

## **CERTIFICATE OF ANALYSIS FOR**

# Zinc Tailings (MMG Rosebery Mine, Tasmania, Australia) CERTIFIED REFERENCE MATERIAL OREAS 630b

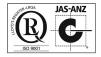
Table 1. Certified Values and Performance Gates for OREAS 630b.

0 17	Certified	P	Absolute	Standard	Deviation	าร	Relative	Standard D	eviations	5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Pb Fire Assay											
Au, ppm	0.358	0.013	0.332	0.385	0.319	0.398	3.65%	7.30%	10.95%	0.341	0.376
Oxidising Fusion	XRF										
Al <sub>2</sub> O <sub>3</sub> , wt.%	9.79	0.231	9.32	10.25	9.09	10.48	2.36%	4.72%	7.08%	9.30	10.28
BaO, wt.%	7.57	0.114	7.34	7.80	7.23	7.91	1.51%	3.02%	4.53%	7.19	7.95
CaO, wt.%	2.20	0.038	2.13	2.28	2.09	2.32	1.71%	3.42%	5.13%	2.09	2.31
Cr, ppm	< 70	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cu, ppm	547	45	457	637	412	683	8.24%	16.48%	24.72%	520	575
Fe, wt.%	6.24	0.132	5.98	6.50	5.85	6.64	2.11%	4.22%	6.33%	5.93	6.55
K <sub>2</sub> O, wt.%	2.23	0.031	2.17	2.29	2.14	2.32	1.39%	2.79%	4.18%	2.12	2.34
MgO, wt.%	1.44	0.027	1.39	1.49	1.36	1.52	1.86%	3.71%	5.57%	1.37	1.51
Mn, wt.%	3.49	0.103	3.28	3.70	3.18	3.80	2.94%	5.88%	8.83%	3.32	3.66
Na₂O, wt.%	0.969	0.097	0.775	1.163	0.678	1.260	10.00%	20.01%	30.01%	0.921	1.018
P <sub>2</sub> O <sub>5</sub> , wt.%	0.104	0.007	0.091	0.117	0.084	0.124	6.32%	12.64%	18.96%	0.099	0.109

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.



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Table I Continued.											
Constituent	Certified	P	Absolute S	Standard	Deviation	าร	Relative	Standard D	eviations	5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Oxidising Fusion	XRF contin	ued							•		•
Pb, wt.%	0.442	0.010	0.423	0.462	0.413	0.472	2.19%	4.38%	6.57%	0.420	0.465
S, wt.%	6.92	0.115	6.69	7.15	6.57	7.26	1.66%	3.32%	4.97%	6.57	7.26
SiO <sub>2</sub> , wt.%	46.32	0.373	45.57	47.06	45.20	47.43	0.80%	1.61%	2.41%	44.00	48.63
Sn, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Sr, ppm	735	61	613	858	552	919	8.32%	16.64%	24.96%	699	772
TiO <sub>2</sub> , wt.%	0.404	0.020	0.364	0.444	0.344	0.464	4.98%	9.95%	14.93%	0.384	0.424
Zn, wt.%	1.11	0.025	1.06	1.16	1.03	1.18	2.24%	4.48%	6.73%	1.05	1.16
Zr, ppm	192	46	101	283	55	329	23.72%	47.44%	71.16%	183	202
Borate / Peroxide	Fusion ICP								•		•
Al, wt.%	5.11	0.120	4.87	5.35	4.75	5.47	2.36%	4.71%	7.07%	4.86	5.37
As, ppm	410	21	368	452	348	473	5.08%	10.17%	15.25%	390	431
Be, ppm	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	3.93	0.196	3.54	4.32	3.35	4.52	4.97%	9.94%	14.91%	3.74	4.13
Ca, wt.%	1.59	0.077	1.44	1.74	1.36	1.82	4.84%	9.68%	14.52%	1.51	1.67
Cd, ppm	28.8	1.80	25.2	32.4	23.4	34.2	6.25%	12.51%	18.76%	27.4	30.2
Cu, ppm	518	19	480	556	461	575	3.66%	7.32%	10.98%	492	544
Fe, wt.%	6.13	0.141	5.85	6.41	5.71	6.55	2.30%	4.59%	6.89%	5.82	6.44
In, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	1.82	0.081	1.66	1.98	1.58	2.07	4.43%	8.87%	13.30%	1.73	1.91
La, ppm	36.9	3.9	29.0	44.7	25.1	48.6	10.67%	21.35%	32.02%	35.0	38.7
Li, ppm	21.9	2.6	16.6	27.2	13.9	29.8	12.08%	24.15%	36.23%	20.8	23.0
Mg, wt.%	0.837	0.020	0.796	0.877	0.775	0.898	2.44%	4.87%	7.31%	0.795	0.878
Mn, wt.%	3.46	0.120	3.22	3.70	3.10	3.82	3.47%	6.94%	10.41%	3.28	3.63
Mo, ppm	14.5	2.2	10.1	18.9	7.9	21.0	15.12%	30.24%	45.37%	13.8	15.2
Nb, ppm	8.60	1.83	4.94	12.26	3.11	14.09	21.29%	42.58%	63.86%	8.17	9.03
Ni, ppm	46.9	9.1	28.6	65.1	19.5	74.3	19.48%	38.95%	58.43%	44.5	49.2
P, wt.%	0.052	0.008	0.037	0.068	0.029	0.076	14.79%	29.57%	44.36%	0.050	0.055
Pb, wt.%	0.432	0.012	0.408	0.457	0.395	0.469	2.85%	5.71%	8.56%	0.411	0.454
Rb, ppm	106	2	101	110	99	112	2.10%	4.19%	6.29%	100	111
S, wt.%	6.16	0.63	4.90	7.42	4.27	8.04	10.21%	20.43%	30.64%	5.85	6.47
Sb, ppm	50	3.6	43	57	39	61	7.19%	14.38%	21.57%	48	53
Si, wt.%	21.62	0.520	20.58	22.66	20.06	23.18	2.40%	4.81%	7.21%	20.54	22.70
Sr, ppm	706	31	644	769	612	801	4.45%	8.89%	13.34%	671	742
Th, ppm	10.8	1.1	8.6	13.0	7.6	14.0	10.00%	20.00%	30.00%	10.3	11.3
Ti, wt.%	0.237	0.008	0.221	0.253	0.213	0.261	3.41%	6.81%	10.22%	0.225	0.249
TI, ppm	24.2	1.33	21.6	26.9	20.2	28.2	5.48%	10.96%	16.45%	23.0	25.4
U, ppm	5.97	0.402	5.17	6.78	4.76	7.18	6.74%	13.47%	20.21%	5.67	6.27
V, ppm	< 50	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
W, ppm	25.4	6.5	12.4	38.3	5.9	44.8	25.52%	51.04%	76.57%	24.1	26.6
Y, ppm	24.7	1.40	21.9	27.5	20.5	28.9	5.67%	11.34%	17.01%	23.5	25.9
Yb, ppm	2.85	0.42	2.00	3.69	1.58	4.11	14.81%	29.62%	44.43%	2.70	2.99
Zn, wt.%	1.11	0.020	1.07	1.15	1.05	1.17	1.77%	3.53%	5.30%	1.06	1.17
SI unit equivalent						·	L				1

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

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Constituent	Certified	<i>F</i>	1	1	Deviation		Relative	Standard D	eviations	5% W	IIIQOW
	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion					1					1	
Ag, ppm	19.0	0.53	18.0	20.1	17.5	20.6	2.76%	5.52%	8.28%	18.1	20.0
Al, wt.%	5.08	0.182	4.72	5.44	4.53	5.62	3.58%	7.15%	10.73%	4.82	5.33
As, ppm	413	18	376	449	358	467	4.41%	8.82%	13.23%	392	433
Be, ppm	1.28	0.098	1.08	1.48	0.99	1.57	7.67%	15.35%	23.02%	1.22	1.34
Bi, ppm	4.14	0.176	3.79	4.49	3.61	4.67	4.25%	8.49%	12.74%	3.93	4.35
Ca, wt.%	1.56	0.024	1.51	1.61	1.49	1.63	1.56%	3.11%	4.67%	1.48	1.64
Cd, ppm	27.0	1.21	24.6	29.4	23.3	30.6	4.50%	8.99%	13.49%	25.6	28.3
Ce, ppm	48.5	15.0	18.5	78.4	3.5	93.4	30.90%	61.81%	92.71%	46.0	50.9
Co, ppm	7.42	0.590	6.24	8.60	5.65	9.18	7.95%	15.90%	23.85%	7.05	7.79
Cr, ppm	11.5	2.4	6.7	16.3	4.3	18.7	20.88%	41.77%	62.65%	10.9	12.0
Cs, ppm	5.03	0.115	4.80	5.26	4.68	5.38	2.29%	4.59%	6.88%	4.78	5.28
Cu, ppm	521	11	498	544	487	556	2.20%	4.40%	6.60%	495	547
Dy, ppm	3.92	0.202	3.51	4.32	3.31	4.52	5.16%	10.31%	15.47%	3.72	4.11
Er, ppm	2.22	0.154	1.91	2.53	1.76	2.68	6.93%	13.87%	20.80%	2.11	2.33
Eu, ppm	2.31	0.28	1.74	2.87	1.46	3.15	12.20%	24.40%	36.61%	2.19	2.42
Fe, wt.%	6.12	0.226	5.67	6.58	5.45	6.80	3.69%	7.38%	11.07%	5.82	6.43
Ga, ppm	15.9	2.7	10.5	21.4	7.8	24.1	17.01%	34.01%	51.02%	15.1	16.7
Gd, ppm	5.36	0.447	4.46	6.25	4.02	6.70	8.34%	16.69%	25.03%	5.09	5.63
Hf, ppm	3.20	0.131	2.94	3.46	2.81	3.59	4.08%	8.17%	12.25%	3.04	3.36
Ho, ppm	0.76	0.037	0.69	0.83	0.65	0.87	4.83%	9.66%	14.49%	0.72	0.80
In, ppm	0.35	0.019	0.32	0.39	0.30	0.41	5.40%	10.79%	16.19%	0.34	0.37
K, wt.%	1.79	0.053	1.68	1.89	1.63	1.94	2.96%	5.93%	8.89%	1.70	1.87
La, ppm	17.1	7.0	3.0	31.1	0.0	38.1	41.08%	82.16%	123.25	16.2	17.9
Li, ppm	20.2	0.70	18.8	21.6	18.2	22.3	3.44%	6.88%	10.32%	19.2	21.3
Lu, ppm	0.32	0.021	0.28	0.36	0.26	0.38	6.47%	12.95%	19.42%	0.30	0.34
Mg, wt.%	0.837	0.019	0.799	0.876	0.779	0.896	2.31%	4.62%	6.93%	0.796	0.879
Mn, wt.%	3.39	0.122	3.15	3.64	3.03	3.76	3.60%	7.21%	10.81%	3.22	3.56
Mo, ppm	12.7	0.66	11.4	14.0	10.7	14.7	5.21%	10.42%	15.63%	12.1	13.4
Na, wt.%	0.721	0.029	0.663	0.780	0.634	0.809	4.05%	8.10%	12.15%	0.685	0.757
Nb, ppm	8.10	0.800	6.50	9.70	5.70	10.50	9.88%	19.75%	29.63%	7.70	8.51
Nd, ppm	30.1	3.3	23.4	36.7	20.1	40.0	11.04%	22.08%	33.12%	28.6	31.6
Ni, ppm	35.1	1.17	32.7	37.4	31.6	38.6	3.34%	6.69%	10.03%	33.3	36.8
P, wt.%	0.045	0.003	0.040	0.051	0.037	0.054	6.05%	12.09%	18.14%	0.043	0.048
Pb, wt.%	0.411	0.018	0.375	0.448	0.357	0.466	4.42%	8.84%	13.25%	0.391	0.432
Pr, ppm	7.27	0.94	5.40	9.15	4.46	10.08	12.89%	25.79%	38.68%	6.91	7.63
Rb, ppm	102	5	92	112	86	117	4.98%	9.95%	14.93%	97	107
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	5.17	0.146	4.87	5.46	4.73	5.61	2.83%	5.66%	8.50%	4.91	5.43
Sb, ppm	47.9	3.84	40.3	55.6	36.4	59.5	8.01%	16.02%	24.03%	45.6	50.3
Sc, ppm	6.29	0.330	5.63	6.96	5.30	7.29	5.25%	10.50%	15.75%	5.98	6.61
Sm, ppm	6.20	0.76	4.68	7.71	3.93	8.47	12.21%	24.42%	36.63%	5.89	6.51
Sn, ppm	2.22	0.077	2.07	2.37	1.99	2.45	3.48%	6.95%	10.43%	2.11	2.33
Sr, ppm	385	110	164	605	54	715	28.64%	57.27%	85.91%	366	404
SI unit equivalent	· , ,		· \ _	" -		004 40	. –				

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Constituent	Certified	<i>F</i>	Absolute S	Standard	Deviation	าร	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 Digestion	continued										
Ta, ppm	0.58	0.054	0.47	0.68	0.42	0.74	9.31%	18.62%	27.93%	0.55	0.61
Tb, ppm	0.71	0.040	0.63	0.79	0.59	0.83	5.64%	11.27%	16.91%	0.68	0.75
Te, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	7.49	1.70	4.09	10.89	2.39	12.59	22.69%	45.38%	68.07%	7.12	7.87
Ti, wt.%	0.200	0.017	0.166	0.235	0.148	0.252	8.61%	17.23%	25.84%	0.190	0.210
TI, ppm	25.1	1.46	22.2	28.0	20.7	29.5	5.83%	11.66%	17.49%	23.8	26.4
Tm, ppm	0.32	0.018	0.28	0.36	0.27	0.38	5.67%	11.35%	17.02%	0.31	0.34
U, ppm	5.87	0.306	5.26	6.49	4.95	6.79	5.22%	10.43%	15.65%	5.58	6.17
V, ppm	27.3	2.58	22.2	32.5	19.6	35.1	9.44%	18.87%	28.31%	26.0	28.7
W, ppm	19.7	1.54	16.6	22.8	15.0	24.3	7.84%	15.67%	23.51%	18.7	20.7
Y, ppm	18.4	0.93	16.6	20.3	15.6	21.2	5.03%	10.06%	15.09%	17.5	19.3
Yb, ppm	2.13	0.189	1.75	2.51	1.56	2.70	8.87%	17.73%	26.60%	2.02	2.24
Zn, wt.%	1.11	0.025	1.06	1.16	1.04	1.19	2.21%	4.42%	6.63%	1.06	1.17
Zr, ppm	113	9	95	130	87	139	7.69%	15.38%	23.07%	107	118
Aqua Regia Diges	stion										
Ag, ppm	19.1	0.72	17.6	20.5	16.9	21.2	3.75%	7.51%	11.26%	18.1	20.0
Al, wt.%	1.09	0.077	0.93	1.24	0.85	1.32	7.13%	14.27%	21.40%	1.03	1.14
As, ppm	413	17	380	447	363	464	4.08%	8.15%	12.23%	393	434
Au, ppm	0.345	0.011	0.323	0.366	0.313	0.376	3.05%	6.10%	9.16%	0.327	0.362
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.27	0.04	0.20	0.34	0.16	0.37	12.98%	25.97%	38.95%	0.26	0.28
Bi, ppm	4.13	0.237	3.66	4.61	3.42	4.84	5.73%	11.46%	17.19%	3.93	4.34
Ca, wt.%	1.46	0.054	1.35	1.57	1.30	1.62	3.73%	7.46%	11.19%	1.39	1.53
Cd, ppm	27.1	1.81	23.5	30.8	21.7	32.6	6.68%	13.37%	20.05%	25.8	28.5
Ce, ppm	33.4	12.0	9.3	57.4	0.0	69.4	35.98%	71.97%	107.95	31.7	35.0
Co, ppm	7.20	0.377	6.45	7.95	6.07	8.33	5.24%	10.48%	15.71%	6.84	7.56
Cr, ppm	9.77	0.850	8.07	11.47	7.22	12.32	8.70%	17.39%	26.09%	9.28	10.26
Cs, ppm	2.86	0.238	2.38	3.33	2.14	3.57	8.32%	16.65%	24.97%	2.72	3.00
Cu, ppm	523	20	484	563	464	583	3.79%	7.59%	11.38%	497	549
Fe, wt.%	5.79	0.192	5.41	6.18	5.21	6.37	3.32%	6.65%	9.97%	5.50	6.08
Ga, ppm	3.98	0.369	3.24	4.72	2.88	5.09	9.27%	18.54%	27.81%	3.78	4.18
Hf, ppm	0.62	0.10	0.43	0.81	0.33	0.90	15.44%	30.89%	46.33%	0.59	0.65
Hg, ppm	1.21	0.067	1.07	1.34	1.00	1.41	5.59%	11.18%	16.77%	1.15	1.27
In, ppm	0.33	0.018	0.29	0.36	0.27	0.38	5.47%	10.95%	16.42%	0.31	0.34
K, wt.%	0.297	0.031	0.235	0.359	0.204	0.390	10.47%	20.94%	31.41%	0.282	0.312
La, ppm	10.3	4.5	1.2	19.4	0.0	23.9	43.95%	87.90%	131.85	9.8	10.8
Li, ppm	7.71	0.82	6.07	9.36	5.25	10.18	10.65%	21.30%	31.95%	7.33	8.10
Mg, wt.%	0.609	0.026	0.557	0.661	0.531	0.687	4.26%	8.52%	12.78%	0.579	0.640
Mn, wt.%	3.18	0.143	2.90	3.47	2.75	3.61	4.49%	8.99%	13.48%	3.02	3.34
Mo, ppm	12.4	0.47	11.4	13.3	11.0	13.8	3.77%	7.55%	11.32%	11.8	13.0
Na, wt.%	0.048	0.007	0.034	0.062	0.027	0.069	14.48%	28.95%	43.43%	0.045	0.050
Nb, ppm	1.08	0.19	0.70	1.46	0.52	1.65	17.42%	34.83%	52.25%	1.03	1.13
Ni, ppm	34.8	1.53	31.7	37.9	30.2	39.4	4.40%	8.81%	13.21%	33.1	36.5
SI unit equivalent	, ,		\ _	" -	/ - 0 0	004 40	/ - 4000				

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Constituent	Certified	P	Absolute S	Standard	Deviation	ns	Relative	Standard D	eviations	5% w	indow
Constituent	Value	1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Aqua Regia Diges	tion contin	ued									
P, wt.%	0.044	0.002	0.039	0.048	0.037	0.051	5.41%	10.82%	16.23%	0.041	0.046
Pb, wt.%	0.408	0.021	0.365	0.451	0.344	0.472	5.23%	10.45%	15.68%	0.388	0.429
Rb, ppm	21.9	1.89	18.2	25.7	16.3	27.6	8.59%	17.18%	25.77%	20.9	23.0
S, wt.%	5.19	0.168	4.86	5.53	4.69	5.70	3.23%	6.46%	9.70%	4.93	5.45
Sb, ppm	40.7	4.6	31.5	49.9	26.9	54.5	11.30%	22.60%	33.90%	38.7	42.7
Sc, ppm	1.68	0.129	1.42	1.94	1.29	2.07	7.67%	15.33%	23.00%	1.60	1.77
Sn, ppm	0.65	0.09	0.47	0.83	0.39	0.91	13.55%	27.11%	40.66%	0.62	0.68
Tb, ppm	0.43	0.07	0.29	0.56	0.22	0.63	15.81%	31.63%	47.44%	0.40	0.45
Th, ppm	3.23	0.82	1.60	4.86	0.78	5.68	25.24%	50.48%	75.72%	3.07	3.39
Ti, wt.%	0.069	0.012	0.044	0.093	0.032	0.105	17.81%	35.62%	53.42%	0.065	0.072
TI, ppm	8.47	0.711	7.05	9.89	6.34	10.60	8.39%	16.79%	25.18%	8.05	8.89
U, ppm	3.34	0.279	2.78	3.90	2.51	4.18	8.35%	16.69%	25.04%	3.18	3.51
V, ppm	6.41	0.89	4.63	8.20	3.74	9.09	13.92%	27.83%	41.75%	6.09	6.73
W, ppm	12.3	0.69	10.9	13.7	10.2	14.4	5.58%	11.16%	16.74%	11.7	12.9
Y, ppm	10.1	0.60	8.9	11.4	8.3	12.0	5.94%	11.89%	17.83%	9.6	10.7
Yb, ppm	0.76	0.10	0.57	0.95	0.47	1.05	12.79%	25.58%	38.37%	0.72	0.80
Zn, wt.%	1.10	0.033	1.03	1.16	1.00	1.20	2.99%	5.98%	8.96%	1.04	1.15
Zr, ppm	23.1	2.5	18.1	28.1	15.6	30.6	10.89%	21.78%	32.68%	21.9	24.3
Thermogravimetry	у										
LOI <sup>1000</sup> , wt.%	11.46	0.746	9.97	12.96	9.22	13.70	6.51%	13.02%	19.54%	10.89	12.04
Infrared Combust	ion										
S, wt.%	6.87	0.200	6.47	7.27	6.27	7.47	2.91%	5.83%	8.74%	6.52	7.21

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.

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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 'Intended Use' should be read carefully.

## SOURCE MATERIAL

OREAS 630b is a zinc tailings certified reference material (CRM) sourced from the Rosebery metallurgical plant owned and operated by MMG Ltd. The Rosebery mine and plant are located in the north-west region of Tasmania, Australia approximately 300 kilometres north-west of Hobart and 125 kilometres south of Burnie. The key minerals of economic importance include sphalerite, galena, pyrite, chalcopyrite and electrum.

# **PERFORMANCE GATES**

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 www.westgard.com/mltirule.htm). A second method utilises a 5% window calculated directly from the certified value.

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

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

# COMMINUTION AND HOMOGENISATION PROCEDURES

The material (1900kg) constituting OREAS 630b was prepared in the following manner:

- Drying to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging in 10g and 60g units sealed under nitrogen in laminated foil pouches.

## PHYSICAL PROPERTIES

OREAS 630b was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 2 presents these findings that should be used for informational purposes only.

Table 2. Physical properties of OREAS 630b.

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
626	0.70	N5	Medium Gray

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

## ANALYTICAL PROGRAM

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

- Au by 30-50g fire assay with ICP-OES finish (10 laboratories) and AAS finish (9 laboratories);
- Majors & base metals by oxidising fusion with X-ray fluorescence finish (11 laboratories);
- Full elemental suite by peroxide fusion with ICP-OES and ICP-MS finish (up to 17 laboratories depending on the element);
- Four acid digestion for full elemental suites with ICP-OES and ICP-MS (up to 18 laboratories depending on the element);
- Aqua regia digestion for full elemental suites with AAS and/or ICP-OES and ICP-MS (up to 18 laboratories depending on the element);
- Total Sulphur by infra-red combustion furnace or C/S analyser.

To confirm homogeneity, gold was also undertaken by instrumental neutron activation analysis (INAA) on 20 x 85mg subsamples by the Australian Nuclear Science and Technology Organisation (ANSTO) located in Lucas Heights, NSW, Australia (see Table 5 in the 'Homogeneity Evaluation' section below).

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. Aqua regia is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions which 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.

For the round robin program ten 1.7kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 120g pulp samples were submitted to each laboratory for analysis. The samples received by each laboratory were obtained by taking two samples from each of three separate 1.7kg test units. This format enabled a nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance (see 'Homogeneity Evaluation' section below).

Table 1 presents the 158 certified values together with performance gate intervals based on their pooled 1SD's. Table 2 provides indicative physical properties and Table 3 shows 71 indicative values (non-certified elements). Table 4 provides 95% confidence and tolerance limits and their associated 1SD's and Table 5 shows the gold instrumental neutron activation analysis (INAA) results.

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 630b DataPack-1.0.191119\_104832.xlsx).

Results are also presented in scatter plots for Zn and lead by 4-acid digestion and Au by fire assay (Figures 1 to 3, respectively) together with ±3SD (magenta), ±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.

Table 3. Indicative Values for OREAS 630b.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assa	ay							
Pd	ppb	< 5	Pt	ppb	3.75			
Oxidising Fu	ısion XR	F						
As	ppm	436	HfO <sub>2</sub>	ppm	< 100	Rb	ppm	105
Bi	ppm	86	La <sub>2</sub> O <sub>3</sub>	ppm	81	Sb	ppm	35.2
CeO <sub>2</sub>	ppm	< 100	Мо	ppm	39.9	V	ppm	120
CI	ppm	38.3	Nb	ppm	< 50	W	ppm	10.3
Со	ppm	< 100	Ni	ppm	22.8	Y <sub>2</sub> O <sub>3</sub>	ppm	61
Borate / Per	oxide Fu	sion ICP						
Ag	ppm	18.5	Ga	ppm	15.9	Sc	ppm	5.50
В	ppm	< 50	Gd	ppm	5.92	Se	ppm	17.7
Ва	wt.%	6.75	Ge	ppm	2.83	Sm	ppm	11.4
Ce	ppm	77	Hf	ppm	4.20	Sn	ppm	36.9
Со	ppm	< 10	Но	ppm	1.04	Та	ppm	1.17
Cr	ppm	55	Lu	ppm	0.56	Tb	ppm	0.79
Cs	ppm	5.03	Na	wt.%	0.715	Te	ppm	0.69
Dy	ppm	4.88	Nd	ppm	34.1	Tm	ppm	0.45

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.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate / Per	oxide Fu	sion ICP con	tinued					
Er	ppm	2.79	Pr	ppm	8.79	Zr	ppm	161
Eu	ppm	3.32	Re	ppm	< 1			
4-Acid Diges	stion							
Ва	wt.%	0.091	Ge	ppm	0.65	Se	ppm	1.20
Aqua Regia	Digestio	n						
Ва	wt.%	0.004	Lu	ppm	0.10	Se	ppm	0.55
Dy	ppm	1.90	Nd	ppm	17.0	Si	wt.%	0.126
Er	ppm	0.89	Pd	ppb	< 10	Sm	ppm	3.95
u	ppm	1.13	Pr	ppm	2.71	Sr	ppm	70
Gd	ppm	3.26	Pt	ppb	< 5	Та	ppm	0.017
Ge	ppm	0.11	Re	ppm	0.001	Te	ppm	< 0.05
Но	ppm	0.30	Ru	ppm	< 0.005	Tm	ppm	0.12
Infrared Con	nbustion	1						
С	wt.%	1.28						

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.

# 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.

**Certified Values** are the means of accepted laboratory means after outlier filtering. The INAA data (see Table 5) are omitted from determination of the certified value for Au and are used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 630b.

**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.** 

Table 4. 95% Confidence & Tolerance Limits for OREAS 630b.

Comptituent	Certified	95% Confid	ence Limits	95% Tolera	ance Limits
Constituent	Value	Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppm)	0.358	0.353	0.364	0.357*	0.360*
Oxidising Fusion XRF					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	9.79	9.63	9.94	9.70	9.87
BaO, Barium oxide (wt.%)	7.57	7.49	7.65	7.51	7.63
CaO, Calcium oxide (wt.%)	2.20	2.18	2.23	2.18	2.22
Cr, Chromium (ppm)	< 70	IND	IND	IND	IND
Cu, Copper (ppm)	547	510	585	524	570
Fe, Iron (wt.%)	6.24	6.15	6.34	6.20	6.28
K <sub>2</sub> O, Potassium oxide (wt.%)	2.23	2.21	2.25	2.21	2.25
MgO, Magnesium oxide (wt.%)	1.44	1.42	1.46	1.42	1.46
Mn, Manganese (wt.%)	3.49	3.42	3.56	3.46	3.52
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.969	0.839	1.100	0.948	0.990
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.104	0.100	0.108	0.099	0.109
Pb, Lead (wt.%)	0.442	0.435	0.450	0.436	0.449
S, Sulphur (wt.%)	6.92	6.83	7.00	6.84	6.99
SiO <sub>2</sub> , Silicon dioxide (wt.%)	46.32	46.02	46.61	46.11	46.52
Sn, Tin (ppm)	< 50	IND	IND	IND	IND
Sr, Strontium (ppm)	735	684	787	IND	IND

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: intervals may appear asymmetric due to rounding.



<sup>\*</sup>Gold Tolerance Limits for typical 30g fire assay are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Table 4 continued.

	Certified	95% Confid	lence Limits	95% Tolera	ance Limits
Constituent	Value	Low	High	Low	High
Oxidising Fusion XRF continued			19		
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.404	0.390	0.418	0.392	0.416
Zn, Zinc (wt.%)	1.11	1.09	1.13	1.10	1.12
Zr, Zirconium (ppm)	192	152	232	IND	IND
Borate / Peroxide Fusion ICP					
Al, Aluminium (wt.%)	5.11	5.05	5.18	5.03	5.20
As, Arsenic (ppm)	410	395	426	389	432
Be, Beryllium (ppm)	< 5	IND	IND	IND	IND
Bi, Bismuth (ppm)	3.93	3.71	4.15	IND	IND
Ca, Calcium (wt.%)	1.59	1.55	1.63	1.54	1.64
Cd, Cadmium (ppm)	28.8	26.9	30.7	27.3	30.3
Cu, Copper (ppm)	518	508	527	506	529
Fe, Iron (wt.%)	6.13	6.07	6.20	6.04	6.23
In, Indium (ppm)	< 0.5	IND	IND	IND	IND
K, Potassium (wt.%)	1.82	1.78	1.86	1.78	1.86
La, Lanthanum (ppm)	36.9	31.8	41.9	35.5	38.2
Li, Lithium (ppm)	21.9	20.9	22.9	18.9	24.9
Mg, Magnesium (wt.%)	0.837	0.826	0.847	0.820	0.853
Mn, Manganese (wt.%)	3.46	3.40	3.52	3.40	3.51
Mo, Molybdenum (ppm)	14.5	12.4	16.5	IND	IND
Nb, Niobium (ppm)	8.60	6.46	10.74	IND	IND
Ni, Nickel (ppm)	46.9	41.7	52.1	IND	IND
P, Phosphorus (wt.%)	0.052	0.044	0.061	IND	IND
Pb, Lead (wt.%)	0.432	0.427	0.438	0.424	0.441
Rb, Rubidium (ppm)	106	104	107	102	109
S, Sulphur (wt.%)	6.16	5.78	6.54	5.99	6.33
Sb, Antimony (ppm)	50	47	54	46	54
Si, Silicon (wt.%)	21.62	21.35	21.89	21.25	21.99
Sr, Strontium (ppm)	706	684	729	692	721
Th, Thorium (ppm)	10.8	9.7	11.9	10.4	11.2
Ti, Titanium (wt.%)	0.237	0.233	0.240	0.233	0.241
TI, Thallium (ppm)	24.2	22.5	26.0	IND	IND
U, Uranium (ppm)	5.97	5.47	6.47	IND	IND
V, Vanadium (ppm)	< 50	IND	IND	IND	IND
W, Tungsten (ppm)	25.4	19.2	31.5	IND	IND
Y, Yttrium (ppm)	24.7	23.6	25.8	23.0	26.3
Yb, Ytterbium (ppm)	2.85	2.33	3.37	IND	IND
Zn, Zinc (wt.%)	1.11	1.11	1.12	1.09	1.14

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: intervals may appear asymmetric due to rounding.

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Table 4 continued.

Table 4 continued.  Certified 95% Confidence Limits 95% Tolerance Limits											
Constituent	Certified		1		ance Limits						
	Value	Low	High	Low	High						
4-Acid Digestion		T			T						
Ag, Silver (ppm)	19.0	18.8	19.3	18.5	19.6						
Al, Aluminium (wt.%)	5.08	4.99	5.16	4.98	5.17						
As, Arsenic (ppm)	413	403	422	403	422						
Be, Beryllium (ppm)	1.28	1.25	1.31	1.22	1.34						
Bi, Bismuth (ppm)	4.14	4.03	4.25	3.98	4.30						
Ca, Calcium (wt.%)	1.56	1.55	1.57	1.54	1.59						
Cd, Cadmium (ppm)	27.0	26.4	27.6	26.3	27.6						
Ce, Cerium (ppm)	48.5	39.6	57.4	46.1	50.8						
Co, Cobalt (ppm)	7.42	7.06	7.77	7.08	7.76						
Cr, Chromium (ppm)	11.5	10.2	12.8	10.8	12.2						
Cs, Caesium (ppm)	5.03	4.99	5.07	4.86	5.20						
Cu, Copper (ppm)	521	516	526	513	529						
Dy, Dysprosium (ppm)	3.92	3.77	4.06	3.74	4.09						
Er, Erbium (ppm)	2.22	2.09	2.35	2.12	2.32						
Eu, Europium (ppm)	2.31	2.05	2.57	2.15	2.47						
Fe, Iron (wt.%)	6.12	6.01	6.24	6.02	6.23						
Ga, Gallium (ppm)	15.9	14.5	17.4	15.4	16.4						
Gd, Gadolinium (ppm)	5.36	4.99	5.72	5.21	5.51						
Hf, Hafnium (ppm)	3.20	3.14	3.26	3.11	3.30						
Ho, Holmium (ppm)	0.76	0.73	0.79	0.73	0.79						
In, Indium (ppm)	0.35	0.35	0.36	0.34	0.37						
K, Potassium (wt.%)	1.79	1.76	1.81	1.75	1.82						
La, Lanthanum (ppm)	17.1	13.1	21.0	15.7	18.4						
Li, Lithium (ppm)	20.2	19.9	20.6	19.4	21.1						
Lu, Lutetium (ppm)	0.32	0.30	0.34	0.31	0.33						
Mg, Magnesium (wt.%)	0.837	0.827	0.847	0.822	0.853						
Mn, Manganese (wt.%)	3.39	3.33	3.46	3.33	3.45						
Mo, Molybdenum (ppm)	12.7	12.4	13.1	12.2	13.2						
Na, Sodium (wt.%)	0.721	0.706	0.737	0.705	0.738						
Nb, Niobium (ppm)	8.10	7.64	8.57	7.84	8.37						
Nd, Neodymium (ppm)	30.1	27.2	33.0	29.4	30.8						
Ni, Nickel (ppm)	35.1	34.5	35.7	33.9	36.3						
P, Phosphorus (wt.%)	0.045	0.044	0.047	0.044	0.047						
Pb, Lead (wt.%)	0.411	0.402	0.421	0.397	0.425						
Pr, Praseodymium (ppm)	7.27	6.45	8.09	6.87	7.67						
Rb, Rubidium (ppm)	102	99	104	99	104						
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND						
S, Sulphur (wt.%)	5.17	5.08	5.25	5.08	5.25						

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: intervals may appear asymmetric due to rounding.



Table 4 continued.

Table 4 continued.							
Constituent	Certified	95% Confidence Limits		95% Tolerance Limits			
Constituent	Value	Low	High	Low	High		
4-Acid Digestion continued							
Sb, Antimony (ppm)	47.9	45.9	50.0	46.3	49.6		
Sc, Scandium (ppm)	6.29	6.12	6.47	6.01	6.58		
Sm, Samarium (ppm)	6.20	5.60	6.79	5.96	6.44		
Sn, Tin (ppm)	2.22	2.20	2.24	2.11	2.33		
Sr, Strontium (ppm)	385	325	444	367	402		
Ta, Tantalum (ppm)	0.58	0.54	0.61	0.55	0.61		
Tb, Terbium (ppm)	0.71	0.68	0.75	0.69	0.73		
Te, Tellurium (ppm)	< 0.05	IND	IND	IND	IND		
Th, Thorium (ppm)	7.49	6.56	8.42	7.08	7.90		
Ti, Titanium (wt.%)	0.200	0.191	0.209	0.194	0.207		
TI, Thallium (ppm)	25.1	24.4	25.8	24.6	25.6		
Tm, Thulium (ppm)	0.32	0.30	0.34	0.31	0.33		
U, Uranium (ppm)	5.87	5.72	6.02	5.71	6.04		
V, Vanadium (ppm)	27.3	26.0	28.6	26.2	28.4		
W, Tungsten (ppm)	19.7	18.9	20.4	19.0	20.4		
Y, Yttrium (ppm)	18.4	17.9	18.9	17.9	18.9		
Yb, Ytterbium (ppm)	2.13	1.99	2.26	2.06	2.20		
Zn, Zinc (wt.%)	1.11	1.10	1.12	1.10	1.13		
Zr, Zirconium (ppm)	113	108	117	110	116		
Aqua Regia Digestion							
Ag, Silver (ppm)	19.1	18.6	19.5	18.7	19.4		
Al, Aluminium (wt.%)	1.09	1.04	1.13	1.05	1.13		
As, Arsenic (ppm)	413	405	422	405	421		
Au, Gold (ppm)	0.345	0.339	0.350	0.332	0.357		
B, Boron (ppm)	< 10	IND	IND	IND	IND		
Be, Beryllium (ppm)	0.27	0.24	0.30	0.24	0.29		
Bi, Bismuth (ppm)	4.13	3.99	4.27	4.05	4.22		
Ca, Calcium (wt.%)	1.46	1.43	1.49	1.42	1.49		
Cd, Cadmium (ppm)	27.1	26.2	28.1	26.5	27.7		
Ce, Cerium (ppm)	33.4	26.2	40.5	32.2	34.5		
Co, Cobalt (ppm)	7.20	6.99	7.41	6.95	7.45		
Cr, Chromium (ppm)	9.77	9.27	10.27	8.93	10.61		
Cs, Caesium (ppm)	2.86	2.71	3.01	2.78	2.94		
Cu, Copper (ppm)	523	513	533	514	532		
Fe, Iron (wt.%)	5.79	5.70	5.89	5.65	5.93		
Ga, Gallium (ppm)	3.98	3.73	4.24	3.83	4.14		
Hf, Hafnium (ppm)	0.62	0.55	0.69	0.58	0.65		
Hg, Mercury (ppm)	1.21	1.17	1.25	1.16	1.25		

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: intervals may appear asymmetric due to rounding.



Table 4 continued.

O a markit a mark	Certified	95% Confidence Limits		95% Tolerance Limits	
Constituent	Value	Low	High	Low	High
Aqua Regia Digestion continued					
In, Indium (ppm)	0.33	0.32	0.34	0.32	0.34
K, Potassium (wt.%)	0.297	0.280	0.313	0.286	0.308
La, Lanthanum (ppm)	10.3	7.8	12.8	9.8	10.9
Li, Lithium (ppm)	7.71	7.24	8.19	7.34	8.09
Mg, Magnesium (wt.%)	0.609	0.596	0.623	0.593	0.626
Mn, Manganese (wt.%)	3.18	3.09	3.27	3.12	3.25
Mo, Molybdenum (ppm)	12.4	12.1	12.6	12.0	12.8
Na, Sodium (wt.%)	0.048	0.044	0.052	0.045	0.050
Nb, Niobium (ppm)	1.08	0.92	1.25	1.02	1.14
Ni, Nickel (ppm)	34.8	34.1	35.5	33.7	35.8
P, Phosphorus (wt.%)	0.044	0.043	0.045	0.042	0.045
Pb, Lead (wt.%)	0.408	0.397	0.419	0.401	0.415
Rb, Rubidium (ppm)	21.9	20.8	23.1	21.2	22.7
S, Sulphur (wt.%)	5.19	5.10	5.29	5.09	5.30
Sb, Antimony (ppm)	40.7	38.1	43.2	39.2	42.1
Sc, Scandium (ppm)	1.68	1.61	1.76	IND	IND
Sn, Tin (ppm)	0.65	0.59	0.71	0.57	0.73
Tb, Terbium (ppm)	0.43	0.33	0.52	0.40	0.45
Th, Thorium (ppm)	3.23	2.62	3.84	3.06	3.40
Ti, Titanium (wt.%)	0.069	0.062	0.075	0.067	0.071
TI, Thallium (ppm)	8.47	8.04	8.90	8.25	8.69
U, Uranium (ppm)	3.34	3.19	3.49	3.23	3.45
V, Vanadium (ppm)	6.41	5.90	6.92	6.09	6.74
W, Tungsten (ppm)	12.3	11.9	12.7	11.9	12.7
Y, Yttrium (ppm)	10.1	9.8	10.5	9.7	10.6
Yb, Ytterbium (ppm)	0.76	0.65	0.87	IND	IND
Zn, Zinc (wt.%)	1.10	1.08	1.11	1.08	1.12
Zr, Zirconium (ppm)	23.1	21.7	24.5	22.1	24.1
Thermogravimetry					
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	11.46	10.87	12.06	11.29	11.63
Infrared Combustion					
S, Sulphur (wt.%)	6.87	6.76	6.97	6.80	6.93

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: intervals may appear asymmetric due to rounding.

## **Homogeneity Evaluation**

For analytes other than gold the tolerance limits (ISO 16269:2014) shown in Table 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 zinc by oxidising fusion with XRF, where 99% of the time  $(1-\alpha=0.99)$  at least

95% of subsamples ( $\rho$ =0.95) will have concentrations lying between 1.10 and 1.12 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 gold INAA data determined on 20 x 85mg subsamples of OREAS 630b. An equivalent scaled version of the results are also provided to demonstrate the level of repeatability that would be achieved if 30g fire assay determinations were undertaken without the normal measurement error associated with this methodology.

Table 5. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples and showing the equivalent results scaled to a 30g sample mass typical of fire assay determination.

Replicate	Au	Au	
No	85mg actual 30g equivaler		
1	0.355 0.357		
2	0.345 0.356		
3	0.340 0.356		
4	0.359	0.357	
5	0.340	0.356	
6	0.352	0.357	
7	0.365	0.357	
8	0.370	0.358	
9	0.360	0.357	
10	0.364	0.357	
11	0.361	0.357	
12	0.345	0.356	
13	0.354	0.357	
14	0.372	0.358	
15	0.368	0.357	
16	0.361	0.357	
17	0.361	0.357	
18	0.353	0.357	
19	0.353	0.357	
20	0.357	0.357	
Mean	0.357	0.357	
Median	0.358	0.357	
Std Dev.	0.009	0.000	
Rel.Std.Dev.	2.55%	0.136%	

<sup>\*</sup>Results calculated for a 30g equivalent sample mass using the formula:  $x^{30g \ Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@85mg} + \bar{X}$  where  $x^{30g \ Eq} =$  equivalent result calculated for a 30g 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 (i.e. sampling error) and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.136% was calculated for a 30g fire assay sample (2.55% at 85mg weights) and confirms the high level of gold homogeneity in OREAS 630b.

The homogeneity of OREAS 630b has also been evaluated in a **nested ANOVA** of the round robin program. Each of the nineteen round robin laboratories received six samples of the CRM and these samples were made up of paired samples from three different, nonadjacent sampling intervals. 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 (1900kg). The test was performed using the following parameters:

- Gold fire assay 114 samples (19 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis, H<sub>0</sub>: Between-unit variance is no greater than within-unit variance (reject  $H_0$  if p-value < 0.05);
- Alternative Hypothesis, H<sub>1</sub>: Between-unit variance is greater than within-unit variance.

P-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of p-values. For gold fire assay a p-value of 0.99 was calculated. Gold by INAA was also considered for ANOVA (10 paired samples) yielding a p-value of 0.51. Both p-values are insignificant and the Null Hypothesis is retained. Additionally, none of the other certified values showed significant p-values except for Li by 4-acid digestion (pvalue = 0.02). This isolated case is most likely due to random\* statistical probability as there is no other supporting evidence to suspect greater between-unit variance compared with within-unit variance.

Note: p-values are calculated at the 95% probability level. Therefore by definition 5% of p-values calculated will naturally fall as 'significant' (false positives). Additionally, only analytes present in concentrations greater than 20 times the lower limit of detection for the various methods undertaken were considered for the objective of using p-values to evaluate 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 630b 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 630b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

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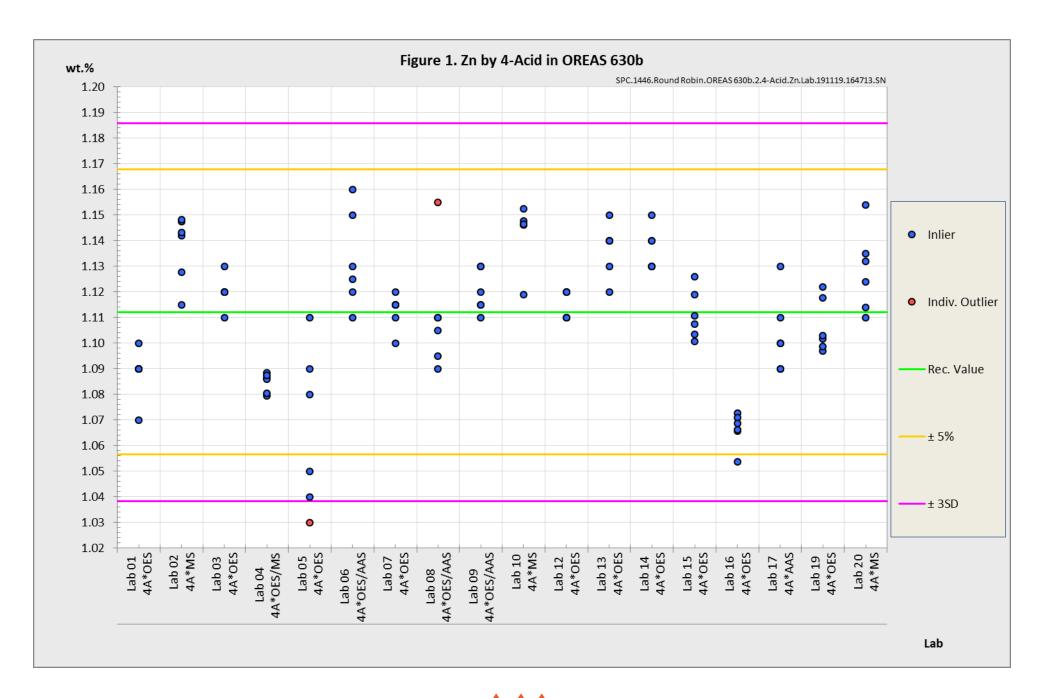


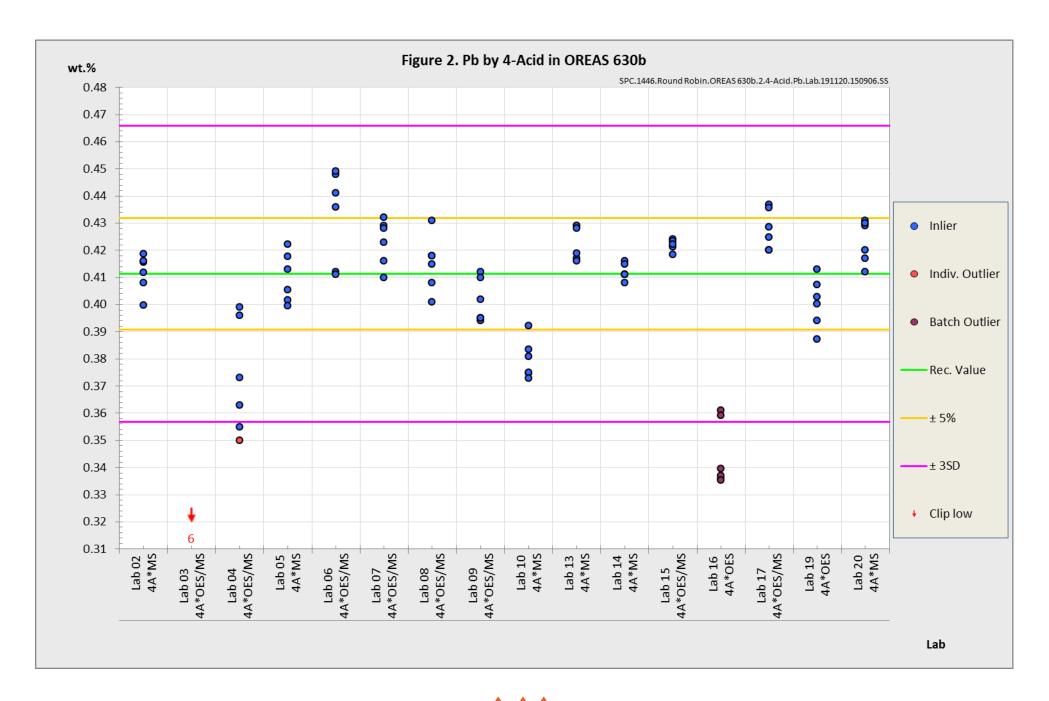
## PARTICIPATING LABORATORIES

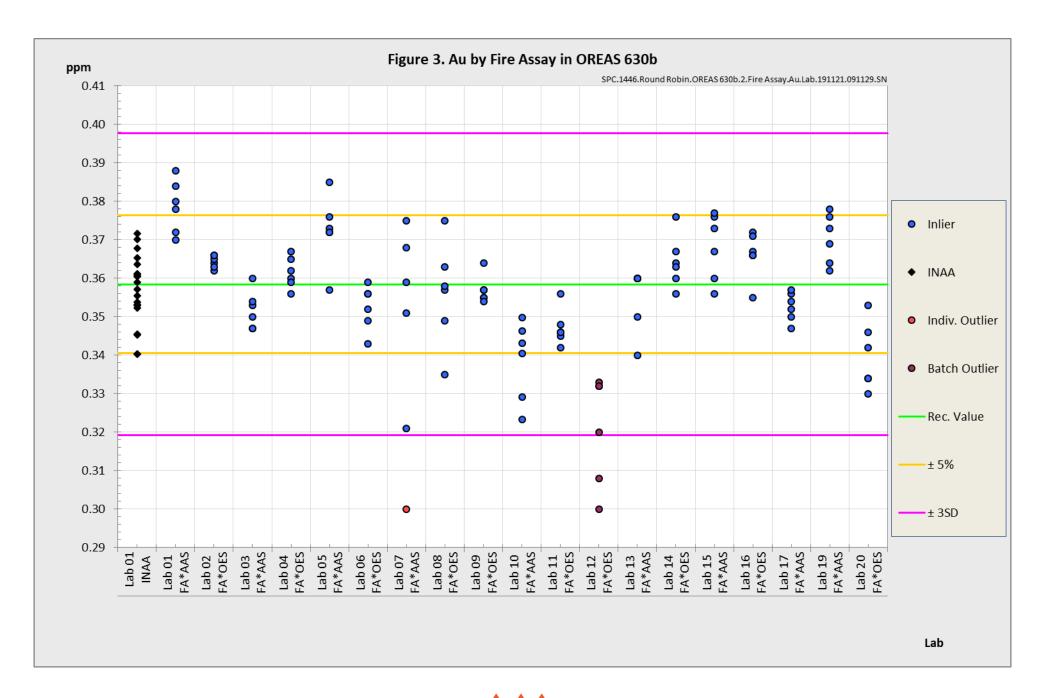
- 1. AGAT Laboratories, Mississauga, Ontario, Canada
- 2. ALS, Brisbane, QLD, Australia
- 3. ALS, Lima, Peru
- 4. ALS, Loughrea, Galway, Ireland
- 5. ALS, Vancouver, BC, Canada
- 6. ANSTO, Lucas Heights, NSW, Australia
- 7. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
- 8. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
- 9. Bureau Veritas Geoanalytical, Perth, WA, Australia
- 10. Intertek Genalysis, Perth, WA, Australia
- 11. Intertek Testing Services, Townsville, QLD, Australia
- 12. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
- 13. Labtium Oy, Kuopio, Rovaniemi, Finland
- 14. Labtium Oy, Sodankyla, Finland
- 15. MSALABS, Vancouver, BC, Canada
- 16. Nagrom, Perth, WA, Australia
- 17. SGS, Randfontein, Gauteng, South Africa
- 18. SGS Canada Inc., Vancouver, BC, Canada
- 19. SGS del Peru, Lima, Peru
- 20. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
- 21. Intertek Tarkwa, Tarkwa, Ghana

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.

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# PREPARER AND SUPPLIER

Certified reference material OREAS 630b was prepared, certified and supplied by:



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## **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 method is possible. In this case, certification takes place on the basis of agreement among independent measurement results (see ISO Guide 35:2006, Clause 10)."

## 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 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 their field samples.

## **INTENDED USE**

OREAS 630b 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 630b may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 630b is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological 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.

## For use with the agua regia digestion method

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. Aqua regia is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions which 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 from 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.

## 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.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 630b has been prepared from primary sulphide bearing ores from the Rosebery deposit. It contains reactive sulphide (6.9% S) and has been packaged under a nitrogen environment (in single-use laminated foil pouches). In its unopened state and under



normal conditions of storage the CRM has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

# INSTRUCTIONS FOR CORRECT USE

The certified values for oxidising fusion with XRF and for LOI at 1000° C are on a dry sample basis while the certified values for all other methods are reported on a 'sample as received' basis.

## 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.

# **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.

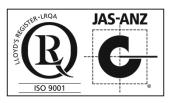
## **DOCUMENT HISTORY**

Revision No.	Date	Changes applied
0	21 <sup>st</sup> November 2019	First publication.

## QMS ACCREDITATION

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





# **CERTIFYING OFFICER**

8/2

21<sup>st</sup> Nov, 2019

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



## REFERENCES

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



