



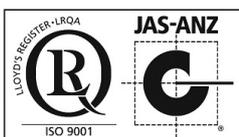
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**CERTIFICATE OF ANALYSIS FOR**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 932b**

**Copper Ore (Cobar, NSW, Australia)**



COA-1652-OREAS932-R0  
BUP-70-10-01 Ver:2.0

30-Jun-2023

**Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 932b.**

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	26.8	26.0	27.6	26.3	27.3
Al, Aluminium (wt.%)	4.91	4.78	5.03	4.80	5.02
As, Arsenic (ppm)	88	85	92	86	91
Be, Beryllium (ppm)	1.79	1.69	1.89	1.74	1.84
Bi, Bismuth (ppm)	331	316	346	322	341
Ca, Calcium (wt.%)	0.125	0.121	0.130	0.122	0.129
Cd, Cadmium (ppm)	0.98	0.89	1.07	0.93	1.03
Ce, Cerium (ppm)	63	59	68	60	66
Co, Cobalt (ppm)	46.0	44.2	47.8	44.9	47.1
Cr, Chromium (ppm)	65	60	69	62	67
Cs, Caesium (ppm)	5.06	4.83	5.30	4.91	5.22
Cu, Copper (wt.%)	6.27	6.12	6.42	6.14	6.40
Dy, Dysprosium (ppm)	2.68	2.45	2.91	2.55	2.80
Er, Erbium (ppm)	1.31	1.21	1.42	1.22	1.40
Eu, Europium (ppm)	1.02	0.95	1.08	0.97	1.06
Fe, Iron (wt.%)	11.81	11.47	12.15	11.60	12.02
Ga, Gallium (ppm)	16.0	15.2	16.8	15.4	16.5
Gd, Gadolinium (ppm)	3.93	3.40	4.46	3.72	4.15
Hf, Hafnium (ppm)	1.63	1.49	1.76	1.54	1.71
Ho, Holmium (ppm)	0.44	0.36	0.53	0.42	0.46
In, Indium (ppm)	5.18	4.89	5.47	5.02	5.34
K, Potassium (wt.%)	1.87	1.83	1.92	1.83	1.92
La, Lanthanum (ppm)	29.2	26.7	31.6	27.7	30.7
Li, Lithium (ppm)	14.6	14.0	15.1	14.1	15.0
Lu, Lutetium (ppm)	0.20	0.17	0.23	IND	IND
Mg, Magnesium (wt.%)	0.380	0.366	0.393	0.371	0.388
Mn, Manganese (wt.%)	0.024	0.023	0.025	0.024	0.025
Mo, Molybdenum (ppm)	3.66	3.47	3.86	3.49	3.83
Na, Sodium (wt.%)	0.068	0.066	0.070	0.066	0.070
Nb, Niobium (ppm)	2.33	2.02	2.65	2.11	2.56
Nd, Neodymium (ppm)	27.9	25.7	30.1	27.1	28.7
Ni, Nickel (ppm)	13.5	12.8	14.1	13.0	13.9
P, Phosphorus (wt.%)	0.026	0.024	0.027	0.025	0.027
Pb, Lead (ppm)	176	170	182	171	181
Pr, Praseodymium (ppm)	7.00	5.91	8.09	6.78	7.22
Rb, Rubidium (ppm)	109	103	115	105	113
Re, Rhenium (ppm)	0.003	0.001	0.004	IND	IND

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

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

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
S, Sulphur (wt.%)	6.55	6.34	6.75	6.42	6.68
Sb, Antimony (ppm)	4.29	3.99	4.59	4.11	4.47
Sc, Scandium (ppm)	8.87	8.43	9.32	8.45	9.30
Se, Selenium (ppm)	71	66	75	68	73
Sm, Samarium (ppm)	5.19	4.64	5.73	4.93	5.45
Sn, Tin (ppm)	58	55	60	56	60
Sr, Strontium (ppm)	42.7	40.6	44.9	41.0	44.5
Ta, Tantalum (ppm)	0.16	0.14	0.19	0.12	0.20
Tb, Terbium (ppm)	0.53	0.50	0.57	0.51	0.56
Te, Tellurium (ppm)	0.22	0.17	0.26	0.19	0.25
Th, Thorium (ppm)	9.51	8.79	10.22	9.04	9.97
Ti, Titanium (wt.%)	0.131	0.123	0.139	0.127	0.135
Tl, Thallium (ppm)	0.64	0.61	0.68	0.63	0.66
Tm, Thulium (ppm)	0.18	0.16	0.21	IND	IND
U, Uranium (ppm)	3.10	2.96	3.24	3.01	3.19
V, Vanadium (ppm)	120	115	124	117	122
W, Tungsten (ppm)	29.5	28.1	30.9	28.4	30.5
Y, Yttrium (ppm)	12.2	11.2	13.3	11.6	12.9
Yb, Ytterbium (ppm)	1.24	1.09	1.39	1.16	1.32
Zn, Zinc (ppm)	426	414	438	419	433
Zr, Zirconium (ppm)	55	53	58	54	57
<b>Aqua Regia Digestion</b>					
Ag, Silver (ppm)	26.9	25.9	27.9	26.3	27.5
Al, Aluminium (wt.%)	0.842	0.804	0.881	0.820	0.865
As, Arsenic (ppm)	84	80	87	82	86
Au, Gold (ppm)	0.018	0.016	0.020	0.016	0.020
B, Boron (ppm)	< 10	IND	IND	IND	IND
Be, Beryllium (ppm)	0.40	0.35	0.45	0.38	0.42
Bi, Bismuth (ppm)	319	302	336	308	329
Ca, Calcium (wt.%)	0.120	0.115	0.125	0.116	0.124
Cd, Cadmium (ppm)	0.95	0.85	1.05	0.91	0.99
Ce, Cerium (ppm)	43.2	40.5	45.9	41.8	44.7
Co, Cobalt (ppm)	45.2	43.8	46.6	44.3	46.1
Cr, Chromium (ppm)	21.1	20.2	22.1	20.3	21.9
Cs, Caesium (ppm)	2.66	2.51	2.81	2.58	2.74
Cu, Copper (wt.%)	6.17	6.05	6.28	6.10	6.24
Fe, Iron (wt.%)	11.59	11.30	11.87	11.40	11.77

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

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Ga, Gallium (ppm)	3.91	3.63	4.19	3.80	4.02
Ge, Germanium (ppm)	0.24	0.19	0.28	0.22	0.26
Hf, Hafnium (ppm)	0.32	0.30	0.35	0.30	0.34
Hg, Mercury (ppm)	0.16	0.14	0.19	0.15	0.18
In, Indium (ppm)	5.21	4.92	5.50	5.06	5.36
K, Potassium (wt.%)	0.185	0.174	0.197	0.177	0.194
La, Lanthanum (ppm)	16.3	15.1	17.5	15.5	17.0
Li, Lithium (ppm)	2.19	1.93	2.45	2.08	2.30
Lu, Lutetium (ppm)	0.086	0.065	0.106	IND	IND
Mg, Magnesium (wt.%)	0.185	0.176	0.195	0.179	0.192
Mn, Manganese (wt.%)	0.021	0.020	0.022	0.020	0.021
Mo, Molybdenum (ppm)	3.71	3.48	3.95	3.61	3.82
Na, Sodium (wt.%)	0.010	0.009	0.011	IND	IND
Nb, Niobium (ppm)	0.082	0.064	0.099	IND	IND
Ni, Nickel (ppm)	11.4	10.8	12.1	11.1	11.7
P, Phosphorus (wt.%)	0.020	0.019	0.021	0.020	0.021
Pb, Lead (ppm)	170	165	174	166	173
Rb, Rubidium (ppm)	14.9	13.5	16.2	14.3	15.4
Re, Rhenium (ppm)	0.002	0.002	0.003	IND	IND
S, Sulphur (wt.%)	6.65	6.24	7.05	6.48	6.81
Sb, Antimony (ppm)	2.94	2.66	3.21	2.82	3.05
Sc, Scandium (ppm)	1.56	1.42	1.71	IND	IND
Se, Selenium (ppm)	71	67	75	69	73
Sn, Tin (ppm)	52	49	54	50	53
Sr, Strontium (ppm)	14.9	13.9	16.0	14.4	15.5
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.32	0.26	0.38	0.31	0.33
Te, Tellurium (ppm)	0.21	0.19	0.24	0.19	0.24
Th, Thorium (ppm)	6.58	6.24	6.91	6.37	6.79
Ti, Titanium (wt.%)	0.008	0.007	0.009	0.007	0.009
Tl, Thallium (ppm)	0.12	0.11	0.13	IND	IND
U, Uranium (ppm)	1.90	1.82	1.97	1.85	1.95
V, Vanadium (ppm)	19.1	17.8	20.4	18.2	20.0
W, Tungsten (ppm)	24.3	22.8	25.8	23.6	25.1
Y, Yttrium (ppm)	5.30	4.99	5.62	5.15	5.45
Yb, Ytterbium (ppm)	0.56	0.50	0.61	IND	IND

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

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 an upper bound/non-detect limit value).

Note: intervals may appear asymmetric due to rounding.

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Zn, Zinc (ppm)	406	394	418	398	414
Zr, Zirconium (ppm)	10.3	9.7	10.9	9.9	10.7
<b>Borate Fusion XRF</b>					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	9.51	9.36	9.66	9.41	9.61
BaO, Barium oxide (ppm)	1668	1555	1781	1623	1713
CaO, Calcium oxide (wt.%)	0.171	0.159	0.184	IND	IND
Cu, Copper (wt.%)	6.22	6.09	6.34	6.16	6.28
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	17.22	17.08	17.37	17.10	17.34
K <sub>2</sub> O, Potassium oxide (wt.%)	2.21	2.03	2.39	2.19	2.23
MgO, Magnesium oxide (wt.%)	0.665	0.563	0.767	0.630	0.701
MnO, Manganese oxide (wt.%)	0.032	0.022	0.042	IND	IND
S, Sulphur (wt.%)	6.82	6.55	7.09	6.74	6.91
SiO <sub>2</sub> , Silicon dioxide (wt.%)	55.45	54.74	56.16	55.08	55.82
Zn, Zinc (ppm)	407	361	453	387	427
<b>Borate / Peroxide Fusion ICP</b>					
Al, Aluminium (wt.%)	5.05	4.94	5.15	4.96	5.13
As, Arsenic (ppm)	87	78	96	82	93
B, Boron (ppm)	103	76	130	94	112
Ba, Barium (ppm)	1478	1437	1518	1445	1511
Be, Beryllium (ppm)	1.98	1.77	2.19	IND	IND
Bi, Bismuth (ppm)	339	322	356	330	347
Ca, Calcium (wt.%)	0.129	0.101	0.157	0.122	0.136
Ce, Cerium (ppm)	69	65	73	66	72
Co, Cobalt (ppm)	44.0	41.9	46.1	42.4	45.6
Cr, Chromium (ppm)	83	72	95	75	92
Cs, Caesium (ppm)	5.16	4.86	5.46	4.87	5.45
Cu, Copper (wt.%)	6.22	6.08	6.35	6.10	6.33
Dy, Dysprosium (ppm)	3.78	3.36	4.19	3.44	4.11
Er, Erbium (ppm)	2.11	1.93	2.29	1.99	2.23
Eu, Europium (ppm)	1.14	1.00	1.28	1.04	1.24
Fe, Iron (wt.%)	12.01	11.78	12.23	11.83	12.18
Ga, Gallium (ppm)	16.3	15.4	17.1	15.4	17.1
Gd, Gadolinium (ppm)	4.67	4.07	5.27	4.20	5.14
Ge, Germanium (ppm)	2.09	1.43	2.75	IND	IND
Ho, Holmium (ppm)	0.71	0.63	0.79	0.66	0.75
In, Indium (ppm)	5.26	4.73	5.79	5.02	5.50
K, Potassium (wt.%)	1.92	1.87	1.97	1.86	1.98

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

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

**Table 1 continued.**

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Borate / Peroxide Fusion ICP continued</b>					
La, Lanthanum (ppm)	33.5	30.6	36.3	31.6	35.3
Li, Lithium (ppm)	16.4	14.1	18.8	IND	IND
Lu, Lutetium (ppm)	0.31	0.27	0.35	0.28	0.34
Mg, Magnesium (wt.%)	0.400	0.387	0.413	0.390	0.410
Mn, Manganese (wt.%)	0.025	0.023	0.026	0.024	0.025
Mo, Molybdenum (ppm)	4.28	3.02	5.53	IND	IND
Nb, Niobium (ppm)	9.56	6.86	12.26	8.50	10.62
Nd, Neodymium (ppm)	28.6	26.2	31.0	26.9	30.3
P, Phosphorus (wt.%)	0.028	0.020	0.036	IND	IND
Pb, Lead (ppm)	179	162	197	171	187
Pr, Praseodymium (ppm)	7.74	7.22	8.26	7.28	8.21
Rb, Rubidium (ppm)	107	102	113	104	111
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND
S, Sulphur (wt.%)	6.96	6.77	7.15	6.84	7.08
Sb, Antimony (ppm)	4.37	3.83	4.92	4.13	4.61
Sc, Scandium (ppm)	7.87	6.53	9.20	IND	IND
Se, Selenium (ppm)	72	63	81	65	79
Si, Silicon (wt.%)	26.63	26.04	27.22	26.29	26.98
Sm, Samarium (ppm)	5.19	4.69	5.69	4.69	5.69
Sn, Tin (ppm)	65	59	70	61	68
Sr, Strontium (ppm)	49.7	46.6	52.9	47.8	51.7
Ta, Tantalum (ppm)	0.72	0.59	0.85	IND	IND
Tb, Terbium (ppm)	0.66	0.61	0.71	0.61	0.71
Th, Thorium (ppm)	10.2	9.4	11.1	9.7	10.7
Ti, Titanium (wt.%)	0.231	0.224	0.238	0.227	0.235
Tl, Thallium (ppm)	0.65	0.54	0.76	IND	IND
Tm, Thulium (ppm)	0.30	0.25	0.35	0.27	0.33
U, Uranium (ppm)	3.42	3.15	3.68	3.19	3.65
V, Vanadium (ppm)	126	119	133	123	129
W, Tungsten (ppm)	28.0	24.4	31.6	26.6	29.5
Y, Yttrium (ppm)	20.6	19.1	22.0	19.7	21.5
Yb, Ytterbium (ppm)	1.99	1.84	2.14	1.80	2.18
Zn, Zinc (ppm)	415	395	434	403	426
<b>Infrared Combustion</b>					
S, Sulphur (wt.%)	7.02	6.90	7.14	6.94	7.10

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

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 an upper bound/non-detect limit value).

Note: intervals may appear asymmetric due to rounding.

**Table 2. Indicative Values for OREAS 932b.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>4-Acid Digestion</b>								
B	ppm	66	Ge	ppm	0.85			
Ba	ppm	306	Hg	ppm	0.22			
<b>Aqua Regia Digestion</b>								
Ba	ppm	67	Gd	ppm	2.54	Pr	ppm	4.60
Dy	ppm	1.53	Ho	ppm	0.26	Pt	ppb	< 5
Er	ppm	0.63	Nd	ppm	14.1	Sm	ppm	3.32
Eu	ppm	0.62	Pd	ppb	193	Tm	ppm	0.083
<b>Borate Fusion XRF</b>								
Bi	ppm	309	Na <sub>2</sub> O	wt. %	0.139	SrO	ppm	65
Ce	ppm	< 80	Nb	ppm	< 53.20112	Ta	ppm	< 80
Cl	ppm	< 498.372	Nd	ppm	< 90	Th	ppm	< 44
Co	ppm	52	Ni	ppm	226	TiO <sub>2</sub>	wt. %	0.394
Cr <sub>2</sub> O <sub>3</sub>	ppm	115	P <sub>2</sub> O <sub>5</sub>	wt. %	0.070	TOT_XRF	wt. %	100.03
F	ppm	< 3000	Pb	ppm	185	U	ppm	< 42
Ga	ppm	< 70	Pr	ppm	< 80	V <sub>2</sub> O <sub>5</sub>	ppm	237
Gd	ppm	< 90	Rb	ppm	115	W	ppm	< 80
Hf	ppm	< 80	Sb	ppm	< 170	Y	ppm	< 39
La	ppm	< 170	Sm	ppm	< 170	ZrO <sub>2</sub>	ppm	< 50
Mo	ppm	< 33	Sn	ppm	< 160			
<b>Borate / Peroxide Fusion ICP</b>								
Ag	ppm	27.6	Na	wt. %	0.058	Zr	ppm	76
Cd	ppm	1.09	Ni	ppm	23.5			
Hf	ppm	2.16	Te	ppm	< 1			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt. %	6.20						
<b>Infrared Combustion</b>								
C	wt. %	0.587						

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 OF CONTENTS

INTRODUCTION .....	9
SOURCE MATERIAL.....	9
COMMUNITION AND HOMOGENISATION PROCEDURES .....	9
PHYSICAL PROPERTIES .....	10
ANALYTICAL PROGRAM.....	10
STATISTICAL ANALYSIS.....	10
PERFORMANCE GATES .....	11
Homogeneity Evaluation .....	16
PARTICIPATING LABORATORIES.....	17
PREPARER AND SUPPLIER.....	19
METROLOGICAL TRACEABILITY .....	19
COMMUTABILITY .....	20
INTENDED USE .....	20
PERIOD OF VALIDITY & STORAGE INSTRUCTIONS.....	20
INSTRUCTIONS FOR HANDLING & CORRECT USE.....	21
LEGAL NOTICE.....	22
DOCUMENT HISTORY .....	22
QMS CERTIFICATION .....	22
CERTIFYING OFFICER.....	23
REFERENCES .....	23

## LIST OF TABLES

Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 932b. ....	2
Table 2. Indicative Values for OREAS 932b. ....	7
Table 3. Physical properties of OREAS 932b. ....	10
Table 4. Performance Gates for OREAS 932b. ....	12

## LIST OF FIGURES

Figure 1. Cu by 4-acid digestion in OREAS 932b .....	18
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## INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself. In evaluating laboratory performance with this CRM, the section headed 'Instructions for correct use' should be read carefully.

Tables 1 provide the certified values and their associated 95% expanded uncertainty and tolerance intervals, Table 2 shows indicative values including major and trace element characterisation, Table 3 provides some indicative physical properties and Table 4 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 932b-DataPack.1.0.230602\_170311.xlsx**).

Results are also presented in scatter plots for copper 4-acid digestion (Figure 1) 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 932b was prepared from a blend of barren black slate and mine waste rock. The barren black slate was sourced from a quarry located in Victoria, Australia and the mine waste rock was sourced from the CSA mine located near the town of Cobar in central western New South Wales, Australia. The copper ore body of the CSA mine is hosted by the Early Devonian CSA Siltstone, a thinly bedded turbiditic sequence of carbonaceous siltstones and mudstones with minor coarser units. Iron-rich chlorite and silica are prominent alterations in the siltstone host.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 932b was prepared in the following manner:

- Drying of black slate and copper ore to constant mass at 105°C and 85°C respectively;
- Crushing and milling of black slate to >98% minus 75 microns;
- Crushing and multi stage milling of the copper ore to 100% passing 30 microns;
- Blending the black slate and copper ore in appropriate proportions to achieve the target Cu grade;
- Homogenisation using OREAS' novel processing technologies;
- Packaging into 10g units sealed under nitrogen in laminated foil pouches.

## PHYSICAL PROPERTIES

OREAS 932b was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

**Table 3. Physical properties of OREAS 932b.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
757	0.74	N3	Dark 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.

## ANALYTICAL PROGRAM

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

- 4-acid (HF-HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) digestion for full ICP-OES and ICP-MS elemental suites (up to 23 laboratories depending on the element);
- Aqua regia digestion for full ICP-OES and ICP-MS elemental suites (up to 23 laboratories depending on the element);
- Peroxide fusion for full ICP-OES and ICP-MS elemental suites with the exception of one laboratory that used borate fusion with ICP-OES (up to 21 laboratories depending on the element)
- Al<sub>2</sub>O<sub>3</sub>, BaO, CaO, Cu, Fe<sub>2</sub>O<sub>3</sub>, Pb, S, SiO<sub>2</sub> and Zn by oxidising fusion XRF (up to 14 laboratories depending on the element);
- Total S by IR combustion furnace (22 laboratories).

For the round robin program ten 350g test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate 350g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e., to ascertain whether between-unit variance is greater than within-unit variance.

## STATISTICAL ANALYSIS

**Standard Deviation** intervals (see Table 1) provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program (see Intended Use section for more detail).

The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e., after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e., the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. ***The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.***

**Certified Values, Standard Deviations, Confidence Limits and Tolerance Limits** (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances, statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

**95% Confidence Limits** are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. ***95% Confidence Limits should not be used as control limits for laboratory performance.***

**Indicative (uncertified) values** (Table 2) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where inter-laboratory consensus is poor.

## PERFORMANCE GATES

Table 4 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 4. Performance Gates for OREAS 932b.**

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</b>											
Ag, ppm	26.8	0.81	25.2	28.4	24.4	29.2	3.02%	6.04%	9.05%	25.5	28.2
Al, wt. %	4.91	0.146	4.62	5.20	4.47	5.35	2.98%	5.96%	8.94%	4.66	5.15
As, ppm	88	4.0	80	96	76	100	4.55%	9.11%	13.66%	84	93
Be, ppm	1.79	0.135	1.52	2.06	1.38	2.19	7.54%	15.08%	22.63%	1.70	1.88
Bi, ppm	331	26	279	383	253	409	7.85%	15.70%	23.56%	315	348
Ca, wt. %	0.125	0.006	0.114	0.137	0.109	0.142	4.48%	8.96%	13.44%	0.119	0.132
Cd, ppm	0.98	0.12	0.74	1.22	0.62	1.34	12.21%	24.42%	36.63%	0.93	1.03
Ce, ppm	63	6.2	51	76	45	82	9.75%	19.51%	29.26%	60	67
Co, ppm	46.0	1.77	42.5	49.6	40.7	51.3	3.86%	7.72%	11.57%	43.7	48.3
Cr, ppm	65	5.8	53	76	47	82	8.95%	17.90%	26.84%	61	68
Cs, ppm	5.06	0.368	4.33	5.80	3.96	6.17	7.26%	14.52%	21.78%	4.81	5.32
Cu, wt. %	6.27	0.172	5.93	6.61	5.76	6.79	2.74%	5.47%	8.21%	5.96	6.58
Dy, ppm	2.68	0.112	2.45	2.90	2.34	3.01	4.19%	8.38%	12.57%	2.54	2.81
Er, ppm	1.31	0.061	1.19	1.43	1.13	1.49	4.62%	9.24%	13.86%	1.25	1.38
Eu, ppm	1.02	0.043	0.93	1.10	0.89	1.15	4.24%	8.49%	12.73%	0.97	1.07
Fe, wt. %	11.81	0.475	10.86	12.76	10.39	13.24	4.02%	8.04%	12.07%	11.22	12.40
Ga, ppm	16.0	0.97	14.0	17.9	13.1	18.9	6.10%	12.19%	18.29%	15.2	16.8
Gd, ppm	3.93	0.40	3.14	4.73	2.74	5.12	10.12%	20.24%	30.35%	3.73	4.13
Hf, ppm	1.63	0.104	1.42	1.83	1.31	1.94	6.38%	12.76%	19.14%	1.54	1.71
Ho, ppm	0.44	0.05	0.33	0.55	0.28	0.60	12.19%	24.38%	36.57%	0.42	0.46
In, ppm	5.18	0.250	4.68	5.68	4.43	5.93	4.83%	9.66%	14.49%	4.92	5.44
K, wt. %	1.87	0.075	1.72	2.02	1.65	2.10	3.98%	7.96%	11.94%	1.78	1.97
La, ppm	29.2	4.0	21.2	37.1	17.3	41.1	13.61%	27.21%	40.82%	27.7	30.6
Li, ppm	14.6	0.78	13.0	16.1	12.2	16.9	5.33%	10.66%	15.99%	13.8	15.3
Lu, ppm	0.20	0.018	0.16	0.24	0.15	0.26	9.14%	18.28%	27.42%	0.19	0.21
Mg, wt. %	0.380	0.025	0.330	0.429	0.306	0.453	6.48%	12.97%	19.45%	0.361	0.399
Mn, wt. %	0.024	0.001	0.022	0.026	0.021	0.027	4.65%	9.31%	13.96%	0.023	0.025
Mo, ppm	3.66	0.258	3.14	4.18	2.89	4.44	7.05%	14.11%	21.16%	3.48	3.84
Na, wt. %	0.068	0.004	0.059	0.077	0.055	0.081	6.41%	12.81%	19.22%	0.064	0.071
Nb, ppm	2.33	0.53	1.27	3.40	0.74	3.93	22.77%	45.54%	68.31%	2.22	2.45
Nd, ppm	27.9	1.39	25.1	30.7	23.7	32.1	4.98%	9.97%	14.95%	26.5	29.3
Ni, ppm	13.5	0.83	11.8	15.1	11.0	16.0	6.13%	12.25%	18.38%	12.8	14.2
Pb, ppm	176	8	161	191	153	199	4.30%	8.60%	12.90%	167	185
Pr, ppm	7.00	1.02	4.96	9.04	3.94	10.05	14.55%	29.09%	43.64%	6.65	7.35
Rb, ppm	109	5	99	119	94	124	4.54%	9.08%	13.62%	104	115
Re, ppm	0.003	0.001	0.001	0.004	0.001	0.004	21.24%	42.49%	63.73%	0.002	0.003
S, wt. %	6.55	0.276	6.00	7.10	5.72	7.38	4.22%	8.44%	12.66%	6.22	6.88
Sb, ppm	4.29	0.373	3.55	5.04	3.17	5.41	8.69%	17.37%	26.06%	4.08	4.51
Sc, ppm	8.87	0.495	7.88	9.86	7.39	10.36	5.57%	11.15%	16.72%	8.43	9.32
Se, ppm	71	7	56	85	49	92	10.12%	20.23%	30.35%	67	74
Sm, ppm	5.19	0.487	4.22	6.16	3.73	6.65	9.38%	18.76%	28.14%	4.93	5.45
Sn, ppm	58	3.2	51	64	48	67	5.56%	11.13%	16.69%	55	61
Sr, ppm	42.7	1.95	38.8	46.6	36.9	48.6	4.56%	9.13%	13.69%	40.6	44.9

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt. %.

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 4 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>											
Ta, ppm	0.16	0.02	0.12	0.21	0.09	0.23	13.95%	27.90%	41.85%	0.15	0.17
Tb, ppm	0.53	0.047	0.44	0.63	0.39	0.67	8.85%	17.69%	26.54%	0.51	0.56
Te, ppm	0.22	0.03	0.16	0.27	0.14	0.29	12.05%	24.10%	36.16%	0.21	0.23
Th, ppm	9.51	0.881	7.74	11.27	6.86	12.15	9.27%	18.55%	27.82%	9.03	9.98
Ti, wt. %	0.131	0.012	0.107	0.154	0.095	0.166	9.02%	18.04%	27.07%	0.124	0.137
Tl, ppm	0.64	0.034	0.58	0.71	0.54	0.74	5.21%	10.43%	15.64%	0.61	0.68
Tm, ppm	0.18	0.02	0.14	0.23	0.12	0.25	12.21%	24.43%	36.64%	0.17	0.19
U, ppm	3.10	0.179	2.74	3.46	2.56	3.64	5.78%	11.55%	17.33%	2.94	3.25
V, ppm	120	6	108	132	102	138	5.08%	10.15%	15.23%	114	126
W, ppm	29.5	1.90	25.6	33.3	23.7	35.2	6.47%	12.93%	19.40%	28.0	30.9
Y, ppm	12.2	1.9	8.4	16.1	6.5	18.0	15.59%	31.18%	46.76%	11.6	12.9
Yb, ppm	1.24	0.14	0.95	1.53	0.81	1.67	11.61%	23.22%	34.83%	1.18	1.30
Zn, ppm	426	19	388	464	369	483	4.45%	8.91%	13.36%	404	447
Zr, ppm	55	3.3	49	62	45	65	5.98%	11.96%	17.94%	53	58
<b>Aqua Regia Digestion</b>											
Ag, ppm	26.9	2.09	22.7	31.1	20.6	33.2	7.78%	15.56%	23.34%	25.6	28.2
Al, wt. %	0.842	0.072	0.699	0.986	0.627	1.058	8.51%	17.02%	25.53%	0.800	0.885
As, ppm	84	5.0	74	94	68	99	6.02%	12.03%	18.05%	79	88
Au, ppm	0.018	0.003	0.012	0.024	0.009	0.027	16.21%	32.41%	48.62%	0.017	0.019
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.40	0.05	0.30	0.50	0.25	0.55	12.43%	24.86%	37.29%	0.38	0.42
Bi, ppm	319	30	259	379	229	409	9.40%	18.80%	28.19%	303	335
Ca, wt. %	0.120	0.006	0.107	0.133	0.101	0.139	5.28%	10.55%	15.83%	0.114	0.126
Cd, ppm	0.95	0.16	0.63	1.27	0.47	1.43	16.90%	33.81%	50.71%	0.90	0.99
Ce, ppm	43.2	4.3	34.5	51.9	30.2	56.3	10.06%	20.11%	30.17%	41.1	45.4
Co, ppm	45.2	1.97	41.2	49.1	39.3	51.1	4.36%	8.73%	13.09%	42.9	47.4
Cr, ppm	21.1	1.16	18.8	23.4	17.6	24.6	5.50%	11.00%	16.49%	20.1	22.2
Cs, ppm	2.66	0.243	2.17	3.14	1.93	3.39	9.14%	18.27%	27.41%	2.53	2.79
Cu, wt. %	6.17	0.162	5.84	6.49	5.68	6.65	2.63%	5.26%	7.90%	5.86	6.47
Fe, wt. %	11.59	0.277	11.03	12.14	10.75	12.42	2.39%	4.79%	7.18%	11.01	12.16
Ga, ppm	3.91	0.368	3.17	4.65	2.80	5.01	9.42%	18.84%	28.27%	3.71	4.10
Ge, ppm	0.24	0.04	0.16	0.32	0.12	0.36	16.85%	33.70%	50.55%	0.22	0.25
Hf, ppm	0.32	0.022	0.28	0.36	0.26	0.39	6.79%	13.59%	20.38%	0.30	0.34
Hg, ppm	0.16	0.03	0.11	0.22	0.08	0.24	16.46%	32.91%	49.37%	0.16	0.17
In, ppm	5.21	0.395	4.42	6.00	4.03	6.40	7.59%	15.17%	22.76%	4.95	5.47
K, wt. %	0.185	0.020	0.146	0.225	0.126	0.245	10.70%	21.40%	32.10%	0.176	0.195
La, ppm	16.3	1.9	12.5	20.1	10.6	21.9	11.58%	23.16%	34.74%	15.5	17.1
Li, ppm	2.19	0.36	1.48	2.90	1.12	3.25	16.24%	32.47%	48.71%	2.08	2.30
Lu, ppm	0.086	0.013	0.059	0.113	0.045	0.126	15.73%	31.45%	47.18%	0.081	0.090
Mg, wt. %	0.185	0.017	0.152	0.219	0.136	0.235	8.95%	17.90%	26.85%	0.176	0.195
Mn, wt. %	0.021	0.002	0.018	0.024	0.016	0.026	7.99%	15.98%	23.96%	0.020	0.022
Mo, ppm	3.71	0.361	2.99	4.44	2.63	4.80	9.73%	19.45%	29.18%	3.53	3.90
Na, wt. %	0.010	0.002	0.007	0.014	0.005	0.016	17.75%	35.49%	53.24%	0.010	0.011

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 4 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>											
Nb, ppm	0.082	0.025	0.032	0.131	0.007	0.156	30.30%	60.60%	90.89%	0.077	0.086
Ni, ppm	11.4	1.00	9.4	13.4	8.4	14.4	8.72%	17.44%	26.17%	10.9	12.0
P, wt. %	0.020	0.002	0.017	0.024	0.015	0.025	8.04%	16.08%	24.12%	0.019	0.021
Pb, ppm	170	8	153	186	145	194	4.80%	9.61%	14.41%	161	178
Rb, ppm	14.9	2.4	10.1	19.6	7.8	21.9	15.90%	31.80%	47.70%	14.1	15.6
Re, ppm	0.002	0.000	0.001	0.003	0.001	0.004	20.07%	40.14%	60.22%	0.002	0.002
S, wt. %	6.65	0.635	5.38	7.92	4.74	8.55	9.56%	19.12%	28.68%	6.31	6.98
Sb, ppm	2.94	0.47	2.00	3.87	1.54	4.33	15.88%	31.75%	47.63%	2.79	3.08
Sc, ppm	1.56	0.18	1.20	1.93	1.02	2.11	11.66%	23.32%	34.98%	1.49	1.64
Se, ppm	71	6.9	57	85	50	92	9.78%	19.56%	29.34%	67	74
Sn, ppm	52	3.1	45	58	42	61	5.91%	11.83%	17.74%	49	54
Sr, ppm	14.9	1.8	11.4	18.4	9.6	20.2	11.80%	23.60%	35.40%	14.2	15.7
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.32	0.04	0.23	0.41	0.19	0.45	13.65%	27.30%	40.95%	0.31	0.34
Te, ppm	0.21	0.020	0.17	0.26	0.15	0.28	9.51%	19.02%	28.53%	0.20	0.23
Th, ppm	6.58	0.529	5.52	7.63	4.99	8.16	8.04%	16.08%	24.11%	6.25	6.90
Ti, wt. %	0.008	0.002	0.004	0.012	0.002	0.014	25.87%	51.74%	77.60%	0.008	0.008
Tl, ppm	0.12	0.009	0.10	0.14	0.09	0.15	7.37%	14.74%	22.10%	0.12	0.13
U, ppm	1.90	0.112	1.67	2.12	1.56	2.23	5.89%	11.78%	17.67%	1.80	1.99
V, ppm	19.1	2.2	14.8	23.5	12.6	25.6	11.33%	22.66%	33.99%	18.2	20.1
W, ppm	24.3	3.0	18.4	30.3	15.4	33.3	12.23%	24.47%	36.70%	23.1	25.5
Y, ppm	5.30	0.53	4.24	6.37	3.71	6.90	10.05%	20.09%	30.14%	5.04	5.57
Yb, ppm	0.56	0.046	0.46	0.65	0.42	0.69	8.20%	16.40%	24.60%	0.53	0.58
Zn, ppm	406	12	382	430	370	442	2.99%	5.99%	8.98%	386	426
Zr, ppm	10.3	1.2	7.8	12.8	6.6	14.0	11.96%	23.91%	35.87%	9.8	10.8
<b>Borate Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	9.51	0.147	9.22	9.80	9.07	9.95	1.54%	3.08%	4.63%	9.04	9.99
BaO, ppm	1668	159	1351	1985	1192	2144	9.51%	19.01%	28.52%	1584	1751
CaO, wt. %	0.171	0.014	0.144	0.199	0.130	0.212	8.01%	16.02%	24.03%	0.163	0.180
Cu, wt. %	6.22	0.152	5.91	6.52	5.76	6.67	2.44%	4.89%	7.33%	5.91	6.53
Fe <sub>2</sub> O <sub>3</sub> , wt. %	17.22	0.145	16.93	17.51	16.79	17.66	0.84%	1.68%	2.53%	16.36	18.09
K <sub>2</sub> O, wt. %	2.21	0.133	1.94	2.48	1.81	2.61	6.03%	12.07%	18.10%	2.10	2.32
MgO, wt. %	0.665	0.072	0.521	0.809	0.449	0.881	10.82%	21.64%	32.47%	0.632	0.699
MnO, wt. %	0.032	0.007	0.018	0.045	0.012	0.051	20.84%	41.67%	62.51%	0.030	0.033
S, wt. %	6.82	0.361	6.10	7.54	5.74	7.91	5.29%	10.57%	15.86%	6.48	7.16
SiO <sub>2</sub> , wt. %	55.45	0.717	54.02	56.88	53.30	57.60	1.29%	2.59%	3.88%	52.68	58.22
Zn, ppm	407	48	311	502	264	550	11.74%	23.48%	35.22%	387	427
<b>Borate / Peroxide Fusion ICP</b>											
Al, wt. %	5.05	0.113	4.82	5.27	4.71	5.39	2.25%	4.49%	6.74%	4.79	5.30
As, ppm	87	7.0	73	101	66	108	7.99%	15.98%	23.98%	83	92
B, ppm	103	12	79	128	66	140	11.89%	23.78%	35.66%	98	108
Ba, ppm	1478	44	1389	1566	1345	1610	2.99%	5.98%	8.97%	1404	1552
Be, ppm	1.98	0.128	1.72	2.23	1.59	2.36	6.48%	12.96%	19.43%	1.88	2.08

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 4 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>Borate / Peroxide Fusion ICP continued</b>											
Bi, ppm	339	17	304	373	287	391	5.09%	10.17%	15.26%	322	356
Ca, wt. %	0.129	0.025	0.080	0.178	0.055	0.203	19.13%	38.25%	57.38%	0.122	0.135
Ce, ppm	69	3.8	61	77	58	80	5.52%	11.03%	16.55%	66	72
Co, ppm	44.0	4.6	34.7	53.3	30.1	57.9	10.56%	21.12%	31.68%	41.8	46.2
Cr, ppm	83	20	44	123	24	143	23.77%	47.54%	71.31%	79	88
Cs, ppm	5.16	0.223	4.71	5.61	4.49	5.83	4.33%	8.66%	12.99%	4.90	5.42
Cu, wt. %	6.22	0.127	5.96	6.47	5.83	6.60	2.04%	4.08%	6.13%	5.91	6.53
Dy, ppm	3.78	0.218	3.34	4.21	3.12	4.43	5.78%	11.56%	17.33%	3.59	3.96
Er, ppm	2.11	0.144	1.82	2.40	1.68	2.54	6.80%	13.61%	20.41%	2.00	2.22
Eu, ppm	1.14	0.12	0.90	1.38	0.78	1.50	10.53%	21.05%	31.58%	1.08	1.20
Fe, wt. %	12.01	0.200	11.61	12.41	11.41	12.61	1.67%	3.33%	5.00%	11.41	12.61
Ga, ppm	16.3	1.04	14.2	18.3	13.1	19.4	6.38%	12.76%	19.14%	15.5	17.1
Gd, ppm	4.67	0.443	3.78	5.55	3.34	6.00	9.48%	18.97%	28.45%	4.44	4.90
Ge, ppm	2.09	0.52	1.05	3.13	0.53	3.65	24.88%	49.76%	74.63%	1.99	2.20
Ho, ppm	0.71	0.062	0.58	0.83	0.52	0.89	8.75%	17.50%	26.24%	0.67	0.74
In, ppm	5.26	0.516	4.23	6.29	3.71	6.81	9.80%	19.60%	29.41%	5.00	5.53
K, wt. %	1.92	0.055	1.81	2.03	1.75	2.09	2.89%	5.78%	8.67%	1.82	2.02
La, ppm	33.5	3.11	27.2	39.7	24.1	42.8	9.29%	18.58%	27.87%	31.8	35.1
Li, ppm	16.4	2.5	11.4	21.4	8.9	23.9	15.20%	30.39%	45.59%	15.6	17.2
Lu, ppm	0.31	0.020	0.27	0.35	0.25	0.37	6.43%	12.86%	19.29%	0.30	0.33
Mg, wt. %	0.400	0.012	0.375	0.424	0.363	0.437	3.06%	6.12%	9.18%	0.380	0.420
Mn, wt. %	0.025	0.003	0.019	0.030	0.016	0.033	11.46%	22.92%	34.38%	0.023	0.026
Mo, ppm	4.28	1.29	1.69	6.86	0.40	8.15	30.21%	60.43%	90.64%	4.06	4.49
Nb, ppm	9.56	3.19	3.18	15.94	0.00	19.13	33.36%	66.72%	100.08%	9.08	10.04
Nd, ppm	28.6	1.92	24.8	32.5	22.9	34.4	6.69%	13.38%	20.07%	27.2	30.1
P, wt. %	0.028	0.004	0.019	0.037	0.015	0.042	15.87%	31.75%	47.62%	0.027	0.030
Pb, ppm	179	21	138	221	117	242	11.61%	23.21%	34.82%	170	188
Pr, ppm	7.74	0.397	6.95	8.54	6.55	8.93	5.13%	10.26%	15.39%	7.36	8.13
Rb, ppm	107	6	96	119	90	125	5.51%	11.02%	16.52%	102	113
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	6.96	0.226	6.51	7.41	6.28	7.64	3.25%	6.51%	9.76%	6.61	7.31
Sb, ppm	4.37	0.52	3.33	5.42	2.81	5.94	11.95%	23.90%	35.85%	4.16	4.59
Sc, ppm	7.87	1.16	5.55	10.18	4.39	11.34	14.72%	29.44%	44.16%	7.47	8.26
Se, ppm	72	5.4	61	83	56	89	7.50%	15.00%	22.50%	69	76
Si, wt. %	26.63	0.869	24.89	28.37	24.03	29.24	3.26%	6.53%	9.79%	25.30	27.96
Sm, ppm	5.19	0.293	4.60	5.78	4.31	6.07	5.65%	11.31%	16.96%	4.93	5.45
Sn, ppm	65	6.2	52	77	46	83	9.59%	19.18%	28.77%	61	68
Sr, ppm	49.7	2.82	44.1	55.4	41.3	58.2	5.68%	11.35%	17.03%	47.3	52.2
Ta, ppm	0.72	0.13	0.45	0.98	0.32	1.11	18.44%	36.87%	55.31%	0.68	0.75
Tb, ppm	0.66	0.048	0.57	0.76	0.52	0.80	7.20%	14.41%	21.61%	0.63	0.69
Th, ppm	10.2	1.02	8.2	12.2	7.2	13.3	9.95%	19.90%	29.85%	9.7	10.7
Ti, wt. %	0.231	0.004	0.223	0.239	0.220	0.243	1.66%	3.32%	4.98%	0.220	0.243
Tl, ppm	0.65	0.07	0.51	0.79	0.44	0.85	10.58%	21.16%	31.74%	0.62	0.68

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 4 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>Borate / Peroxide Fusion ICP continued</b>											
Tm, ppm	0.30	0.03	0.24	0.36	0.21	0.40	10.37%	20.74%	31.12%	0.29	0.32
U, ppm	3.42	0.35	2.72	4.11	2.38	4.46	10.15%	20.30%	30.44%	3.25	3.59
V, ppm	126	7	112	140	105	147	5.55%	11.10%	16.65%	120	132
W, ppm	28.0	4.8	18.5	37.6	13.7	42.4	17.07%	34.15%	51.22%	26.6	29.4
Y, ppm	20.6	1.17	18.2	22.9	17.1	24.1	5.68%	11.37%	17.05%	19.6	21.6
Yb, ppm	1.99	0.127	1.74	2.24	1.61	2.37	6.36%	12.73%	19.09%	1.89	2.09
Zn, ppm	415	19	377	452	359	470	4.50%	8.99%	13.49%	394	435
<b>Infrared Combustion</b>											
S, wt. %	7.02	0.151	6.72	7.32	6.57	7.47	2.15%	4.29%	6.44%	6.67	7.37

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

### Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Tables 4 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for copper by 4-acid digestion, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 6.14 and 6.40 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 (ISO Guide 35).

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

The homogeneity of OREAS 932b has also been evaluated in a **nested ANOVA** of the round robin program. Each of the twenty-three round robin laboratories received six samples per CRM and these samples were made up of paired samples from three different, non-adjacent sampling intervals selected from the pool of ten 350g test units. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between-units to that of the variance within-units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 932b. The test was performed using the following parameters:

- 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 datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of  $p$ -values. This process derived no significant  $p$ -values across the entire extent of certified values. The null hypothesis is retained.

Only results for constituents present in concentrations well above the detection levels (i.e.,  $>20 \times$  Lower Limit of Detection) for the various methods undertaken were considered for the

objective of evaluating homogeneity. It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 932b and whether the variance between two subsamples from the same unit is statistically distinguishable from the variance of two subsamples taken from any two separate units. A reference material therefore can possess poor absolute homogeneity yet still pass a relative homogeneity (ANOVA) test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 932b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

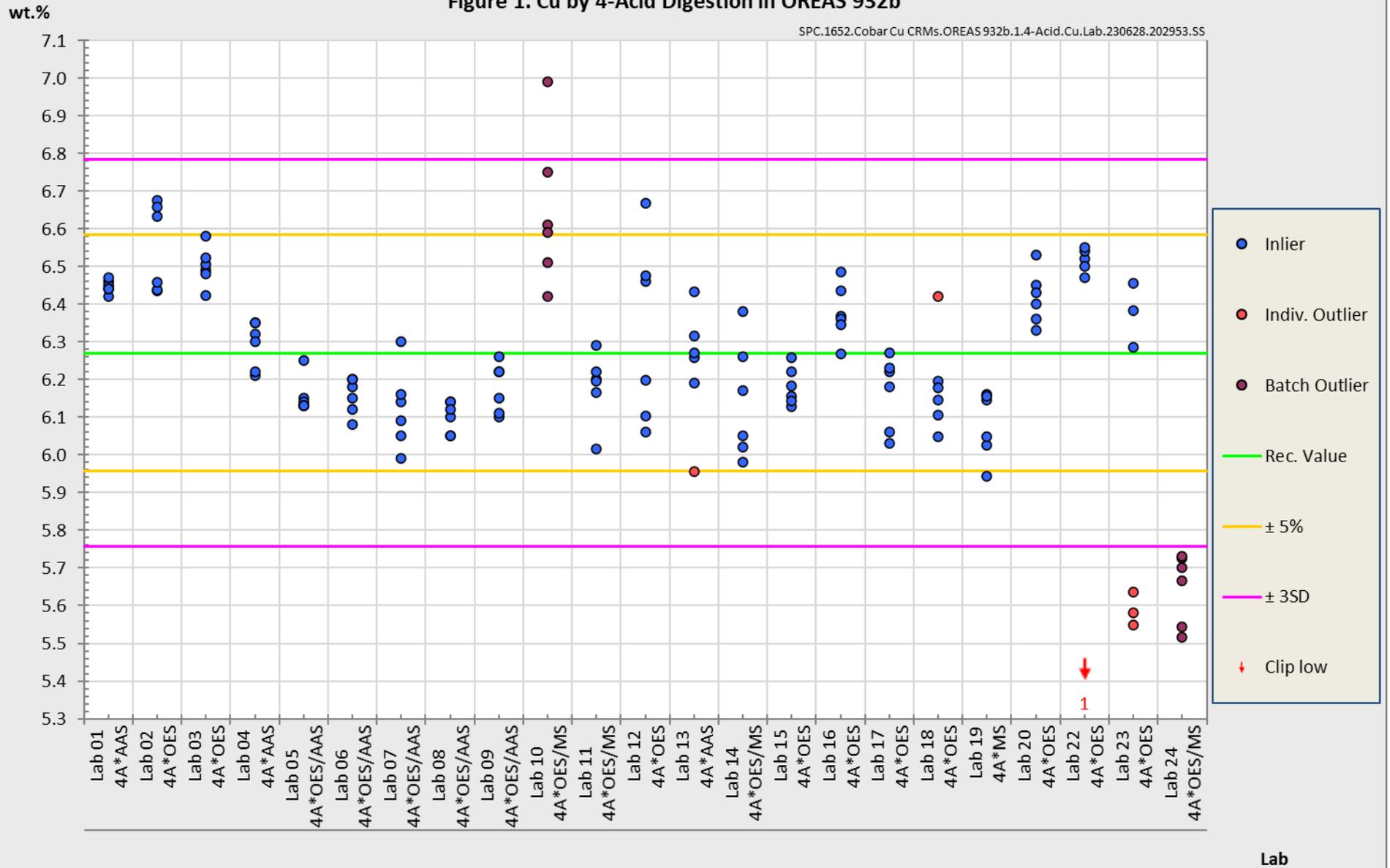
## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Perth, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
10. ESAN Istanbul, Istanbul, Turkey
11. Inspectorate (BV), Lima, Peru
12. Intertek Genalysis, Perth, WA, Australia
13. Intertek Testing Services, Townsville, QLD, Australia
14. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
15. MSALABS, Vancouver, BC, Canada
16. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
17. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
18. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
19. SGS, Randfontein, Gauteng, South Africa
20. SGS Canada Inc., Vancouver, BC, Canada
21. SGS de Mexico SA de CV, Cd. Industrial, Durango, Mexico
22. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
23. UIS Analytical Services, Centurion, South Africa

***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. Cu by 4-Acid Digestion in OREAS 932b

SPC.1652.Cobar Cu CRMs.OREAS 932b.1.4-Acid.Cu.Lab.230628.202953.SS



## PREPARER AND SUPPLIER

Certified reference material OREAS 932b 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. The operationally defined measurands characterised in this certificate are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 35:2017, 9.2.4c)."* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

## COMMUTABILITY

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

## INTENDED USE

OREAS 932b 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 932b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 932b 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).

## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 932b remains valid, within the specified measurement uncertainties, until March 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 931b is sulphide rich (7 wt.% S) and is packaged in single-use sachets sealed under nitrogen. 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. The certified values contained in this report refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis.

### **Minimum sample size**

As a practical guide, 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 sample masses should be used depending on the operationally defined methodology.

- 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25\text{g}$ ;
- Aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5\text{g}$ ;
- Peroxide fusion for full elemental suite with ICP-OES and/or MS finish:  $\geq 0.1\text{g}$ ;
- Oxidising fusion with X-ray fluorescence finish:  $\geq 0.2\text{g}$ ;
- S by infrared combustion furnace/CS analyser:  $\geq 0.1\text{g}$ .

### **QC monitoring using multiples of the Standard Deviation (SD)**

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-laboratory bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

The performance gates shown in Table 4 are intended only to be used as an initial 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, SDs 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% confidence interval 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

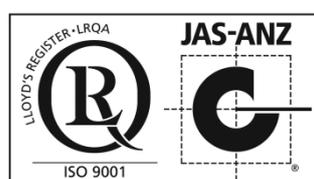
Revision No.	Date	Changes applied
0	28 <sup>th</sup> June, 2023	First publication.

## QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034.



ORE Pty Ltd is ISO 9001:2015 certified by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.



## CERTIFYING OFFICER



28<sup>th</sup> June, 2023

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

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