

**CERTIFICATE OF ANALYSIS FOR**

**PORPHYRY COPPER-GOLD ORE**

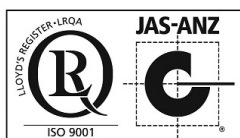
**(Ridgeway/Northparkes Mines, New South Wales, Australia)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 507b**



Accredited for compliance with ISO 17034



COA-1839-OREAS507b-R0  
BUP-70-10-01 Ver:2.0

3-Oct-2024

**Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 507b.**

Constituent	Certified Value <sup>†</sup>	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppm)	0.175	0.172	0.178	0.172*	0.177*

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 [10] and all participating laboratories comply with the requirements of ISO 17025 [9].

\*Gold Tolerance Limits for typical 30g fire assay are determined from 20 x 1 g INAA results and the Sampling Constant (Ingamells & Switzer, 1973) [2].

**Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 507b.**

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion (sample weights 10-50g)					
Au, Gold (ppm)	0.178	0.173	0.182	0.175	0.181
4-Acid Digestion					
Ag, Silver (ppm)	1.60	1.50	1.70	1.54	1.66
Al, Aluminium (wt.%)	7.63	7.36	7.90	7.49	7.77
As, Arsenic (ppm)	21.1	19.7	22.5	20.0	22.1
Ba, Barium (ppm)	976	928	1023	949	1002
Be, Beryllium (ppm)	2.33	2.21	2.46	2.25	2.42
Bi, Bismuth (ppm)	1.72	1.53	1.90	1.56	1.87
Ca, Calcium (wt.%)	1.63	1.58	1.68	1.60	1.67
Cd, Cadmium (ppm)	0.47	0.42	0.51	0.44	0.50
Ce, Cerium (ppm)	69	64	73	65	72
Co, Cobalt (ppm)	9.06	8.58	9.54	8.82	9.29
Cr, Chromium (ppm)	40.2	37.2	43.3	38.6	41.9
Cs, Caesium (ppm)	9.52	9.04	9.99	9.14	9.89
Cu, Copper (wt.%)	0.618	0.600	0.636	0.609	0.626
Dy, Dysprosium (ppm)	3.47	3.13	3.81	3.31	3.63
Er, Erbium (ppm)	1.37	1.19	1.54	1.26	1.47
Eu, Europium (ppm)	1.28	1.18	1.37	1.20	1.35
Fe, Iron (wt.%)	3.07	2.97	3.16	3.02	3.11
Ga, Gallium (ppm)	20.0	19.1	20.8	19.3	20.6
Gd, Gadolinium (ppm)	5.30	4.79	5.82	5.03	5.58
Ge, Germanium (ppm)	0.17	0.11	0.23	0.13	0.21
Hf, Hafnium (ppm)	1.95	1.80	2.10	1.82	2.07
Ho, Holmium (ppm)	0.53	0.47	0.58	0.48	0.57
In, Indium (ppm)	0.14	0.13	0.15	0.13	0.15
K, Potassium (wt.%)	3.13	3.03	3.23	3.03	3.22
La, Lanthanum (ppm)	34.1	32.2	36.1	32.5	35.8
Li, Lithium (ppm)	45.5	43.8	47.1	44.3	46.6
Lu, Lutetium (ppm)	0.17	0.15	0.19	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.

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Mg, Magnesium (wt.%)	0.687	0.662	0.712	0.670	0.703
Mn, Manganese (wt.%)	0.034	0.033	0.035	0.033	0.035
Mo, Molybdenum (ppm)	116	112	119	113	118
Na, Sodium (wt.%)	2.03	1.98	2.09	1.99	2.08
Nb, Niobium (ppm)	11.8	11.3	12.4	11.4	12.3
Nd, Neodymium (ppm)	30.3	25.7	34.9	28.8	31.9
Ni, Nickel (ppm)	15.7	14.9	16.6	15.3	16.2
P, Phosphorus (wt.%)	0.083	0.080	0.087	0.082	0.085
Pb, Lead (ppm)	54	52	57	52	57
Pr, Praseodymium (ppm)	7.79	6.71	8.87	7.49	8.09
Rb, Rubidium (ppm)	160	153	167	155	165
Re, Rhenium (ppm)	0.083	0.079	0.087	0.078	0.087
S, Sulphur (wt.%)	0.829	0.798	0.860	0.814	0.844
Sb, Antimony (ppm)	2.74	2.59	2.88	2.63	2.85
Sc, Scandium (ppm)	8.24	7.82	8.67	7.96	8.53
Se, Selenium (ppm)	3.98	3.34	4.62	3.77	4.19
Sm, Samarium (ppm)	6.15	5.32	6.99	5.77	6.54
Sn, Tin (ppm)	4.87	4.58	5.16	4.69	5.04
Sr, Strontium (ppm)	220	213	228	216	225
Ta, Tantalum (ppm)	1.01	0.94	1.07	0.95	1.06
Tb, Terbium (ppm)	0.69	0.64	0.74	0.66	0.72
Te, Tellurium (ppm)	0.37	0.30	0.43	0.32	0.42
Th, Thorium (ppm)	13.4	12.7	14.1	12.8	14.0
Ti, Titanium (wt.%)	0.338	0.327	0.349	0.328	0.348
Tl, Thallium (ppm)	0.94	0.89	0.98	0.91	0.96
Tm, Thulium (ppm)	0.17	0.14	0.20	IND	IND
U, Uranium (ppm)	3.85	3.32	4.38	3.56	4.14
V, Vanadium (ppm)	64	62	67	63	66
W, Tungsten (ppm)	11.9	10.7	13.2	11.2	12.6
Y, Yttrium (ppm)	14.5	13.8	15.3	13.9	15.1
Yb, Ytterbium (ppm)	1.14	1.01	1.27	1.06	1.21
Zn, Zinc (ppm)	126	121	131	123	128
Zr, Zirconium (ppm)	64	59	68	61	66
Aqua Regia Digestion					
Ag, Silver (ppm)	1.58	1.51	1.66	1.54	1.63
Al, Aluminium (wt.%)	1.78	1.73	1.82	1.74	1.82
As, Arsenic (ppm)	20.1	19.1	21.2	19.4	20.9

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

Note: intervals may appear asymmetric due to rounding.

IND = indeterminate (due to limited reading resolution of the methods employed).

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion continued					
B, Boron (ppm)	< 10	IND	IND	IND	IND
Ba, Barium (ppm)	398	361	435	384	412
Be, Beryllium (ppm)	1.30	1.25	1.35	1.27	1.34
Bi, Bismuth (ppm)	1.76	1.55	1.97	1.64	1.89
Ca, Calcium (wt.%)	0.554	0.540	0.569	0.542	0.567
Cd, Cadmium (ppm)	0.36	0.32	0.40	0.34	0.38
Ce, Cerium (ppm)	30.1	28.4	31.8	29.2	31.0
Co, Cobalt (ppm)	8.81	8.49	9.14	8.61	9.02
Cr, Chromium (ppm)	45.6	44.1	47.2	44.5	46.8
Cs, Caesium (ppm)	7.42	7.04	7.80	7.27	7.57
Cu, Copper (wt.%)	0.610	0.598	0.622	0.602	0.618
Fe, Iron (wt.%)	2.95	2.89	3.02	2.89	3.01
Ga, Gallium (ppm)	8.52	8.18	8.87	8.26	8.79
Ge, Germanium (ppm)	0.10	0.07	0.13	IND	IND
Hf, Hafnium (ppm)	0.29	0.27	0.32	0.28	0.31
Hg, Mercury (ppm)	0.046	0.033	0.059	IND	IND
In, Indium (ppm)	0.13	0.12	0.14	0.12	0.13
K, Potassium (wt.%)	0.886	0.857	0.915	0.873	0.898
La, Lanthanum (ppm)	13.7	13.0	14.4	13.2	14.2
Li, Lithium (ppm)	37.6	36.3	38.8	36.6	38.6
Mg, Magnesium (wt.%)	0.641	0.620	0.661	0.628	0.653
Mn, Manganese (wt.%)	0.029	0.028	0.029	0.028	0.029
Mo, Molybdenum (ppm)	111	107	114	109	113
Na, Sodium (wt.%)	0.143	0.130	0.156	0.138	0.147
Nb, Niobium (ppm)	0.81	0.60	1.01	0.75	0.87
Ni, Nickel (ppm)	15.3	14.7	16.0	15.0	15.7
P, Phosphorus (wt.%)	0.063	0.060	0.065	0.061	0.064
Pb, Lead (ppm)	35.3	33.9	36.7	34.2	36.4
Rb, Rubidium (ppm)	86	82	91	84	88
Re, Rhenium (ppm)	0.083	0.075	0.090	0.078	0.087
S, Sulphur (wt.%)	0.813	0.791	0.834	0.800	0.825
Sb, Antimony (ppm)	1.89	1.72	2.06	1.83	1.95
Sc, Scandium (ppm)	6.97	6.68	7.27	6.79	7.15
Se, Selenium (ppm)	3.85	3.50	4.20	3.62	4.08
Sn, Tin (ppm)	3.31	3.16	3.46	3.19	3.44
Sr, Strontium (ppm)	56	54	58	55	58
Ta, Tantalum (ppm)	< 0.01	IND	IND	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. 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
Aqua Regia Digestion continued					
Tb, Terbium (ppm)	0.47	0.44	0.50	0.45	0.49
Te, Tellurium (ppm)	0.33	0.28	0.38	0.31	0.35
Th, Thorium (ppm)	5.95	5.49	6.42	5.72	6.19
Ti, Titanium (wt.%)	0.227	0.218	0.236	0.222	0.232
Tl, Thallium (ppm)	0.60	0.57	0.63	0.58	0.61
U, Uranium (ppm)	3.19	2.80	3.57	2.92	3.45
V, Vanadium (ppm)	54	52	56	53	55
W, Tungsten (ppm)	7.13	6.34	7.93	6.69	7.58
Y, Yttrium (ppm)	9.62	9.19	10.06	9.42	9.83
Yb, Ytterbium (ppm)	0.69	0.63	0.75	IND	IND
Zn, Zinc (ppm)	120	116	123	117	122
Zr, Zirconium (ppm)	7.83	7.19	8.47	7.53	8.13

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 3. Indicative Values for OREAS 507b.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppb	2.25	Pt	ppb	< 10			
<b>Sequential Leach</b>								
Cu-Res(4A)	wt. %	0.233	Cu-Sol(CN)	wt. %	0.264	Cu-Sol(H <sub>2</sub> SO <sub>4</sub> )	wt. %	0.120
<b>Gas / Liquid Pycnometry</b>								
SG	Unity	2.71						
<b>Infrared Combustion</b>								
C	wt. %	0.083	S	wt. %	0.763			
<b>Alkaline Leach</b>								
S-(Sulphate)	wt. %	0.443	S-(Sulphide)	wt. %	0.242			
<b>Peroxide Fusion ICP</b>								
Ag	ppm	0.813	Hg	ppm	< 5	Si	wt. %	32.36
Al	wt. %	6.38	Ho	ppm	0.63	Sm	ppm	6.00
As	ppm	19.4	K	wt. %	3.14	Sn	ppm	< 5
Ba	ppm	688	La	ppm	31.7	Sr	ppm	158
Be	ppm	5.00	Li	ppm	50	Ta	ppm	1.27
Bi	ppm	< 5	Lu	ppm	< 0.5	Tb	ppm	0.77
Ca	wt. %	1.56	Mg	wt. %	0.679	Th	ppm	6.18
Cd	ppm	< 1	Mn	wt. %	0.038	Ti	wt. %	0.295
Ce	ppm	62	Mo	ppm	117	Tl	ppm	< 1
Co	ppm	13.0	Na	wt. %	0.137	Tm	ppm	< 1
Cr	ppm	48.0	Nb	ppm	12.7	U	ppm	4.18
Cu	wt. %	0.634	Nd	ppm	28.3	V	ppm	62

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>Peroxide Fusion ICP continued</b>								
Dy	ppm	3.77	Ni	ppm	13.3	W	ppm	11.4
Er	ppm	1.68	P	wt. %	0.072	Y	ppm	22.8
Eu	ppm	1.35	Pb	ppm	42.9	Yb	ppm	1.40
Fe	wt. %	3.16	Pr	ppm	7.42	Zn	ppm	128
Ga	ppm	19.8	S	wt. %	0.750	Zr	ppm	8.42
Gd	ppm	5.62	Sb	ppm	< 5			
Hf	ppm	2.52	Sc	ppm	9.43			
<b>4-Acid Digestion</b>								
B	ppm	< 10	Hg	ppm	0.093	Pt	ppb	< 5
<b>Aqua Regia Digestion</b>								
Dy	ppm	2.33	Ho	ppm	0.40	Pr	ppm	4.48
Er	ppm	0.89	Lu	ppm	0.091	Pt	ppb	2.08
Eu	ppm	0.40	Nd	ppm	17.6	Sm	ppm	3.92
Gd	ppm	3.18	Pd	ppb	< 10	Tm	ppm	0.11
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt. %	14.98	Fe <sub>2</sub> O <sub>3</sub>	wt. %	4.46	S	wt. %	0.851
As	ppm	20.0	K <sub>2</sub> O	wt. %	3.78	SiO <sub>2</sub>	wt. %	67.04
BaO	ppm	1105	MgO	wt. %	1.19	Sn	ppm	< 10
CaO	wt. %	2.29	MnO	wt. %	0.050	Sr	ppm	210
Cl	ppm	100	Na <sub>2</sub> O	wt. %	2.76	TiO <sub>2</sub>	wt. %	0.590
Co	ppm	10.0	Ni	ppm	20.0	V <sub>2</sub> O <sub>5</sub>	ppm	134
Cr <sub>2</sub> O <sub>3</sub>	ppm	73	P <sub>2</sub> O <sub>5</sub>	wt. %	0.188	Zn	ppm	120
Cu	wt. %	0.616	Pb	ppm	50	Zr	ppm	230
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt. %	1.56						
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	1.55	Hf	ppm	6.44	Sm	ppm	7.31
As	ppm	19.6	Ho	ppm	1.07	Sn	ppm	4.80
Ba	ppm	951	In	ppm	0.10	Sr	ppm	215
Be	ppm	2.60	La	ppm	35.4	Ta	ppm	1.09
Bi	ppm	1.67	Lu	ppm	0.38	Tb	ppm	0.98
Cd	ppm	0.35	Mn	wt. %	0.033	Te	ppm	0.30
Ce	ppm	71	Mo	ppm	113	Th	ppm	13.9
Co	ppm	10.1	Nb	ppm	12.2	Ti	wt. %	0.363
Cr	ppm	45.0	Nd	ppm	33.3	Tl	ppm	0.60
Cs	ppm	9.46	Ni	ppm	17.0	Tm	ppm	0.42
Cu	wt. %	0.627	Pb	ppm	54	U	ppm	4.46
Dy	ppm	5.46	Pr	ppm	8.71	V	ppm	68
Er	ppm	2.85	Rb	ppm	154	W	ppm	12.8
Eu	ppm	1.35	Re	ppm	0.070	Y	ppm	27.7
Ga	ppm	19.5	Sb	ppm	2.85	Yb	ppm	2.58
Gd	ppm	6.37	Sc	ppm	8.05	Zn	ppm	118
Ge	ppm	1.40	Se	ppm	< 5	Zr	ppm	229

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.

## TABLE OF CONTENTS

INTRODUCTION .....	8
SOURCE MATERIAL .....	8
COMMUNUTION AND HOMOGENISATION PROCEDURES .....	9
PHYSICAL PROPERTIES.....	9
MINERALOGY .....	9
ANALYTICAL PROGRAM .....	10
STATISTICAL ANALYSIS .....	10
Certified Values and their uncertainty intervals .....	11
Indicative (uncertified) values .....	11
Homogeneity Evaluation.....	11
PERFORMANCE GATES .....	13
PARTICIPATING LABORATORIES.....	18
PREPARER AND SUPPLIER .....	14
METROLOGICAL TRACEABILITY .....	24
COMMUTABILITY .....	24
INTENDED USE.....	25
MINIMUM SAMPLE SIZE.....	25
PERIOD OF VALIDITY & STORAGE INSTRUCTIONS .....	25
INSTRUCTIONS FOR HANDLING & CORRECT USE .....	26
LEGAL NOTICE.....	27
DOCUMENT HISTORY .....	27
CERTIFYING OFFICER.....	27
QMS CERTIFICATION.....	27
REFERENCES .....	28

## LIST OF TABLES

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 507b.....	2
Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 507b. ....	2
Table 3. Indicative Values for OREAS 507b.....	6
Table 4. Physical properties of OREAS 507b.....	9
Table 5. Indicative mineralogy of OREAS 507b based on semi-quantitative XRD analysis.....	10
Table 6. Neutron Activation Analysis of Au on 1 g samples .....	12
Table 7. Performance Gates for OREAS 507b.....	14

## LIST OF FIGURES

Figure 1. Au by Fire Assay in OREAS 501 .....	19
Figure 2. Au by Aqua Regia Digestion in OREAS 507b .....	20
Figure 3. Ag by 4-Acid digestion in OREAS 507b .....	21
Figure 4. Cu by 4-Acid digestion in OREAS 507b .....	22
Figure 5. Mo by 4-Acid digestion in OREAS 507b.....	23

## 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 [9]) 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 507b-DataPack.1.0.240923\_140854.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 507b was prepared from a blend of porphyry copper-gold ores, barren granodiorite and minor quantities of Cu and Mo concentrates. The ores were sourced from both the Ridgeway Mine and Northparkes Mine. Both mines are located in the Central West of New South Wales, Australia. The barren granodiorite was sourced from the mafic, S-Type, Late Devonian Bulla Granodiorite complex located in northern Melbourne, Australia.

Mineralisations in the Northparkes and Ridgeway regions are quite similar and hosted by a sequence of late Ordovician to Early Silurian volcanics, intrusives and sediments that occur within the Bogan Gate Synclinal Zone of the Lachlan Fold Belt. The western portion of this zone is dominated by volcanics and host to the Late Ordovician Goonumbra porphyry copper-gold deposits. These volcanics are interpreted to have erupted from shallow water to partly emergent volcanic centres and show a broad range in composition from shoshonite through to latite to trachyte. Coeval sub-volcanic quartz monzonite porphyries (and attendant mineralisation) have intruded the volcanics. They are generally small, sub-vertical, pipe-like intrusives. Typically, the mineralised porphyries contain plagioclase and quartz



phenocrysts in a matrix of fine-grained potassium feldspar and quartz with minor biotite and hornblende.

Copper-gold mineralisation occurs as stockwork quartz veins and disseminations associated with potassic alteration. This alteration is intimately associated spatially and temporally with the small finger-like quartz monzonite porphyries that intrude the Goonumbla Volcanics. Sulphides are zoned laterally from the centres of mineralisation. The central portions are bornite-rich with minor chalcopyrite, zoning outward through equal concentrations of bornite and chalcopyrite, to an outermost chalcopyrite-rich zone. Pyrite increases outward at the expense of bornite.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 507b was prepared in the following manner:

- Drying of ores and barren granodiorite to constant mass at 105 °C;
- Drying of Cu-Mo concentrate to constant mass at 85 °C;
- Multi-stage milling of ores and concentrate to 100 % minus 30 µm;
- Milling of barren granodiorite to > 98 % minus 75 µm;
- Combining ore, granodiorite and concentrate in appropriate proportions to achieve target grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging into 10 and 60 g units in laminated foil pouches and 500 g units in plastic jars.

## PHYSICAL PROPERTIES

OREAS 507b 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 507b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Colour <sup>‡</sup>
617	0.36	N7	Light Gray

<sup>‡</sup>The Munsell Rock Colour Chart helps geologists and archaeologists 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 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. The most representative minerals in the sample are annite-biotite-phlogopite group, followed by plagioclase, quartz, K-feldspar and chlorite. Clay mineral is mainly vermiculite, while the Kandite group appears to be mainly kaolinite. A presence of some amorphous material is very likely. A trace amount of siderite including a trace of zeolite and boehmite might be present in the sample.

**Table 5. Indicative mineralogy of OREAS 507b based on semi-quantitative XRD analysis.**

Mineral / Mineral Group	% (mass ratio)
Clay mineral	< 1
Chlorite	2
Kandite group	1
Annite - biotite - phlogopite	40
Muscovite	2
Talc	0
Na-Ca amphibole	1
Plagioclase	20
K-feldspar	9
Quartz	25
Calcite and/or chalcopryrite	< 1
Pyrite	1
Bassanite	0
Gypsum	< 1
Alunite	0
Hematite	0
Magnetite	1
Goethite	0

## 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 25-50 g charge weight with AAS (12 laboratories), ICP-OES (10 laboratories) or ICP-MS (1 laboratory) finish;
- Gold by aqua regia digestion using a 10-50 g sample mass with ICP-OES/MS (15 laboratories) or AAS (5 laboratories) finish;
- 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).

To evaluate homogeneity, instrumental neutron activation analysis (INAA) of Au on 20 x 1 g subsamples was undertaken at Actlabs Ancaster in Canada (see 'Homogeneity Evaluation' section and Table 6 below).

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 (non-certified) for other elements where interlaboratory consensus or the quantity of data was insufficient and did not meet OREAS' criteria for certification.

For the round robin program twelve 4 kg 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 120 g 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 10 g 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 [6] and [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 [7]) 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 ( $p=0.95$ ) will have concentrations lying between 0.609 and 0.626 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 Actlabs Ancaster in Canada 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 1 g subsamples of OREAS 507b. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30 g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.49 % calculated for a 30 g fire assay sample (2.61 % at 1 g weights) confirms the high level of gold homogeneity in OREAS 507b.

The homogeneity of OREAS 507b 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 507b. 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 0.32, 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 507b 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 507b is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

**Table 6. Neutron Activation Analysis of Au (in ppm) on 20 x 1 g subsamples and showing the equivalent results scaled to a 30 g sample mass typical of fire assay determination.**

Replicate No	Au 1 g actual	Au 30 g equivalent*
1	0.171	0.171
2	0.170	0.171
3	0.167	0.170
4	0.170	0.171
5	0.166	0.170
6	0.163	0.169
7	0.171	0.171
8	0.165	0.170
9	0.172	0.171
10	0.174	0.171
11	0.169	0.170
12	0.164	0.169
13	0.182	0.173
14	0.171	0.171
15	0.172	0.171
16	0.173	0.171
17	0.172	0.171
18	0.177	0.172
19	0.171	0.171
20	0.174	0.171
Mean	0.171	0.171
Median	0.171	0.171
Std Dev.	0.004	0.001
Rel.Std.Dev.	2.61%	0.49%

\*Results calculated for a 30 g equivalent sample mass using the formula:  $x^{30g Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@1g} + \bar{X}$

where  $x^{30g Eq}$  = equivalent result calculated for a 30 g sample mass

$(x^{INAA})$  = raw INAA result at 1 g

$\bar{X}$  = mean of 1 g INAA results

## 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 507b.**

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
Pb Fire Assay											
Au, ppm	0.175	0.006	0.163	0.187	0.156	0.193	3.46%	6.93%	10.39%	0.166	0.183
Aqua Regia Digestion (sample weights 10-50g)											
Au, ppm	0.178	0.008	0.161	0.194	0.153	0.202	4.69%	9.37%	14.06%	0.169	0.186
4-Acid Digestion											
Ag, ppm	1.60	0.122	1.36	1.85	1.24	1.97	7.59%	15.18%	22.77%	1.52	1.68
Al, wt. %	7.63	0.313	7.00	8.26	6.69	8.57	4.11%	8.22%	12.32%	7.25	8.01
As, ppm	21.1	1.19	18.7	23.4	17.5	24.6	5.64%	11.28%	16.92%	20.0	22.1
Ba, ppm	976	61	853	1098	792	1160	6.29%	12.58%	18.86%	927	1025
Be, ppm	2.33	0.106	2.12	2.54	2.02	2.65	4.53%	9.06%	13.59%	2.22	2.45
Bi, ppm	1.72	0.17	1.37	2.06	1.20	2.23	10.03%	20.06%	30.09%	1.63	1.80
Ca, wt. %	1.63	0.068	1.50	1.77	1.43	1.84	4.19%	8.37%	12.56%	1.55	1.71
Cd, ppm	0.47	0.06	0.35	0.59	0.29	0.65	12.86%	25.72%	38.58%	0.44	0.49
Ce, ppm	69	5.7	57	80	52	86	8.26%	16.52%	24.79%	65	72
Co, ppm	9.06	0.653	7.75	10.36	7.10	11.02	7.21%	14.42%	21.63%	8.60	9.51
Cr, ppm	40.2	4.1	32.1	48.4	28.0	52.5	10.13%	20.26%	30.39%	38.2	42.3
Cs, ppm	9.52	0.342	8.83	10.20	8.49	10.54	3.59%	7.18%	10.77%	9.04	9.99
Cu, wt. %	0.618	0.022	0.575	0.661	0.553	0.683	3.49%	6.97%	10.46%	0.587	0.649
Dy, ppm	3.47	0.183	3.11	3.84	2.92	4.02	5.27%	10.54%	15.80%	3.30	3.64

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
4-Acid Digestion continued											
Er, ppm	1.37	0.097	1.17	1.56	1.07	1.66	7.10%	14.20%	21.30%	1.30	1.43
Eu, ppm	1.28	0.075	1.13	1.43	1.05	1.50	5.85%	11.71%	17.56%	1.21	1.34
Fe, wt. %	3.07	0.094	2.88	3.25	2.79	3.35	3.06%	6.11%	9.17%	2.91	3.22
Ga, ppm	20.0	0.70	18.6	21.4	17.9	22.1	3.52%	7.05%	10.57%	19.0	21.0
Gd, ppm	5.30	0.376	4.55	6.06	4.18	6.43	7.08%	14.17%	21.25%	5.04	5.57
Ge, ppm	0.17	0.06	0.05	0.29	0.00	0.34	34.48%	68.95%	103.4%	0.16	0.18
Hf, ppm	1.95	0.160	1.63	2.27	1.47	2.42	8.21%	16.41%	24.62%	1.85	2.04
Ho, ppm	0.53	0.035	0.46	0.60	0.42	0.63	6.66%	13.32%	19.99%	0.50	0.55
In, ppm	0.14	0.010	0.12	0.16	0.11	0.17	7.47%	14.93%	22.40%	0.13	0.15
K, wt. %	3.13	0.106	2.92	3.34	2.81	3.45	3.38%	6.76%	10.14%	2.97	3.29
La, ppm	34.1	2.06	30.0	38.3	28.0	40.3	6.04%	12.08%	18.12%	32.4	35.9
Li, ppm	45.5	2.49	40.5	50.4	38.0	52.9	5.48%	10.96%	16.44%	43.2	47.7
Lu, ppm	0.17	0.013	0.14	0.19	0.13	0.21	7.78%	15.56%	23.34%	0.16	0.18
Mg, wt. %	0.687	0.032	0.623	0.750	0.592	0.782	4.62%	9.25%	13.87%	0.652	0.721
Mn, wt. %	0.034	0.001	0.031	0.037	0.030	0.038	4.05%	8.10%	12.15%	0.032	0.036
Mo, ppm	116	6	104	127	98	133	4.99%	9.98%	14.98%	110	121
Na, wt. %	2.03	0.060	1.92	2.15	1.86	2.21	2.92%	5.85%	8.77%	1.93	2.14
Nb, ppm	11.8	0.67	10.5	13.2	9.8	13.8	5.64%	11.28%	16.91%	11.3	12.4
Nd, ppm	30.3	3.1	24.1	36.6	21.0	39.7	10.27%	20.54%	30.81%	28.8	31.9
Ni, ppm	15.7	0.65	14.4	17.1	13.8	17.7	4.14%	8.29%	12.43%	15.0	16.5
P, wt. %	0.083	0.004	0.076	0.091	0.072	0.095	4.44%	8.87%	13.31%	0.079	0.088
Pb, ppm	54	2.6	49	60	47	62	4.69%	9.39%	14.08%	52	57
Pr, ppm	7.79	0.87	6.05	9.53	5.18	10.40	11.15%	22.31%	33.46%	7.40	8.18
Rb, ppm	160	6	148	172	141	178	3.85%	7.70%	11.55%	152	168
Re, ppm	0.083	0.004	0.075	0.091	0.071	0.095	4.87%	9.74%	14.60%	0.079	0.087
S, wt. %	0.829	0.045	0.739	0.920	0.693	0.965	5.46%	10.92%	16.38%	0.788	0.871
Sb, ppm	2.74	0.183	2.37	3.10	2.19	3.28	6.67%	13.35%	20.02%	2.60	2.87
Sc, ppm	8.24	0.545	7.15	9.33	6.61	9.88	6.61%	13.23%	19.84%	7.83	8.66
Se, ppm	3.98	0.47	3.04	4.93	2.56	5.40	11.86%	23.73%	35.59%	3.78	4.18
Sm, ppm	6.15	0.65	4.86	7.45	4.22	8.09	10.50%	21.00%	31.49%	5.85	6.46
Sn, ppm	4.87	0.312	4.25	5.49	3.93	5.80	6.40%	12.79%	19.19%	4.63	5.11
Sr, ppm	220	8	205	236	197	244	3.56%	7.12%	10.68%	209	231
Ta, ppm	1.01	0.076	0.85	1.16	0.78	1.23	7.54%	15.07%	22.61%	0.96	1.06
Tb, ppm	0.69	0.052	0.58	0.79	0.53	0.85	7.61%	15.22%	22.84%	0.65	0.72
Te, ppm	0.37	0.04	0.29	0.45	0.25	0.48	10.64%	21.28%	31.92%	0.35	0.39
Th, ppm	13.4	0.72	12.0	14.8	11.2	15.6	5.37%	10.74%	16.11%	12.7	14.1
Ti, wt. %	0.338	0.013	0.313	0.364	0.300	0.376	3.78%	7.55%	11.33%	0.321	0.355
Tl, ppm	0.94	0.040	0.86	1.02	0.82	1.06	4.27%	8.54%	12.81%	0.89	0.98
Tm, ppm	0.17	0.017	0.14	0.20	0.12	0.22	9.88%	19.76%	29.64%	0.16	0.18
U, ppm	3.85	0.320	3.21	4.49	2.89	4.81	8.30%	16.60%	24.91%	3.66	4.04
V, ppm	64	2.6	59	70	57	72	4.07%	8.13%	12.20%	61	68
W, ppm	11.9	0.83	10.3	13.6	9.4	14.4	6.93%	13.85%	20.78%	11.3	12.5
Y, ppm	14.5	0.80	12.9	16.1	12.1	16.9	5.51%	11.03%	16.54%	13.8	15.3

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
4-Acid Digestion continued											
Yb, ppm	1.14	0.12	0.89	1.38	0.77	1.50	10.72%	21.45%	32.17%	1.08	1.19
Zn, ppm	126	5	116	135	111	140	3.90%	7.80%	11.70%	119	132
Zr, ppm	64	5.7	52	75	46	81	8.97%	17.95%	26.92%	60	67
Aqua Regia Digestion											
Ag, ppm	1.58	0.108	1.37	1.80	1.26	1.91	6.81%	13.62%	20.42%	1.50	1.66
Al, wt. %	1.78	0.047	1.68	1.87	1.64	1.92	2.66%	5.32%	7.98%	1.69	1.87
As, ppm	20.1	0.91	18.3	22.0	17.4	22.9	4.51%	9.01%	13.52%	19.1	21.1
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	398	68	263	533	195	600	16.98%	33.96%	50.94%	378	418
Be, ppm	1.30	0.065	1.17	1.43	1.11	1.50	5.02%	10.04%	15.06%	1.24	1.37
Bi, ppm	1.76	0.164	1.43	2.09	1.27	2.25	9.31%	18.62%	27.93%	1.67	1.85
Ca, wt. %	0.554	0.022	0.510	0.599	0.488	0.621	4.00%	8.01%	12.01%	0.527	0.582
Cd, ppm	0.36	0.04	0.28	0.44	0.25	0.48	10.68%	21.35%	32.03%	0.34	0.38
Ce, ppm	30.1	2.36	25.4	34.8	23.0	37.2	7.85%	15.70%	23.54%	28.6	31.6
Co, ppm	8.81	0.501	7.81	9.82	7.31	10.32	5.69%	11.38%	17.07%	8.37	9.26
Cr, ppm	45.6	2.07	41.5	49.8	39.4	51.8	4.53%	9.06%	13.59%	43.4	47.9
Cs, ppm	7.42	0.502	6.42	8.43	5.91	8.93	6.77%	13.54%	20.31%	7.05	7.79
Cu, wt. %	0.610	0.019	0.573	0.647	0.555	0.666	3.04%	6.08%	9.12%	0.580	0.641
Fe, wt. %	2.95	0.082	2.79	3.11	2.70	3.20	2.78%	5.55%	8.33%	2.80	3.10
Ga, ppm	8.52	0.411	7.70	9.34	7.29	9.75	4.83%	9.65%	14.48%	8.09	8.95
Ge, ppm	0.10	0.02	0.06	0.14	0.04	0.16	20.11%	40.23%	60.34%	0.10	0.11
Hf, ppm	0.29	0.028	0.24	0.35	0.21	0.38	9.46%	18.92%	28.38%	0.28	0.31
Hg, ppm	0.046	0.016	0.015	0.077	0.000	0.092	33.86%	67.72%	101.6%	0.044	0.048
In, ppm	0.13	0.005	0.12	0.14	0.11	0.14	4.23%	8.45%	12.68%	0.12	0.13
K, wt. %	0.886	0.052	0.781	0.990	0.729	1.042	5.88%	11.76%	17.65%	0.841	0.930
La, ppm	13.7	1.00	11.7	15.7	10.7	16.7	7.29%	14.58%	21.88%	13.0	14.4
Li, ppm	37.6	1.64	34.3	40.8	32.7	42.5	4.36%	8.72%	13.07%	35.7	39.4
Mg, wt. %	0.641	0.024	0.592	0.690	0.567	0.714	3.82%	7.64%	11.45%	0.609	0.673
Mn, wt. %	0.029	0.001	0.026	0.031	0.025	0.032	4.05%	8.11%	12.16%	0.027	0.030
Mo, ppm	111	3	105	117	102	120	2.75%	5.49%	8.24%	105	116
Na, wt. %	0.143	0.018	0.108	0.178	0.090	0.195	12.26%	24.52%	36.79%	0.136	0.150
Nb, ppm	0.81	0.25	0.32	1.30	0.07	1.54	30.37%	60.73%	91.10%	0.77	0.85
Ni, ppm	15.3	0.62	14.1	16.6	13.5	17.2	4.03%	8.06%	12.09%	14.6	16.1
P, wt. %	0.063	0.003	0.057	0.068	0.054	0.071	4.37%	8.75%	13.12%	0.059	0.066
Pb, ppm	35.3	1.56	32.2	38.4	30.6	40.0	4.43%	8.86%	13.28%	33.5	37.1
Rb, ppm	86	6.1	74	98	68	105	7.09%	14.18%	21.26%	82	91
Re, ppm	0.083	0.005	0.073	0.092	0.068	0.097	5.76%	11.51%	17.27%	0.078	0.087
S, wt. %	0.813	0.035	0.743	0.882	0.709	0.916	4.25%	8.51%	12.76%	0.772	0.853
Sb, ppm	1.89	0.28	1.33	2.45	1.05	2.73	14.83%	29.66%	44.49%	1.80	1.99
Sc, ppm	6.97	0.412	6.15	7.80	5.74	8.21	5.91%	11.82%	17.72%	6.62	7.32
Se, ppm	3.85	0.254	3.34	4.36	3.09	4.61	6.59%	13.17%	19.76%	3.66	4.04
Sn, ppm	3.31	0.107	3.10	3.53	2.99	3.63	3.24%	6.48%	9.71%	3.15	3.48
Sr, ppm	56	2.5	52	61	49	64	4.40%	8.80%	13.20%	54	59

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
Aqua Regia Digestion continued											
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.47	0.019	0.43	0.51	0.41	0.52	3.96%	7.92%	11.87%	0.45	0.49
Te, ppm	0.33	0.025	0.28	0.38	0.25	0.41	7.71%	15.42%	23.13%	0.31	0.35
Th, ppm	5.95	0.68	4.59	7.32	3.90	8.01	11.50%	23.00%	34.50%	5.66	6.25
Ti, wt. %	0.227	0.013	0.202	0.252	0.189	0.265	5.57%	11.13%	16.70%	0.215	0.238
Tl, ppm	0.60	0.035	0.53	0.67	0.49	0.70	5.91%	11.83%	17.74%	0.57	0.63
U, ppm	3.19	0.259	2.67	3.70	2.41	3.96	8.14%	16.28%	24.41%	3.03	3.34
V, ppm	54	1.5	51	57	50	59	2.73%	5.46%	8.19%	51	57
W, ppm	7.13	1.21	4.72	9.55	3.51	10.76	16.93%	33.86%	50.80%	6.78	7.49
Y, ppm	9.62	0.548	8.53	10.72	7.98	11.27	5.69%	11.38%	17.07%	9.14	10.11
Yb, ppm	0.69	0.032	0.63	0.75	0.59	0.78	4.59%	9.18%	13.77%	0.66	0.72
Zn, ppm	120	5	110	129	105	134	3.94%	7.89%	11.83%	114	126
Zr, ppm	7.83	0.96	5.92	9.74	4.96	10.70	12.21%	24.42%	36.63%	7.44	8.22

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.

## PREPARER AND SUPPLIER

Certified reference material OREAS 507b is prepared, certified and supplied by:



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2. Alex Stewart International, Mendoza, Argentina
3. ALS, Johannesburg, South Africa
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Malaga, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
11. Bureau Veritas Geoanalytical, Perth, WA, Australia
12. Bureau Veritas Minerals, Ankara, Central Anatolia, Turkey
13. CERTIMIN, Lima, Peru
14. Inspectorate (BV), Lima, Peru
15. Intertek, Cupang, Muntinlupa, Philippines
16. Intertek, Perth, WA, Australia
17. Intertek Minerals Ltd, Tarkwa, Western Region, Ghana
18. Labwest Minerals Analysis, Perth, WA, Australia
19. Paragon Geochemical Laboratories, Sparks, Nevada, USA
20. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
21. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
22. SGS, Ankara, Anatolia, Turkey
23. SGS, Randfontein, Gauteng, South Africa
24. SGS Canada Inc., Vancouver, BC, Canada
25. SGS de Mexico SA de CV, Cd. Industrial, Durango, Mexico
26. SGS del Peru, Lima, Peru
27. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
28. 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 2. Au by Aqua Regia Digestion (sample weights 10-50g) in OREAS 507b

SPC.1839.RR1.OREAS 507b.3.AR Digest 10-50g.Au.Lab.240913.102353.SN

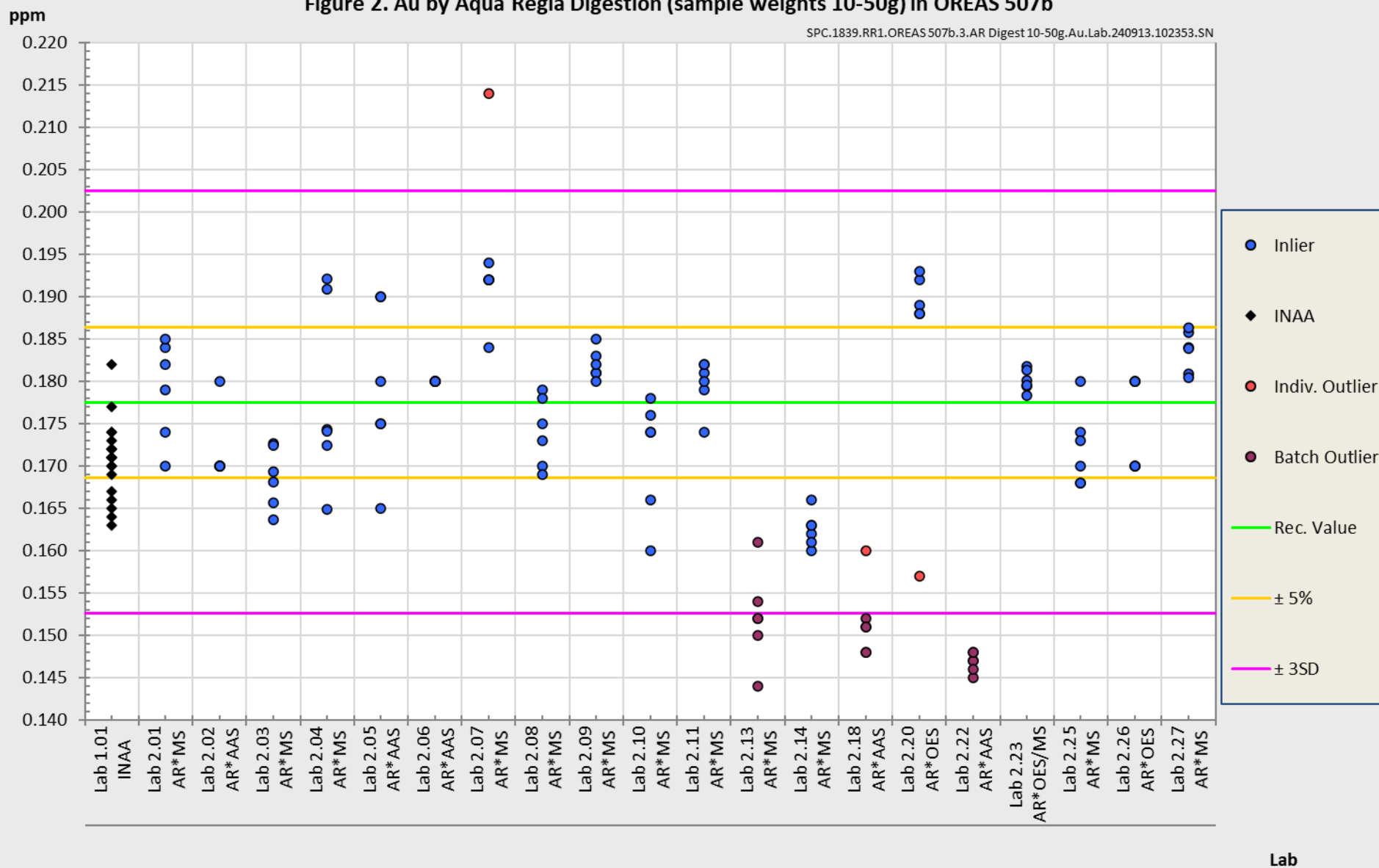


Figure 3. Ag by 4-Acid Digestion in OREAS 507b

SPC.1839.RR1.OREAS 507b.3.4-Acid.Ag.Lab.240917.143043.SN

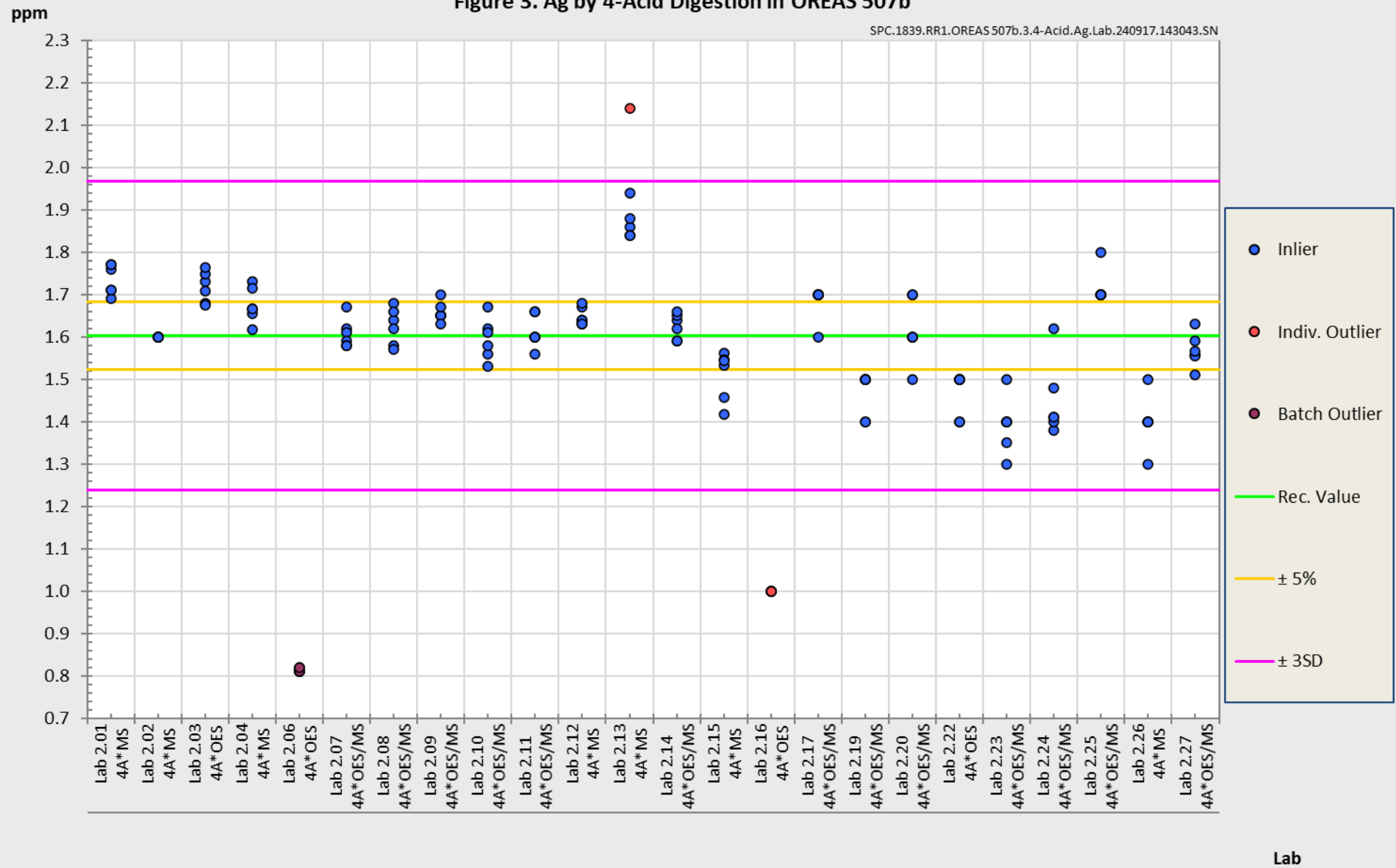
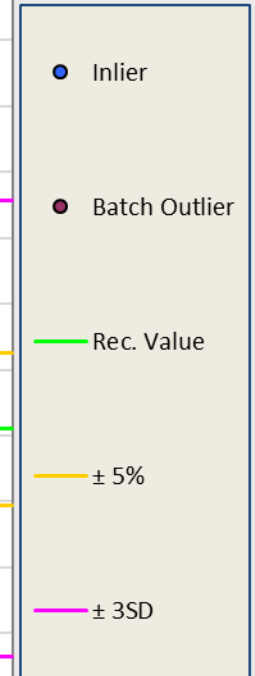
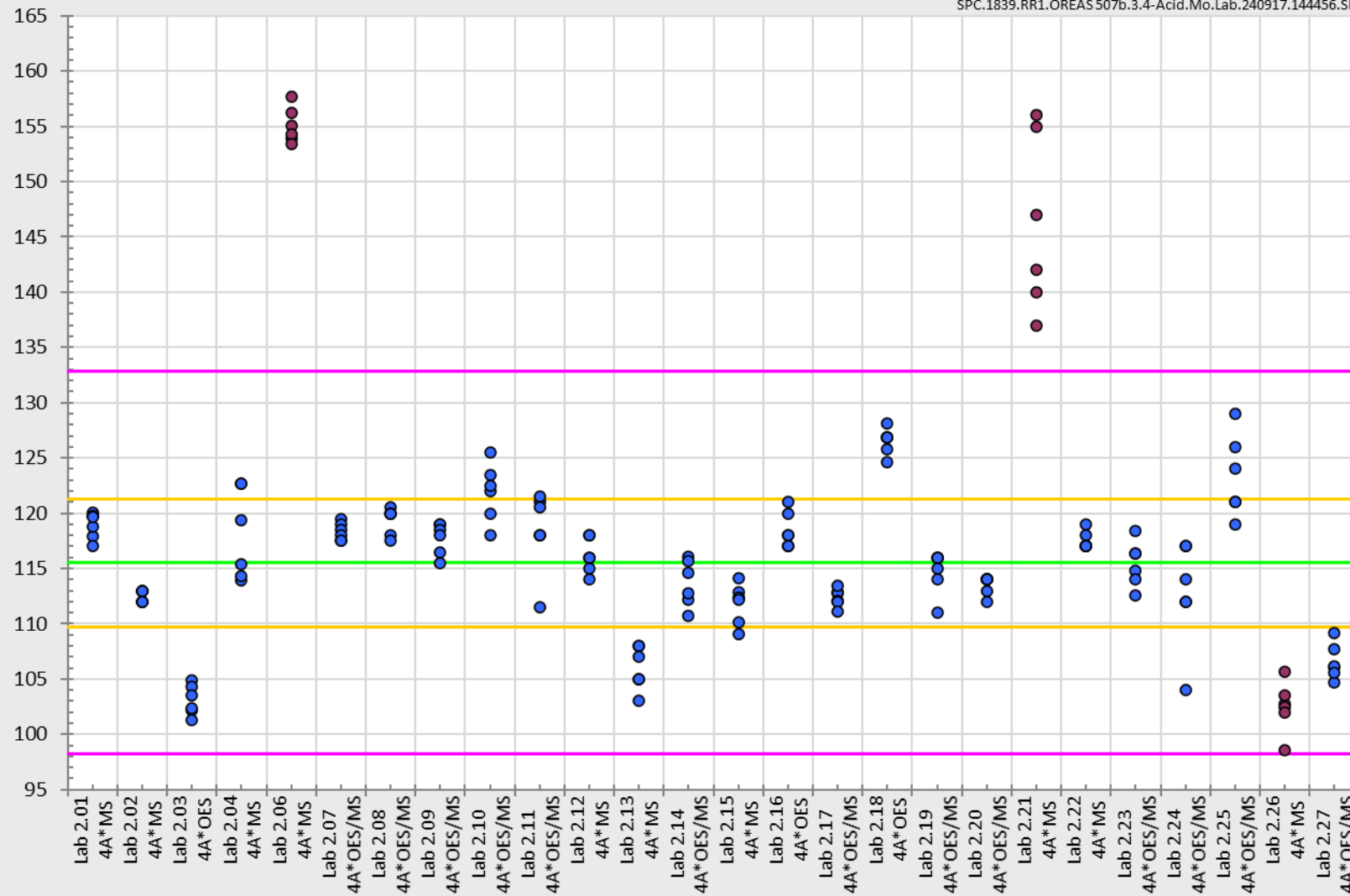




Figure 5. Mo by 4-Acid Digestion in OREAS 507b

SPC.1839.RR1.OREAS 507b.3,4-Acid.Mo.Lab.240917.144456.SN

ppm



Lab

## 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 representativeness was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [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 [8], section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *“Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 33405:2024-05, 9.2.4c) [5].”* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

## COMMUTABILITY

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



in the 'Source Material' section and users should select appropriate CRMs matching these attributes to the field samples being analysed.

## INTENDED USE

OREAS 507b 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 507b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 507b 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:  $\geq 15$  g;
- Gold by aqua regia digestion:  $\geq 10$  g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25$  g;
- Multi-elements by aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5$  g.

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 507b remains valid, within the specified measurement uncertainties, until April 2029, 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 507b contains a low level of Sulphur (0.8 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.

## Repeat-use packaging (500 g unit)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 507b 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.

\*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 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, particularly 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

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## DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	3 <sup>rd</sup> October, 2024	First publication.

## CERTIFYING OFFICER



3<sup>rd</sup> October, 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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