

## **CERTIFICATE OF ANALYSIS FOR**

# **COPPER-SILVER CONCENTRATE**

(Various sources, Australia)

# OREAS 994

Table 1. Certified Values and Performance Gates for OREAS 994.

Constituent Certified Value	Certified		Absolute	Standard	Deviations	5	Relative	Standard D	eviations	5% w	indow
	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High	
Umpire Labs (dry sample basis)											
Classical Wet	Classical Wet Chemistry										
Cu, wt.%	30.00	0.086	29.83	30.18	29.75	30.26	0.29%	0.57%	0.86%	28.50	31.50
Acid Digestion	Acid Digestion (no HF)										
Ag, ppm	181	4	174	189	170	192	1.99%	3.98%	5.97%	172	190

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

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.



Document: COA-1514-OREAS994-R0

## Table 1 continued.

			Absolute Standard Deviations Relative Standard Deviations						eviations	5% window	
Constituent	Certified Value	1SD	2SD	2SD	3SD	3SD	1RSD	2RSD	3RSD	Low	High
Geoanalytic	al Lahe ("		Low	High	Low ie)	High					
Peroxide Fus	<u> </u>	as receiv	veu saii	iipie bas	)is)						
Al, wt.%	0.706	0.017	0.672	0.740	0.654	0.757	2.43%	4.87%	7.30%	0.671	0.741
	3607	135	3338	3877	3203	4012	3.74%	7.47%	11.21%	3427	3788
As, ppm	213	9	195	231	185	240	4.30%	8.60%	12.89%	202	224
Ba, ppm	< 1	IND	IND	IND	IND	IND	4.30 % IND	IND	12.09 70 IND	IND	IND
Be, ppm Bi, ppm	115	6	104	126	98	132	4.84%	9.68%	14.52%	109	121
Ca, wt.%	0.542	0.018	0.507	0.578	0.489	0.596	3.28%	6.56%	9.84%	0.515	0.570
	29.9									28.4	31.4
Cd, ppm	14.5	2.75	24.4	35.4 16.8	21.6	38.2	9.21%	18.42%	27.63%		15.3
Ce, ppm		1.13	12.3		11.2	17.9	7.74%	15.47%	23.21%	13.8	
Co, ppm	353	8	337	369	329	377	2.27%	4.54%	6.80%	335	370
Cr, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, wt.%	29.62	0.426	28.77	30.47	28.34	30.90	1.44%	2.87%	4.31%	28.14	31.10
Dy, ppm	1.02	0.076	0.87	1.17	0.79	1.25	7.46%	14.92%	22.39%	0.97	1.07
Er, ppm	0.57	0.08	0.41	0.72	0.34	0.79	13.47%	26.95%	40.42%	0.54	0.59
Eu, ppm	0.35	0.06	0.24	0.47	0.18	0.53	16.41%	32.82%	49.23%	0.33	0.37
Fe, wt.%	21.48	0.498	20.49	22.48	19.99	22.98	2.32%	4.63%	6.95%	20.41	22.56
Ga, ppm	4.30	0.358	3.58	5.01	3.22	5.37	8.33%	16.66%	24.98%	4.08	4.51
In, ppm	7.71	0.547	6.61	8.80	6.07	9.35	7.10%	14.20%	21.30%	7.32	8.09
K, wt.%	0.193	0.019	0.154	0.231	0.135	0.250	9.90%	19.79%	29.69%	0.183	0.202
Mg, wt.%	0.317	0.016	0.285	0.348	0.269	0.364	5.00%	9.99%	14.99%	0.301	0.333
Mn, wt.%	0.023	0.001	0.021	0.024	0.021	0.025	3.27%	6.54%	9.82%	0.022	0.024
Mo, ppm	677	25	626	728	601	753	3.75%	7.51%	11.26%	643	711
Nd, ppm	5.71	0.361	4.99	6.43	4.63	6.79	6.32%	12.64%	18.95%	5.42	6.00
Ni, ppm	3548	103	3342	3755	3238	3858	2.91%	5.82%	8.73%	3371	3726
Pb, wt.%	0.227	0.013	0.200	0.254	0.187	0.267	5.92%	11.84%	17.76%	0.216	0.239
Pr, ppm	1.59	0.106	1.37	1.80	1.27	1.90	6.69%	13.38%	20.06%	1.51	1.66
Rb, ppm	8.81	0.569	7.67	9.94	7.10	10.51	6.46%	12.91%	19.37%	8.37	9.25
S, wt.%	31.44	0.675	30.10	32.79	29.42	33.47	2.15%	4.29%	6.44%	29.87	33.02
Sb, ppm	499	44	411	587	367	631	8.81%	17.62%	26.43%	474	524
Se, ppm	83	9	65	102	56	111	11.07%	22.14%	33.21%	79	88
Si, wt.%	3.15	0.075	3.00	3.30	2.92	3.37	2.39%	4.78%	7.16%	2.99	3.30
Sm, ppm	1.23	0.22	0.80	1.67	0.58	1.89	17.76%	35.51%	53.27%	1.17	1.30
Sn, ppm	50.0	2.24	45.5	54.4	43.2	56.7	4.49%	8.98%	13.47%	47.5	52.5
Sr, ppm	28.3	3.5	21.3	35.3	17.9	38.8	12.31%	24.62%	36.93%	26.9	29.7
Te, ppm	13.8	2.2	9.3	18.2	7.1	20.4	16.14%	32.29%	48.43%	13.1	14.4
Th, ppm	2.86	0.138	2.58	3.13	2.44	3.27	4.83%	9.65%	14.48%	2.71	3.00
Ti, wt.%	0.050	0.001	0.048	0.051	0.047	0.052	1.64%	3.28%	4.91%	0.047	0.052
TI, ppm	27.7	1.16	25.4	30.0	24.2	31.2	4.19%	8.37%	12.56%	26.3	29.1
U, ppm	4.26	0.48	3.29	5.23	2.80	5.71	11.38%	22.77%	34.15%	4.04	4.47
W, ppm	10.9	1.9	7.0	14.7	5.1	16.6	17.64%	35.28%	52.92%	10.3	11.4
SI unit equival					l	l	l		l	I .	l

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

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 1 continued.

		. Absolute Standard Deviations Relative Standard Deviation						eviations	5% window		
Constituent	Certified Value			T		1	rtolativo			0 70 11	l
	value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Peroxide Fus	ion ICP con	tinued									
Y, ppm	5.42	0.497	4.42	6.41	3.93	6.91	9.17%	18.33%	27.50%	5.15	5.69
Zn, wt.%	0.611	0.024	0.564	0.658	0.540	0.681	3.85%	7.71%	11.56%	0.580	0.641
4-Acid Digest	ion										
Ag, ppm	183	7	169	197	161	204	3.88%	7.77%	11.65%	174	192
AI, wt.%	0.696	0.020	0.656	0.737	0.635	0.757	2.91%	5.83%	8.74%	0.661	0.731
As, ppm	3483	135	3214	3753	3079	3887	3.87%	7.74%	11.61%	3309	3657
Be, ppm	0.23	0.04	0.15	0.30	0.11	0.34	17.42%	34.84%	52.26%	0.21	0.24
Bi, ppm	111	5	102	121	97	125	4.18%	8.37%	12.55%	106	117
Ca, wt.%	0.522	0.019	0.484	0.559	0.465	0.578	3.60%	7.20%	10.80%	0.496	0.548
Cd, ppm	27.7	1.53	24.6	30.7	23.1	32.3	5.54%	11.09%	16.63%	26.3	29.1
Ce, ppm	13.6	0.52	12.5	14.6	12.0	15.1	3.86%	7.73%	11.59%	12.9	14.3
Co, ppm	348	9	330	366	321	375	2.58%	5.16%	7.75%	331	366
Cr, ppm	30.9	2.81	25.2	36.5	22.4	39.3	9.12%	18.23%	27.35%	29.3	32.4
Cu, wt.%	30.22	0.841	28.54	31.90	27.70	32.75	2.78%	5.57%	8.35%	28.71	31.73
Dy, ppm	0.92	0.11	0.71	1.14	0.60	1.25	11.71%	23.42%	35.13%	0.88	0.97
Er, ppm	0.47	0.08	0.31	0.62	0.24	0.70	16.44%	32.88%	49.31%	0.44	0.49
Eu, ppm	0.30	0.012	0.27	0.32	0.26	0.33	4.08%	8.16%	12.24%	0.28	0.31
Fe, wt.%	21.45	0.679	20.09	22.81	19.41	23.49	3.17%	6.33%	9.50%	20.38	22.52
Ga, ppm	4.12	0.402	3.32	4.93	2.92	5.33	9.75%	19.50%	29.24%	3.92	4.33
Gd, ppm	1.10	0.12	0.86	1.34	0.74	1.46	10.90%	21.79%	32.69%	1.05	1.16
Hf, ppm	0.59	0.06	0.47	0.71	0.41	0.78	10.44%	20.87%	31.31%	0.56	0.62
In, ppm	7.12	0.221	6.68	7.56	6.46	7.78	3.11%	6.21%	9.32%	6.76	7.48
K, wt.%	0.181	0.013	0.155	0.206	0.142	0.219	7.09%	14.18%	21.27%	0.172	0.190
La, ppm	7.57	0.453	6.66	8.47	6.21	8.92	5.98%	11.97%	17.95%	7.19	7.94
Li, ppm	2.12	0.38	1.35	2.88	0.97	3.27	18.09%	36.17%	54.26%	2.01	2.22
Mg, wt.%	0.307	0.016	0.275	0.338	0.259	0.354	5.13%	10.27%	15.40%	0.291	0.322
Mn, wt.%	0.022	0.001	0.020	0.025	0.019	0.026	5.33%	10.66%	15.99%	0.021	0.023
Mo, ppm	661	33	594	728	561	762	5.06%	10.13%	15.19%	628	694
Na, wt.%	0.111	0.004	0.103	0.119	0.099	0.123	3.60%	7.19%	10.79%	0.105	0.116
Nb, ppm	1.10	0.15	0.79	1.41	0.63	1.56	14.09%	28.17%	42.26%	1.04	1.15
Nd, ppm	5.63	0.417	4.80	6.46	4.38	6.88	7.40%	14.81%	22.21%	5.35	5.91
Ni, ppm	3498	130	3238	3758	3108	3888	3.71%	7.43%	11.14%	3323	3673
P, wt.%	0.025	0.004	0.017	0.034	0.013	0.038	16.30%	32.60%	48.91%	0.024	0.027
Pb, wt.%	0.225	0.008	0.210	0.240	0.202	0.248	3.41%	6.82%	10.23%	0.214	0.236
Pr, ppm	1.53	0.083	1.36	1.70	1.28	1.78	5.45%	10.90%	16.35%	1.45	1.61
Rb, ppm	8.35	0.581	7.19	9.52	6.61	10.10	6.96%	13.92%	20.88%	7.94	8.77
Re, ppm	0.44	0.029	0.38	0.50	0.36	0.53	6.53%	13.07%	19.60%	0.42	0.46
Sb, ppm	489	24	441	538	417	562	4.94%	9.88%	14.82%	465	514
Sc, ppm	1.34	0.18	0.98	1.70	0.80	1.88	13.41%	26.83%	40.24%	1.28	1.41
Se, ppm	83	6.7	69	96	63	103	8.11%	16.22%	24.34%	79	87

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

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 1 continued.

0 11	Certified		Absolute	Standard	Deviations	5	Relative	Standard D	eviations	5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digesti	4-Acid Digestion continued										
Sm, ppm	1.12	0.11	0.90	1.35	0.78	1.46	10.03%	20.07%	30.10%	1.06	1.18
Sn, ppm	48.7	2.98	42.7	54.6	39.7	57.6	6.13%	12.26%	18.39%	46.2	51.1
Sr, ppm	25.5	1.39	22.7	28.3	21.3	29.7	5.47%	10.94%	16.41%	24.2	26.8
Tb, ppm	0.15	0.03	0.10	0.20	0.07	0.22	17.37%	34.73%	52.10%	0.14	0.15
Te, ppm	11.5	0.48	10.6	12.5	10.1	12.9	4.16%	8.32%	12.49%	10.9	12.1
Th, ppm	2.77	0.189	2.39	3.14	2.20	3.33	6.84%	13.68%	20.52%	2.63	2.90
Ti, wt.%	0.040	0.005	0.031	0.049	0.026	0.054	11.62%	23.25%	34.87%	0.038	0.042
TI, ppm	28.1	1.03	26.1	30.2	25.0	31.2	3.67%	7.35%	11.02%	26.7	29.5
Tm, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
U, ppm	4.22	0.233	3.75	4.69	3.52	4.92	5.53%	11.06%	16.59%	4.01	4.43
V, ppm	14.9	1.8	11.3	18.6	9.5	20.4	12.20%	24.39%	36.59%	14.2	15.7
W, ppm	9.75	0.617	8.52	10.99	7.90	11.60	6.32%	12.65%	18.97%	9.26	10.24
Y, ppm	4.48	0.54	3.40	5.56	2.86	6.10	12.09%	24.17%	36.26%	4.26	4.70
Yb, ppm	0.47	0.09	0.30	0.64	0.21	0.72	18.23%	36.45%	54.68%	0.44	0.49
Zn, wt.%	0.602	0.029	0.544	0.661	0.514	0.690	4.88%	9.76%	14.63%	0.572	0.632
Zr, ppm	20.6	2.3	16.0	25.3	13.7	27.6	11.25%	22.50%	33.74%	19.6	21.7
Infrared Comb	oustion										
S, wt.%	31.61	0.767	30.08	33.15	29.31	33.91	2.43%	4.85%	7.28%	30.03	33.19

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion).

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 OF CONTENTS**

INTRODUCTION	6
SOURCE MATERIAL	6
PERFORMANCE GATES	6
COMMINUTION AND HOMOGENISATION PROCEDURES	7
ANALYTICAL PROGRAM	7
PHYSICAL PROPERTIES	9
STATISTICAL ANALYSIS	9
Homogeneity Evaluation	12
PARTICIPATING LABORATORIES	13
PREPARER AND SUPPLIER	14
METROLOGICAL TRACEABILITY	14
COMMUTABILITY	17
INTENDED USE	17
HANDLING INSTRUCTIONS	17
STABILITY AND STORAGE INSTRUCTIONS	17
INSTRUCTIONS FOR CORRECT USE	17
LEGAL NOTICE	18
DOCUMENT HISTORY	18
QMS CERTIFICATION	18
CERTIFYING OFFICER	19
REFERENCES	19
LIST OF TABLES	
Table 1. Certified Values and Performance Gates for OREAS 994	1
Table 2. Indicative Values for OREAS 994.	8
Table 3. Physical properties of OREAS 994.	9
Table 4. 95% Confidence & Tolerance Limits for OREAS 994	10
LIST OF FIGURES	
Figure 1. Cu by classical wet chemistry in OREAS 994	15
Figure 2. Ag by acid digestion in OREAS 994	16

## INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological and sulphide concentrate 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 inhouse 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.

# **SOURCE MATERIAL**

OREAS 994 is a certified reference material (CRM) prepared from a blend of copper concentrate laboratory reject samples sourced from several Australian mine site metallurgical plants. Copper, Iron and Sulphur by weight account for 82.5% of the total chemical composition of OREAS 994.

#### **PERFORMANCE GATES**

The standard deviations (SD's) reported in 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 (see 'Homogeneity Evaluation' for verification of OREAS 994's high level of homogeneity).

Table 1 above 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 <a href="www.westgard.com/mltirule.htm">www.westgard.com/mltirule.htm</a>). A second method utilises a 5% window calculated directly from the certified value. For information on the calculation of standard deviations see the 'Statistical Analysis' section below.

Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL)  $\pm$  10%.

i.e. Certified Value ± 10% ± 2DL (adapted from Govett, 1983).



## COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 994 were prepared in the following manner:

- Drying of concentrate samples to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging under nitrogen in 10g units in laminated foil pouches.

# ANALYTICAL PROGRAM

For the interlaboratory 'round robin' certification program, samples were taken at 10 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire prepared batch of OREAS 994.

# **Umpire Laboratories**

Fifteen 'umpire' laboratories each received a single 60g sample and undertook copper, silver and moisture analysis on the sample as received. The term 'umpire' here refers to the routine analysis of these laboratories using classical methodologies for precious and base metals.

Strict, pre-assay instructions were provided to ensure proper handling of moisture including:

- Equilibration of sample material to laboratory atmosphere for a minimum of 2 hours;
- Hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Au, Cu and Ag as per ISO 9599.

The laboratories were requested to report analyte concentrations on both a dry (using the moisture value to correct the sample to dry basis) and moisture-bearing basis and include all results for moisture determinations. The 'Umpire Lab' certified values shown in Table 1 are on a dry sample basis (see 'Instructions for correct use' section).

The following analytical methods were undertaken:

- Copper (3 trials on undried sample) by classical wet chemistry (predominantly by short iodide titration with one laboratory employing electrogravimetry and one laboratory used a potentiometric titration);
- Silver (3 trials on undried sample) by the laboratory's own preferred acid digestion method. This resulted in 2 acid or aqua regia digestion with AAS finish (9 laboratories), 3-acid digestion with AAS (1 laboratory) or ICP-OES finish (1 laboratory) and 4-acid digestion with AAS (1 laboratory) or ICP-OES finish (1 laboratory).

# **Geoanalytical Laboratories**

Fifteen geoanalytical laboratories also participated in the analytical program for OREAS 994. Each laboratory was sent 6 x 30g samples to undertake the following:

- Full elemental suites by peroxide fusion with ICP-OES and MS finish (up to 14 laboratories depending on the element);
- Full elemental suites by 4-acid digestion with ICP-OES and MS finish (up to 15 laboratories depending on the element);
- Total S by IR combustion furnace (15 laboratories).

Table 1 provides performance gate intervals for the certified values and Table 2 shows indicative values. Table 3 provides some indicative physical properties and Table 4 presents the 95% confidence and tolerance limits for all certified values.

Table 2. Indicative Values for OREAS 994.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value		
Thermogravir	Thermogravimetry									
H <sub>2</sub> O-	wt.%	1.13								
Peroxide Fusion ICP										
Ag	ppm	182	La	ppm	8.00	Та	ppm	0.39		
В	ppm	43.2	Li	ppm	2.33	Tb	ppm	0.17		
Cs	ppm	0.53	Lu	ppm	0.10	Tm	ppm	0.086		
Gd	ppm	1.14	Nb	ppm	7.42	<b>V</b>	ppm	30.3		
Ge	ppm	2.57	Р	wt.%	0.030	Yb	ppm	0.50		
Hf	ppm	0.90	Re	ppm	0.44	Zr	ppm	30.2		
Но	ppm	0.17	Sc	ppm	< 10					
4-Acid Digest	ion									
Ва	ppm	171	Hg	ppm	1.44	S	wt.%	31.26		
Cs	ppm	0.40	Но	ppm	0.20	Та	ppm	0.076		
Ge	ppm	0.71	Lu	ppm	0.059					
Infrared Com	Infrared Combustion									
С	wt.%	0.247				_				

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb (parts per billion). 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.

Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM³) are presented in the detailed certification data for this CRM (OREAS 994-DataPack.1.0.200902\_133657.xlsx).

Results are also presented in scatter plots for Cu (wt.%) by classical wet chemistry and Ag by acid digestion in Figures 1 and 2 respectively, 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.

## PHYSICAL PROPERTIES

OREAS 994 was tested at ORE Research & Exploration Pty Ltd's onsite laboratory for various physical properties. Table 3 presents these findings which should be used for informational purposes only.

Table 3. Physical properties of OREAS 994.

CRM Name	Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color‡
OREAS 994	795	1.93	N3	Dark Gray

<sup>&</sup>lt;sup>‡</sup>The Munsell Rock Color Chart helps geologists and archeologists communicate with color more effectively by cross-referencing ISCC-NBS color names with unique Munsell alpha-numeric color notations for rock color samples.

# 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 4) 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.

**Certified Values** are the means of accepted laboratory means after outlier filtering.

**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 interlaboratory consensus is poor.

Table 4. 95% Confidence & Tolerance Limits for OREAS 994.

Constituent	Certified	95% Confid	lence Limits	95% Tolerance Limits					
Constituent	Value	Low	High	Low	High				
Classical Wet Chemistry									
Cu, Copper (wt.%)	30.00	29.96	30.05	29.96	30.05				
Acid Digestion (no HF)									
Ag, Silver (ppm)	181	179	184	180	183				
Peroxide Fusion ICP									
Al, Aluminium (wt.%)	0.706	0.695	0.717	0.684	0.727				
As, Arsenic (ppm)	3607	3521	3693	3514	3700				
Ba, Barium (ppm)	213	204	222	205	221				
Be, Beryllium (ppm)	< 1	IND	IND	IND	IND				
Bi, Bismuth (ppm)	115	110	120	110	120				
Ca, Calcium (wt.%)	0.542	0.528	0.557	0.518	0.567				
Cd, Cadmium (ppm)	29.9	26.9	32.9	28.3	31.5				
Ce, Cerium (ppm)	14.5	13.3	15.8	13.8	15.3				
Co, Cobalt (ppm)	353	349	356	341	364				
Cr, Chromium (ppm)	< 100	IND	IND	IND	IND				
Cu, Copper (wt.%)	29.62	29.36	29.88	29.35	29.89				
Dy, Dysprosium (ppm)	1.02	0.95	1.09	IND	IND				
Er, Erbium (ppm)	0.57	0.51	0.63	IND	IND				
Eu, Europium (ppm)	0.35	0.31	0.40	IND	IND				
Fe, Iron (wt.%)	21.48	21.24	21.72	21.10	21.87				
Ga, Gallium (ppm)	4.30	4.07	4.52	IND	IND				
In, Indium (ppm)	7.71	7.12	8.30	7.28	8.14				
K, Potassium (wt.%)	0.193	0.181	0.204	IND	IND				
Mg, Magnesium (wt.%)	0.317	0.308	0.326	0.306	0.328				
Mn, Manganese (wt.%)	0.023	0.023	0.023	0.022	0.024				
Mo, Molybdenum (ppm)	677	657	697	655	700				
Nd, Neodymium (ppm)	5.71	5.40	6.03	IND	IND				
Ni, Nickel (ppm)	3548	3489	3608	3453	3644				
Pb, Lead (wt.%)	0.227	0.220	0.235	0.220	0.235				

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$  µg/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note 1: intervals may appear asymmetric due to rounding.

# Table 4 continued.

Table 4 continued.						
Constituent	Certified		lence Limits	95% Tolerance Limits		
	Value	Low	High	Low	High	
Peroxide Fusion ICP continued					1	
Pr, Praseodymium (ppm)	1.59	1.49	1.68	IND	IND	
Rb, Rubidium (ppm)	8.81	8.29	9.32	8.12	9.49	
S, Sulphur (wt.%)	31.44	31.09	31.80	30.68	32.21	
Sb, Antimony (ppm)	499	451	547	482	516	
Se, Selenium (ppm)	83	76	91	76	91	
Si, Silicon (wt.%)	3.15	3.11	3.18	3.07	3.22	
Sm, Samarium (ppm)	1.23	1.06	1.41	IND	IND	
Sn, Tin (ppm)	50.0	47.6	52.4	41.4	58.5	
Sr, Strontium (ppm)	28.3	24.8	31.8	27.0	29.7	
Te, Tellurium (ppm)	13.8	10.8	16.7	IND	IND	
Th, Thorium (ppm)	2.86	2.76	2.95	2.61	3.10	
Ti, Titanium (wt.%)	0.050	0.049	0.050	IND	IND	
Tl, Thallium (ppm)	27.7	26.7	28.6	26.8	28.6	
U, Uranium (ppm)	4.26	3.83	4.68	3.96	4.56	
W, Tungsten (ppm)	10.9	9.1	12.6	IND	IND	
Y, Yttrium (ppm)	5.42	4.98	5.85	5.20	5.64	
Zn, Zinc (wt.%)	0.611	0.598	0.624	0.598	0.624	
4-Acid Digestion					I.	
Ag, Silver (ppm)	183	178	187	180	186	
Al, Aluminium (wt.%)	0.696	0.685	0.708	0.680	0.713	
As, Arsenic (ppm)	3483	3402	3564	3404	3562	
Be, Beryllium (ppm)	0.23	0.19	0.26	IND	IND	
Bi, Bismuth (ppm)	111	109	114	108	115	
Ca, Calcium (wt.%)	0.522	0.510	0.533	0.511	0.532	
Cd, Cadmium (ppm)	27.7	26.8	28.6	26.8	28.5	
Ce, Cerium (ppm)	13.6	13.2	14.0	13.0	14.1	
Co, Cobalt (ppm)	348	343	353	341	356	
Cr, Chromium (ppm)	30.9	29.0	32.7	29.4	32.3	
Cu, Copper (wt.%)	30.22	29.59	30.85	29.57	30.87	
Dy, Dysprosium (ppm)	0.92	0.84	1.01	IND	IND	
Er, Erbium (ppm)	0.47	0.41	0.53	IND	IND	
Eu, Europium (ppm)	0.30	0.29	0.31	IND	IND	
Fe, Iron (wt.%)	21.45	21.11	21.80	21.13	21.77	
Ga, Gallium (ppm)	4.12	3.85	4.40	3.91	4.34	
Gd, Gadolinium (ppm)	1.10	1.01	1.19	IND	IND	
Hf, Hafnium (ppm)	0.59	0.56	0.62	IND	IND	
In, Indium (ppm)	7.12	6.98	7.25	6.89	7.34	
K, Potassium (wt.%)	0.181	0.174	0.188	0.176	0.185	
La, Lanthanum (ppm)	7.57	7.27	7.86	7.21	7.92	
Li, Lithium (ppm)	2.12	1.82	2.42	1.92	2.32	
Mg, Magnesium (wt.%)	0.307	0.298	0.316	0.300	0.314	
<u> </u>	l .	0.298	1		0.014	

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion.

Note 1: intervals may appear asymmetric due to rounding.



#### Table 4 continued.

	Table 4 Continued.							
Constituent	Certified	95% Confid	lence Limits	95% Toler	95% Tolerance Limits			
Condition	Value	Low	High	Low	High			
4-Acid Digestion continued								
Mn, Manganese (wt.%)	0.022	0.022	0.023	0.022	0.023			
Mo, Molybdenum (ppm)	661	643	680	645	678			
Na, Sodium (wt.%)	0.111	0.109	0.113	0.108	0.114			
Nb, Niobium (ppm)	1.10	0.99	1.21	IND	IND			
Nd, Neodymium (ppm)	5.63	5.31	5.95	5.31	5.95			
Ni, Nickel (ppm)	3498	3422	3574	3451	3545			
P, Phosphorus (wt.%)	0.025	0.023	0.028	0.024	0.027			
Pb, Lead (wt.%)	0.225	0.221	0.229	0.219	0.231			
Pr, Praseodymium (ppm)	1.53	1.46	1.59	IND	IND			
Rb, Rubidium (ppm)	8.35	8.02	8.68	7.96	8.74			
Re, Rhenium (ppm)	0.44	0.42	0.46	0.41	0.47			
Sb, Antimony (ppm)	489	474	504	476	502			
Sc, Scandium (ppm)	1.34	1.14	1.54	IND	IND			
Se, Selenium (ppm)	83	79	87	79	87			
Sm, Samarium (ppm)	1.12	1.10	1.14	IND	IND			
Sn, Tin (ppm)	48.7	46.8	50.5	46.7	50.6			
Sr, Strontium (ppm)	25.5	24.6	26.4	24.4	26.5			
Tb, Terbium (ppm)	0.15	0.14	0.16	IND	IND			
Te, Tellurium (ppm)	11.5	11.2	11.8	10.9	12.1			
Th, Thorium (ppm)	2.77	2.66	2.88	2.66	2.87			
Ti, Titanium (wt.%)	0.040	0.037	0.042	0.038	0.041			
TI, Thallium (ppm)	28.1	27.5	28.7	27.1	29.1			
Tm, Thulium (ppm)	< 0.1	IND	IND	IND	IND			
U, Uranium (ppm)	4.22	4.11	4.33	4.00	4.44			
V, Vanadium (ppm)	14.9	13.7	16.2	IND	IND			
W, Tungsten (ppm)	9.75	9.36	10.14	9.24	10.26			
Y, Yttrium (ppm)	4.48	4.15	4.81	4.31	4.65			
Yb, Ytterbium (ppm)	0.47	0.39	0.54	IND	IND			
Zn, Zinc (wt.%)	0.602	0.585	0.619	0.592	0.613			
Zr, Zirconium (ppm)	20.6	19.1	22.1	19.5	21.7			
Infrared Combustion	•	•	•	•	•			
S, Sulphur (wt.%)	31.61	31.20	32.03	31.21	32.02			
	•	•	•	•				

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note 1: intervals may appear asymmetric due to rounding.

# **Homogeneity Evaluation**

The tolerance limits (ISO 16269:2014) shown in Table 1 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 classical wet chemistry, where 99% of the time  $(1-\alpha=0.99)$  at least 95% of subsamples (p=0.95) will have concentrations lying between 29.96 and 30.05 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 994 has also been evaluated in an ANOVA study for all certified analytes present in concentrations that are at least 20 times the lower limit of detection. No significant p-values were found indicating that no evidence exists that between-unit variance is greater than within-unit variance.

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 994 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is large and similar across all units.

# PARTICIPATING LABORATORIES

- 1. \*Actlabs, Ancaster, Ontario, Canada
- 2. \*AGAT Laboratories, Mississauga, Ontario, Canada
- 3. \*AH Knight, Tianjin, China
- 4. \*ALS, Perth, WA, Australia
- 5. \*ALS, Ulaanbaatar, Khan-Uul District, Mongolia
- 6. \*ALS, Vancouver, BC, Canada
- 7. \*ALS Inspection, Prescot, Merseyside, UK
- 8. \*Bachelet, Angleur, Liege, Belgium
- 9. \*Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 10. \*Bureau Veritas Geoanalytical, Perth, WA, Australia
- 11. Customs Central Laboratory of Mongolia, Ulaanbaatar, Sükhbaatar District, Mongolia
- 12. \*Erdenet Central Chemical Laboratory, Erdenet, Orkhon province, Mongolia
- 13. \*Inspectorate (BV), Lima, Peru
- 14. Inspectorate (BV), Shanghai, Bao Shan District, China
- 15. Inspectorate (BV), Witham, Essex, UK
- 16. \*Intertek Genalysis, Perth, WA, Australia
- 17. Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
- 18. \*Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 19. \*MinAnalytical Services, Perth, WA, Australia
- 20. \*MSALABS, Vancouver, BC, Canada
- 21. \*Nagrom, Perth, WA, Australia
- 22. \*PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 23. \*RC Inspection, Rotterdam, Netherlands





- 24. \*RC Inspection, Ulaanbaatar, Khan-Uul District, Mongolia
- 25. \*Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
- 26. \*SGS Australia Mineral Services, Perth, WA, Australia
- 27. \*SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 28. \*SGS Nederland B.V., Spijkenisse, Zuid-Holland, Netherlands
- 29. \*SRL, Perth, WA, Australia
  - ♦ = Umpire laboratory (classical methods); \* = Geoanalytical laboratory (instrumental methods).

Please note: Above numbered alphabetical list of participating laboratories does not reflect the Lab ID numbering on the scatter plots below.

# PREPARER AND SUPPLIER

Certified reference material OREAS 994 was prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd

Tel: +613-9729 0333
37A Hosie Street

Fax: +613-9729 8338
Bayswater North VIC 3153

Web: www.ore.com.au
AUSTRALIA

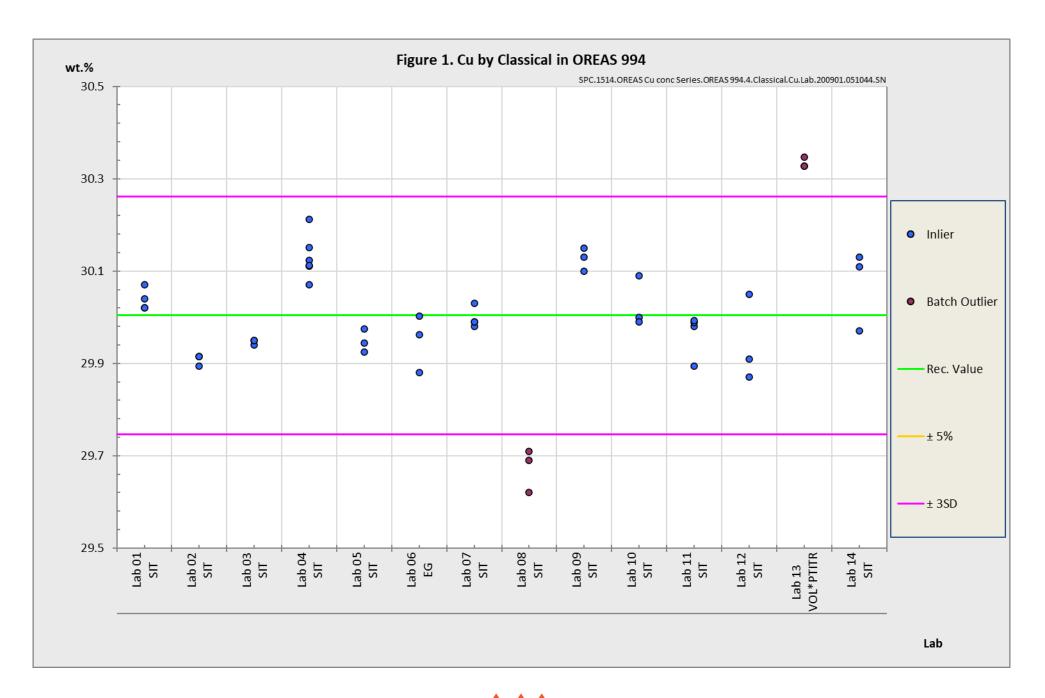
Email: info@ore.com.au

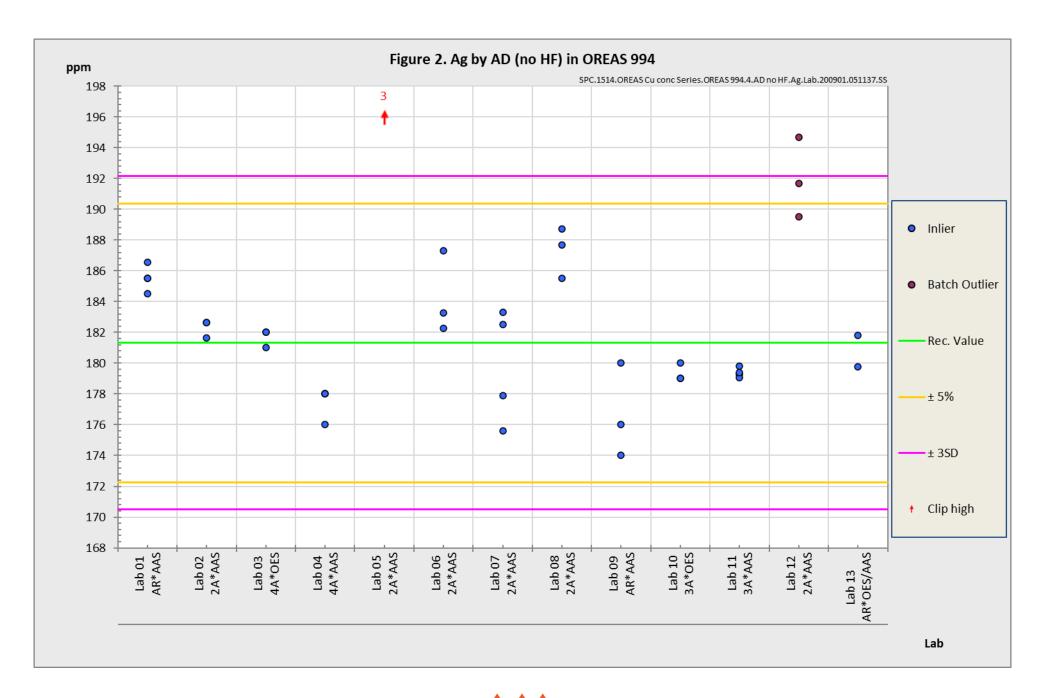
# **METROLOGICAL TRACEABILITY**

The analytical samples were selected in a manner representative of the entire batch of the prepared 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 that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment, 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 (digestion/fusion) 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/concentrate samples 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 their field samples.

# **INTENDED USE**

OREAS 994 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 994 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 994 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in sulphide concentrate samples;
- For the verification of analytical methods for analytes reported in Table 1;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Table 1.

# HANDLING INSTRUCTIONS

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised. Sulphur is a known transitory upper respiratory irritant. Close exposure may cause coughing or throat irritation.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 994 was prepared from a blend of various sulphide bearing copper concentrate samples. To ensure a long shelf life it has been sealed under nitrogen in robust laminated foil pouches. In its unopened state under normal conditions of storage it has a shelf life beyond five years.

\*normal storage conditions: not in direct sunlight in a dry, clean, well ventilated area at temperatures between -10° and 50°C.

## INSTRUCTIONS FOR CORRECT USE

The 'umpire lab' Cu, Au and Ag certified values for OREAS 994 refer to the concentration levels on a dry sample basis. All analyses were performed on the samples as received after



equilibration with the laboratory atmosphere for a minimum of 2 hours and hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Cu and Ag as per ISO 9599. The results were then corrected to dry basis using the moisture value. Moisture content varied amongst the laboratories between 0.21 and 2.77 wt.% but with an average of 1.13 wt.%.

The 'geoanalytical lab' certified values for OREAS 994 are on a 'sample as received' basis. The CRM should not be dried prior to analysis.

# 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. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

# **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
0	2 <sup>nd</sup> Sep, 2020	First publication.

## **QMS CERTIFICATION**

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**



2<sup>nd</sup> September, 2020

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

## **REFERENCES**

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

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

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

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

ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.

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

ISO 17025:2005, General requirements for the competence of testing and calibration laboratories.

Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), 4300 44th Street SE, Grand Rapids, MI 49512.