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

CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 921b

Copper-bearing siltstone (Cobar, NSW, Australia)

Table 1. Certified Values, Uncertainty & Tolerance Intervals for OREAS 921b.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion					
Ag, Silver (ppm)	0.371	0.349	0.392	0.342	0.400
Al, Aluminium (wt.%)	6.82	6.65	7.00	6.67	6.98
As, Arsenic (ppm)	104	99	109	101	108
Ba, Barium (ppm)	1979	1923	2035	1927	2031
Be, Beryllium (ppm)	2.56	2.40	2.72	2.48	2.64
Bi, Bismuth (ppm)	1.30	1.21	1.39	1.25	1.35
Ca, Calcium (wt.%)	0.074	0.069	0.079	0.072	0.076
Cd, Cadmium (ppm)	0.068	0.048	0.088	IND	IND
Ce, Cerium (ppm)	89	84	93	85	92
Co, Cobalt (ppm)	3.36	3.17	3.55	3.21	3.50
Cr, Chromium (ppm)	88	82	94	85	91
Cs, Caesium (ppm)	4.50	4.33	4.67	4.36	4.64
Cu, Copper (ppm)	254	246	262	245	262
Dy, Dysprosium (ppm)	3.78	3.41	4.15	3.63	3.94
Er, Erbium (ppm)	1.78	1.67	1.89	1.71	1.85
Eu, Europium (ppm)	1.29	1.13	1.45	1.24	1.35
Fe, Iron (wt.%)	3.30	3.22	3.38	3.23	3.37
Ga, Gallium (ppm)	20.1	18.9	21.2	19.2	20.9
Gd, Gadolinium (ppm)	5.45	4.73	6.16	5.23	5.66
Ge, Germanium (ppm)	0.12	0.10	0.14	IND	IND
Hf, Hafnium (ppm)	2.36	2.23	2.48	2.21	2.51
Ho, Holmium (ppm)	0.64	0.60	0.68	0.60	0.67
In, Indium (ppm)	0.100	0.092	0.108	0.093	0.107
K, Potassium (wt.%)	2.69	2.61	2.76	2.61	2.76
La, Lanthanum (ppm)	43.2	41.5	44.9	42.0	44.4
Li, Lithium (ppm)	22.2	21.4	23.1	21.5	22.9
Lu, Lutetium (ppm)	0.27	0.24	0.30	0.25	0.29
Mg, Magnesium (wt.%)	0.501	0.485	0.517	0.489	0.513
Mn, Manganese (wt.%)	0.015	0.015	0.016	0.015	0.016
Mo, Molybdenum (ppm)	2.86	2.72	3.00	2.73	2.99
Na, Sodium (wt.%)	0.114	0.106	0.121	0.110	0.117
Nb, Niobium (ppm)	5.85	5.06	6.63	5.49	6.21
Nd, Neodymium (ppm)	35.9	32.5	39.2	34.6	37.1
Ni, Nickel (ppm)	14.4	13.7	15.1	13.9	15.0
P, Phosphorus (wt.%)	0.034	0.033	0.035	0.033	0.035
Pb, Lead (ppm)	31.0	29.5	32.6	29.8	32.2
Pr, Praseodymium (ppm)	9.92	8.73	11.11	9.60	10.24

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
4-Acid Digestion continued					
Rb, Rubidium (ppm)	156	147	165	150	162
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.061	0.059	0.062	0.059	0.063
Sb, Antimony (ppm)	3.71	3.53	3.88	3.56	3.85
Sc, Scandium (ppm)	12.5	12.0	13.0	12.1	12.9
Se, Selenium (ppm)	2.40	1.67	3.14	2.24	2.56
Sm, Samarium (ppm)	6.96	6.41	7.52	6.69	7.23
Sn, Tin (ppm)	4.54	4.30	4.78	4.33	4.76
Sr, Strontium (ppm)	54	52	56	52	56
Ta, Tantalum (ppm)	0.47	0.40	0.54	0.43	0.51
Tb, Terbium (ppm)	0.68	0.60	0.76	0.65	0.71
Te, Tellurium (ppm)	0.080	0.059	0.101	IND	IND
Th, Thorium (ppm)	15.7	15.0	16.4	15.1	16.2
Ti, Titanium (wt.%)	0.234	0.217	0.252	0.225	0.244
Tl, Thallium (ppm)	0.85	0.80	0.90	0.82	0.89
Tm, Thulium (ppm)	0.26	0.21	0.31	0.24	0.27
U, Uranium (ppm)	3.92	3.75	4.09	3.76	4.08
V, Vanadium (ppm)	160	155	165	156	164
W, Tungsten (ppm)	2.06	1.88	2.24	1.92	2.20
Y, Yttrium (ppm)	16.0	15.0	17.0	15.2	16.7
Yb, Ytterbium (ppm)	1.72	1.52	1.91	1.60	1.83
Zn, Zinc (ppm)	82	80	85	80	85
Aqua Regia Digestion					
Ag, Silver (ppm)	0.244	0.228	0.261	0.229	0.259
Al, Aluminium (wt.%)	0.921	0.883	0.958	0.888	0.953
As, Arsenic (ppm)	102	98	105	99	105
Au, Gold (ppm)	< 0.02	IND	IND	IND	IND
B, Boron (ppm)	< 10	IND	IND	IND	IND
Ba, Barium (ppm)	167	151	182	159	175
Be, Beryllium (ppm)	0.54	0.48	0.61	0.52	0.57
Bi, Bismuth (ppm)	1.13	1.07	1.18	1.08	1.18
Ca, Calcium (wt.%)	0.064	0.058	0.069	0.061	0.067
Cd, Cadmium (ppm)	0.064	0.054	0.074	IND	IND
Ce, Cerium (ppm)	76	73	80	74	78
Co, Cobalt (ppm)	3.16	2.99	3.33	3.04	3.28
Cr, Chromium (ppm)	25.2	24.1	26.3	24.2	26.2
Cs, Caesium (ppm)	1.13	1.03	1.23	1.07	1.18

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					
Cu, Copper (ppm)	252	243	261	245	258
Fe, Iron (wt.%)	3.02	2.93	3.12	2.95	3.10
Ga, Gallium (ppm)	3.08	2.81	3.35	2.97	3.18
Hf, Hafnium (ppm)	0.41	0.37	0.45	0.38	0.44
In, Indium (ppm)	0.044	0.039	0.049	0.041	0.046
K, Potassium (wt.%)	0.267	0.245	0.288	0.254	0.280
La, Lanthanum (ppm)	32.6	31.1	34.1	31.6	33.7
Li, Lithium (ppm)	5.05	4.57	5.52	4.90	5.20
Lu, Lutetium (ppm)	0.12	0.10	0.14	IND	IND
Mg, Magnesium (wt.%)	0.212	0.201	0.223	0.204	0.220
Mn, Manganese (wt.%)	0.013	0.012	0.013	0.012	0.013
Mo, Molybdenum (ppm)	2.68	2.56	2.80	2.54	2.81
Ni, Nickel (ppm)	11.3	10.9	11.7	10.9	11.7
P, Phosphorus (wt.%)	0.029	0.028	0.031	0.029	0.030
Pb, Lead (ppm)	23.1	21.8	24.3	22.1	24.1
Rb, Rubidium (ppm)	16.9	15.2	18.6	16.1	17.8
Re, Rhenium (ppm)	< 0.001	IND	IND	IND	IND
S, Sulphur (wt.%)	0.059	0.056	0.062	0.057	0.062
Sb, Antimony (ppm)	2.51	2.27	2.75	2.37	2.65
Sc, Scandium (ppm)	1.73	1.60	1.87	1.52	1.95
Se, Selenium (ppm)	2.31	2.06	2.56	2.14	2.48
Sn, Tin (ppm)	0.90	0.79	1.00	0.83	0.96
Sr, Strontium (ppm)	14.3	12.3	16.2	13.4	15.1
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.53	0.46	0.61	0.49	0.58
Te, Tellurium (ppm)	0.068	0.053	0.084	IND	IND
Th, Thorium (ppm)	10.7	10.2	11.3	10.4	11.1
Ti, Titanium (wt.%)	0.024	0.021	0.027	0.023	0.025
Tl, Thallium (ppm)	0.11	0.10	0.12	IND	IND
U, Uranium (ppm)	2.34	2.23	2.45	2.26	2.41
V, Vanadium (ppm)	19.8	18.5	21.2	19.0	20.6
W, Tungsten (ppm)	0.22	0.18	0.25	0.20	0.23
Y, Yttrium (ppm)	8.19	7.81	8.56	7.94	8.44
Zn, Zinc (ppm)	73	71	75	71	75
Zr, Zirconium (ppm)	14.0	13.1	15.0	13.6	14.5
Borate / Peroxide Fusion ICP					
Ag, Silver (ppm)	< 5	IND	IND	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 the upper bound/non-detect limit value).

Table 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued					
Al, Aluminium (wt.%)	7.00	6.87	7.14	6.88	7.13
As, Arsenic (ppm)	103	94	112	97	109
B, Boron (ppm)	131	114	147	117	145
Ba, Barium (ppm)	1978	1931	2024	1943	2012
Be, Beryllium (ppm)	2.43	1.86	2.99	2.19	2.66
Bi, Bismuth (ppm)	1.35	1.13	1.57	1.18	1.52
Ca, Calcium (wt.%)	0.094	0.066	0.122	IND	IND
Ce, Cerium (ppm)	91	85	97	88	94
Co, Cobalt (ppm)	3.58	3.12	4.03	3.31	3.84
Cr, Chromium (ppm)	105	91	120	97	114
Cs, Caesium (ppm)	4.63	4.38	4.89	4.40	4.86
Cu, Copper (ppm)	255	244	265	244	265
Dy, Dysprosium (ppm)	4.97	4.47	5.47	4.63	5.31
Er, Erbium (ppm)	2.80	2.59	3.01	2.60	3.00
Eu, Europium (ppm)	1.36	1.10	1.62	1.28	1.43
Fe, Iron (wt.%)	3.35	3.27	3.43	3.30	3.39
Ga, Gallium (ppm)	20.7	19.6	21.8	19.4	22.0
Gd, Gadolinium (ppm)	5.85	5.06	6.64	5.49	6.21
Ge, Germanium (ppm)	2.22	1.71	2.72	1.86	2.58
Ho, Holmium (ppm)	0.95	0.87	1.03	0.90	1.01
In, Indium (ppm)	< 0.2	IND	IND	IND	IND
K, Potassium (wt.%)	2.77	2.70	2.83	2.71	2.82
La, Lanthanum (ppm)	43.4	40.5	46.4	42.0	44.9
Li, Lithium (ppm)	22.5	19.2	25.7	20.8	24.1
Lu, Lutetium (ppm)	0.40	0.34	0.46	0.37	0.43
Mg, Magnesium (wt.%)	0.507	0.492	0.522	0.496	0.518
Mn, Manganese (wt.%)	0.017	0.016	0.018	0.017	0.017
Mo, Molybdenum (ppm)	3.35	2.47	4.24	IND	IND
Nb, Niobium (ppm)	12.4	10.9	13.8	11.6	13.1
Nd, Neodymium (ppm)	37.1	34.2	40.0	35.6	38.6
P, Phosphorus (wt.%)	0.034	0.030	0.037	IND	IND
Pb, Lead (ppm)	33.4	28.5	38.2	31.6	35.2
Pr, Praseodymium (ppm)	9.76	8.97	10.54	9.45	10.06
Rb, Rubidium (ppm)	156	149	163	151	161
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND
S, Sulphur (wt.%)	0.067	0.055	0.078	IND	IND
Sb, Antimony (ppm)	3.82	3.37	4.27	3.54	4.11

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
Borate / Peroxide Fusion ICP continued					
Sc, Scandium (ppm)	11.9	10.7	13.1	IND	IND
Si, Silicon (wt.%)	34.87	34.03	35.70	34.18	35.56
Sm, Samarium (ppm)	6.80	6.23	7.36	6.36	7.23
Sr, Strontium (ppm)	58	55	62	56	61
Ta, Tantalum (ppm)	1.01	0.69	1.33	0.72	1.30
Tb, Terbium (ppm)	0.83	0.75	0.92	0.76	0.91
Te, Tellurium (ppm)	< 1	IND	IND	IND	IND
Th, Thorium (ppm)	14.4	13.3	15.6	13.9	14.9
Ti, Titanium (wt.%)	0.338	0.328	0.347	0.329	0.346
Tl, Thallium (ppm)	0.89	0.81	0.97	0.84	0.94
Tm, Thulium (ppm)	0.41	0.37	0.46	0.38	0.45
U, Uranium (ppm)	4.34	4.00	4.68	4.17	4.51
V, Vanadium (ppm)	170	164	176	166	174
W, Tungsten (ppm)	2.99	2.48	3.50	2.76	3.21
Y, Yttrium (ppm)	27.3	25.8	28.7	26.2	28.3
Yb, Ytterbium (ppm)	2.75	2.40	3.09	2.60	2.90
Zn, Zinc (ppm)	83	77	89	80	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; 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 921b.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
B	ppm	*21.2*	Hg	ppm	*0.012*			
Aqua Regia Digestion								
Dy	ppm	*2.36*	Ho	ppm	*0.39*	Pt	ppb	*< 5*
Er	ppm	*0.95*	Na	wt.%	*0.011*	Sm	ppm	*5.47*
Eu	ppm	*0.98*	Nb	ppm	*0.16*	Tm	ppm	*0.12*
Gd	ppm	*4.14*	Nd	ppm	*29.2*	Yb	ppm	*0.80*
Ge	ppm	*0.10*	Pd	ppb	*70.2*			
Hg	ppm	*0.015*	Pr	ppm	*7.98*			
Borate Fusion XRF								
Cd	ppm	*< 0.8*	Ni	ppm	*21.3*	Zr	ppm	*107*
Hf	ppm	*3.10*	Se	ppm	*3.73*			
Na	wt.%	*0.131*	Sn	ppm	*6.79*			
Thermogravimetry								
LOI^{1000}	wt.%	*3.62*						
Infrared Combustion								
C	wt.%	*0.728*	S	wt.%	*0.057*			

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.

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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[3]) are presented in the detailed certification data for this CRM (**OREAS 921b-DataPack.1.0.230602_165040.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 921b 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 921b was prepared in the following manner:

- Drying of black slate and waste rock to constant mass at 105°C;
- Crushing and milling of black slate and waste rock to >98% minus 75 microns;
- Blending the black slate and waste rock in appropriate proportions to achieve the target Cu grade;
- Homogenisation using OREAS' novel processing technologies;
- Packaging into 10g units in laminated foil pouches.

PHYSICAL PROPERTIES

OREAS 921b 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 921b.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
664	0.57	N6	Medium Light 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).

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 921b.

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	0.371	0.027	0.318	0.424	0.291	0.451	7.19%	14.38%	21.57%	0.352	0.389
Al, wt.%	6.82	0.265	6.30	7.35	6.03	7.62	3.88%	7.76%	11.64%	6.48	7.17
As, ppm	104	4	96	113	91	117	4.11%	8.22%	12.33%	99	109
Ba, ppm	1979	85	1809	2149	1724	2233	4.29%	8.57%	12.86%	1880	2078
Be, ppm	2.56	0.215	2.13	2.99	1.91	3.20	8.41%	16.82%	25.24%	2.43	2.69
Bi, ppm	1.30	0.091	1.12	1.48	1.03	1.57	6.98%	13.95%	20.93%	1.24	1.37
Ca, wt.%	0.074	0.005	0.064	0.084	0.059	0.089	6.62%	13.25%	19.87%	0.071	0.078
Cd, ppm	0.068	0.016	0.036	0.100	0.020	0.116	23.42%	46.84%	70.26%	0.065	0.071
Ce, ppm	89	3.7	81	96	78	100	4.17%	8.33%	12.50%	84	93
Co, ppm	3.36	0.169	3.02	3.69	2.85	3.86	5.02%	10.05%	15.07%	3.19	3.52
Cr, ppm	88	11	65	111	54	122	12.92%	25.85%	38.77%	84	93
Cs, ppm	4.50	0.217	4.07	4.94	3.85	5.15	4.82%	9.63%	14.45%	4.28	4.73
Cu, ppm	254	10	233	274	223	285	4.06%	8.12%	12.17%	241	266
Dy, ppm	3.78	0.365	3.05	4.51	2.69	4.88	9.66%	19.32%	28.98%	3.59	3.97
Er, ppm	1.78	0.081	1.62	1.94	1.54	2.02	4.55%	9.09%	13.64%	1.69	1.87
Eu, ppm	1.29	0.123	1.05	1.54	0.92	1.66	9.52%	19.05%	28.57%	1.23	1.36
Fe, wt.%	3.30	0.098	3.10	3.49	3.00	3.59	2.97%	5.93%	8.90%	3.13	3.46
Ga, ppm	20.1	1.19	17.7	22.4	16.5	23.6	5.94%	11.89%	17.83%	19.0	21.1
Gd, ppm	5.45	0.60	4.25	6.64	3.65	7.24	10.98%	21.95%	32.93%	5.17	5.72
Ge, ppm	0.12	0.03	0.07	0.17	0.04	0.20	22.66%	45.32%	67.98%	0.11	0.13
Hf, ppm	2.36	0.147	2.06	2.65	1.92	2.80	6.23%	12.47%	18.70%	2.24	2.48
Ho, ppm	0.64	0.026	0.59	0.69	0.56	0.71	4.00%	8.00%	12.01%	0.61	0.67
In, ppm	0.100	0.010	0.079	0.121	0.069	0.131	10.48%	20.96%	31.44%	0.095	0.105
K, wt.%	2.69	0.097	2.49	2.88	2.39	2.98	3.62%	7.25%	10.87%	2.55	2.82
La, ppm	43.2	1.82	39.5	46.8	37.7	48.7	4.22%	8.44%	12.66%	41.0	45.3
Li, ppm	22.2	1.09	20.0	24.4	19.0	25.5	4.90%	9.81%	14.71%	21.1	23.3
Lu, ppm	0.27	0.022	0.23	0.32	0.21	0.34	8.18%	16.35%	24.53%	0.26	0.29
Mg, wt.%	0.501	0.022	0.457	0.545	0.435	0.567	4.42%	8.84%	13.26%	0.476	0.526
Mn, wt.%	0.015	0.001	0.014	0.017	0.014	0.017	4.01%	8.01%	12.02%	0.015	0.016
Mo, ppm	2.86	0.152	2.56	3.16	2.41	3.32	5.30%	10.60%	15.91%	2.72	3.00
Na, wt.%	0.114	0.014	0.086	0.141	0.073	0.154	11.97%	23.95%	35.92%	0.108	0.119
Nb, ppm	5.85	1.19	3.46	8.23	2.27	9.43	20.40%	40.80%	61.19%	5.55	6.14
Nd, ppm	35.9	3.14	29.6	42.1	26.4	45.3	8.75%	17.50%	26.24%	34.1	37.6
Ni, ppm	14.4	0.60	13.2	15.6	12.6	16.2	4.16%	8.32%	12.48%	13.7	15.1
P, wt.%	0.034	0.002	0.031	0.037	0.029	0.039	4.55%	9.10%	13.65%	0.032	0.036
Pb, ppm	31.0	2.45	26.1	35.9	23.7	38.4	7.90%	15.80%	23.70%	29.5	32.6
Pr, ppm	9.92	1.14	7.65	12.19	6.51	13.33	11.45%	22.90%	34.36%	9.42	10.41
Rb, ppm	156	8	140	172	132	180	5.08%	10.16%	15.24%	148	164
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.061	0.003	0.055	0.067	0.052	0.070	4.81%	9.63%	14.44%	0.058	0.064
Sb, ppm	3.71	0.202	3.30	4.11	3.10	4.31	5.44%	10.87%	16.31%	3.52	3.89
Sc, ppm	12.5	0.52	11.5	13.6	10.9	14.1	4.18%	8.36%	12.54%	11.9	13.1
Se, ppm	2.40	0.42	1.56	3.24	1.15	3.66	17.43%	34.87%	52.30%	2.28	2.52

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; 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
4-Acid Digestion continued											
Sm, ppm	6.96	0.575	5.81	8.11	5.24	8.69	8.25%	16.51%	24.76%	6.61	7.31
Sn, ppm	4.54	0.238	4.07	5.02	3.83	5.26	5.24%	10.47%	15.71%	4.32	4.77
Sr, ppm	54	2.8	49	60	46	62	5.09%	10.18%	15.27%	51	57
Ta, ppm	0.47	0.08	0.30	0.64	0.22	0.72	17.77%	35.54%	53.31%	0.45	0.49
Tb, ppm	0.68	0.059	0.56	0.79	0.50	0.85	8.66%	17.33%	25.99%	0.64	0.71
Te, ppm	0.080	0.017	0.046	0.113	0.029	0.130	21.05%	42.10%	63.14%	0.076	0.084
Th, ppm	15.7	0.93	13.8	17.5	12.9	18.5	5.96%	11.92%	17.88%	14.9	16.5
Ti, wt.%	0.234	0.024	0.187	0.282	0.163	0.306	10.17%	20.35%	30.52%	0.223	0.246
Tl, ppm	0.85	0.037	0.78	0.93	0.74	0.96	4.36%	8.71%	13.07%	0.81	0.89
Tm, ppm	0.26	0.03	0.19	0.32	0.16	0.35	12.63%	25.26%	37.88%	0.24	0.27
U, ppm	3.92	0.235	3.45	4.39	3.22	4.62	5.98%	11.96%	17.94%	3.72	4.12
V, ppm	160	7	145	175	138	182	4.57%	9.15%	13.72%	152	168
W, ppm	2.06	0.23	1.60	2.52	1.37	2.75	11.20%	22.40%	33.59%	1.96	2.16
Y, ppm	16.0	1.53	12.9	19.0	11.4	20.6	9.58%	19.16%	28.73%	15.2	16.8
Yb, ppm	1.72	0.20	1.32	2.11	1.12	2.31	11.52%	23.04%	34.56%	1.63	1.80
Zn, ppm	82	2.6	77	88	75	90	3.20%	6.40%	9.60%	78	87
Zr, ppm	82	4.8	72	92	67	97	5.91%	11.82%	17.73%	78	86
Aqua Regia Digestion											
Ag, ppm	0.244	0.012	0.219	0.269	0.207	0.281	5.09%	10.17%	15.26%	0.232	0.256
Al, wt.%	0.921	0.062	0.796	1.045	0.734	1.108	6.77%	13.54%	20.31%	0.875	0.967
As, ppm	102	6	90	114	84	120	5.84%	11.67%	17.51%	97	107
Au, ppm	< 0.02	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	167	29	108	226	79	255	17.64%	35.27%	52.91%	159	175
Be, ppm	0.54	0.09	0.36	0.72	0.27	0.81	16.47%	32.95%	49.42%	0.52	0.57
Bi, ppm	1.13	0.094	0.94	1.32	0.84	1.41	8.38%	16.76%	25.14%	1.07	1.18
Ca, wt.%	0.064	0.007	0.051	0.077	0.044	0.083	10.30%	20.61%	30.91%	0.060	0.067
Cd, ppm	0.064	0.011	0.041	0.087	0.030	0.098	17.74%	35.49%	53.23%	0.061	0.067
Ce, ppm	76	4.4	68	85	63	90	5.80%	11.60%	17.40%	73	80
Co, ppm	3.16	0.254	2.65	3.67	2.40	3.92	8.04%	16.09%	24.13%	3.00	3.32
Cr, ppm	25.2	1.38	22.4	27.9	21.0	29.3	5.47%	10.94%	16.41%	23.9	26.4
Cs, ppm	1.13	0.16	0.81	1.44	0.65	1.60	14.12%	28.25%	42.37%	1.07	1.18
Cu, ppm	252	11	230	274	218	286	4.44%	8.87%	13.31%	239	265
Fe, wt.%	3.02	0.158	2.71	3.34	2.55	3.50	5.23%	10.46%	15.69%	2.87	3.18
Ga, ppm	3.08	0.38	2.31	3.85	1.93	4.23	12.47%	24.94%	37.41%	2.93	3.23
Hf, ppm	0.41	0.05	0.31	0.51	0.26	0.56	12.02%	24.04%	36.07%	0.39	0.43
In, ppm	0.044	0.004	0.036	0.051	0.032	0.055	8.96%	17.93%	26.89%	0.041	0.046
K, wt.%	0.267	0.044	0.180	0.354	0.136	0.397	16.31%	32.62%	48.93%	0.253	0.280
La, ppm	32.6	2.52	27.6	37.7	25.1	40.2	7.72%	15.43%	23.15%	31.0	34.2
Li, ppm	5.05	0.84	3.37	6.72	2.54	7.56	16.59%	33.18%	49.77%	4.80	5.30
Lu, ppm	0.12	0.02	0.09	0.16	0.07	0.17	13.89%	27.78%	41.67%	0.12	0.13
Mg, wt.%	0.212	0.016	0.179	0.245	0.163	0.261	7.71%	15.42%	23.12%	0.201	0.223
Mn, wt.%	0.013	0.001	0.011	0.014	0.010	0.015	6.40%	12.81%	19.21%	0.012	0.013

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
Aqua Regia Digestion continued											
Mo, ppm	2.68	0.148	2.38	2.97	2.23	3.12	5.53%	11.05%	16.58%	2.54	2.81
Ni, ppm	11.3	0.69	9.9	12.7	9.2	13.4	6.07%	12.14%	18.21%	10.7	11.9
P, wt.%	0.029	0.002	0.025	0.034	0.022	0.036	8.01%	16.02%	24.04%	0.028	0.031
Pb, ppm	23.1	2.27	18.5	27.6	16.3	29.9	9.84%	19.69%	29.53%	21.9	24.2
Rb, ppm	16.9	2.9	11.2	22.7	8.4	25.5	16.87%	33.74%	50.61%	16.1	17.8
Re, ppm	< 0.001	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.059	0.003	0.054	0.065	0.051	0.068	4.69%	9.38%	14.07%	0.056	0.062
Sb, ppm	2.51	0.44	1.62	3.40	1.18	3.84	17.67%	35.34%	53.01%	2.39	2.64
Sc, ppm	1.73	0.25	1.23	2.24	0.97	2.50	14.64%	29.28%	43.92%	1.65	1.82
Se, ppm	2.31	0.25	1.82	2.80	1.58	3.05	10.61%	21.23%	31.84%	2.20	2.43
Sn, ppm	0.90	0.13	0.64	1.15	0.51	1.28	14.30%	28.60%	42.91%	0.85	0.94
Sr, ppm	14.3	3.4	7.5	21.0	4.1	24.4	23.72%	47.44%	71.17%	13.5	15.0
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.53	0.051	0.43	0.64	0.38	0.69	9.54%	19.08%	28.61%	0.51	0.56
Te, ppm	0.068	0.008	0.053	0.084	0.045	0.092	11.46%	22.93%	34.39%	0.065	0.072
Th, ppm	10.7	0.77	9.2	12.3	8.4	13.0	7.18%	14.36%	21.53%	10.2	11.3
Ti, wt.%	0.024	0.005	0.014	0.034	0.009	0.039	20.76%	41.51%	62.27%	0.023	0.025
Tl, ppm	0.11	0.02	0.08	0.14	0.06	0.15	14.11%	28.22%	42.33%	0.10	0.11
U, ppm	2.34	0.146	2.05	2.63	1.90	2.78	6.26%	12.51%	18.77%	2.22	2.46
V, ppm	19.8	1.95	15.9	23.7	14.0	25.7	9.85%	19.69%	29.54%	18.8	20.8
W, ppm	0.22	0.04	0.13	0.30	0.09	0.34	18.81%	37.62%	56.43%	0.21	0.23
Y, ppm	8.19	0.531	7.13	9.25	6.59	9.78	6.49%	12.97%	19.46%	7.78	8.60
Zn, ppm	73	3.5	66	80	63	83	4.73%	9.45%	14.18%	69	77
Zr, ppm	14.0	1.40	11.2	16.8	9.8	18.3	9.99%	19.99%	29.98%	13.3	14.7
Borate / Peroxide Fusion ICP											
Ag, ppm	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	7.00	0.162	6.68	7.33	6.52	7.49	2.32%	4.63%	6.95%	6.65	7.35
As, ppm	103	9	85	121	75	131	8.95%	17.91%	26.86%	98	108
B, ppm	131	12	107	155	94	167	9.27%	18.54%	27.81%	124	137
Ba, ppm	1978	51	1875	2080	1824	2131	2.59%	5.19%	7.78%	1879	2076
Be, ppm	2.43	0.38	1.67	3.19	1.29	3.57	15.66%	31.32%	46.97%	2.31	2.55
Bi, ppm	1.35	0.23	0.89	1.80	0.67	2.03	16.84%	33.68%	50.51%	1.28	1.42
Ca, wt.%	0.094	0.024	0.047	0.142	0.024	0.165	24.91%	49.81%	74.72%	0.090	0.099
Ce, ppm	91	6.0	79	103	73	109	6.55%	13.11%	19.66%	86	95
Co, ppm	3.58	0.332	2.91	4.24	2.58	4.57	9.28%	18.55%	27.83%	3.40	3.75
Cr, ppm	105	20	65	146	45	166	19.11%	38.23%	57.34%	100	111
Cs, ppm	4.63	0.182	4.27	5.00	4.09	5.18	3.93%	7.85%	11.78%	4.40	4.86
Cu, ppm	255	19	217	292	198	311	7.36%	14.72%	22.07%	242	267
Dy, ppm	4.97	0.494	3.98	5.96	3.49	6.45	9.95%	19.90%	29.84%	4.72	5.22
Er, ppm	2.80	0.204	2.39	3.21	2.19	3.41	7.28%	14.57%	21.85%	2.66	2.94
Eu, ppm	1.36	0.26	0.83	1.89	0.57	2.15	19.40%	38.79%	58.19%	1.29	1.43
Fe, wt.%	3.35	0.107	3.13	3.56	3.03	3.67	3.18%	6.36%	9.55%	3.18	3.52
Ga, ppm	20.7	1.28	18.1	23.3	16.9	24.5	6.19%	12.37%	18.56%	19.7	21.7

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 / Peroxide Fusion ICP continued											
Gd, ppm	5.85	0.70	4.45	7.25	3.75	7.95	11.97%	23.94%	35.91%	5.56	6.14
Ge, ppm	2.22	0.31	1.60	2.83	1.29	3.14	13.87%	27.75%	41.62%	2.11	2.33
Ho, ppm	0.95	0.061	0.83	1.07	0.77	1.13	6.41%	12.81%	19.22%	0.90	1.00
In, ppm	< 0.2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	2.77	0.084	2.60	2.94	2.51	3.02	3.05%	6.09%	9.14%	2.63	2.90
La, ppm	43.4	3.87	35.7	51.2	31.8	55.1	8.92%	17.84%	26.77%	41.3	45.6
Li, ppm	22.5	2.8	16.9	28.0	14.2	30.7	12.25%	24.51%	36.76%	21.3	23.6
Lu, ppm	0.40	0.037	0.33	0.47	0.29	0.51	9.23%	18.47%	27.70%	0.38	0.42
Mg, wt.%	0.507	0.027	0.454	0.561	0.427	0.587	5.28%	10.55%	15.83%	0.482	0.533
Mn, wt.%	0.017	0.002	0.014	0.020	0.012	0.022	9.40%	18.81%	28.21%	0.016	0.018
Mo, ppm	3.35	0.72	1.92	4.78	1.21	5.50	21.34%	42.68%	64.01%	3.18	3.52
Nb, ppm	12.4	1.18	10.0	14.7	8.8	15.9	9.53%	19.06%	28.59%	11.8	13.0
Nd, ppm	37.1	2.93	31.2	43.0	28.3	45.9	7.90%	15.80%	23.70%	35.2	38.9
P, wt.%	0.034	0.006	0.021	0.046	0.014	0.053	18.99%	37.98%	56.97%	0.032	0.035
Pb, ppm	33.4	4.6	24.1	42.6	19.5	47.3	13.89%	27.78%	41.66%	31.7	35.0
Pr, ppm	9.76	0.888	7.98	11.53	7.09	12.42	9.11%	18.21%	27.32%	9.27	10.24
Rb, ppm	156	7	143	169	137	176	4.17%	8.34%	12.51%	148	164
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.067	0.011	0.045	0.088	0.034	0.099	16.37%	32.73%	49.10%	0.063	0.070
Sb, ppm	3.82	0.291	3.24	4.40	2.95	4.69	7.60%	15.20%	22.80%	3.63	4.01
Sc, ppm	11.9	1.01	9.9	13.9	8.9	14.9	8.49%	16.98%	25.48%	11.3	12.5
Si, wt.%	34.87	0.887	33.10	36.64	32.21	37.53	2.54%	5.09%	7.63%	33.13	36.61
Sm, ppm	6.80	0.524	5.75	7.84	5.22	8.37	7.71%	15.42%	23.13%	6.46	7.13
Sr, ppm	58	3.9	51	66	47	70	6.65%	13.30%	19.94%	55	61
Ta, ppm	1.01	0.20	0.61	1.41	0.40	1.61	19.95%	39.89%	59.84%	0.96	1.06
Tb, ppm	0.83	0.061	0.71	0.96	0.65	1.02	7.25%	14.51%	21.76%	0.79	0.88
Te, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	14.4	1.7	11.1	17.8	9.4	19.5	11.67%	23.35%	35.02%	13.7	15.1
Ti, wt.%	0.338	0.013	0.311	0.364	0.298	0.378	3.94%	7.89%	11.83%	0.321	0.355
Tl, ppm	0.89	0.072	0.75	1.04	0.68	1.11	8.07%	16.15%	24.22%	0.85	0.94
Tm, ppm	0.41	0.033	0.35	0.48	0.31	0.51	7.92%	15.85%	23.77%	0.39	0.43
U, ppm	4.34	0.423	3.49	5.19	3.07	5.61	9.75%	19.51%	29.26%	4.12	4.56
V, ppm	170	9	153	187	144	196	5.02%	10.04%	15.06%	162	179
W, ppm	2.99	0.54	1.90	4.08	1.36	4.62	18.18%	36.36%	54.54%	2.84	3.14
Y, ppm	27.3	1.94	23.4	31.1	21.4	33.1	7.11%	14.22%	21.33%	25.9	28.6
Yb, ppm	2.75	0.235	2.28	3.22	2.04	3.45	8.54%	17.08%	25.62%	2.61	2.89
Zn, ppm	83	8.3	66	99	58	108	9.97%	19.94%	29.91%	79	87

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 245 and 262 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

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

The homogeneity of OREAS 921b 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 921b. 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 921b 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 921b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

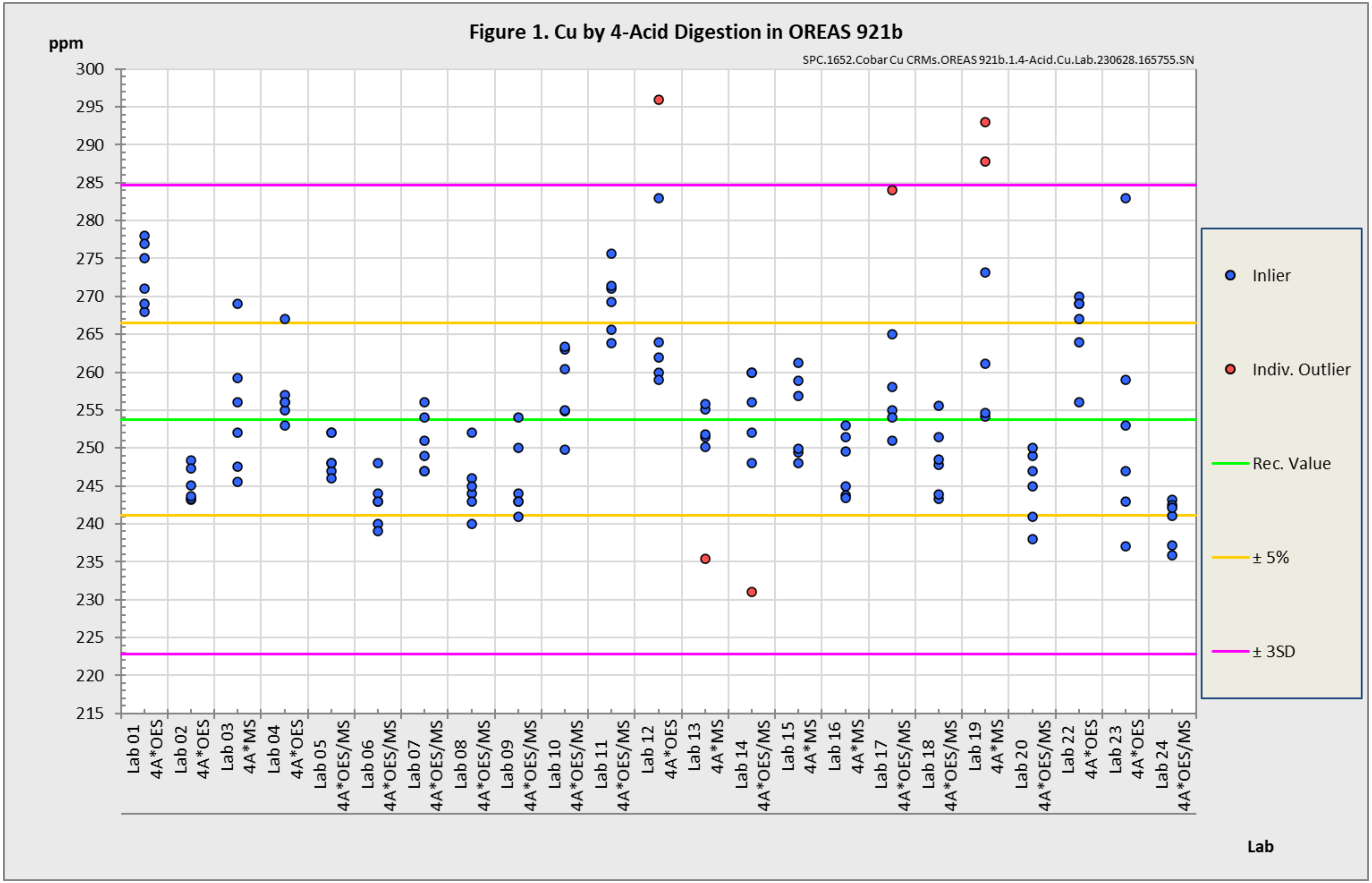

Figure 1. Cu by 4-Acid Digestion in OREAS 921b
ppm
SPC.1652.Cobar Cu CRMs.OREAS 921b.1.4-Acid.Cu.Lab.230628.165755.SN
300
295
290
285
280
275
270
265
260
255
250
245
240
235
230
225
220
215
Lab 01 4A*OES
Lab 02 4A*OES
Lab 03 4A*MS
Lab 04 4A*OES
Lab 05 4A*OES/MS
Lab 06 4A*OES/MS
Lab 07 4A*OES/MS
Lab 08 4A*OES/MS
Lab 09 4A*OES/MS
Lab 10 4A*OES/MS
Lab 11 4A*OES/MS
Lab 12 4A*OES
Lab 13 4A*MS
Lab 14 4A*OES/MS
Lab 15 4A*MS
Lab 16 4A*MS
Lab 17 4A*OES/MS
Lab 18 4A*OES/MS
Lab 19 4A*MS
Lab 20 4A*OES/MS
Lab 22 4A*OES
Lab 23 4A*OES
Lab 24 4A*OES/MS
Lab
Inlier
Indiv. Outlier
Rec. Value
± 5%
± 3SD

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 <u>does not</u> correspond with the Lab ID numbering on the scatter plots below.

PREPARER AND SUPPLIER

Certified reference material OREAS 921b is prepared, certified and supplied by:

ORE RESEARCH & EXPLORATION

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

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 921b 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 921b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution. OREAS 921b 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 921b 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

Following analysis, it is the manufacturer's expectation that any remaining material is discarded unless the sachet is promptly resealed. It is the user's responsibility to prevent contamination and minimise exposure to the atmosphere.

Repeat-use packaging (e.g., 500g plastic jars)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 921b contains a non-hygroscopic* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 3 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (0.06 wt.% S).

*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorped moisture (weakly held H_2O- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2mm thick) layer and left exposed for a period of 2 hours.

INSTRUCTIONS FOR HANDLING & CORRECT USE

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [13]. Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised. 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.

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.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
1	2nd August, 2023	Changed the description on the title page from 'Copper ore' to 'Copper-bearing siltstone'.
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.