

CERTIFICATE OF ANALYSIS FOR
CERTIFIED REFERENCE MATERIAL
OREAS 625

Volcanogenic Massive Sulphide Polymetallic Ore
Rosebery Mine, Tasmania, Australia

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 625.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppm)	0.667	0.659	0.674	0.665*	0.668*

SI unit equivalents: ppm (parts per million; 1×10^{-6}) \equiv mg/kg.

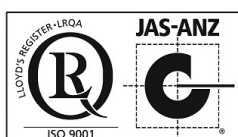
†This operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

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

Note: intervals may appear asymmetric due to rounding.



Accredited for compliance with ISO 17034



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Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 625.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion (sample weights 10-50g)					
Au, Gold (ppm)	0.667	0.656	0.677	0.665*	0.668*
4-Acid Digestion					
Ag, Silver (ppm)	11.7	11.2	12.2	11.3	12.1
Al, Aluminium (wt.%)	6.68	6.46	6.90	6.56	6.80
As, Arsenic (ppm)	143	138	148	139	148
Be, Beryllium (ppm)	2.57	2.45	2.69	2.47	2.66
Bi, Bismuth (ppm)	8.61	8.26	8.97	8.39	8.84
Ca, Calcium (wt.%)	1.14	1.11	1.18	1.12	1.17
Cd, Cadmium (ppm)	83	80	86	81	85
Ce, Cerium (ppm)	72	67	78	70	75
Co, Cobalt (ppm)	4.21	3.99	4.44	4.03	4.40
Cr, Chromium (ppm)	13.3	11.6	14.9	12.3	14.2
Cs, Caesium (ppm)	5.94	5.68	6.21	5.76	6.12
Cu, Copper (wt.%)	0.171	0.166	0.176	0.169	0.173
Dy, Dysprosium (ppm)	3.52	3.22	3.82	3.36	3.67
Er, Erbium (ppm)	1.24	1.14	1.35	1.17	1.31
Eu, Europium (ppm)	1.36	1.19	1.54	1.31	1.41
Fe, Iron (wt.%)	3.98	3.87	4.08	3.91	4.05
Ga, Gallium (ppm)	20.6	19.8	21.3	20.1	21.0
Gd, Gadolinium (ppm)	5.46	5.13	5.79	5.27	5.66
Hf, Hafnium (ppm)	6.36	6.03	6.68	6.19	6.53
Ho, Holmium (ppm)	0.54	0.49	0.58	0.50	0.57
In, Indium (ppm)	2.12	2.01	2.22	2.05	2.18
K, Potassium (wt.%)	2.86	2.80	2.93	2.81	2.92
La, Lanthanum (ppm)	34.7	31.0	38.5	33.4	36.0
Li, Lithium (ppm)	17.9	17.2	18.6	17.4	18.4
Lu, Lutetium (ppm)	0.14	0.12	0.15	IND	IND
Mg, Magnesium (wt.%)	0.261	0.251	0.272	0.257	0.266
Mn, Manganese (wt.%)	0.443	0.433	0.453	0.436	0.451
Mo, Molybdenum (ppm)	12.3	11.6	13.0	12.0	12.6
Na, Sodium (wt.%)	1.91	1.86	1.96	1.88	1.95
Nb, Niobium (ppm)	14.8	13.9	15.7	14.4	15.2
Nd, Neodymium (ppm)	37.3	33.0	41.7	36.3	38.4
Ni, Nickel (ppm)	5.98	5.55	6.42	5.60	6.37
P, Phosphorus (wt.%)	0.030	0.029	0.031	0.029	0.031
Pb, Lead (wt.%)	0.822	0.804	0.840	0.811	0.833

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

*Gold Tolerance Limits are calculated for a typical 25g aqua regia digestion and determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

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

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Pr, Praseodymium (ppm)	9.74	8.57	10.91	9.41	10.06
Rb, Rubidium (ppm)	135	130	140	132	139
Re, Rhenium (ppm)	0.004	0.002	0.006	IND	IND
S, Sulphur (wt.%)	3.80	3.69	3.92	3.73	3.87
Sb, Antimony (ppm)	35.1	33.4	36.9	34.1	36.1
Sc, Scandium (ppm)	4.60	4.29	4.92	4.45	4.76
Sm, Samarium (ppm)	6.80	6.22	7.38	6.40	7.20
Sn, Tin (ppm)	3.57	3.37	3.76	3.43	3.71
Sr, Strontium (ppm)	130	125	135	126	135
Ta, Tantalum (ppm)	1.11	1.02	1.20	1.05	1.16
Tb, Terbium (ppm)	0.72	0.65	0.80	0.69	0.76
Te, Tellurium (ppm)	0.063	0.017	0.110	IND	IND
Th, Thorium (ppm)	12.7	11.7	13.7	12.3	13.1
Ti, Titanium (wt.%)	0.120	0.115	0.125	0.117	0.123
Tl, Thallium (ppm)	9.79	9.40	10.18	9.55	10.03
Tm, Thulium (ppm)	0.17	0.14	0.19	IND	IND
U, Uranium (ppm)	5.52	5.27	5.77	5.37	5.67
V, Vanadium (ppm)	11.5	10.6	12.3	11.0	12.0
W, Tungsten (ppm)	3.99	3.67	4.30	3.84	4.14
Y, Yttrium (ppm)	14.9	13.9	15.8	14.4	15.3
Yb, Ytterbium (ppm)	0.96	0.85	1.07	0.92	1.00
Zn, Zinc (wt.%)	3.17	3.10	3.24	3.12	3.21
Zr, Zirconium (ppm)	229	221	237	224	234
Aqua Regia Digestion					
Ag, Silver (ppm)	11.7	11.3	12.0	11.4	11.9
Al, Aluminium (wt.%)	0.565	0.531	0.599	0.550	0.581
As, Arsenic (ppm)	134	127	140	130	137
B, Boron (ppm)	< 10	IND	IND	IND	IND
Be, Beryllium (ppm)	0.61	0.58	0.64	0.59	0.63
Bi, Bismuth (ppm)	8.81	8.48	9.15	8.53	9.09
Ca, Calcium (wt.%)	0.966	0.936	0.996	0.948	0.984
Cd, Cadmium (ppm)	83	81	86	82	85
Ce, Cerium (ppm)	36.8	34.2	39.4	35.9	37.7
Co, Cobalt (ppm)	3.54	3.35	3.72	3.39	3.68
Cr, Chromium (ppm)	13.1	12.2	13.9	12.5	13.7
Cs, Caesium (ppm)	1.14	1.07	1.22	1.11	1.18
Cu, Copper (wt.%)	0.174	0.168	0.179	0.171	0.176

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

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion continued					
Fe, Iron (wt.%)	3.27	3.15	3.40	3.21	3.34
Ga, Gallium (ppm)	2.63	2.34	2.91	2.55	2.70
Hf, Hafnium (ppm)	1.46	1.38	1.54	1.41	1.52
Hg, Mercury (ppm)	1.72	1.58	1.85	1.66	1.77
In, Indium (ppm)	2.07	1.95	2.18	1.97	2.16
K, Potassium (wt.%)	0.276	0.261	0.290	0.267	0.284
La, Lanthanum (ppm)	17.7	16.2	19.1	17.2	18.1
Li, Lithium (ppm)	3.40	3.10	3.70	3.21	3.59
Lu, Lutetium (ppm)	0.045	0.036	0.054	IND	IND
Mg, Magnesium (wt.%)	0.119	0.113	0.125	0.115	0.123
Mn, Manganese (wt.%)	0.434	0.421	0.447	0.427	0.441
Mo, Molybdenum (ppm)	11.6	11.1	12.2	11.3	12.0
Na, Sodium (wt.%)	0.059	0.056	0.062	0.057	0.061
Nb, Niobium (ppm)	0.70	0.60	0.79	0.66	0.74
Ni, Nickel (ppm)	5.32	4.99	5.65	5.10	5.54
P, Phosphorus (wt.%)	0.025	0.024	0.026	0.024	0.026
Pb, Lead (wt.%)	0.826	0.807	0.845	0.816	0.836
Rb, Rubidium (ppm)	14.3	13.3	15.2	13.7	14.8
Re, Rhenium (ppm)	0.004	0.003	0.005	IND	IND
S, Sulphur (wt.%)	3.77	3.64	3.89	3.69	3.85
Sb, Antimony (ppm)	28.5	27.0	30.0	27.5	29.5
Sc, Scandium (ppm)	1.12	1.00	1.24	IND	IND
Sn, Tin (ppm)	0.95	0.83	1.07	0.87	1.02
Sr, Strontium (ppm)	20.7	19.8	21.6	20.2	21.2
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.38	0.32	0.44	0.36	0.40
Te, Tellurium (ppm)	0.050	0.028	0.073	IND	IND
Th, Thorium (ppm)	7.49	7.04	7.94	7.28	7.70
Ti, Titanium (wt.%)	0.023	0.021	0.026	0.023	0.024
Tl, Thallium (ppm)	3.03	2.89	3.17	2.92	3.14
U, Uranium (ppm)	2.77	2.60	2.94	2.70	2.84
V, Vanadium (ppm)	3.11	2.88	3.35	2.82	3.40
W, Tungsten (ppm)	1.47	1.35	1.59	1.42	1.52
Y, Yttrium (ppm)	6.40	6.04	6.75	6.22	6.58
Yb, Ytterbium (ppm)	0.34	0.31	0.38	IND	IND
Zn, Zinc (wt.%)	3.18	3.11	3.25	3.13	3.23
Zr, Zirconium (ppm)	55	50	60	53	57

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

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Peroxide Fusion ICP					
Al, Aluminium (wt.%)	6.83	6.65	7.00	6.73	6.92
As, Arsenic (ppm)	144	134	155	139	149
Ba, Barium (ppm)	3415	3249	3581	3351	3480
Bi, Bismuth (ppm)	8.61	7.93	9.29	8.32	8.90
Ca, Calcium (wt.%)	1.15	1.09	1.21	1.11	1.19
Cd, Cadmium (ppm)	83	77	88	80	85
Ce, Cerium (ppm)	86	80	92	83	90
Co, Cobalt (ppm)	4.50	3.80	5.21	4.07	4.94
Cs, Caesium (ppm)	6.00	5.40	6.60	5.78	6.22
Cu, Copper (wt.%)	0.170	0.166	0.175	0.167	0.174
Dy, Dysprosium (ppm)	3.66	3.22	4.09	3.50	3.82
Er, Erbium (ppm)	1.41	1.25	1.58	1.35	1.48
Eu, Europium (ppm)	1.58	1.23	1.93	1.49	1.67
Fe, Iron (wt.%)	4.01	3.91	4.11	3.95	4.08
Ga, Gallium (ppm)	20.4	18.8	22.1	19.7	21.2
Gd, Gadolinium (ppm)	5.53	4.99	6.07	5.28	5.78
Ge, Germanium (ppm)	1.31	0.43	2.18	IND	IND
Ho, Holmium (ppm)	0.60	0.58	0.63	0.58	0.63
In, Indium (ppm)	2.11	1.85	2.36	1.85	2.36
K, Potassium (wt.%)	2.89	2.80	2.98	2.83	2.95
La, Lanthanum (ppm)	43.4	40.9	45.9	41.7	45.0
Li, Lithium (ppm)	18.2	15.9	20.4	IND	IND
Lu, Lutetium (ppm)	0.14	0.11	0.17	IND	IND
Mg, Magnesium (wt.%)	0.267	0.257	0.276	0.256	0.277
Mn, Manganese (wt.%)	0.447	0.438	0.456	0.440	0.454
Mo, Molybdenum (ppm)	13.7	12.1	15.3	IND	IND
Nb, Niobium (ppm)	14.7	12.4	17.0	IND	IND
Nd, Neodymium (ppm)	37.7	35.9	39.5	36.9	38.5
P, Phosphorus (wt.%)	0.030	0.028	0.032	IND	IND
Pb, Lead (wt.%)	0.806	0.783	0.829	0.792	0.819
Pr, Praseodymium (ppm)	9.95	8.98	10.93	9.59	10.32
Rb, Rubidium (ppm)	136	127	146	131	142
S, Sulphur (wt.%)	3.85	3.73	3.98	3.76	3.94
Sb, Antimony (ppm)	36.2	33.1	39.3	35.2	37.1
Si, Silicon (wt.%)	29.02	28.25	29.79	28.58	29.46
Sm, Samarium (ppm)	7.04	6.21	7.86	6.66	7.41
Sr, Strontium (ppm)	142	135	149	139	146

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

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

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Peroxide Fusion ICP continued					
Tb, Terbium (ppm)	0.77	0.68	0.87	0.75	0.79
Th, Thorium (ppm)	13.7	12.9	14.5	13.4	14.1
Ti, Titanium (wt.%)	0.129	0.122	0.136	0.124	0.135
Tl, Thallium (ppm)	9.99	9.21	10.76	9.72	10.26
Tm, Thulium (ppm)	0.18	0.16	0.20	IND	IND
U, Uranium (ppm)	5.78	5.28	6.29	5.49	6.07
V, Vanadium (ppm)	11.5	8.3	14.7	IND	IND
W, Tungsten (ppm)	4.39	3.04	5.74	4.25	4.53
Y, Yttrium (ppm)	17.6	16.5	18.7	17.4	17.8
Yb, Ytterbium (ppm)	1.05	0.93	1.18	IND	IND
Zn, Zinc (wt.%)	3.20	3.11	3.29	3.15	3.25
Infrared Combustion					
S, Sulphur (wt.%)	3.91	3.83	4.00	3.87	3.95

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

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

Table 3. Indicative Values for OREAS 625.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Ag	ppm	6.67	Pd	ppb	0.750	Pt	ppb	< 10
4-Acid Digestion								
B	ppm	20.8	Ge	ppm	0.23	Se	ppm	2.07
Ba	ppm	528	Hg	ppm	1.35			
Aqua Regia Digestion								
Ba	ppm	77	Ge	ppm	0.084	Pt	ppb	< 5
Dy	ppm	1.69	Ho	ppm	0.24	Se	ppm	1.55
Er	ppm	0.55	Nd	ppm	17.2	Sm	ppm	3.30
Eu	ppm	0.67	Pd	ppb	11.5	Tm	ppm	0.059
Gd	ppm	2.90	Pr	ppm	4.52			
Ba	ppm	77	Ge	ppm	0.084	Pt	ppb	< 5
Peroxide Fusion ICP								
Ag	ppm	12.6	Hg	ppm	< 5	Se	ppm	< 5
B	ppm	25.3	Na	wt.%	1.88	Sn	ppm	3.48
Be	ppm	2.91	Ni	ppm	20.7	Ta	ppm	1.43
Cr	ppm	57	Re	ppm	< 0.05	Te	ppm	< 1
Hf	ppm	6.29	Sc	ppm	4.84	Zr	ppm	248
Infrared Combustion								
C	wt.%	0.384						

SI unit equivalents: ppb (parts per billion; 1×10^{-9}) \equiv μ g/kg; ppm (parts per million; 1×10^{-6}) \equiv mg/kg; wt.% (weight per cent) \equiv % (mass fraction).

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

Table 3 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
Al ₂ O ₃	wt. %	13.16	MgO	wt. %	0.467	Sn	ppm	27.5
As	ppm	3156	Mn	wt. %	0.454	Sr	ppm	99
Ba	ppm	3588	Na ₂ O	wt. %	2.69	TiO ₂	wt. %	0.211
CaO	wt. %	1.61	Ni	ppm	10.6	V	ppm	18.0
Cr	ppm	27.4	P	wt. %	0.029	Zn	wt. %	3.32
Cu	wt. %	0.169	Pb	wt. %	0.871	Zr	ppm	296
Fe	wt. %	4.05	S	wt. %	3.33			
K ₂ O	wt. %	3.52	SiO ₂	wt. %	62.85			
Thermogravimetry								
LOI ¹⁰⁰⁰	wt. %	4.37						
Laser Ablation ICP-MS								
Ag	ppm	10.9	Hf	ppm	6.80	Se	ppm	< 5
As	ppm	142	Ho	ppm	0.60	Sm	ppm	6.89
Ba	ppm	3450	In	ppm	1.95	Sn	ppm	3.40
Be	ppm	2.60	La	ppm	42.7	Sr	ppm	134
Bi	ppm	8.90	Lu	ppm	0.13	Ta	ppm	1.18
Ce	ppm	82	Mn	wt. %	0.454	Tb	ppm	0.74
Co	ppm	5.05	Mo	ppm	11.6	Te	ppm	0.15
Cr	ppm	16.0	Nb	ppm	15.6	Th	ppm	14.3
Cs	ppm	6.07	Nd	ppm	36.2	Ti	wt. %	0.134
Cu	wt. %	0.207	Ni	ppm	8.00	Tm	ppm	0.16
Dy	ppm	3.83	Pb	wt. %	0.737	U	ppm	5.60
Er	ppm	1.37	Pr	ppm	10.2	V	ppm	11.1
Eu	ppm	1.43	Rb	ppm	136	W	ppm	4.50
Ga	ppm	19.8	Re	ppm	< 0.01	Y	ppm	16.6
Gd	ppm	5.36	Sb	ppm	36.4	Yb	ppm	1.07
Ge	ppm	1.08	Sc	ppm	5.10	Zr	ppm	241

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

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

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INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself. In evaluating laboratory performance with this CRM, the section headed 'Instructions for correct use' should be read carefully.

Table 1 (all laboratories accredited to ISO 17025) and Table 2 (most laboratories accredited to ISO 17025 for the operationally defined measurands shown) provide the certified values and their associated 95% expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation and Table 4 provides some indicative physical properties. Gold homogeneity (via INAA) is shown in Table 5 and is also demonstrated by a nested ANOVA (see 'Homogeneity Evaluation' section). Finally, Table 6 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM³) are presented in the detailed certification data for this CRM (**OREAS 625-DataPack.1.0.240118_151640.xlsx**). Results are also presented in scatter plots for Au by fire assay, Cu, Pb and Zn by 4-acid digestion (Figures 1 to 4, respectively) together with $\pm 3SD$ (magenta) and $\pm 5\%$ (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

SOURCE MATERIAL

OREAS 625 was prepared from a blend of polymetallic (Au-Zn-Cu-Pb-Ag) ore, barren rhyodacite and minor additions of Cu, Zn and Pb concentrates. The ore was sourced from the Rosebery mine 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. The barren rhyodacite was sourced from the rhyodacitic unit of the Mt Dandenong Igneous Complex located approximately 34km east of Melbourne (Victoria), Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 625 was prepared in the following manner:

- Drying of ore and barren materials to constant mass at 105° C;
- Drying of Cu, Zn and Pb concentrates to constant mass at 85° C;
- Crushing and milling of the ore and concentrate materials to 100% minus 30 microns;
- Crushing and milling of the barren rhyodacite to >98% minus 75 microns;

- Blending ores, rhyodacite and concentrates in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging under nitrogen in 10g and 60g units in laminated foil pouches.

PHYSICAL PROPERTIES

OREAS 625 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

Table 4. Physical properties of OREAS 625.

Bulk Density (kg/m ³)	Moisture (wt.%)	Munsell Notation [‡]	Munsell Color [‡]
800.5	0.31	N6	Medium Light Gray

[‡]The Munsell Rock Color Chart helps geologists and archeologists communicate with colour more effectively by cross-referencing ISCC-NBS colour names with unique Munsell alpha-numeric colour notations for rock colour samples.

ANALYTICAL PROGRAM

Thirty-one commercial analytical laboratories participated in the program to certify the elements reported in Tables 1 and 2. The following methods were employed:

- Gold by fire assay (25-50g charge weight) with AAS (19 laboratories) or ICP-OES (10 laboratories) finish;
- Gold by aqua regia digestion (10-50g sample weight) with AAS (5 laboratories) or ICP-MS (16 laboratories) finish;
- Full ICP-OES and ICP-MS elemental suites by 4-acid (HNO₃-HF-HClO₄-HCl) digestion (up to 29 laboratories depending on the element) with 10 laboratories reporting over-range Zn by AAS finish and 1 laboratory reporting Pb by AAS finish;
- Full ICP-OES and ICP-MS elemental suites by aqua regia digestion (up to 29 laboratories depending on the element) with 10 laboratories reporting over-range Zn by AAS finish;
- Full ICP-OES and ICP-MS elemental suites by peroxide fusion (up to 23 laboratories depending on the element) and
- Total Sulphur by infrared combustion furnace (27 laboratories).

Instrumental neutron activation analysis for Au on 20 x 85mg subsamples was also undertaken at ANSTO, Lucas Heights to confirm homogeneity (see Table 5).

Table 3 shows indicative values including major and trace element characterisation based on two samples analysed at Bureau Veritas in Perth, Western Australia which includes:

- Major oxides by lithium borate fusion with X-ray fluorescence;
- LOI at 1000° C by thermogravimetric analyser;
- Trace elements by laser ablation (on the fused bead) with ICP-MS finish and
- Total Carbon by infrared combustion furnace.

For the round robin program, ten 3kg test units (lots) were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered

representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking a 110g scoop split from six different 3kg lots.

The 20 individual INAA results upon which much of the homogeneity evaluation is based, included paired 10g samples taken from each of the 10 different test units. This format enabled a nested ANOVA treatment of the INAA results to evaluate homogeneity (see 'Homogeneity Evaluation' section below).

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Tables 1 and 2) 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. 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. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

Certified Values are the means of accepted laboratory means after outlier filtering and are the present best estimate of the true value. 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.

95% Expanded Uncertainty provides a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits and is calculated according to the method outlined in ISO 98-3:2008 [6]. All known or suspected sources of bias have been investigated or taken into account.

Indicative (uncertified) values (Table 3) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor.

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Tables 1 and 2 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for copper by 4-acid digestion, where 99% of the time ($1-\alpha=0.99$) at least 95% of subsamples ($\rho=0.95$) will have concentrations lying between 0.169 and 0.173 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 35:2017). **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 625. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.078% calculated for a 30g fire assay sample (1.46% at 85mg weights) confirms the high level of gold homogeneity in OREAS 625.

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 No	Au 85mg actual	Au 30g equivalent*
1	0.70	0.692
2	0.69	0.691
3	0.70	0.692
4	0.68	0.690
5	0.70	0.691
6	0.69	0.691
7	0.69	0.691
8	0.68	0.690
9	0.70	0.691
10	0.69	0.691
11	0.69	0.691
12	0.70	0.691
13	0.68	0.691
14	0.68	0.690
15	0.69	0.691
16	0.69	0.691
17	0.69	0.691
18	0.71	0.692
19	0.70	0.691
20	0.67	0.690
Mean	0.691	0.691
Median	0.691	0.691
Std Dev.	0.010	0.001
Rel.Std.Dev.	1.46%	0.078%

*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 at ANSTO using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973 [2]). 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.

The homogeneity of OREAS 625 has also been evaluated in an Analysis of Variance (**ANOVA**) of the INAA data. The 20 samples were comprised of paired samples from each of 10 sampling lot intervals (representative of the prepared batch) and were randomised

prior to assigning sample numbers. The duplicate samples enabled an ANOVA by comparison of within- and between-unit variances across the 10 pairs. The purpose of the ANOVA is to test that no statistically significant difference exists in the variance between units to that of the variance within units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 625. The test was performed using the following parameters:

- Gold INAA – 20 results (1 laboratory providing duplicate analyses on 10 samples where each sample can be viewed as a ‘unit’);
- Null Hypothesis, H_0 : Between-unit variance is no greater than within-unit variance (reject H_0 if p -value < 0.05);
- Alternative Hypothesis, H_1 : Between-unit variance is greater than within-unit variance.

The data was not filtered for outliers prior to the calculation of the p -value. This process derived a p -value of 0.984 a statistically insignificant result so the Null Hypothesis is accepted.

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 625 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 ANOVA and the results of the interlaboratory certification program, it can be concluded that OREAS 625 is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

PERFORMANCE GATES

The standard deviations (SD’s) intervals reported in Table 6 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 ‘Instructions for handling and correct 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.

Table 6 below shows intervals calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit www.westgard.com/mltirule.htm). A second method utilises a 5% window calculated directly from the certified value. Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL) \pm 10%.

i.e., Certified Value \pm 10% \pm 2DL [1].

Table 6. Performance Gates for OREAS 625.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Pb Fire Assay											
Au, ppm	0.667	0.020	0.627	0.706	0.607	0.726	2.98%	5.96%	8.93%	0.633	0.700
Aqua Regia Digestion (sample weights 10-50g)											
Au, ppm	0.667	0.024	0.619	0.714	0.596	0.738	3.55%	7.10%	10.65%	0.633	0.700
4-Acid Digestion											
Ag, ppm	11.7	0.61	10.5	12.9	9.9	13.5	5.19%	10.37%	15.56%	11.1	12.3
Al, wt. %	6.68	0.273	6.13	7.22	5.86	7.50	4.09%	8.18%	12.27%	6.34	7.01
As, ppm	143	4	135	152	131	156	2.84%	5.69%	8.53%	136	151
Be, ppm	2.57	0.157	2.25	2.88	2.10	3.04	6.11%	12.22%	18.34%	2.44	2.70
Bi, ppm	8.61	0.483	7.65	9.58	7.16	10.06	5.60%	11.21%	16.81%	8.18	9.04
Ca, wt. %	1.14	0.053	1.04	1.25	0.99	1.30	4.62%	9.23%	13.85%	1.09	1.20
Cd, ppm	83	5.3	72	94	67	99	6.37%	12.73%	19.10%	79	87
Ce, ppm	72	10	53	92	43	102	13.64%	27.28%	40.92%	69	76
Co, ppm	4.21	0.236	3.74	4.69	3.51	4.92	5.60%	11.20%	16.80%	4.00	4.42
Cr, ppm	13.3	1.6	10.0	16.5	8.4	18.1	12.19%	24.38%	36.57%	12.6	13.9
Cs, ppm	5.94	0.209	5.52	6.36	5.31	6.57	3.53%	7.05%	10.58%	5.65	6.24
Cu, wt. %	0.171	0.006	0.158	0.184	0.152	0.190	3.72%	7.43%	11.15%	0.162	0.180
Dy, ppm	3.52	0.175	3.17	3.87	2.99	4.04	4.96%	9.93%	14.89%	3.34	3.69
Er, ppm	1.24	0.049	1.14	1.34	1.10	1.39	3.92%	7.84%	11.75%	1.18	1.30
Eu, ppm	1.36	0.109	1.14	1.58	1.04	1.69	7.99%	15.99%	23.98%	1.29	1.43
Fe, wt. %	3.98	0.131	3.72	4.24	3.58	4.37	3.30%	6.60%	9.89%	3.78	4.18
Ga, ppm	20.6	0.63	19.3	21.8	18.7	22.5	3.09%	6.18%	9.26%	19.5	21.6
Gd, ppm	5.46	0.206	5.05	5.88	4.84	6.08	3.78%	7.55%	11.33%	5.19	5.74
Hf, ppm	6.36	0.377	5.60	7.11	5.22	7.49	5.93%	11.87%	17.80%	6.04	6.67
Ho, ppm	0.54	0.024	0.49	0.58	0.46	0.61	4.54%	9.09%	13.63%	0.51	0.56
In, ppm	2.12	0.098	1.92	2.31	1.82	2.41	4.63%	9.25%	13.88%	2.01	2.22

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

Note 1: intervals may appear asymmetric due to rounding.

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
4-Acid Digestion continued											
K, wt. %	2.86	0.087	2.69	3.04	2.60	3.13	3.03%	6.07%	9.10%	2.72	3.01
La, ppm	34.7	5.9	22.9	46.5	17.0	52.4	17.01%	34.02%	51.02%	33.0	36.5
Li, ppm	17.9	0.72	16.4	19.3	15.7	20.0	4.02%	8.05%	12.07%	17.0	18.8
Lu, ppm	0.14	0.006	0.12	0.15	0.12	0.15	4.77%	9.54%	14.31%	0.13	0.14
Mg, wt. %	0.261	0.012	0.237	0.286	0.224	0.298	4.72%	9.44%	14.16%	0.248	0.274
Mn, wt. %	0.443	0.015	0.413	0.474	0.397	0.490	3.47%	6.95%	10.42%	0.421	0.466
Mo, ppm	12.3	0.90	10.5	14.1	9.6	15.0	7.28%	14.57%	21.85%	11.7	12.9
Na, wt. %	1.91	0.058	1.80	2.03	1.74	2.09	3.03%	6.07%	9.10%	1.82	2.01
Nb, ppm	14.8	1.34	12.1	17.5	10.8	18.8	9.06%	18.12%	27.17%	14.0	15.5
Nd, ppm	37.3	3.72	29.9	44.8	26.2	48.5	9.97%	19.94%	29.91%	35.5	39.2
Ni, ppm	5.98	0.534	4.92	7.05	4.38	7.59	8.92%	17.83%	26.75%	5.69	6.28
P, wt. %	0.030	0.001	0.028	0.032	0.027	0.033	3.45%	6.90%	10.34%	0.028	0.031
Pb, wt. %	0.822	0.019	0.784	0.860	0.765	0.879	2.29%	4.59%	6.88%	0.781	0.863
Pr, ppm	9.74	0.99	7.76	11.71	6.78	12.70	10.14%	20.27%	30.41%	9.25	10.22
Rb, ppm	135	6	123	148	117	154	4.54%	9.08%	13.63%	129	142
Re, ppm	0.004	0.001	0.002	0.006	0.001	0.007	27.59%	55.18%	82.77%	0.004	0.004
S, wt. %	3.80	0.120	3.56	4.04	3.45	4.16	3.14%	6.29%	9.43%	3.61	3.99
Sb, ppm	35.1	2.30	30.5	39.7	28.2	42.0	6.56%	13.12%	19.68%	33.4	36.9
Sc, ppm	4.60	0.324	3.95	5.25	3.63	5.57	7.05%	14.09%	21.14%	4.37	4.83
Sm, ppm	6.80	0.357	6.09	7.51	5.73	7.87	5.25%	10.50%	15.75%	6.46	7.14
Sn, ppm	3.57	0.171	3.23	3.91	3.05	4.08	4.79%	9.57%	14.36%	3.39	3.75
Sr, ppm	130	7	116	144	109	151	5.37%	10.74%	16.11%	124	137
Ta, ppm	1.11	0.081	0.94	1.27	0.86	1.35	7.34%	14.67%	22.01%	1.05	1.16
Tb, ppm	0.72	0.064	0.60	0.85	0.53	0.92	8.83%	17.66%	26.50%	0.69	0.76
Te, ppm	0.063	0.030	0.003	0.124	0.000	0.154	47.79%	95.58%	143.37	0.060	0.066
Th, ppm	12.7	1.3	10.1	15.2	8.9	16.5	10.03%	20.07%	30.10%	12.0	13.3
Ti, wt. %	0.120	0.004	0.112	0.128	0.109	0.132	3.23%	6.45%	9.68%	0.114	0.126
Tl, ppm	9.79	0.413	8.96	10.62	8.55	11.03	4.22%	8.44%	12.67%	9.30	10.28
Tm, ppm	0.17	0.02	0.12	0.21	0.10	0.23	13.60%	27.21%	40.81%	0.16	0.17
U, ppm	5.52	0.263	4.99	6.05	4.73	6.31	4.77%	9.54%	14.31%	5.24	5.80
V, ppm	11.5	1.06	9.3	13.6	8.3	14.6	9.22%	18.43%	27.65%	10.9	12.0
W, ppm	3.99	0.41	3.17	4.80	2.77	5.21	10.20%	20.40%	30.60%	3.79	4.19
Y, ppm	14.9	1.03	12.8	17.0	11.8	18.0	6.93%	13.87%	20.80%	14.1	15.6
Yb, ppm	0.96	0.055	0.85	1.07	0.79	1.12	5.75%	11.50%	17.25%	0.91	1.01
Zn, wt. %	3.17	0.098	2.97	3.36	2.88	3.46	3.09%	6.18%	9.26%	3.01	3.33
Zr, ppm	229	10	210	249	200	259	4.27%	8.53%	12.80%	218	241
Aqua Regia Digestion											
Ag, ppm	11.7	0.40	10.9	12.4	10.5	12.8	3.40%	6.81%	10.21%	11.1	12.2
Al, wt. %	0.565	0.064	0.437	0.693	0.373	0.757	11.32%	22.63%	33.95%	0.537	0.593
As, ppm	134	10	115	153	105	162	7.14%	14.29%	21.43%	127	140
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.61	0.035	0.54	0.68	0.50	0.72	5.75%	11.51%	17.26%	0.58	0.64
Bi, ppm	8.81	0.295	8.22	9.40	7.93	9.70	3.35%	6.70%	10.04%	8.37	9.25
Ca, wt. %	0.966	0.035	0.895	1.036	0.860	1.071	3.65%	7.30%	10.94%	0.918	1.014

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

Note 1: intervals may appear asymmetric due to rounding.

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Aqua Regia Digestion continued											
Cd, ppm	83	3.8	76	91	72	95	4.53%	9.06%	13.59%	79	87
Ce, ppm	36.8	3.8	29.1	44.5	25.3	48.3	10.44%	20.89%	31.33%	34.9	38.6
Co, ppm	3.54	0.302	2.93	4.14	2.63	4.44	8.54%	17.07%	25.61%	3.36	3.71
Cr, ppm	13.1	0.78	11.5	14.6	10.7	15.4	5.93%	11.87%	17.80%	12.4	13.7
Cs, ppm	1.14	0.108	0.93	1.36	0.82	1.47	9.44%	18.88%	28.31%	1.09	1.20
Cu, wt.%	0.174	0.006	0.162	0.185	0.156	0.191	3.28%	6.57%	9.85%	0.165	0.182
Fe, wt.%	3.27	0.228	2.82	3.73	2.59	3.96	6.95%	13.91%	20.86%	3.11	3.44
Ga, ppm	2.63	0.44	1.75	3.50	1.31	3.94	16.71%	33.43%	50.14%	2.49	2.76
Hf, ppm	1.46	0.095	1.27	1.65	1.18	1.74	6.48%	12.96%	19.43%	1.39	1.53
Hg, ppm	1.72	0.137	1.44	1.99	1.30	2.13	8.01%	16.02%	24.03%	1.63	1.80
In, ppm	2.07	0.135	1.79	2.34	1.66	2.47	6.55%	13.09%	19.64%	1.96	2.17
K, wt.%	0.276	0.026	0.224	0.328	0.198	0.354	9.44%	18.88%	28.32%	0.262	0.289
La, ppm	17.7	2.5	12.6	22.7	10.1	25.2	14.33%	28.65%	42.98%	16.8	18.5
Li, ppm	3.40	0.46	2.48	4.33	2.01	4.79	13.59%	27.17%	40.76%	3.23	3.57
Lu, ppm	0.045	0.005	0.035	0.055	0.030	0.060	11.18%	22.35%	33.53%	0.043	0.047
Mg, wt.%	0.119	0.009	0.101	0.137	0.092	0.147	7.70%	15.39%	23.09%	0.113	0.125
Mn, wt.%	0.434	0.012	0.409	0.459	0.397	0.471	2.83%	5.66%	8.49%	0.412	0.456
Mo, ppm	11.6	0.66	10.3	13.0	9.7	13.6	5.69%	11.39%	17.08%	11.1	12.2
Na, wt.%	0.059	0.006	0.046	0.072	0.040	0.078	10.96%	21.93%	32.89%	0.056	0.062
Nb, ppm	0.70	0.15	0.40	0.99	0.26	1.14	20.98%	41.96%	62.94%	0.66	0.73
Ni, ppm	5.32	0.457	4.41	6.23	3.95	6.69	8.59%	17.19%	25.78%	5.05	5.59
P, wt.%	0.025	0.002	0.022	0.029	0.020	0.030	6.88%	13.76%	20.64%	0.024	0.026
Pb, wt.%	0.826	0.021	0.784	0.868	0.763	0.889	2.54%	5.07%	7.61%	0.785	0.868
Rb, ppm	14.3	1.5	11.4	17.2	9.9	18.6	10.19%	20.38%	30.57%	13.6	15.0
Re, ppm	0.004	0.001	0.003	0.005	0.002	0.006	15.81%	31.62%	47.42%	0.004	0.004
S, wt.%	3.77	0.184	3.40	4.13	3.22	4.32	4.87%	9.75%	14.62%	3.58	3.95
Sb, ppm	28.5	2.58	23.3	33.7	20.8	36.2	9.04%	18.08%	27.13%	27.1	29.9
Sc, ppm	1.12	0.15	0.81	1.43	0.65	1.58	13.83%	27.66%	41.49%	1.06	1.17
Sn, ppm	0.95	0.17	0.61	1.28	0.45	1.45	17.57%	35.14%	52.71%	0.90	0.99
Sr, ppm	20.7	1.61	17.5	24.0	15.9	25.6	7.79%	15.57%	23.36%	19.7	21.8
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.38	0.05	0.28	0.48	0.23	0.53	13.23%	26.47%	39.70%	0.36	0.40
Te, ppm	0.050	0.015	0.021	0.079	0.007	0.094	28.97%	57.94%	86.90%	0.048	0.053
Th, ppm	7.49	0.607	6.28	8.71	5.67	9.31	8.11%	16.22%	24.33%	7.12	7.87
Ti, wt.%	0.023	0.005	0.014	0.032	0.010	0.037	19.28%	38.56%	57.84%	0.022	0.025
Tl, ppm	3.03	0.144	2.74	3.32	2.60	3.46	4.75%	9.50%	14.25%	2.88	3.18
U, ppm	2.77	0.29	2.19	3.35	1.90	3.64	10.49%	20.98%	31.48%	2.63	2.91
V, ppm	3.11	0.48	2.15	4.07	1.67	4.56	15.49%	30.97%	46.46%	2.95	3.27
W, ppm	1.47	0.130	1.21	1.73	1.08	1.86	8.82%	17.63%	26.45%	1.40	1.54
Y, ppm	6.40	0.505	5.39	7.41	4.88	7.91	7.89%	15.78%	23.68%	6.08	6.72
Yb, ppm	0.34	0.04	0.26	0.42	0.22	0.46	11.95%	23.90%	35.84%	0.32	0.36
Zn, wt.%	3.18	0.090	3.00	3.36	2.91	3.45	2.82%	5.63%	8.45%	3.02	3.34
Zr, ppm	55	9	37	73	28	82	16.51%	33.03%	49.54%	52	57

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

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

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Peroxide Fusion ICP											
Al, wt.%	6.83	0.171	6.49	7.17	6.31	7.34	2.50%	5.01%	7.51%	6.49	7.17
As, ppm	144	22	99	189	77	212	15.60%	31.19%	46.79%	137	151
Ba, ppm	3415	243	2930	3901	2687	4144	7.11%	14.22%	21.34%	3245	3586
Bi, ppm	8.61	0.684	7.24	9.98	6.56	10.66	7.94%	15.88%	23.82%	8.18	9.04
Ca, wt.%	1.15	0.055	1.04	1.26	0.98	1.31	4.82%	9.64%	14.47%	1.09	1.21
Cd, ppm	83	4.6	74	92	69	96	5.55%	11.11%	16.66%	79	87
Ce, ppm	86	2.5	81	91	79	93	2.88%	5.76%	8.63%	82	90
Co, ppm	4.50	0.406	3.69	5.32	3.29	5.72	9.01%	18.01%	27.02%	4.28	4.73
Cs, ppm	6.00	0.281	5.44	6.56	5.16	6.84	4.68%	9.36%	14.04%	5.70	6.30
Cu, wt.%	0.170	0.005	0.161	0.180	0.156	0.185	2.84%	5.68%	8.51%	0.162	0.179
Dy, ppm	3.66	0.284	3.09	4.23	2.81	4.51	7.76%	15.52%	23.28%	3.48	3.84
Er, ppm	1.41	0.092	1.23	1.60	1.14	1.69	6.53%	13.06%	19.59%	1.34	1.49
Eu, ppm	1.58	0.30	0.97	2.19	0.66	2.49	19.29%	38.57%	57.86%	1.50	1.66
Fe, wt.%	4.01	0.109	3.79	4.23	3.68	4.34	2.73%	5.45%	8.18%	3.81	4.21
Ga, ppm	20.4	1.34	17.8	23.1	16.4	24.5	6.54%	13.07%	19.61%	19.4	21.5
Gd, ppm	5.53	0.245	5.04	6.02	4.79	6.27	4.44%	8.87%	13.31%	5.25	5.81
Ge, ppm	1.31	0.45	0.41	2.21	0.00	2.66	34.48%	68.95%	103.43%	1.24	1.37
Ho, ppm	0.60	0.023	0.56	0.65	0.53	0.67	3.86%	7.72%	11.58%	0.57	0.63
In, ppm	2.11	0.187	1.73	2.48	1.54	2.67	8.88%	17.76%	26.64%	2.00	2.21
K, wt.%	2.89	0.075	2.74	3.04	2.66	3.11	2.61%	5.22%	7.83%	2.74	3.03
La, ppm	43.4	1.72	39.9	46.8	38.2	48.5	3.97%	7.94%	11.91%	41.2	45.5
Li, ppm	18.2	1.73	14.7	21.6	13.0	23.4	9.50%	18.99%	28.49%	17.3	19.1
Lu, ppm	0.14	0.03	0.09	0.20	0.06	0.23	19.30%	38.60%	57.90%	0.14	0.15
Mg, wt.%	0.267	0.013	0.241	0.292	0.229	0.305	4.74%	9.48%	14.22%	0.253	0.280
Mn, wt.%	0.447	0.009	0.428	0.466	0.418	0.475	2.12%	4.25%	6.37%	0.424	0.469
Mo, ppm	13.7	1.7	10.4	17.0	8.7	18.6	12.09%	24.19%	36.28%	13.0	14.4
Nb, ppm	14.7	2.2	10.2	19.2	8.0	21.4	15.26%	30.52%	45.78%	14.0	15.4
Nd, ppm	37.7	1.09	35.5	39.9	34.5	41.0	2.89%	5.77%	8.66%	35.8	39.6
P, wt.%	0.030	0.001	0.028	0.033	0.026	0.035	4.53%	9.06%	13.59%	0.029	0.032
Pb, wt.%	0.806	0.034	0.739	0.873	0.705	0.907	4.18%	8.36%	12.54%	0.766	0.846
Pr, ppm	9.95	0.941	8.07	11.84	7.13	12.78	9.46%	18.92%	28.37%	9.46	10.45
Rb, ppm	136	9	118	155	109	164	6.74%	13.48%	20.22%	130	143
S, wt.%	3.85	0.150	3.55	4.15	3.40	4.30	3.89%	7.78%	11.67%	3.66	4.04
Sb, ppm	36.2	1.39	33.4	39.0	32.0	40.4	3.85%	7.71%	11.56%	34.4	38.0
Si, wt.%	29.02	0.756	27.51	30.53	26.75	31.29	2.61%	5.21%	7.82%	27.57	30.47
Sm, ppm	7.04	0.454	6.13	7.94	5.68	8.40	6.45%	12.90%	19.35%	6.68	7.39
Sr, ppm	142	9	124	161	114	171	6.60%	13.19%	19.79%	135	149
Tb, ppm	0.77	0.065	0.64	0.90	0.58	0.97	8.36%	16.71%	25.07%	0.73	0.81
Th, ppm	13.7	0.94	11.8	15.6	10.9	16.6	6.85%	13.70%	20.55%	13.0	14.4
Ti, wt.%	0.129	0.006	0.118	0.141	0.112	0.147	4.58%	9.15%	13.73%	0.123	0.136
Tl, ppm	9.99	0.702	8.58	11.39	7.88	12.09	7.03%	14.05%	21.08%	9.49	10.49
Tm, ppm	0.18	0.017	0.15	0.22	0.13	0.24	9.56%	19.12%	28.68%	0.17	0.19
U, ppm	5.78	0.506	4.77	6.79	4.26	7.30	8.75%	17.51%	26.26%	5.49	6.07
V, ppm	11.5	2.2	7.1	15.9	4.8	18.2	19.30%	38.59%	57.89%	10.9	12.1
W, ppm	4.39	0.56	3.27	5.52	2.70	6.08	12.82%	25.64%	38.47%	4.17	4.61

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

Note 1: intervals may appear asymmetric due to rounding.

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Peroxide Fusion ICP continued											
Y, ppm	17.6	0.95	15.7	19.5	14.7	20.4	5.43%	10.86%	16.28%	16.7	18.4
Yb, ppm	1.05	0.065	0.92	1.19	0.86	1.25	6.19%	12.38%	18.56%	1.00	1.11
Zn, wt.%	3.20	0.082	3.04	3.36	2.95	3.45	2.56%	5.12%	7.68%	3.04	3.36
Infrared Combustion											
S, wt.%	3.91	0.112	3.69	4.13	3.58	4.25	2.85%	5.70%	8.56%	3.72	4.11

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

Note 1: intervals may appear asymmetric due to rounding.

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

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Calgary, Alberta, Canada
3. Alex Stewart International, Mendoza, Argentina
4. ALS, Johannesburg, South Africa
5. ALS, Lima, Peru
6. ALS, Loughrea, Galway, Ireland
7. ALS, Malaga, WA, Australia
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ANSTO, Lucas Heights, NSW, Australia
11. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
12. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
13. Bureau Veritas Geoscientific, Perth, WA, Australia
14. Bureau Veritas Minerals, Ankara, Central Anatolia, Turkey
15. CERTIMIN, Lima, Peru
16. ESAN Istanbul, Istanbul, Turkey
17. Inspectorate (BV), Lima, Peru
18. Intertek Genalysis, Perth, WA, Australia
19. Intertek Testing Services, Townsville, QLD, Australia
20. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
21. Koza Gold (Ovacik Gold Mine), Bergama, Izmir, Turkey
22. MSALABS, Vancouver, BC, Canada
23. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
24. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
25. SGS, Ankara, Anatolia, Turkey
26. SGS Canada Inc., Vancouver, BC, Canada
27. SGS de Mexico SA de CV, Cd. Industrial, Durango, Mexico
28. SGS del Peru, Lima, Peru
29. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
30. Skyline Assayers & Laboratories, Tucson, Arizona, USA
31. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

Please note: To preserve anonymity, the above numbered alphabetical list of participating laboratories does not correspond with the Lab ID numbering on the scatter plots below.

Figure 1. Au by Pb Fire Assay in OREAS 625

SPC.1616.RR1.OREAS 625.2.Fire Assay.Au.Lab.240118.221817.SS

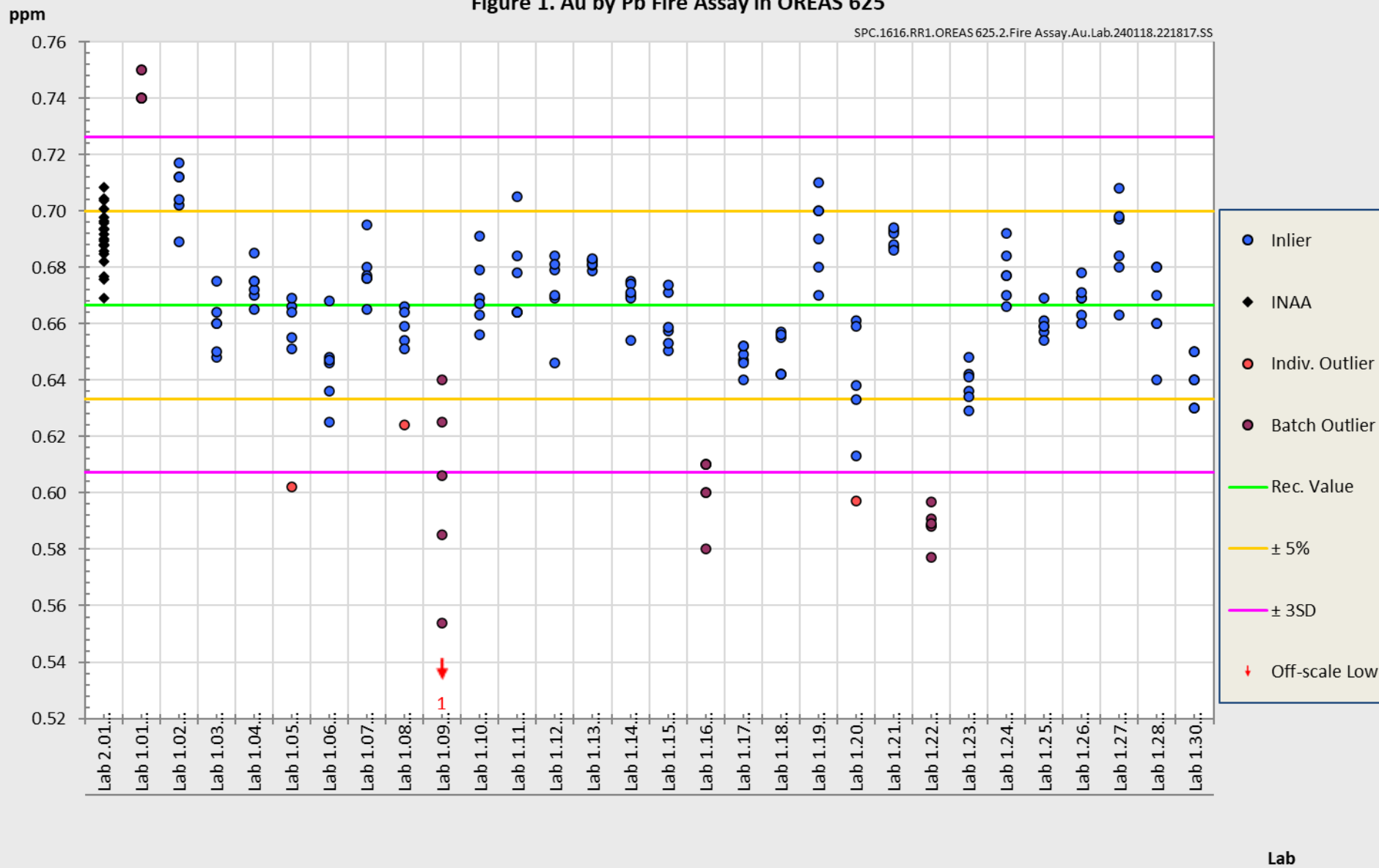


Figure 2. Cu by 4-Acid Digestion in OREAS 625

SPC.1616.RR1.OREAS 625.2.4-Acid.Cu.Lab.231215.171647.SN

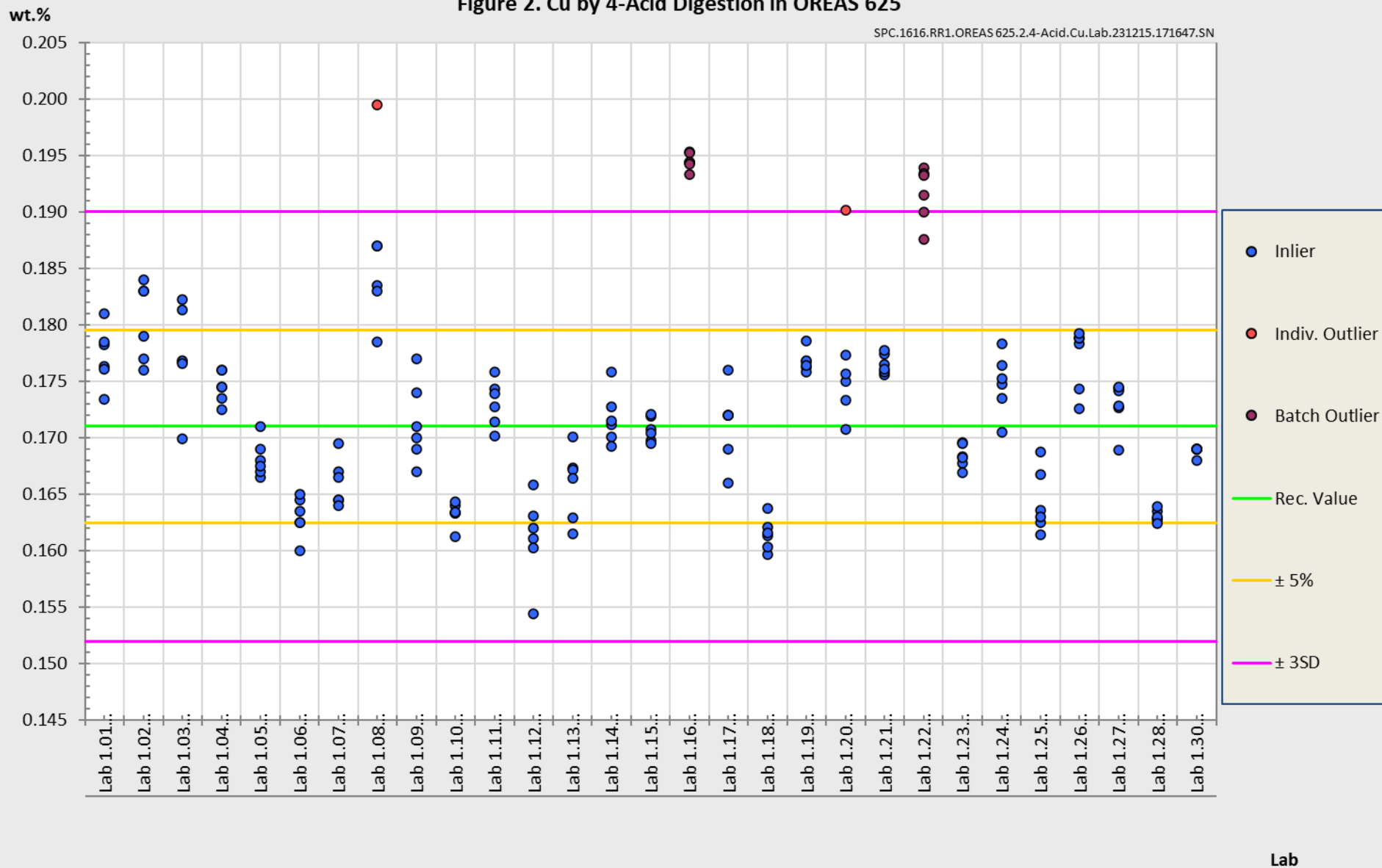


Figure 3. Pb by 4-Acid Digestion in OREAS 625

SPC.1616.RR1.OREAS 625.2.4-Acid.Pb.Lab.240118.222432.SS

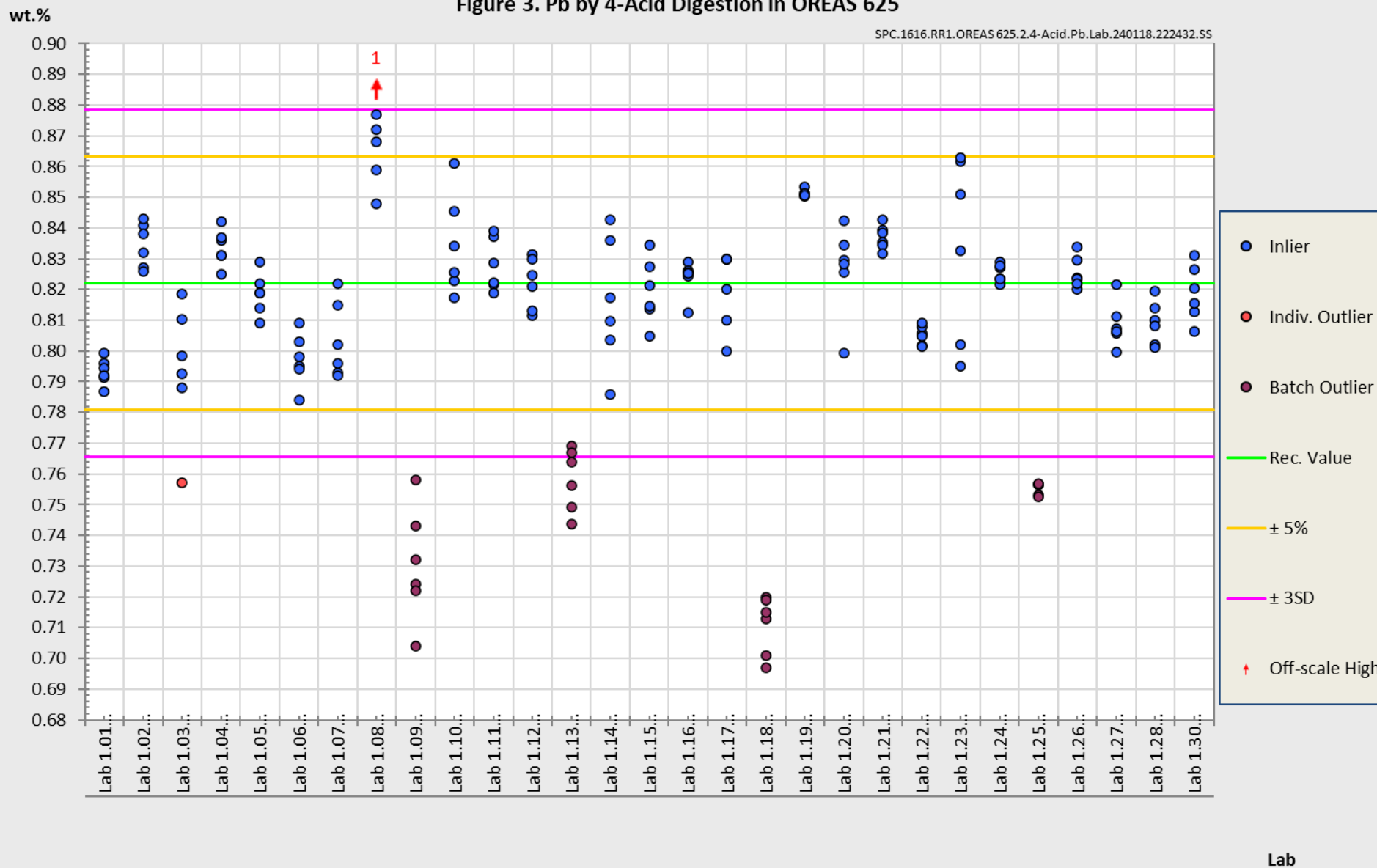
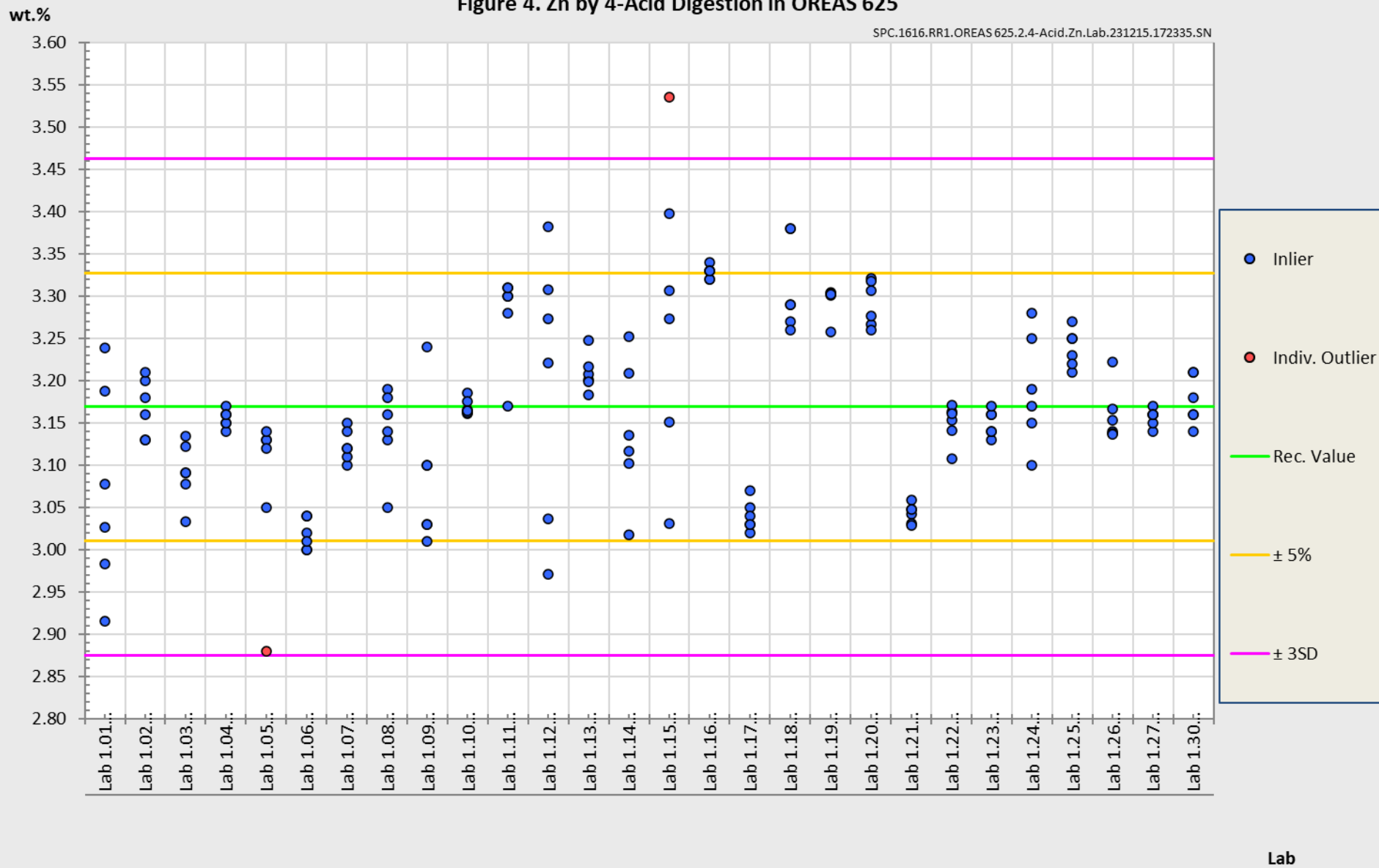


Figure 4. Zn by 4-Acid Digestion in OREAS 625

SPC.1616.RR1.OREAS 625.2.4-Acid.Zn.Lab.231215.172335.SN



PREPARER AND SUPPLIER

Certified reference material OREAS 625 is prepared, certified and supplied by:



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

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

METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified values are metrologically traceable to the international measurement scale (SI) of mass (either as a % mass fraction or as milligrams per kilogram (mg/kg)). In line with popular use, all data within tables in this certificate are expressed as the mass fraction in either weight percent (wt.%) or parts per million (ppm).

The analytical samples sent to participating laboratories were selected in a manner to be representative of the entire prepared batch of CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [10], each analytical data set received from the participating laboratories has been validated by its assayer through the inclusion of internal reference materials and QC checks during and post analysis.

The participating laboratories were chosen on the basis of their competence (from past performance in interlaboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. These laboratories are accredited to ISO 17025 for Au by fire assay (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 35:2017, 9.2.4c)."* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (fusion/digestion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to the field samples being analysed.

INTENDED USE

OREAS 625 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 625 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 625 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 and 2 in geological samples;
- For the verification of analytical methods for analytes reported in Tables 1 and 2;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 and 2. When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. Users can determine an approximation of the standard uncertainty by calculating one fourth of the width of the Expanded Uncertainty interval given in this certificate (Expanded Uncertainty intervals are provided in Tables 1 and 2).

MINIMUM SAMPLE SIZE

To relate analytical determinations to the values in this certificate, the minimum mass of sample used should match the typical mass that the laboratories used in the interlaboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Au by fire assay: $\geq 25\text{g}$;
- Au by aqua regia digestion: $\geq 15\text{g}$;
- 4-acid digestion with ICP-OES and/or MS finish: $\geq 0.25\text{g}$;
- Aqua regia digestion with ICP-OES and/or MS finish: $\geq 0.5\text{g}$;
- Peroxide fusion with ICP-OES and/or MS finish: $\geq 0.1\text{g}$;
- Total Sulphur by infrared combustion furnace/CS analyser: $\geq 0.1\text{g}$.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 625 remains valid, within the specified measurement uncertainties, until November 2033, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

Store in a clean and cool dry place away from direct sunlight.

Long-term stability will be monitored at appropriate intervals and purchasers notified if any changes are observed. The period of validity may well be indefinite and will be reassessed prior to expiry with the aim of extending the validity if possible.

Single-use sachets sealed under nitrogen (10g and 60g units)

OREAS 625 contains sulphides (3.91 wt.% Total Sulphur by infrared combustion furnace) and is packaged in single-use sachets sealed under nitrogen. Following analysis, it is the manufacturer's expectation that any remaining material is discarded unless the sachet is promptly resealed under nitrogen or vacuum. It is the user's responsibility to prevent contamination and minimise exposure to the atmosphere to avoid oxidation of the sulphide minerals.

INSTRUCTIONS FOR HANDLING & CORRECT USE

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [13].

Fine powders pose a risk to eyes and lungs and therefore standard precautions including the use of safety glasses and dust masks are advised.

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

The performance gates shown in Table 6 are intended only to be used as a preliminary guide as to what a laboratory may be able to achieve. Over a period of time monitoring your own laboratory's data for this CRM, SD's should be calculated directly from your own laboratory's process. This will enable you to establish more specific performance gates that are fit for purpose for your application as well as the ability to monitor bias. If your long-term trend analysis shows an average value that is within the 95% expanded uncertainty then generally there is no cause for concern in regard to bias.

For use with the aqua regia digestion method

It is important to note that in the analytical industry there is no standardisation of the aqua regia digestion process. This method is a partial empirical digest and differences in recoveries for various analytes are commonplace. These are caused by variations in the digest conditions and can include the ratio of nitric to hydrochloric acids, acid strength, temperatures, leach times and secondary digestions. Recoveries for sulphide-hosted base metal sulphides approach total values, however, other analytes, in particular the lithophile elements, show greater sensitivity to method parameters. This can result in lack of consensus in an inter-laboratory certification program for these elements.

The approach applied here is to report certified values in those instances where reasonable agreement exists amongst a majority of participating laboratories. The results of specific laboratories may differ significantly from the certified values, but will, nonetheless, be valid and reproducible in the context of the specifics of the aqua regia method in use. Users of this reference material should, therefore, be mindful of this limitation when applying the certified values in a quality control program.

LEGAL NOTICE

Ore Research & Exploration Pty Ltd has prepared and statistically evaluated the property values of this reference material to the best of its ability. The Purchaser by receipt hereof releases and indemnifies Ore Research & Exploration Pty Ltd from and against all liability and costs arising from the use of this material and information.

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DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	25 th January, 2024	First publication.

CERTIFYING OFFICER



25th January, 2024

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

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016.



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.



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