ORE RESEARCH & EXPLORATION P/L ABN 28 006 859 856
37A Hosie Street · Bayswater North · VIC 3153 · AUSTRALIA
61 3 9729 0333 Fx 61 3 9729 8338
info@ore.com.au www.oreas.com

CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 931b

Copper Ore (Cobar, NSW, Australia)

Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 931b.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion					
Ag, Silver (ppm)	16.9	16.3	17.5	16.5	17.2
Al, Aluminium (wt.%)	5.54	5.42	5.67	5.42	5.66
As, Arsenic (ppm)	99	94	103	96	101
Be, Beryllium (ppm)	2.08	1.94	2.22	2.03	2.14
Bi, Bismuth (ppm)	211	201	222	205	217
Ca, Calcium (wt.%)	0.083	0.079	0.088	0.081	0.086
Cd, Cadmium (ppm)	0.64	0.58	0.70	0.61	0.68
Ce, Cerium (ppm)	71	66	75	68	74
Co, Cobalt (ppm)	29.6	28.3	30.9	28.9	30.2
Cr, Chromium (ppm)	72	67	77	69	75
Cs, Caesium (ppm)	4.75	4.52	4.97	4.61	4.88
Cu, Copper (wt.%)	3.95	3.84	4.06	3.88	4.02
Dy, Dysprosium (ppm)	3.06	2.74	3.38	2.91	3.21
Er, Erbium (ppm)	1.37	1.26	1.48	1.30	1.45
Eu, Europium (ppm)	1.09	0.96	1.22	1.04	1.14
Fe, Iron (wt.%)	8.55	8.27	8.83	8.40	8.70
Ga, Gallium (ppm)	17.7	16.7	18.7	17.1	18.3
Gd, Gadolinium (ppm)	4.43	3.86	5.00	4.20	4.65
Ge, Germanium (ppm)	0.21	0.17	0.25	0.18	0.23
Hf, Hafnium (ppm)	1.82	1.73	1.90	1.75	1.88
Ho, Holmium (ppm)	0.49	0.44	0.53	0.46	0.51
In, Indium (ppm)	3.32	3.17	3.46	3.21	3.42
K, Potassium (wt.%)	2.18	2.10	2.25	2.13	2.22
La, Lanthanum (ppm)	32.3	30.0	34.7	31.0	33.7
Li, Lithium (ppm)	17.0	16.1	17.9	16.5	17.5
Lu, Lutetium (ppm)	0.22	0.19	0.25	0.21	0.24
Mg, Magnesium (wt.%)	0.354	0.339	0.369	0.344	0.364
Mn, Manganese (wt.%)	0.016	0.016	0.017	0.016	0.017
Mo, Molybdenum (ppm)	3.38	3.22	3.55	3.24	3.53
Na, Sodium (wt.%)	0.076	0.074	0.079	0.073	0.080
Nb, Niobium (ppm)	2.72	2.37	3.08	2.55	2.90
Nd, Neodymium (ppm)	29.9	27.0	32.7	29.0	30.8
Ni, Nickel (ppm)	12.6	11.9	13.3	12.1	13.1
P, Phosphorus (wt.%)	0.027	0.026	0.028	0.026	0.028
Pb, Lead (ppm)	121	115	127	118	125
Pr, Praseodymium (ppm)	8.12	7.28	8.97	7.89	8.35
Rb, Rubidium (ppm)	127	122	131	124	129

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
Note: intervals may appear asymmetric due to rounding.

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Re, Rhenium (ppm)	0.002	0.001	0.003	IND	IND
S, Sulphur (wt.%)	4.30	4.20	4.40	4.22	4.38
Sb, Antimony (ppm)	4.22	3.97	4.46	4.04	4.39
Sc, Scandium (ppm)	10.1	9.6	10.6	9.8	10.4
Se, Selenium (ppm)	45.9	43.2	48.7	43.9	48.0
Sm, Samarium (ppm)	5.77	5.28	6.27	5.50	6.05
Sn, Tin (ppm)	37.4	35.6	39.2	36.5	38.3
Sr, Strontium (ppm)	46.6	44.3	48.9	45.0	48.3
Ta, Tantalum (ppm)	0.20	0.16	0.24	0.16	0.24
Tb, Terbium (ppm)	0.59	0.52	0.65	0.56	0.62
Te, Tellurium (ppm)	0.17	0.14	0.21	IND	IND
Th, Thorium (ppm)	11.3	10.6	12.0	10.9	11.7
Ti, Titanium (wt.%)	0.150	0.141	0.159	0.146	0.155
Tl, Thallium (ppm)	0.71	0.67	0.75	0.69	0.73
Tm, Thulium (ppm)	0.20	0.19	0.21	IND	IND
U, Uranium (ppm)	3.46	3.28	3.63	3.35	3.56
V, Vanadium (ppm)	138	133	143	135	141
W, Tungsten (ppm)	18.8	18.0	19.6	18.2	19.4
Y, Yttrium (ppm)	13.5	12.4	14.7	13.0	14.1
Yb, Ytterbium (ppm)	1.38	1.23	1.53	1.29	1.47
Zn, Zinc (ppm)	287	276	298	282	292
Zr, Zirconium (ppm)	62	59	65	60	64
Aqua Regia Digestion					
Ag, Silver (ppm)	17.1	16.4	17.8	16.6	17.6
Al, Aluminium (wt.%)	0.727	0.689	0.766	0.708	0.747
As, Arsenic (ppm)	97	93	101	95	100
Au, Gold (ppm)	0.018	0.015	0.021	0.016	0.020
B, Boron (ppm)	< 10	IND	IND	IND	IND
Be, Beryllium (ppm)	0.44	0.37	0.50	0.41	0.46
Bi, Bismuth (ppm)	210	198	222	204	216
Ca, Calcium (wt.%)	0.079	0.076	0.081	0.076	0.081
Cd, Cadmium (ppm)	0.62	0.55	0.68	0.59	0.65
Ce, Cerium (ppm)	49.9	47.7	52.1	48.9	51.0
Co, Cobalt (ppm)	29.6	28.3	30.8	28.8	30.3
Cr, Chromium (ppm)	21.1	19.7	22.5	20.3	21.8
Cs, Caesium (ppm)	2.06	1.93	2.19	2.01	2.12
Cu, Copper (wt.%)	3.95	3.85	4.05	3.89	4.01

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
Aqua Regia Digestion continued					
Fe, Iron (wt.%)	8.29	8.06	8.52	8.14	8.44
Ga, Gallium (ppm)	3.25	3.00	3.50	3.12	3.38
Ge, Germanium (ppm)	0.18	0.14	0.21	IND	IND
Hf, Hafnium (ppm)	0.34	0.31	0.37	0.32	0.36
Hg, Mercury (ppm)	0.11	0.09	0.14	0.09	0.14
In, Indium (ppm)	3.24	3.13	3.34	3.16	3.32
K, Potassium (wt.%)	0.190	0.177	0.204	0.182	0.198
La, Lanthanum (ppm)	19.5	18.1	21.0	18.7	20.3
Li, Lithium (ppm)	2.03	1.74	2.32	1.86	2.20
Lu, Lutetium (ppm)	0.092	0.071	0.112	IND	IND
Mg, Magnesium (wt.%)	0.128	0.118	0.137	0.124	0.132
Mn, Manganese (wt.%)	0.014	0.013	0.014	0.013	0.014
Mo, Molybdenum (ppm)	3.40	3.22	3.58	3.26	3.54
Na, Sodium (wt.%)	0.010	0.009	0.011	IND	IND
Nb, Niobium (ppm)	0.056	0.043	0.068	IND	IND
Ni, Nickel (ppm)	10.2	9.6	10.8	9.8	10.6
P, Phosphorus (wt.%)	0.022	0.020	0.023	0.021	0.022
Pb, Lead (ppm)	115	111	119	112	118
Rb, Rubidium (ppm)	13.7	12.9	14.6	13.1	14.4
Re, Rhenium (ppm)	0.001	0.000	0.002	IND	IND
S, Sulphur (wt.%)	4.25	4.11	4.39	4.15	4.35
Sb, Antimony (ppm)	2.94	2.64	3.25	2.82	3.06
Sc, Scandium (ppm)	1.47	1.34	1.61	IND	IND
Se, Selenium (ppm)	46.3	44.2	48.5	44.8	47.8
Sn, Tin (ppm)	34.6	32.4	36.8	33.5	35.7
Sr, Strontium (ppm)	13.5	12.0	15.0	13.0	14.0
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.36	0.29	0.43	0.33	0.39
Te, Tellurium (ppm)	0.16	0.14	0.19	0.10	0.23
Th, Thorium (ppm)	7.68	7.15	8.21	7.44	7.92
Ti, Titanium (wt.%)	0.007	0.006	0.008	0.006	0.007
Tl, Thallium (ppm)	0.11	0.10	0.12	IND	IND
U, Uranium (ppm)	2.08	1.98	2.19	2.02	2.15
V, Vanadium (ppm)	18.8	17.5	20.1	17.9	19.6
W, Tungsten (ppm)	13.7	12.7	14.6	13.1	14.2
Y, Yttrium (ppm)	5.53	5.20	5.86	5.35	5.70
Yb, Ytterbium (ppm)	0.61	0.58	0.64	IND	IND

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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 an upper bound/non-detect limit value).

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion continued					
Zn, Zinc (ppm)	272	265	279	267	277
Zr, Zirconium (ppm)	10.8	10.0	11.5	10.4	11.1
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	10.81	10.67	10.95	10.68	10.94
BaO, Barium oxide (ppm)	2009	1877	2140	1879	2138
CaO, Calcium oxide (wt.%)	0.115	0.104	0.126	IND	IND
Cu, Copper (wt.%)	3.95	3.86	4.04	3.90	4.00
Fe_2O_3, Iron(III) oxide (wt.%)	12.42	12.24	12.61	12.29	12.55
K_2O, Potassium oxide (wt.%)	2.55	2.41	2.70	2.52	2.59
MgO, Magnesium oxide (wt.%)	0.632	0.534	0.730	0.590	0.673
MnO, Manganese oxide (wt.%)	0.022	0.015	0.029	IND	IND
P_2O_5, Phosphorus(V) oxide (wt.%)	0.070	0.049	0.090	IND	IND
S, Sulphur (wt.%)	4.35	4.18	4.52	4.28	4.42
SiO_2, Silicon dioxide (wt.%)	62.36	61.66	63.07	61.77	62.95
TiO_2, Titanium dioxide (wt.%)	0.441	0.411	0.471	0.426	0.456
Zn, Zinc (ppm)	290	268	312	274	306
Borate / Peroxide Fusion ICP					
Al, Aluminium (wt.%)	5.73	5.60	5.87	5.64	5.83
As, Arsenic (ppm)	97	88	106	93	101
B, Boron (ppm)	114	97	130	101	127
Ba, Barium (ppm)	1739	1672	1806	1691	1788
Be, Beryllium (ppm)	2.06	1.98	2.13	IND	IND
Bi, Bismuth (ppm)	216	203	228	209	223
Ca, Calcium (wt.%)	0.096	0.071	0.121	IND	IND
Ce, Cerium (ppm)	76	71	81	73	80
Co, Cobalt (ppm)	28.4	26.9	29.8	27.1	29.7
Cr, Chromium (ppm)	91	82	100	84	98
Cs, Caesium (ppm)	4.92	4.60	5.23	4.67	5.17
Cu, Copper (wt.%)	3.96	3.84	4.07	3.88	4.04
Dy, Dysprosium (ppm)	4.12	3.90	4.33	3.88	4.35
Er, Erbium (ppm)	2.36	2.06	2.66	2.24	2.48
Eu, Europium (ppm)	1.26	1.10	1.41	1.14	1.37
Fe, Iron (wt.%)	8.68	8.49	8.86	8.55	8.80
Ga, Gallium (ppm)	18.3	16.7	19.9	17.2	19.4
Gd, Gadolinium (ppm)	4.94	4.48	5.41	4.64	5.24
Ge, Germanium (ppm)	2.33	1.69	2.96	1.95	2.71
Ho, Holmium (ppm)	0.81	0.74	0.88	0.76	0.86

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

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

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued					
In, Indium (ppm)	3.32	3.08	3.56	3.15	3.49
K, Potassium (wt.%)	2.24	2.18	2.31	2.19	2.30
La, Lanthanum (ppm)	37.2	34.4	39.9	35.4	38.9
Li, Lithium (ppm)	17.8	14.6	21.0	IND	IND
Lu, Lutetium (ppm)	0.34	0.30	0.38	0.31	0.37
Mg, Magnesium (wt.%)	0.376	0.360	0.391	0.363	0.389
Mn, Manganese (wt.%)	0.018	0.017	0.018	0.017	0.018
Mo, Molybdenum (ppm)	3.74	2.89	4.60	IND	IND
Nb, Niobium (ppm)	8.37	6.70	10.04	7.00	9.74
Nd, Neodymium (ppm)	31.6	29.7	33.6	30.3	33.0
P, Phosphorus (wt.%)	0.030	0.024	0.035	IND	IND
Pb, Lead (ppm)	121	106	136	114	127
Pr, Praseodymium (ppm)	8.45	8.01	8.89	8.04	8.86
Rb, Rubidium (ppm)	124	118	130	119	129
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND
S, Sulphur (wt.%)	4.42	4.28	4.57	4.32	4.53
Sb, Antimony (ppm)	3.99	3.14	4.84	3.72	4.26
Sc, Scandium (ppm)	9.44	7.97	10.92	IND	IND
Se, Selenium (ppm)	46.2	41.7	50.8	42.4	50.1
Si, Silicon (wt.%)	29.73	29.11	30.34	29.01	30.45
Sm, Samarium (ppm)	5.85	5.56	6.14	5.59	6.12
Sn, Tin (ppm)	45.1	39.8	50.3	41.5	48.6
Sr, Strontium (ppm)	54	50	58	52	56
Ta, Tantalum (ppm)	0.94	0.68	1.20	IND	IND
Tb, Terbium (ppm)	0.72	0.67	0.77	0.68	0.75
Th, Thorium (ppm)	11.7	10.7	12.7	11.0	12.4
Ti, Titanium (wt.%)	0.265	0.258	0.273	0.257	0.273
Tl, Thallium (ppm)	0.74	0.65	0.83	IND	IND
Tm, Thulium (ppm)	0.34	0.30	0.39	0.32	0.37
U, Uranium (ppm)	3.80	3.51	4.09	3.65	3.96
V, Vanadium (ppm)	144	138	150	139	149
W, Tungsten (ppm)	17.8	15.5	20.1	16.4	19.2
Y, Yttrium (ppm)	23.6	22.6	24.6	22.6	24.6
Yb, Ytterbium (ppm)	2.25	2.00	2.49	2.15	2.34
Zn, Zinc (ppm)	283	269	298	272	295
Infrared Combustion					
S, Sulphur (wt.%)	4.43	4.35	4.51	4.38	4.48

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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. Indicative Values for OREAS 931b.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
B	ppm	*51*	Ba	ppm	*581*	Hg	ppm	*0.16*
Aqua Regia Digestion								
Ba	ppm	*92*	Gd	ppm	*2.90*	Pr	ppm	*5.43*
Dy	ppm	*1.68*	Ho	ppm	*0.28*	Pt	ppb	*< 5*
Er	ppm	*0.67*	Nd	ppm	*17.0*	Sm	ppm	*3.83*
Eu	ppm	*0.60*	Pd	ppb	*150*	Tm	ppm	*0.090*
Borate Fusion XRF								
Bi	ppm	*212*	Mo	ppm	*< 33*	Sn	ppm	*< 160*
Ce	ppm	*< 80*	Na_2O	wt.%	*0.154*	SrO	ppm	*51*
Cl	ppm	*< 331.815*	Nb	ppm	*< 59.95051*	Ta	ppm	*< 80*
Co	ppm	*< 60*	Nd	ppm	*< 90*	Th	ppm	*< 44*
Cr_2O_3	ppm	*129*	Ni	ppm	*201*	TOT_XRF	wt.%	*99.92*
F	ppm	*< 3000*	Pb	ppm	*136*	U	ppm	*< 42*
Ga	ppm	*< 70*	Pr	ppm	*< 80*	V_2O_5	ppm	*282*
Gd	ppm	*< 90*	Rb	ppm	*140*	W	ppm	*< 80*
Hf	ppm	*< 80*	Sb	ppm	*< 170*	Y	ppm	*< 39*
La	ppm	*< 170*	Sm	ppm	*< 170*	ZrO_2	ppm	*45.0*
Borate / Peroxide Fusion ICP								
Ag	ppm	*17.2*	Na	wt.%	*0.080*	Zr	ppm	*82*
Cd	ppm	*0.64*	Ni	ppm	*21.9*			
Hf	ppm	*2.29*	Te	ppm	*< 1*			
Thermogravimetry								
LOI^{1000}	wt.%	*5.21*						
Infrared Combustion								
C	wt.%	*0.679*						

SI unit equivalents: ppb (parts per billion; 1 x 10^{-9}) ≡ µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
COMMINUTION 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 18
METROLOGICAL TRACEABILITY 20
COMMUTABILITY 20
INTENDED USE 21
PERIOD OF VALIDITY & STORAGE INSTRUCTIONS 21
INSTRUCTIONS FOR HANDLING & CORRECT USE 21
LEGAL NOTICE 23
DOCUMENT HISTORY 23
QMS CERTIFICATION 23
CERTIFYING OFFICER 23
REFERENCES 24

LIST OF TABLES

Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 931b. 2
Table 2. Indicative Values for OREAS 931b. 7
Table 3. Physical properties of OREAS 931b. 10
Table 4. Performance Gates for OREAS 931b. 12

LIST OF FIGURES

Figure 1. Cu by 4-acid digestion in OREAS 931b 19

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[3]) are presented in the detailed certification data for this CRM (**OREAS 931b-DataPack.1.0.230602_165625.xlsx**).

Results are also presented in scatter plots for copper 4-acid digestion (Figure 1) together with ±3SD (magenta) and ±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 931b was prepared from a blend of barren black slate and copper ore. The barren black slate was sourced from a quarry located in Victoria, Australia and the copper ore 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. The CSA Siltstone is part of the Cobar Supergroup, consisting of lower syn-rift sediments and upper post-rift sag phase sediments. The mineralisation is structurally controlled and confined to a number of steeply dipping bodies within a major shear zone on the eastern margin of the Early Devonian Cobar Basin. It is characterised by low-grade greenschist alteration and epigenetic low-grade mineralisation enveloping higher-grade shoots of vein complexes or sub-massive to massive sulphides. The sulphides include chalcopyrite, pyrrhotite, pyrite, sphalerite, galena, bornite and cubanite. Iron-rich chlorite and silica are prominent alterations in the siltstone host.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 931b 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 931b 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 931b.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
700	0.59	N3	Dark Gray

‡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_3-$HClO_4$-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_2O_3, BaO, CaO, Cu, Fe_2O_3, Pb, S, SiO_2 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). 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) ± 10%.

I.e., Certified Value ± 10% ± 2DL [1].

Table 4. Performance Gates for OREAS 931b.

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											
Ag, ppm	16.9	0.54	15.8	18.0	15.2	18.5	3.21%	6.43%	9.64%	16.0	17.7
Al, wt.%	5.54	0.155	5.23	5.85	5.08	6.01	2.80%	5.60%	8.40%	5.27	5.82
As, ppm	99	4.5	89	108	85	112	4.61%	9.22%	13.83%	94	103
Be, ppm	2.08	0.184	1.71	2.45	1.53	2.63	8.86%	17.71%	26.57%	1.98	2.19
Bi, ppm	211	15	181	241	166	256	7.09%	14.18%	21.27%	201	222
Ca, wt.%	0.083	0.005	0.074	0.092	0.070	0.097	5.43%	10.85%	16.28%	0.079	0.087
Cd, ppm	0.64	0.06	0.51	0.77	0.45	0.84	10.08%	20.16%	30.24%	0.61	0.68
Ce, ppm	71	5.1	60	81	55	86	7.20%	14.39%	21.59%	67	74
Co, ppm	29.6	1.41	26.7	32.4	25.3	33.8	4.77%	9.53%	14.30%	28.1	31.0
Cr, ppm	72	6.2	60	85	54	91	8.60%	17.20%	25.80%	69	76
Cs, ppm	4.75	0.261	4.22	5.27	3.96	5.53	5.49%	10.99%	16.48%	4.51	4.98
Cu, wt.%	3.95	0.124	3.70	4.20	3.57	4.32	3.15%	6.30%	9.45%	3.75	4.14
Dy, ppm	3.06	0.32	2.42	3.70	2.10	4.02	10.45%	20.91%	31.36%	2.91	3.21
Er, ppm	1.37	0.091	1.19	1.56	1.10	1.65	6.65%	13.30%	19.95%	1.31	1.44
Eu, ppm	1.09	0.106	0.88	1.30	0.77	1.41	9.71%	19.41%	29.12%	1.04	1.15
Fe, wt.%	8.55	0.341	7.87	9.23	7.53	9.58	3.99%	7.99%	11.98%	8.12	8.98
Ga, ppm	17.7	1.23	15.3	20.2	14.0	21.4	6.92%	13.85%	20.77%	16.8	18.6
Gd, ppm	4.43	0.411	3.61	5.25	3.20	5.66	9.28%	18.55%	27.83%	4.21	4.65
Ge, ppm	0.21	0.04	0.14	0.28	0.10	0.32	17.71%	35.43%	53.14%	0.20	0.22
Hf, ppm	1.82	0.080	1.65	1.98	1.57	2.06	4.42%	8.85%	13.27%	1.72	1.91
Ho, ppm	0.49	0.044	0.40	0.58	0.36	0.62	8.95%	17.91%	26.86%	0.46	0.51
In, ppm	3.32	0.145	3.03	3.60	2.88	3.75	4.36%	8.72%	13.08%	3.15	3.48
K, wt.%	2.18	0.083	2.01	2.34	1.93	2.42	3.81%	7.62%	11.43%	2.07	2.28
La, ppm	32.3	3.7	24.9	39.8	21.2	43.5	11.48%	22.95%	34.43%	30.7	34.0
Li, ppm	17.0	0.65	15.7	18.3	15.1	18.9	3.80%	7.60%	11.40%	16.1	17.8
Lu, ppm	0.22	0.017	0.19	0.26	0.17	0.27	7.46%	14.92%	22.38%	0.21	0.23
Mg, wt.%	0.354	0.019	0.315	0.393	0.296	0.412	5.46%	10.92%	16.38%	0.336	0.372
Mn, wt.%	0.016	0.001	0.015	0.017	0.014	0.018	4.00%	8.00%	11.99%	0.015	0.017
Mo, ppm	3.38	0.175	3.03	3.73	2.86	3.91	5.17%	10.35%	15.52%	3.21	3.55
Na, wt.%	0.076	0.005	0.067	0.086	0.062	0.091	6.27%	12.55%	18.82%	0.073	0.080
Nb, ppm	2.72	0.63	1.47	3.98	0.85	4.60	22.97%	45.95%	68.92%	2.59	2.86
Nd, ppm	29.9	2.52	24.8	34.9	22.3	37.4	8.44%	16.88%	25.32%	28.4	31.4
Ni, ppm	12.6	0.68	11.2	13.9	10.6	14.6	5.39%	10.78%	16.18%	12.0	13.2

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg ≡ µg/g ≡ 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
4-Acid Digestion continued											
P, wt.%	0.027	0.002	0.024	0.030	0.023	0.032	5.64%	11.29%	16.93%	0.026	0.029
Pb, ppm	121	7	108	134	101	141	5.45%	10.89%	16.34%	115	127
Pr, ppm	8.12	0.780	6.56	9.68	5.78	10.46	9.60%	19.20%	28.81%	7.72	8.53
Rb, ppm	127	4	118	135	114	139	3.23%	6.45%	9.68%	120	133
Re, ppm	0.002	0.001	0.001	0.003	0.000	0.004	36.63%	73.27%	109.90%	0.002	0.002
S, wt.%	4.30	0.159	3.98	4.62	3.82	4.77	3.69%	7.38%	11.07%	4.08	4.51
Sb, ppm	4.22	0.249	3.72	4.71	3.47	4.96	5.92%	11.84%	17.75%	4.00	4.43
Sc, ppm	10.1	0.57	8.9	11.2	8.4	11.8	5.68%	11.36%	17.05%	9.6	10.6
Se, ppm	45.9	3.47	39.0	52.9	35.5	56.4	7.55%	15.10%	22.65%	43.6	48.2
Sm, ppm	5.77	0.485	4.80	6.74	4.32	7.23	8.40%	16.80%	25.21%	5.48	6.06
Sn, ppm	37.4	1.13	35.1	39.7	34.0	40.8	3.03%	6.05%	9.08%	35.5	39.3
Sr, ppm	46.6	2.48	41.7	51.6	39.2	54.0	5.31%	10.62%	15.93%	44.3	48.9
Ta, ppm	0.20	0.05	0.10	0.30	0.04	0.35	25.83%	51.67%	77.50%	0.19	0.21
Tb, ppm	0.59	0.041	0.51	0.67	0.47	0.71	6.92%	13.85%	20.77%	0.56	0.62
Te, ppm	0.17	0.02	0.12	0.22	0.10	0.24	13.68%	27.37%	41.05%	0.16	0.18
Th, ppm	11.3	0.95	9.4	13.2	8.4	14.1	8.38%	16.77%	25.15%	10.7	11.9
Ti, wt.%	0.150	0.015	0.121	0.179	0.107	0.194	9.71%	19.41%	29.12%	0.143	0.158
Tl, ppm	0.71	0.036	0.64	0.78	0.60	0.82	5.04%	10.07%	15.11%	0.67	0.75
Tm, ppm	0.20	0.008	0.18	0.21	0.17	0.22	3.89%	7.77%	11.66%	0.19	0.21
U, ppm	3.46	0.195	3.07	3.85	2.87	4.04	5.64%	11.28%	16.93%	3.28	3.63
V, ppm	138	6	125	151	119	157	4.60%	9.20%	13.81%	131	145
W, ppm	18.8	0.85	17.1	20.5	16.2	21.3	4.53%	9.07%	13.60%	17.8	19.7
Y, ppm	13.5	1.9	9.7	17.4	7.8	19.3	14.11%	28.21%	42.32%	12.9	14.2
Yb, ppm	1.38	0.15	1.08	1.67	0.94	1.82	10.64%	21.29%	31.93%	1.31	1.45
Zn, ppm	287	11	265	309	254	320	3.78%	7.56%	11.34%	273	301
Zr, ppm	62	4.6	53	71	48	76	7.37%	14.74%	22.11%	59	65
Aqua Regia Digestion											
Ag, ppm	17.1	1.33	14.5	19.8	13.1	21.1	7.75%	15.51%	23.26%	16.3	18.0
Al, wt.%	0.727	0.059	0.609	0.846	0.549	0.906	8.17%	16.33%	24.50%	0.691	0.764
As, ppm	97	6.0	85	109	79	115	6.15%	12.30%	18.46%	92	102
Au, ppm	0.018	0.003	0.012	0.024	0.009	0.027	17.25%	34.49%	51.74%	0.017	0.019
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.44	0.08	0.27	0.61	0.18	0.69	19.51%	39.02%	58.53%	0.41	0.46
Bi, ppm	210	16	179	241	163	257	7.48%	14.95%	22.43%	199	220
Ca, wt.%	0.079	0.003	0.073	0.085	0.070	0.088	3.84%	7.68%	11.53%	0.075	0.083
Cd, ppm	0.62	0.09	0.44	0.79	0.36	0.88	14.14%	28.28%	42.42%	0.59	0.65
Ce, ppm	49.9	2.62	44.7	55.2	42.1	57.8	5.24%	10.48%	15.72%	47.4	52.4
Co, ppm	29.6	2.07	25.4	33.7	23.4	35.8	6.99%	13.99%	20.98%	28.1	31.0
Cr, ppm	21.1	0.70	19.7	22.5	19.0	23.2	3.31%	6.62%	9.93%	20.0	22.1
Cs, ppm	2.06	0.193	1.68	2.45	1.48	2.64	9.36%	18.72%	28.09%	1.96	2.17
Cu, wt.%	3.95	0.121	3.71	4.19	3.59	4.31	3.06%	6.13%	9.19%	3.75	4.15
Fe, wt.%	8.29	0.309	7.67	8.91	7.36	9.22	3.72%	7.45%	11.17%	7.88	8.70
Ga, ppm	3.25	0.40	2.45	4.04	2.06	4.44	12.22%	24.44%	36.66%	3.09	3.41

SI unit equivalents: ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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 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
Aqua Regia Digestion continued											
Ge, ppm	0.18	0.03	0.12	0.23	0.09	0.26	16.94%	33.89%	50.83%	0.17	0.18
Hf, ppm	0.34	0.022	0.30	0.38	0.27	0.40	6.36%	12.72%	19.08%	0.32	0.36
Hg, ppm	0.11	0.03	0.06	0.17	0.04	0.19	22.60%	45.21%	67.81%	0.11	0.12
In, ppm	3.24	0.124	2.99	3.49	2.87	3.61	3.83%	7.66%	11.49%	3.08	3.40
K, wt.%	0.190	0.020	0.149	0.231	0.129	0.252	10.77%	21.53%	32.30%	0.181	0.200
La, ppm	19.5	2.1	15.4	23.6	13.4	25.7	10.52%	21.04%	31.56%	18.5	20.5
Li, ppm	2.03	0.35	1.32	2.74	0.97	3.09	17.42%	34.85%	52.27%	1.93	2.13
Lu, ppm	0.092	0.012	0.068	0.115	0.057	0.126	12.63%	25.27%	37.90%	0.087	0.096
Mg, wt.%	0.128	0.012	0.104	0.152	0.092	0.164	9.43%	18.86%	28.29%	0.121	0.134
Mn, wt.%	0.014	0.001	0.012	0.015	0.011	0.016	5.99%	11.99%	17.98%	0.013	0.014
Mo, ppm	3.40	0.236	2.93	3.88	2.69	4.11	6.95%	13.90%	20.84%	3.23	3.57
Na, wt.%	0.010	0.002	0.007	0.014	0.005	0.015	16.71%	33.42%	50.13%	0.010	0.011
Nb, ppm	0.056	0.009	0.038	0.073	0.029	0.082	15.68%	31.36%	47.03%	0.053	0.058
Ni, ppm	10.2	0.77	8.7	11.7	7.9	12.5	7.52%	15.05%	22.57%	9.7	10.7
P, wt.%	0.022	0.003	0.017	0.027	0.014	0.029	11.53%	23.07%	34.60%	0.021	0.023
Pb, ppm	115	6	103	127	97	133	5.24%	10.47%	15.71%	109	121
Rb, ppm	13.7	1.06	11.6	15.9	10.6	16.9	7.72%	15.44%	23.17%	13.1	14.4
Re, ppm	0.001	0.000	0.000	0.002	0.000	0.003	35.53%	71.06%	106.59%	0.001	0.001
S, wt.%	4.25	0.216	3.82	4.68	3.60	4.90	5.09%	10.17%	15.26%	4.04	4.46
Sb, ppm	2.94	0.56	1.82	4.07	1.26	4.63	19.04%	38.07%	57.11%	2.80	3.09
Sc, ppm	1.47	0.17	1.13	1.82	0.95	1.99	11.75%	23.50%	35.26%	1.40	1.55
Se, ppm	46.3	3.07	40.2	52.5	37.1	55.5	6.63%	13.26%	19.89%	44.0	48.6
Sn, ppm	34.6	3.7	27.1	42.0	23.4	45.8	10.77%	21.54%	32.31%	32.8	36.3
Sr, ppm	13.5	2.5	8.6	18.4	6.1	20.9	18.23%	36.45%	54.68%	12.8	14.2
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.36	0.05	0.26	0.46	0.21	0.50	13.41%	26.82%	40.24%	0.34	0.38
Te, ppm	0.16	0.014	0.14	0.19	0.12	0.20	8.30%	16.60%	24.90%	0.16	0.17
Th, ppm	7.68	0.578	6.52	8.83	5.94	9.41	7.53%	15.06%	22.58%	7.29	8.06
Ti, wt.%	0.007	0.002	0.003	0.010	0.001	0.012	26.43%	52.85%	79.28%	0.006	0.007
Tl, ppm	0.11	0.010	0.09	0.13	0.08	0.14	9.15%	18.29%	27.44%	0.11	0.12
U, ppm	2.08	0.114	1.85	2.31	1.74	2.43	5.50%	11.00%	16.50%	1.98	2.19
V, ppm	18.8	2.3	14.2	23.4	11.9	25.7	12.20%	24.41%	36.61%	17.8	19.7
W, ppm	13.7	1.31	11.1	16.3	9.7	17.6	9.55%	19.10%	28.65%	13.0	14.3
Y, ppm	5.53	0.471	4.59	6.47	4.11	6.94	8.52%	17.05%	25.57%	5.25	5.81
Yb, ppm	0.61	0.027	0.55	0.66	0.53	0.69	4.41%	8.82%	13.23%	0.58	0.64
Zn, ppm	272	7	257	286	250	294	2.68%	5.35%	8.03%	258	286
Zr, ppm	10.8	1.3	8.3	13.3	7.0	14.5	11.62%	23.23%	34.85%	10.2	11.3
Borate Fusion XRF											
Al_2O_3, wt.%	10.81	0.137	10.54	11.08	10.40	11.22	1.27%	2.54%	3.81%	10.27	11.35
BaO, ppm	2009	144	1720	2297	1576	2441	7.18%	14.35%	21.53%	1908	2109
CaO, wt.%	0.115	0.011	0.094	0.136	0.083	0.147	9.22%	18.43%	27.65%	0.109	0.121
Cu, wt.%	3.95	0.109	3.73	4.17	3.62	4.28	2.76%	5.51%	8.27%	3.75	4.15
Fe_2O_3, wt.%	12.42	0.191	12.04	12.80	11.85	12.99	1.54%	3.07%	4.61%	11.80	13.04

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
Borate Fusion XRF continued											
K_2O, wt.%	2.55	0.102	2.35	2.76	2.25	2.86	4.01%	8.02%	12.03%	2.43	2.68
MgO, wt.%	0.632	0.068	0.496	0.768	0.428	0.836	10.75%	21.50%	32.25%	0.600	0.663
MnO, wt.%	0.022	0.005	0.011	0.033	0.006	0.039	24.62%	49.24%	73.86%	0.021	0.023
P_2O_5, wt.%	0.070	0.014	0.043	0.097	0.029	0.111	19.45%	38.91%	58.36%	0.066	0.073
S, wt.%	4.35	0.234	3.88	4.82	3.65	5.05	5.37%	10.73%	16.10%	4.13	4.57
SiO_2, wt.%	62.36	0.708	60.95	63.78	60.24	64.49	1.13%	2.27%	3.40%	59.24	65.48
TiO_2, wt.%	0.441	0.023	0.396	0.486	0.373	0.509	5.11%	10.22%	15.33%	0.419	0.463
Zn, ppm	290	25	240	340	215	365	8.61%	17.22%	25.83%	275	304
Borate / Peroxide Fusion ICP											
Al, wt.%	5.73	0.160	5.41	6.05	5.25	6.21	2.79%	5.57%	8.36%	5.45	6.02
As, ppm	97	9.5	78	116	68	125	9.80%	19.60%	29.41%	92	102
B, ppm	114	10	93	134	83	144	8.95%	17.89%	26.84%	108	119
Ba, ppm	1739	54	1632	1847	1578	1901	3.10%	6.20%	9.29%	1652	1826
Be, ppm	2.06	0.199	1.66	2.46	1.46	2.65	9.68%	19.37%	29.05%	1.95	2.16
Bi, ppm	216	11	193	239	182	250	5.31%	10.62%	15.94%	205	227
Ca, wt.%	0.096	0.025	0.047	0.145	0.022	0.170	25.58%	51.17%	76.75%	0.091	0.101
Ce, ppm	76	4.1	68	84	64	88	5.35%	10.69%	16.04%	72	80
Co, ppm	28.4	1.72	24.9	31.8	23.2	33.5	6.07%	12.14%	18.21%	27.0	29.8
Cr, ppm	91	18	54	128	36	146	20.17%	40.34%	60.51%	87	96
Cs, ppm	4.92	0.248	4.42	5.41	4.18	5.66	5.04%	10.08%	15.12%	4.67	5.16
Cu, wt.%	3.96	0.092	3.77	4.14	3.68	4.23	2.31%	4.62%	6.94%	3.76	4.16
Dy, ppm	4.12	0.193	3.73	4.50	3.54	4.69	4.68%	9.35%	14.03%	3.91	4.32
Er, ppm	2.36	0.152	2.06	2.67	1.91	2.82	6.42%	12.83%	19.25%	2.24	2.48
Eu, ppm	1.26	0.120	1.02	1.50	0.90	1.62	9.58%	19.15%	28.73%	1.19	1.32
Fe, wt.%	8.68	0.138	8.40	8.95	8.26	9.09	1.60%	3.19%	4.79%	8.24	9.11
Ga, ppm	18.3	2.1	14.2	22.4	12.1	24.5	11.25%	22.50%	33.75%	17.4	19.2
Gd, ppm	4.94	0.333	4.28	5.61	3.94	5.94	6.73%	13.46%	20.19%	4.69	5.19
Ge, ppm	2.33	0.48	1.36	3.29	0.88	3.78	20.75%	41.50%	62.25%	2.21	2.44
Ho, ppm	0.81	0.054	0.70	0.92	0.65	0.97	6.68%	13.37%	20.05%	0.77	0.85
In, ppm	3.32	0.192	2.94	3.70	2.74	3.90	5.79%	11.57%	17.36%	3.15	3.49
K, wt.%	2.24	0.072	2.10	2.39	2.03	2.46	3.20%	6.41%	9.61%	2.13	2.35
La, ppm	37.2	2.84	31.5	42.9	28.7	45.7	7.63%	15.25%	22.88%	35.3	39.0
Li, ppm	17.8	2.0	13.9	21.7	11.9	23.7	11.07%	22.13%	33.20%	16.9	18.7
Lu, ppm	0.34	0.023	0.29	0.38	0.27	0.41	6.72%	13.43%	20.15%	0.32	0.35
Mg, wt.%	0.376	0.015	0.345	0.406	0.330	0.421	4.06%	8.12%	12.18%	0.357	0.394
Mn, wt.%	0.018	0.002	0.014	0.021	0.012	0.023	11.24%	22.47%	33.71%	0.017	0.018
Mo, ppm	3.74	0.81	2.13	5.35	1.33	6.16	21.53%	43.05%	64.58%	3.55	3.93
Nb, ppm	8.37	1.36	5.64	11.10	4.28	12.46	16.29%	32.59%	48.88%	7.95	8.79
Nd, ppm	31.6	1.07	29.5	33.8	28.4	34.9	3.39%	6.78%	10.16%	30.1	33.2
P, wt.%	0.030	0.004	0.021	0.038	0.017	0.042	13.88%	27.76%	41.64%	0.028	0.031
Pb, ppm	121	20	80	162	60	182	16.83%	33.66%	50.49%	115	127
Pr, ppm	8.45	0.360	7.73	9.17	7.37	9.53	4.26%	8.51%	12.77%	8.03	8.88
Rb, ppm	124	6	112	136	106	142	4.83%	9.66%	14.49%	118	130

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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 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
Borate / Peroxide Fusion ICP continued											
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	4.42	0.171	4.08	4.77	3.91	4.94	3.87%	7.74%	11.61%	4.20	4.64
Sb, ppm	3.99	0.73	2.54	5.44	1.81	6.17	18.18%	36.37%	54.55%	3.79	4.19
Sc, ppm	9.44	1.23	6.99	11.90	5.76	13.13	13.00%	26.00%	39.00%	8.97	9.92
Se, ppm	46.2	4.22	37.8	54.7	33.6	58.9	9.12%	18.25%	27.37%	43.9	48.6
Si, wt.%	29.73	0.851	28.03	31.43	27.17	32.28	2.86%	5.73%	8.59%	28.24	31.21
Sm, ppm	5.85	0.278	5.29	6.41	5.02	6.69	4.76%	9.51%	14.27%	5.56	6.14
Sn, ppm	45.1	5.0	35.0	55.1	30.0	60.1	11.12%	22.25%	33.37%	42.8	47.3
Sr, ppm	54	5.2	44	64	38	70	9.62%	19.24%	28.87%	51	57
Ta, ppm	0.94	0.35	0.24	1.64	0.00	1.99	37.20%	74.40%	111.60%	0.89	0.99
Tb, ppm	0.72	0.040	0.64	0.80	0.60	0.84	5.62%	11.24%	16.86%	0.68	0.75
Th, ppm	11.7	1.2	9.2	14.2	7.9	15.4	10.69%	21.37%	32.06%	11.1	12.3
Ti, wt.%	0.265	0.008	0.250	0.280	0.242	0.288	2.86%	5.72%	8.58%	0.252	0.279
Tl, ppm	0.74	0.052	0.64	0.85	0.59	0.90	6.93%	13.87%	20.80%	0.71	0.78
Tm, ppm	0.34	0.030	0.28	0.40	0.25	0.43	8.65%	17.31%	25.96%	0.33	0.36
U, ppm	3.80	0.321	3.16	4.44	2.84	4.77	8.46%	16.92%	25.37%	3.61	3.99
V, ppm	144	6	132	156	127	161	4.04%	8.08%	12.12%	137	151
W, ppm	17.8	2.6	12.6	23.1	9.9	25.7	14.78%	29.56%	44.35%	16.9	18.7
Y, ppm	23.6	0.92	21.8	25.4	20.8	26.3	3.88%	7.76%	11.64%	22.4	24.8
Yb, ppm	2.25	0.184	1.88	2.61	1.69	2.80	8.19%	16.37%	24.56%	2.13	2.36
Zn, ppm	283	23	238	329	215	352	8.02%	16.03%	24.05%	269	298
Infrared Combustion											
S, wt.%	4.43	0.113	4.20	4.66	4.09	4.77	2.54%	5.09%	7.63%	4.21	4.65

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

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

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Tables 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-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 3.88 and 4.02 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 931b 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 931b. 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 x Lower Limit of Detection) for the various methods undertaken were considered for the objective of evaluating homogeneity. It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 931b 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 931b 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.

PREPARER AND SUPPLIER

Certified reference material OREAS 931b is prepared, certified and supplied by:

ORE Research & Exploration Pty Ltd
37A Hosie Street
Bayswater North VIC 3153
AUSTRALIA

Tel: +613-9729 0333
Fax: +613-9729 8338
Web: www.oreas.com
Email: info@ore.com.au

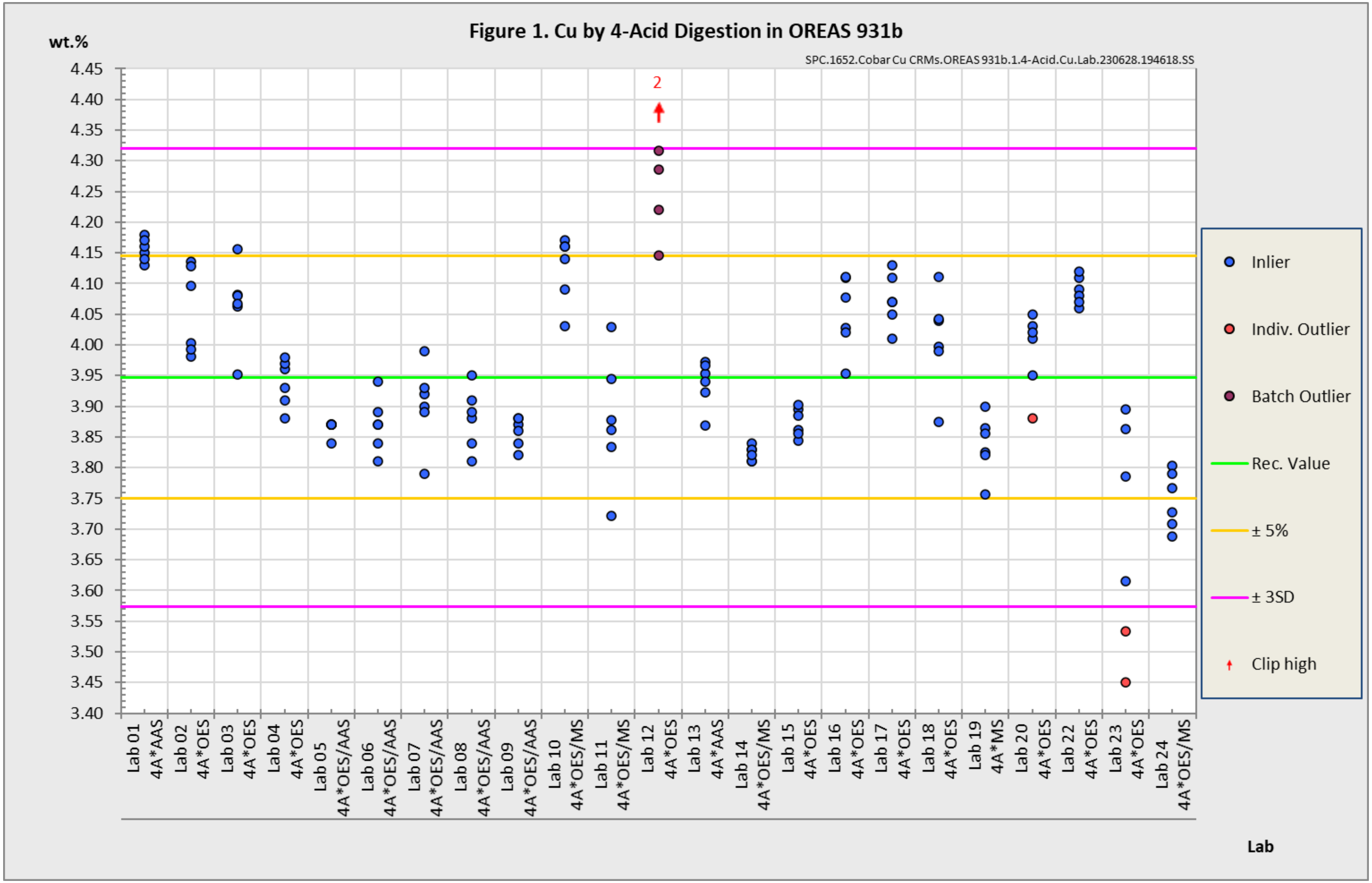

Figure 1. Cu by 4-Acid Digestion in OREAS 931b
wt.%
SPC.1652.Cobar Cu CRMs.OREAS 931b.1.4-Acid.Cu.Lab.230628.194618.SS
4.45
4.40
4.35
4.30
4.25
4.20
4.15
4.10
4.05
4.00
3.95
3.90
3.85
3.80
3.75
3.70
3.65
3.60
3.55
3.50
3.45
3.40
2
Lab 01 4A*AAS
Lab 02 4A*OES
Lab 03 4A*OES
Lab 04 4A*OES
Lab 05 4A*OES/AAS
Lab 06 4A*OES/AAS
Lab 07 4A*OES/AAS
Lab 08 4A*OES/AAS
Lab 09 4A*OES/AAS
Lab 10 4A*OES/MS
Lab 11 4A*OES/MS
Lab 12 4A*OES
Lab 13 4A*AAS
Lab 14 4A*OES/MS
Lab 15 4A*OES
Lab 16 4A*OES
Lab 17 4A*OES
Lab 18 4A*OES
Lab 19 4A*MS
Lab 20 4A*OES
Lab 22 4A*OES
Lab 23 4A*OES
Lab 24 4A*OES/MS
Lab
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Clip high

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 931b 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 931b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 931b 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 931b 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 (4.3 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: ≥0.25g;
- Aqua regia digestion with ICP-OES and/or MS finish: ≥0.5g;
- Peroxide fusion for full elemental suite with ICP-OES and/or MS finish: ≥0.1g;
- Oxidising fusion with X-ray fluorescence finish: ≥0.2g;
- S by infrared combustion furnace/CS analyser: ≥0.1g.

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

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	28th 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

28th June, 2023

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

REFERENCES

[1] Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).

[2] Ingamells, C. O. and Switzer, P. (1973). A Proposed Sampling Constant for Use in Geochemical Analysis, Talanta 20, 547-568.

[3] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.

[4] ISO Guide 31:2015. Reference materials – Contents of certificates and labels.

[5] ISO Guide 35:2017. Certification of reference materials - General and statistical principals.

[6] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[7] ISO 16269-6:2014, Statistical interpretation of data – Determination of statistical tolerance intervals.

[8] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.

[9] ISO 17025:2017, General requirements for the competence of testing and calibration laboratories.

[10] ISO Guide 17034:2016. General requirements for the competence of reference material producers.

[11] Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), Minnesota (USA).

[12] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.

[13] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.

[14] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.

[15] Thompson, A.; Taylor, B.N.; Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Nov 2021).

[16] Van der Veen AMH and Pauwels, J. (2001), Accred Qual Assur 6: 290-294.