

### CERTIFICATE OF ANALYSIS FOR

# COPPER-SILVER-GOLD-PALLADIUM-PLATINUM CONCENTRATE

### (Nickel matte by-product, Western Australia)

# CERTIFIED REFERENCE MATERIAL OREAS 992b

Constituent Certified Value	Certified		Absolute	Standard	Deviations	6	Relative	Standard D	eviations	5% w	indow
	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Umpire Labs (dry sample basis)											
Classical Wet Chemistry											
Cu, wt.%	45.98	0.227	45.52	46.43	45.30	46.66	0.49%	0.99%	1.48%	43.68	48.28
Pb Fire Assay	(Grav)										
Ag, ppm	344	7	331	357	324	364	1.93%	3.86%	5.79%	327	361
Au, ppm	15.00	0.851	13.30	16.70	12.44	17.55	5.67%	11.35%	17.02%	14.25	15.75
Pb Fire Assay	,										
Pd, ppm	127.9	3.0	121.8	133.9	118.8	136.9	2.36%	4.72%	7.08%	121.5	134.3
Pt, ppm	21.90	0.695	20.51	23.29	19.82	23.99	3.17%	6.35%	9.52%	20.81	23.00

#### Table 1. Certified Values and Performance Gates for OREAS 992b.

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.



Constituent	Certified		Absolute	Standard	Deviation	3	Relative	Standard D	eviations	5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Geoanalytical	Labs ('as r	eceived' s	ample ba	asis)							
Peroxide Fusi	ion ICP										
AI, wt.%	0.047	0.007	0.032	0.061	0.025	0.068	15.61%	31.21%	46.82%	0.044	0.049
As, ppm	594	38	517	670	478	709	6.48%	12.96%	19.43%	564	623
Ba, ppm	584	40	503	665	463	706	6.92%	13.83%	20.75%	555	614
Be, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	3.89	0.233	3.42	4.36	3.19	4.59	6.00%	11.99%	17.99%	3.70	4.09
Ca, wt.%	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cd, ppm	26.8	2.17	22.5	31.2	20.3	33.3	8.09%	16.18%	24.27%	25.5	28.2
Co, ppm	750	31	688	812	657	843	4.14%	8.28%	12.42%	712	787
Cr, ppm	< 100	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, wt.%	44.52	1.603	41.32	47.73	39.71	49.34	3.60%	7.20%	10.80%	42.30	46.75
Dy, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Er, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Eu, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Fe, wt.%	1.07	0.037	1.00	1.15	0.96	1.18	3.42%	6.84%	10.26%	1.02	1.13
Ga, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
In, ppm	0.38	0.06	0.27	0.50	0.22	0.55	14.62%	29.25%	43.87%	0.36	0.40
K, wt.%	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Mg, wt.%	0.013	0.004	0.005	0.022	0.000	0.026	32.07%	64.14%	96.21%	0.013	0.014
Mn, wt.%	0.013	0.002	0.008	0.017	0.006	0.019	16.50%	33.00%	49.50%	0.012	0.013
Mo, ppm	9.22	1.02	7.19	11.25	6.17	12.27	11.02%	22.04%	33.06%	8.76	9.68
Ni, wt.%	1.48	0.028	1.42	1.53	1.39	1.56	1.90%	3.81%	5.71%	1.40	1.55
P, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pb, wt.%	0.379	0.015	0.350	0.409	0.335	0.423	3.89%	7.79%	11.68%	0.360	0.398
Pr, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Rb, ppm	1.97	0.54	0.90	3.05	0.36	3.58	27.17%	54.35%	81.52%	1.87	2.07
Re, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	38.33	1.015	36.30	40.36	35.28	41.38	2.65%	5.30%	7.95%	36.41	40.25
Sb, ppm	841	78	685	997	607	1075	9.28%	18.55%	27.83%	799	883
Se, ppm	74	6.4	61	87	55	93	8.60%	17.20%	25.81%	70	78
Si, wt.%	0.220	0.028	0.164	0.275	0.137	0.303	12.61%	25.22%	37.83%	0.209	0.231
Te, ppm	1.90	0.62	0.66	3.14	0.03	3.77	32.73%	65.46%	98.19%	1.81	2.00
Th, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ti, wt.%	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
TI, ppm	0.58	0.07	0.45	0.72	0.39	0.78	11.25%	22.50%	33.75%	0.56	0.61
Y, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Zn, wt.%	0.861	0.034	0.793	0.929	0.759	0.963	3.94%	7.88%	11.83%	0.818	0.904
4-Acid Digest	ion										
Ag, ppm	340	13	314	367	301	380	3.82%	7.65%	11.47%	323	358
AI, wt.%	0.040	0.003	0.034	0.047	0.031	0.050	7.70%	15.40%	23.10%	0.038	0.042
SI unit equival	lents: nnm (i	narte nor	million) =	ma/ka =	ua/a = 0.0		$= 1000  \mathrm{n}$	nh (narte n	or hillion)	•	•

#### Table 1 continued.

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.



Constituent	Certified		Absolute	Standard	Deviation	S	Relative	Standard D	eviations	viations 5% window		
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High	
4-Acid Digest	ion continue	ed										
As, ppm	582	36	510	653	475	689	6.12%	12.25%	18.37%	553	611	
Be, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Bi, ppm	3.75	0.159	3.43	4.07	3.27	4.23	4.24%	8.48%	12.72%	3.56	3.94	
Ca, wt.%	0.017	0.004	0.009	0.025	0.006	0.029	22.57%	45.14%	67.71%	0.016	0.018	
Cd, ppm	25.5	1.80	21.9	29.1	20.1	30.9	7.05%	14.10%	21.15%	24.3	26.8	
Ce, ppm	0.65	0.15	0.35	0.94	0.20	1.09	23.05%	46.09%	69.14%	0.61	0.68	
Co, ppm	749	21	707	791	686	812	2.81%	5.63%	8.44%	712	786	
Cs, ppm	0.082	0.016	0.050	0.115	0.034	0.131	19.58%	39.16%	58.74%	0.078	0.086	
Cu, wt.%	44.73	1.393	41.94	47.51	40.55	48.91	3.11%	6.23%	9.34%	42.49	46.96	
Er, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Eu, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Fe, wt.%	1.05	0.054	0.95	1.16	0.89	1.22	5.10%	10.20%	15.30%	1.00	1.11	
Ga, ppm	0.26	0.04	0.17	0.34	0.13	0.38	16.25%	32.49%	48.74%	0.24	0.27	
Gd, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Hf, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
In, ppm	0.36	0.033	0.29	0.42	0.26	0.45	9.24%	18.49%	27.73%	0.34	0.37	
K, wt.%	0.020	0.004	0.012	0.027	0.008	0.031	20.17%	40.34%	60.51%	0.019	0.021	
Mg, wt.%	0.010	0.001	0.008	0.012	0.007	0.013	9.57%	19.14%	28.71%	0.009	0.010	
Mn, wt.%	0.012	0.001	0.011	0.014	0.011	0.014	4.87%	9.73%	14.60%	0.012	0.013	
Mo, ppm	7.29	0.78	5.73	8.85	4.95	9.64	10.72%	21.44%	32.16%	6.93	7.66	
Na, wt.%	0.010	0.001	0.007	0.012	0.006	0.014	13.42%	26.83%	40.25%	0.009	0.010	
Nb, ppm	0.093	0.010	0.072	0.113	0.062	0.123	11.04%	22.08%	33.12%	0.088	0.097	
Nd, ppm	0.29	0.021	0.25	0.33	0.23	0.35	7.07%	14.13%	21.20%	0.28	0.31	
Ni, wt.%	1.48	0.021	1.44	1.53	1.42	1.55	1.42%	2.85%	4.27%	1.41	1.56	
Pb, wt.%	0.374	0.010	0.354	0.393	0.345	0.403	2.58%	5.16%	7.74%	0.355	0.392	
Pr, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Rb, ppm	0.97	0.12	0.73	1.22	0.61	1.34	12.60%	25.20%	37.80%	0.93	1.02	
Re, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Sb, ppm	818	35	748	887	714	922	4.24%	8.48%	12.71%	777	859	
Sc, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Se, ppm	71	9	53	89	44	98	12.60%	25.20%	37.80%	67	74	
Sn, ppm	0.48	0.06	0.36	0.60	0.30	0.66	12.57%	25.14%	37.71%	0.45	0.50	
Sr, ppm	4.96	0.87	3.21	6.70	2.34	7.58	17.60%	35.21%	52.81%	4.71	5.21	
Tb, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
Te, ppm	1.63	0.130	1.37	1.89	1.24	2.02	7.99%	15.99%	23.98%	1.55	1.71	
Ti, wt.%	0.003	0.000	0.002	0.003	0.001	0.004	17.91%	35.81%	53.72%	0.002	0.003	
TI, ppm	0.60	0.032	0.53	0.66	0.50	0.70	5.39%	10.79%	16.18%	0.57	0.63	
Tm, ppm	< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
U, ppm	0.26	0.05	0.17	0.35	0.12	0.39	17.77%	35.53%	53.30%	0.24	0.27	
V, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
SI unit oquiva												

#### Table 1 continued.

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 1 continued.

Constituent Certified Value		Absolute	Standard	Deviations	6	Relative	Standard D	eviations	5% window		
	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High	
4-Acid Digestion continued											
1.12	0.23	0.66	1.58	0.43	1.81	20.42%	40.85%	61.27%	1.07	1.18	
0.26	0.05	0.17	0.36	0.12	0.41	18.79%	37.57%	56.36%	0.25	0.28	
< 0.1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND	
0.862	0.036	0.790	0.935	0.754	0.971	4.20%	8.40%	12.60%	0.819	0.905	
1.33	0.087	1.15	1.50	1.07	1.59	6.54%	13.08%	19.63%	1.26	1.39	
Infrared Combustion											
37.79	0.701	36.39	39.19	35.69	39.89	1.85%	3.71%	5.56%	35.90	39.68	
	Value ion continue 1.12 0.26 < 0.1 0.862 1.33 oustion	Certified Value         1SD           ion continue         1           1.12         0.23           0.26         0.05           < 0.1	Certified Value         1SD         2SD Low           ion continue         1         0.23         0.66           0.26         0.05         0.17           < 0.1	Certified Value         1SD         2SD Low         2SD High           ion continue         2SD         2SD           1.12         0.23         0.66         1.58           0.26         0.05         0.17         0.36           < 0.1	Certified Value         1SD         2SD Low         2SD High         3SD Low           ion continued         0.23         0.66         1.58         0.43           0.26         0.05         0.17         0.36         0.12           < 0.1	Value         1SD         2SD Low         2SD High         3SD Low         3SD High         3SD High           ion continue         0.23         0.66         1.58         0.43         1.81           0.26         0.05         0.17         0.36         0.12         0.41           < 0.1	Certified Value         1SD         2SD Low         2SD High         3SD Low         3SD High         1RSD           ion continue         0.23         0.66         1.58         0.43         1.81         20.42%           0.26         0.05         0.17         0.36         0.12         0.41         18.79%           < 0.1	Certified Value         1SD         2SD Low         2SD High         3SD Low         3SD High         1RSD         2RSD           ion continue         0.23         0.66         1.58         0.43         1.81         20.42%         40.85%           0.26         0.05         0.17         0.36         0.12         0.41         18.79%         37.57%           < 0.1	Certified Value         1SD         2SD Low         2SD High         3SD Low         3SD High         1RSD         2RSD         3RSD           ion continue         1         0.23         0.66         1.58         0.43         1.81         20.42%         40.85%         61.27%           0.26         0.05         0.17         0.36         0.12         0.41         18.79%         37.57%         56.36%           < 0.1	Certified Value         1SD         2SD Low         2SD High         3SD Low         3SD High         1RSD         2RSD         3RSD         Low           ion continued           1.12         0.23         0.66         1.58         0.43         1.81         20.42%         40.85%         61.27%         1.07           0.26         0.05         0.17         0.36         0.12         0.41         18.79%         37.57%         56.36%         0.25           < 0.1	

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.



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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 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 992b is a copper sulphide certified reference material (CRM) prepared from a nickel matte by-product during the refining process at a nickel refinery. It has been prepared, packaged and certified by Ore Research & Exploration P/L.

## 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 992b'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 <u>www.westgard.com/mltirule.htm</u>). 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 material constituting OREAS 992b was prepared in the following manner:

- Drying of ore materials to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging into 10g and 50g units sealed under nitrogen 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 992b.

#### Umpire Laboratories

Fifteen 'umpire' laboratories each received a single 140g sample and undertook copper, gold, silver, platinum, palladium and moisture analysis on the sample as received. The term 'umpire' here refers to the routine analysis by 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 <u>and</u> weighed for analysis at the same time as the sample aliquots for Au, Cu, Pt, Pd 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 employing a potentiometric titration);
- Gold and Silver (3 trials on undried sample) by reduced charge weight (1-30g) fire assay with gravimetric finish and full corrections for slag, cupel and silver losses;
- Palladium and Platinum (3 trials on undried sample) by reduced charge weight (1-30g) fire assay with ICP-OES finish (10 laboratories).

#### Geoanalytical Laboratories

Fifteen geoanalytical laboratories also participated in the analytical program for OREAS 992b. 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);
- Gold by instrumental neutron activation analysis (INAA) on 20 x 85mg subsamples to confirm homogeneity (undertaken by ANSTO, Lucas Heights).

Table 1 provides performance gate intervals for the certified values and Table 2 shows indicative values. Table 3 provides some indicative physical properties, Table 4 presents the 95% confidence and tolerance limits for all certified values and gold homogeneity (via INAA) is shown in Table 5. Gold homogeneity is also demonstrated by a nested ANOVA program using fire assay (see 'Homogeneity Evaluation' section).

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value			
Thermogravin	Thermogravimetry										
H <sub>2</sub> O-	wt.%	3.13									
Peroxide Fusion ICP											
Ag	ppm	344	Li	ppm	< 10	Tb	ppm	< 0.1			
В	ppm	< 50	Lu	ppm	< 0.2	Tm	ppm	< 0.1			
Ce	ppm	0.83	Nb	ppm	< 5	U	ppm	0.62			
Cs	ppm	0.21	Nd	ppm	0.31	V	ppm	< 10			
Gd	ppm	< 0.1	Sc	ppm	< 10	W	ppm	6.41			
Ge	ppm	< 1	Sm	ppm	< 0.5	Yb	ppm	< 0.1			
Hf	ppm	< 10	Sn	ppm	28.4	Zr	ppm	< 5			
Но	ppm	< 0.1	Sr	ppm	11.0						
La	ppm	< 1	Та	ppm	< 0.1						
4-Acid Digest	ion										
Ba	ppm	204	Но	ppm	< 0.1	S	wt.%	31.19			
Cr	ppm	4.30	La	ppm	0.30	Sm	ppm	0.10			
Dy	ppm	< 0.1	Li	ppm	0.23	Та	ppm	< 0.1			
Ge	ppm	0.21	Lu	ppm	< 0.1	Th	ppm	0.10			
Hg	ppm	0.93	Р	wt.%	0.033						
Infrared Com	oustion										
С	wt.%	0.083									

Table 2. Indicative Values for OREAS 992b.

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$  µg/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.

Comparisons of inter-laboratory bias and precision are graphically presented in scatter plots for copper, silver, gold, palladium and platinum by the umpire laboratories (Figures 1 to 5, respectively) together with ±3SD (magenta) and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

Gold homogeneity has been evaluated and confirmed by INAA on 20 x 85 milligram subsamples. Tabulated results of all elements (including Au INAA analyses) together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in an Excel-compatible file for this CRM (**OREAS 992b-DataPack.2.0.200909\_205328.xlsx**).



# PHYSICAL PROPERTIES

OREAS 992b 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.

		elea preperties e				
CRM Name Bulk Density (g/L)		Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>		
OREAS 992b	620	2.7	N2	Grayish Black		

#### Table 3. Physical properties of OREAS 992b.

<sup>‡</sup>The Munsell Rock Color Chart helps geologists and archeologists communicate with color more effectively by crossreferencing 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. The INAA data (see Table 5) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 992b (see 'Homogeneity Evaluation' section below).

**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.

Constituent	Certified	95% Confic	lence Limits	95% Toler	ance Limits
Constituent	Value	Low	High	Low	High
Umpire Labs (dry sample basis)	•				
Classical Wet Chemistry					
Cu, Copper (wt.%)	45.98	45.81	46.15	45.91	46.05
Pb Fire Assay (Grav)					
Ag, Silver (ppm)	344	338	350	337	351
Au, Gold (ppm)	15.00	14.55	15.44	14.94*	15.05*
Pb Fire Assay					
Pd, Palladium (ppm)	127.9	125.7	130.0	127.1	128.7
Pt, Platinum (ppm)	21.90	21.36	22.44	21.85	21.96
Geoanalytical Labs ('as received' s	ample basis)				
Peroxide Fusion ICP					
AI, Aluminium (wt.%)	0.047	0.041	0.052	IND	IND
As, Arsenic (ppm)	594	571	617	574	613
Ba, Barium (ppm)	584	540	629	558	611
Be, Beryllium (ppm)	< 1	IND	IND	IND	IND
Bi, Bismuth (ppm)	3.89	3.68	4.10	IND	IND
Ca, Calcium (wt.%)	< 0.05	IND	IND	IND	IND
Cd, Cadmium (ppm)	26.8	24.8	28.8	25.6	28.0
Co, Cobalt (ppm)	750	732	768	729	771
Cr, Chromium (ppm)	< 100	IND	IND	IND	IND
Cu, Copper (wt.%)	44.52	43.06	45.99	43.80	45.25
Dy, Dysprosium (ppm)	< 0.5	IND	IND	IND	IND
Er, Erbium (ppm)	< 0.1	IND	IND	IND	IND
Eu, Europium (ppm)	< 0.1	IND	IND	IND	IND
Fe, Iron (wt.%)	1.07	1.05	1.09	1.04	1.10

#### Table 4. 95% Confidence & Tolerance Limits for OREAS 992b.

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

\*Gold Tolerance Limits for typical 15g fire assay methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note 1: intervals may appear asymmetric due to rounding.



	Table 4	continued.			
Constituent	Certified	95% Confid	dence Limits	95% Tolera	ance Limits
Constituent	Value	Low	High	Low	High
Peroxide Fusion ICP continued	t k				-
Ga, Gallium (ppm)	< 1	IND	IND	IND	IND
In, Indium (ppm)	0.38	0.35	0.42	IND	IND
K, Potassium (wt.%)	< 0.1	IND	IND	IND	IND
Mg, Magnesium (wt.%)	0.013	0.011	0.015	IND	IND
Mn, Manganese (wt.%)	0.013	0.012	0.013	0.012	0.013
Mo, Molybdenum (ppm)	9.22	8.38	10.06	IND	IND
Ni, Nickel (wt.%)	1.48	1.46	1.49	1.45	1.50
P, Phosphorus (wt.%)	< 0.01	IND	IND	IND	IND
Pb, Lead (wt.%)	0.379	0.371	0.388	0.370	0.388
Pr, Praseodymium (ppm)	< 0.5	IND	IND	IND	IND
Rb, Rubidium (ppm)	1.97	1.35	2.60	IND	IND
Re, Rhenium (ppm)	< 0.1	IND	IND	IND	IND
S, Sulphur (wt.%)	38.33	37.62	39.04	37.53	39.13
Sb, Antimony (ppm)	841	755	927	808	874
Se, Selenium (ppm)	74	68	80	67	81
Si, Silicon (wt.%)	0.220	0.198	0.242	0.188	0.252
Te, Tellurium (ppm)	1.90	1.38	2.42	IND	IND
Th, Thorium (ppm)	< 0.5	IND	IND	IND	IND
Ti, Titanium (wt.%)	< 0.01	IND	IND	IND	IND
TI, Thallium (ppm)	0.58	0.54	0.63	IND	IND
Y, Yttrium (ppm)	< 0.5	IND	IND	IND	IND
Zn, Zinc (wt.%)	0.861	0.841	0.881	0.846	0.876
4-Acid Digestion			1	<u> </u>	
Ag, Silver (ppm)	340	330	351	331	350
Al, Aluminium (wt.%)	0.040	0.039	0.042	IND	IND
As, Arsenic (ppm)	582	560	604	568	595
Be, Beryllium (ppm)	< 0.05	IND	IND	IND	IND
Bi, Bismuth (ppm)	3.75	3.67	3.83	3.61	3.88
Ca, Calcium (wt.%)	0.017	0.015	0.019	IND	IND
Cd, Cadmium (ppm)	25.5	24.5	26.5	24.5	26.5
Ce, Cerium (ppm)	0.65	0.53	0.76	0.51	0.78
Co, Cobalt (ppm)	749	735	763	741	757
Cs, Caesium (ppm)	0.082	0.064	0.101	IND	IND
Cu, Copper (wt.%)	44.73	43.46	46.00	44.26	45.19
Er, Erbium (ppm)	< 0.1	IND	IND	IND	IND
Eu, Europium (ppm)	< 0.1	IND	IND	IND	IND
Fe, Iron (wt.%)	1.05	1.02	1.08	1.03	1.08
Ga, Gallium (ppm)	0.26	0.22	0.29	IND	IND
Gd, Gadolinium (ppm)	< 0.1	IND	IND	IND	IND
Hf, Hafnium (ppm)	< 0.1	IND	IND	IND	IND
In, Indium (ppm)	0.36	0.34	0.37	0.33	0.38

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.			
Constituent	Certified	95% Confic	lence Limits	95% Tolera	ance Limits
Constituent	Value	Low	High	Low	High
4-Acid Digestion continued					
K, Potassium (wt.%)	0.020	0.017	0.022	IND	IND
Mg, Magnesium (wt.%)	0.010	0.010	0.010	IND	IND
Mn, Manganese (wt.%)	0.012	0.012	0.013	0.012	0.013
Mo, Molybdenum (ppm)	7.29	6.69	7.89	6.96	7.62
Na, Sodium (wt.%)	0.010	0.009	0.011	IND	IND
Nb, Niobium (ppm)	0.093	0.080	0.105	IND	IND
Nd, Neodymium (ppm)	0.29	0.28	0.31	IND	IND
Ni, Nickel (wt.%)	1.48	1.47	1.50	1.47	1.50
Pb, Lead (wt.%)	0.374	0.368	0.380	0.368	0.380
Pr, Praseodymium (ppm)	< 0.1	IND	IND	IND	IND
Rb, Rubidium (ppm)	0.97	0.90	1.05	IND	IND
Re, Rhenium (ppm)	< 0.05	IND	IND	IND	IND
Sb, Antimony (ppm)	818	793	843	792	843
Sc, Scandium (ppm)	< 1	IND	IND	IND	IND
Se, Selenium (ppm)	71	65	76	67	75
Sn, Tin (ppm)	0.48	0.44	0.52	IND	IND
Sr, Strontium (ppm)	4.96	4.15	5.76	4.60	5.31
Tb, Terbium (ppm)	< 0.05	IND	IND	IND	IND
Te, Tellurium (ppm)	1.63	1.57	1.69	1.47	1.79
Ti, Titanium (wt.%)	0.003	0.002	0.003	IND	IND
Tl, Thallium (ppm)	0.60	0.58	0.62	0.57	0.63
Tm, Thulium (ppm)	< 0.1	IND	IND	IND	IND
U, Uranium (ppm)	0.26	0.23	0.28	IND	IND
V, Vanadium (ppm)	< 1	IND	IND	IND	IND
W, Tungsten (ppm)	1.12	0.92	1.33	IND	IND
Y, Yttrium (ppm)	0.26	0.24	0.29	IND	IND
Yb, Ytterbium (ppm)	< 0.1	IND	IND	IND	IND
Zn, Zinc (wt.%)	0.862	0.842	0.882	0.850	0.875
Zr, Zirconium (ppm)	1.33	1.28	1.38	IND	IND
Infrared Combustion					
S, Sulphur (wt.%)	37.79	37.37	38.21	37.33	38.26

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 ( $\rho$ =0.95) will have concentrations lying between 45.91 and 46.05 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.

Table 5 below shows the INAA data determined on 20 x 85mg subsamples of OREAS 992b. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 15g fire assay determinations were undertaken without the normal measurement error associated with this methodology.

Replicate	Au	Au
No	85mg actual	15g equivalent*
1	14.49	14.54
2	14.72	14.55
3	14.62	14.55
4	14.32	14.52
5	14.77	14.56
6	14.53	14.54
7	14.04	14.50
8	14.48	14.54
9	14.27	14.52
10	14.65	14.55
11	14.44	14.53
12	14.39	14.53
13	14.82	14.56
14	14.58	14.54
15	14.80	14.56
16	14.38	14.53
17	14.37	14.53
18	14.43	14.53
19	14.79	14.56
20	14.91	14.57
Mean	14.54	14.54
Median	14.51	14.54
Std Dev.	0.22	0.02
Rel.Std.Dev.	1.52%	0.115%

# Table 5. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples and showing the equivalent results scaled to a 15g sample mass typical of fire assay determination.

\*Results calculated for a 15g equivalent sample mass using the formula:  $x^{15g Eq} = \frac{(x^{INAA} - \bar{x}) \times RSD@15g}{RSD@85mg} + \bar{X}$ 

where  $x^{15g Eq}$  = equivalent result calculated for a 15g sample mass  $(x^{INAA})$  = raw INAA result at 85mg

 $\bar{X}$  = mean of 85mg INAA results

The homogeneity of gold has been determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.115% calculated for a 15g fire assay sample (1.52% at 85mg weights) confirms the high level of gold homogeneity in OREAS 992b.



Homogeneity has also been evaluated using a nested ANOVA within the data provided by the geoanalytical laboratories for elements 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 entire prepared batch of OREAS 992b 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.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 992b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

# 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. \*ANSTO, Lucas Heights, NSW, Australia
- 8. \*ALS Inspection, Prescot, Merseyside, UK
- 9. \*Bachelet, Angleur, Liege, Belgium
- 10. \*Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 11. \*Bureau Veritas Geoanalytical, Perth, WA, Australia
- 12. \*Customs Central Laboratory of Mongolia, Ulaanbaatar, Sükhbaatar District, Mongolia
- 13. \*Erdenet Central Chemical Laboratory, Erdenet, Orkhon province, Mongolia
- 14. \*Inspectorate (BV), Lima, Peru
- 15. Inspectorate (BV), Shanghai, Bao Shan District, China
- 16. \*Inspectorate (BV), Witham, Essex, UK
- 17. \*Intertek Genalysis, Perth, WA, Australia
- 18. Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
- 19. \*Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 20. \*Labtium Oy, Kuopio, Rovaniemi, Finland
- 21. \*MinAnalytical Services, Perth, WA, Australia
- 22. \*MSALABS, Vancouver, BC, Canada
- 23. \*Nagrom, Perth, WA, Australia
- 24. \*PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
- 25. \*RC Inspection, Rotterdam, Netherlands
- 26. \*RC Inspection, Ulaanbaatar, Khan-Uul District, Mongolia



- 27. \*Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
- 28. \*SGS Australia Mineral Services, Perth, WA, Australia
- 29. \*SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
- 30. \*SGS Nederland B.V., Spijkenisse, Zuid-Holland, Netherlands
- 31. \*SRL, Perth, WA, Australia

♦ = Umpire laboratory (classical methods); \* = Geoanalytical laboratory (instrumental methods).

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

## PREPARER AND SUPPLIER

Certified reference material OREAS 992b 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 992b 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 992b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 992b 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 992b is a sulphide-rich reference material (S = 37.8%) and is reactive under normal atmospheric conditions. To inhibit oxidation and prolong its shelf life it has been sealed under nitrogen in single-use, 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 laboratory' Cu, Ag, Au, Pd and Pt certified values for OREAS 992b 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 Ag, Au, Pd and Pt as per ISO 9599. The results were then corrected to dry basis using the moisture value. Moisture content varied amongst the laboratories between 0.14 and 7.2 wt.% but with an average of 3.13 wt.%.

The 'geoanalytical lab' certified values for OREAS 992b 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.

# **DOCUMENT HISTORY**

Revision No.	Date	Changes applied	
1	9 <sup>th</sup> Sep, 2020	Statistics amended for 'Au', 'Pd' and 'Pt' by fire assay.	
0	3 <sup>rd</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**



9<sup>th</sup> September, 2020

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



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