.45%	18.90%	28.36%	1.22	1.35
Cu, wt. %	0.122	0.005	0.112	0.132	0.107	0.136	3.96%	7.92%	11.88%	0.116	0.128
Dy, ppm	1.76	0.102	1.55	1.96	1.45	2.06	5.83%	11.66%	17.49%	1.67	1.84
Er, ppm	0.43	0.05	0.32	0.53	0.27	0.59	12.44%	24.89%	37.33%	0.41	0.45
Eu, ppm	0.59	0.07	0.45	0.73	0.38	0.81	12.15%	24.30%	36.45%	0.56	0.62
Fe, wt. %	2.02	0.086	1.85	2.19	1.76	2.28	4.28%	8.57%	12.85%	1.92	2.12
Ga, ppm	5.78	0.408	4.96	6.59	4.56	7.00	7.05%	14.11%	21.16%	5.49	6.07
Gd, ppm	3.16	0.304	2.56	3.77	2.25	4.08	9.60%	19.19%	28.79%	3.01	3.32

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.



Table 7 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>											
Ge, ppm	0.10	0.03	0.05	0.16	0.02	0.19	28.40%	56.80%	85.20%	0.10	0.11
Hf, ppm	1.12	0.076	0.96	1.27	0.89	1.34	6.82%	13.63%	20.45%	1.06	1.17
Hg, ppm	0.15	0.011	0.12	0.17	0.11	0.18	7.81%	15.61%	23.42%	0.14	0.15
Ho, ppm	0.20	0.03	0.15	0.25	0.13	0.28	12.29%	24.57%	36.86%	0.19	0.21
In, ppm	0.26	0.014	0.23	0.29	0.22	0.30	5.35%	10.70%	16.05%	0.25	0.27
K, wt. %	0.240	0.014	0.212	0.269	0.198	0.283	5.90%	11.80%	17.70%	0.228	0.252
La, ppm	23.0	1.59	19.9	26.2	18.3	27.8	6.90%	13.81%	20.71%	21.9	24.2
Li, ppm	17.7	1.71	14.2	21.1	12.5	22.8	9.68%	19.36%	29.04%	16.8	18.5
Lu, ppm	0.027	0.003	0.020	0.034	0.016	0.037	12.92%	25.85%	38.77%	0.025	0.028
Mg, wt. %	0.078	0.005	0.068	0.089	0.062	0.094	6.83%	13.66%	20.49%	0.074	0.082
Mn, wt. %	0.022	0.001	0.021	0.024	0.020	0.025	3.43%	6.87%	10.30%	0.021	0.024
Mo, ppm	5.20	0.291	4.62	5.78	4.33	6.07	5.60%	11.20%	16.80%	4.94	5.46
Na, wt. %	0.069	0.007	0.056	0.082	0.050	0.089	9.38%	18.76%	28.14%	0.066	0.073
Nb, ppm	0.78	0.14	0.49	1.06	0.35	1.20	18.37%	36.74%	55.11%	0.74	0.81
Nd, ppm	20.5	2.5	15.6	25.4	13.1	27.8	11.99%	23.97%	35.96%	19.4	21.5
Ni, ppm	3.96	0.288	3.38	4.53	3.09	4.82	7.29%	14.57%	21.86%	3.76	4.15
P, wt. %	0.020	0.001	0.018	0.021	0.018	0.022	3.74%	7.48%	11.21%	0.019	0.021
Pb, ppm	323	11	300	345	289	357	3.53%	7.06%	10.59%	307	339
Pr, ppm	5.44	0.61	4.22	6.66	3.61	7.27	11.24%	22.47%	33.71%	5.17	5.71
Rb, ppm	11.6	1.4	8.9	14.4	7.5	15.7	11.87%	23.73%	35.60%	11.0	12.2
Re, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	0.682	0.035	0.612	0.752	0.577	0.787	5.15%	10.31%	15.46%	0.648	0.716
Sb, ppm	40.4	3.35	33.7	47.1	30.3	50.5	8.30%	16.61%	24.91%	38.4	42.4
Sc, ppm	1.45	0.21	1.04	1.86	0.84	2.07	14.16%	28.33%	42.49%	1.38	1.53
Se, ppm	3.43	0.34	2.74	4.11	2.40	4.46	10.00%	20.01%	30.01%	3.26	3.60
Sm, ppm	3.89	0.342	3.21	4.58	2.87	4.92	8.80%	17.59%	26.39%	3.70	4.09
Sn, ppm	1.23	0.13	0.97	1.49	0.83	1.62	10.68%	21.37%	32.05%	1.17	1.29
Sr, ppm	29.7	2.02	25.6	33.7	23.6	35.8	6.81%	13.63%	20.44%	28.2	31.2
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.37	0.05	0.27	0.47	0.22	0.52	13.78%	27.57%	41.35%	0.35	0.39
Te, ppm	2.29	0.25	1.79	2.79	1.54	3.03	10.87%	21.73%	32.60%	2.17	2.40
Th, ppm	9.23	0.602	8.03	10.44	7.43	11.04	6.52%	13.03%	19.55%	8.77	9.69
Ti, wt. %	0.017	0.003	0.011	0.023	0.008	0.026	18.36%	36.72%	55.08%	0.016	0.018
Tl, ppm	0.50	0.038	0.42	0.57	0.38	0.61	7.60%	15.21%	22.81%	0.47	0.52
U, ppm	2.31	0.107	2.09	2.52	1.99	2.63	4.62%	9.24%	13.86%	2.19	2.42
V, ppm	2.94	0.39	2.15	3.72	1.75	4.12	13.44%	26.88%	40.32%	2.79	3.08
W, ppm	0.79	0.058	0.67	0.90	0.61	0.96	7.31%	14.62%	21.93%	0.75	0.83
Y, ppm	6.58	0.386	5.81	7.35	5.42	7.74	5.86%	11.73%	17.59%	6.25	6.91
Yb, ppm	0.22	0.011	0.20	0.24	0.19	0.26	5.05%	10.11%	15.16%	0.21	0.23
Zn, ppm	620	33	553	686	520	720	5.37%	10.74%	16.10%	589	651
Zr, ppm	37.7	4.2	29.3	46.2	25.1	50.4	11.16%	22.33%	33.49%	35.9	39.6
<b>Alkaline Leach</b>											
S-(Sulphide), wt. %	0.543	0.156	0.230	0.856	0.074	1.013	28.81%	57.63%	86.44%	0.516	0.570

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.

## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. Alex Stewart International, Mendoza, Argentina
4. ALS, Johannesburg, South Africa
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. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
12. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. CERTIMIN, Lima, Peru
15. ESAN Istanbul, Istanbul, Turkey
16. Inspectorate (BV), Lima, Peru
17. Intertek, Cupang, Muntinlupa, Philippines
18. Intertek Genalysis, Perth, WA, Australia
19. Intertek Minerals Ltd, Tarkwa, Western Region, Ghana
20. Intertek Testing Services, Townsville, QLD, Australia
21. Koza Gold (Ovacik Gold Mine), Bergama, Izmir, Turkey
22. Paragon Geochemical Laboratories, Sparks, Nevada, USA
23. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
24. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
25. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
26. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
27. SGS Australia Mineral Services, Perth, WA, Australia
28. SGS Mineral Services, Townsville, QLD, Australia
29. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
30. 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 608b

SPC.1656.RR1.OREAS 608b.3.Fire Assay.Au.Lab.240510.105437.SN

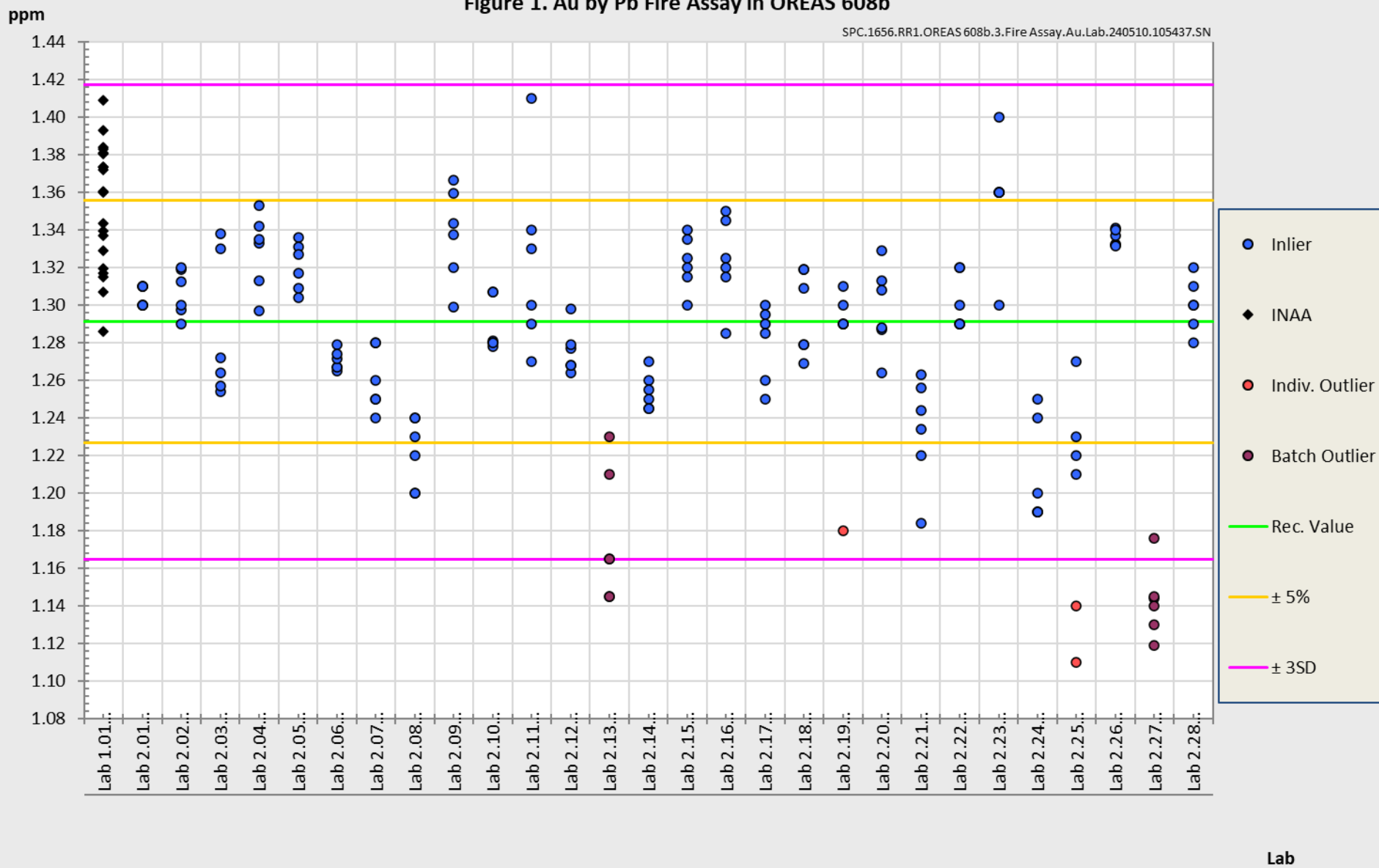


Figure 2. Cu by 4-Acid Digestion in OREAS 608b

SPC.1656.RR1.OREAS 608b.3.4-Acid.Cu.Lab.240510.111745.SN

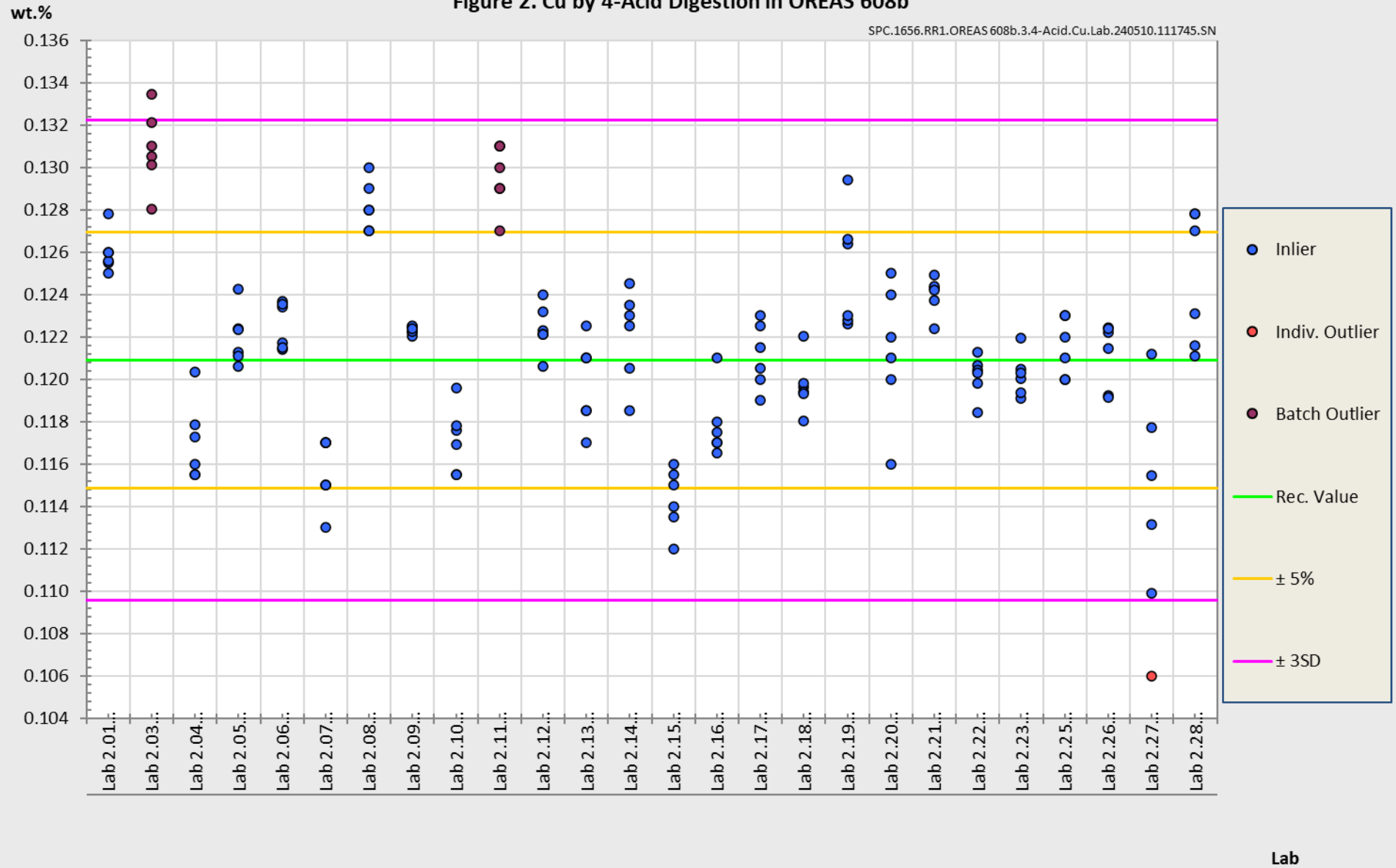
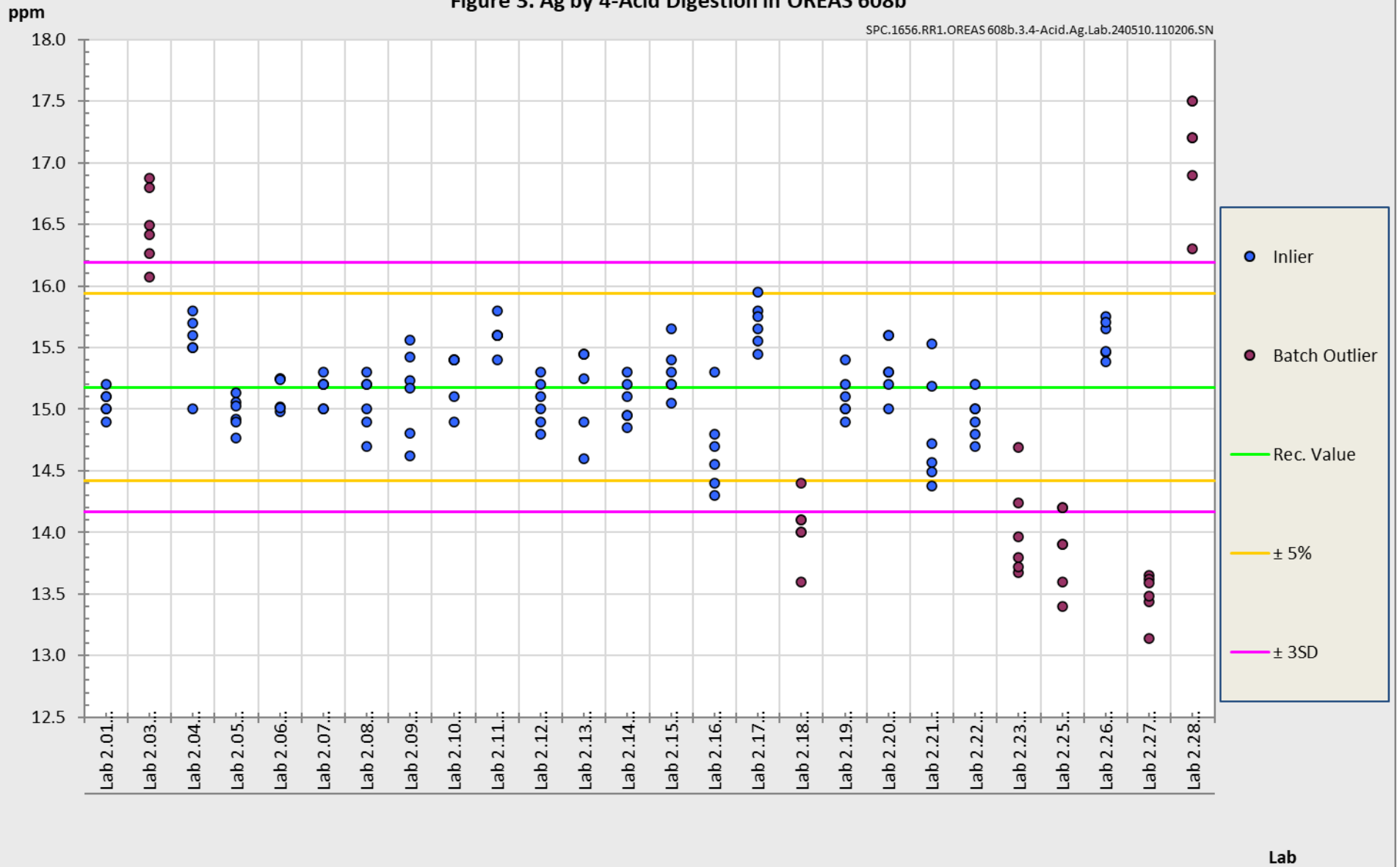


Figure 3. Ag by 4-Acid Digestion in OREAS 608b

SPC.1656.RR1.OREAS 608b.3.4-Acid.Ag.Lab.240510.110206.SN



## PREPARER AND SUPPLIER

Certified reference material OREAS 608b 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 [9], 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. These laboratories are accredited to ISO 17025 for Au by fire assay (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) 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 33405:2024-05, 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 608b 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 608b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 608b 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:

- Gold by fire assay:  $\geq 15\text{g}$ ;
- Gold by aqua regia digestion:  $\geq 10\text{g}$ ;
- Total S by infrared combustion furnace/CS analyser:  $\geq 0.1\text{g}$ ;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25\text{g}$ ;
- Multi-elements by aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5\text{g}$ ;
- Sulphate Sulphur via various leaching methods:  $\geq 0.1\text{g}$ ;
- Sulphide Sulphur via various leaching methods:  $\geq 0.1\text{g}$ .

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 608b remains valid, within the specified measurement uncertainties, until December 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

OREAS 608b contains a moderate level of Sulphur (1.33 wt.% S) and is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

## 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 7 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

Revision No.	Date	Changes applied
0	19 <sup>th</sup> June, 2024	First publication.

## CERTIFYING OFFICER



19<sup>th</sup> June, 2024

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

## QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016.



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.



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