

CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 601d

High Sulphidation Epithermal Ag-Cu-Au Ore (Mt Carlton Mine, Queensland, Australia)

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by fire assay in OREAS 601d.

Constituent	Certified Value [†]	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Pb Fire assay					
Au, Gold (ppm)	0.817	0.804	0.831	0.803*	0.831*

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 30 g fire assay are determined from 20 x 1 g INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.



Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 601d.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion (sample weights 10-50 g)					
Au, Gold (ppm)	0.815	0.796	0.834	0.800	0.830
4-Acid Digestion					
Ag, Silver (ppm)	50.4	48.7	52.1	49.5	51.3
Al, Aluminium (wt.%)	7.13	6.89	7.38	7.00	7.27
As, Arsenic (ppm)	683	658	708	669	698
Ba, Barium (wt.%)	0.205	0.196	0.213	0.200	0.209
Be, Beryllium (ppm)	2.38	2.28	2.48	2.30	2.46
Bi, Bismuth (ppm)	23.0	22.0	24.0	22.4	23.6
Ca, Calcium (wt.%)	0.738	0.712	0.764	0.720	0.755
Cd, Cadmium (ppm)	9.70	9.34	10.07	9.47	9.94
Ce, Cerium (ppm)	74	70	78	72	76
Co, Cobalt (ppm)	4.02	3.80	4.25	3.88	4.17
Cr, Chromium (ppm)	18.2	16.4	19.9	17.0	19.3
Cs, Caesium (ppm)	5.81	5.57	6.05	5.67	5.95
Cu, Copper (wt.%)	0.107	0.104	0.109	0.105	0.109
Dy, Dysprosium (ppm)	3.10	2.82	3.38	3.02	3.18
Er, Erbium (ppm)	1.14	1.09	1.18	1.12	1.15
Eu, Europium (ppm)	1.30	1.19	1.41	1.26	1.35
Fe, Iron (wt.%)	2.39	2.32	2.46	2.33	2.45
Ga, Gallium (ppm)	20.1	19.4	20.8	19.6	20.7
Gd, Gadolinium (ppm)	4.93	4.68	5.18	4.74	5.13
Ge, Germanium (ppm)	0.16	0.11	0.20	IND	IND
Hf, Hafnium (ppm)	4.71	4.50	4.92	4.57	4.85
Ho, Holmium (ppm)	0.45	0.41	0.49	0.44	0.46
In, Indium (ppm)	0.52	0.49	0.56	0.51	0.54
K, Potassium (wt.%)	4.30	4.14	4.47	4.17	4.43
La, Lanthanum (ppm)	37.6	35.7	39.5	36.3	39.0
Li, Lithium (ppm)	26.8	25.7	27.8	26.2	27.3
Lu, Lutetium (ppm)	0.12	0.10	0.14	IND	IND
Mg, Magnesium (wt.%)	0.168	0.161	0.176	0.166	0.171
Mn, Manganese (wt.%)	0.025	0.024	0.026	0.025	0.026
Mo, Molybdenum (ppm)	7.32	6.85	7.79	7.05	7.59
Na, Sodium (wt.%)	1.63	1.58	1.68	1.60	1.66
Nb, Niobium (ppm)	14.1	13.5	14.8	13.7	14.5
Nd, Neodymium (ppm)	31.9	29.4	34.3	30.8	33.0
Ni, Nickel (ppm)	7.22	6.75	7.68	6.91	7.53
P, Phosphorus (wt.%)	0.039	0.037	0.040	0.038	0.040

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
4-Acid Digestion continued					
Pb, Lead (ppm)	986	958	1015	970	1003
Pr, Praseodymium (ppm)	8.52	7.77	9.27	8.33	8.71
Rb, Rubidium (ppm)	201	195	207	196	206
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.991	0.963	1.019	0.976	1.006
Sb, Antimony (ppm)	40.7	39.0	42.4	39.6	41.9
Sc, Scandium (ppm)	4.88	4.59	5.18	4.73	5.04
Se, Selenium (ppm)	4.05	3.40	4.71	3.67	4.44
Sm, Samarium (ppm)	6.13	5.78	6.49	5.86	6.40
Sn, Tin (ppm)	4.86	4.63	5.08	4.66	5.05
Sr, Strontium (ppm)	224	215	233	220	229
Ta, Tantalum (ppm)	1.06	0.98	1.15	1.02	1.11
Tb, Terbium (ppm)	0.62	0.56	0.67	0.59	0.64
Te, Tellurium (ppm)	4.58	4.26	4.91	4.36	4.81
Th, Thorium (ppm)	12.3	11.7	13.0	12.0	12.7
Ti, Titanium (wt.%)	0.181	0.175	0.186	0.178	0.184
Tl, Thallium (ppm)	3.96	3.80	4.13	3.87	4.05
Tm, Thulium (ppm)	0.15	0.12	0.17	IND	IND
U, Uranium (ppm)	4.24	4.07	4.40	4.10	4.37
V, Vanadium (ppm)	23.5	22.4	24.6	22.5	24.4
W, Tungsten (ppm)	2.77	2.60	2.94	2.65	2.89
Y, Yttrium (ppm)	13.4	12.7	14.1	13.0	13.8
Yb, Ytterbium (ppm)	0.90	0.81	1.00	0.88	0.93
Zn, Zinc (ppm)	973	946	999	958	988
Zr, Zirconium (ppm)	164	158	169	160	167
Aqua Regia Digestion					
Ag, Silver (ppm)	50.0	48.7	51.4	49.3	50.7
Al, Aluminium (wt.%)	0.633	0.607	0.660	0.620	0.647
As, Arsenic (ppm)	678	662	695	668	689
B, Boron (ppm)	< 10	IND	IND	IND	IND
Ba, Barium (ppm)	193	179	207	189	197
Be, Beryllium (ppm)	0.58	0.55	0.60	0.55	0.60
Bi, Bismuth (ppm)	23.6	22.7	24.6	23.0	24.2
Ca, Calcium (wt.%)	0.575	0.559	0.590	0.565	0.584
Cd, Cadmium (ppm)	9.59	9.21	9.98	9.34	9.84
Ce, Cerium (ppm)	38.3	35.8	40.9	37.3	39.3
Co, Cobalt (ppm)	3.60	3.41	3.80	3.46	3.74

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					
Cr, Chromium (ppm)	17.0	15.9	18.0	16.5	17.4
Cs, Caesium (ppm)	1.48	1.41	1.55	1.42	1.53
Cu, Copper (wt.%)	0.107	0.105	0.110	0.106	0.109
Dy, Dysprosium (ppm)	1.51	1.31	1.70	1.48	1.53
Er, Erbium (ppm)	0.48	0.42	0.53	0.45	0.50
Eu, Europium (ppm)	0.55	0.42	0.68	0.53	0.57
Fe, Iron (wt.%)	1.92	1.87	1.97	1.89	1.96
Ga, Gallium (ppm)	3.17	2.94	3.39	3.04	3.29
Gd, Gadolinium (ppm)	2.55	2.22	2.89	2.49	2.62
Hf, Hafnium (ppm)	0.88	0.83	0.94	0.85	0.92
Hg, Mercury (ppm)	0.16	0.14	0.18	0.13	0.18
Ho, Holmium (ppm)	0.22	0.19	0.25	0.21	0.23
In, Indium (ppm)	0.46	0.43	0.49	0.44	0.48
K, Potassium (wt.%)	0.352	0.337	0.367	0.340	0.364
La, Lanthanum (ppm)	19.1	17.7	20.5	18.5	19.7
Li, Lithium (ppm)	6.07	5.70	6.44	5.89	6.26
Lu, Lutetium (ppm)	0.044	0.037	0.051	IND	IND
Mg, Magnesium (wt.%)	0.086	0.082	0.089	0.083	0.088
Mn, Manganese (wt.%)	0.020	0.020	0.021	0.020	0.021
Mo, Molybdenum (ppm)	6.72	6.39	7.05	6.52	6.92
Na, Sodium (wt.%)	0.046	0.043	0.048	0.044	0.047
Nd, Neodymium (ppm)	16.3	13.0	19.5	15.5	17.0
Ni, Nickel (ppm)	6.91	6.48	7.34	6.63	7.19
P, Phosphorus (wt.%)	0.027	0.026	0.028	0.027	0.028
Pb, Lead (ppm)	930	904	956	916	944
Rb, Rubidium (ppm)	17.6	16.5	18.8	17.1	18.1
Re, Rhenium (ppm)	0.001	0.000	0.002	IND	IND
S, Sulphur (wt.%)	0.848	0.823	0.872	0.835	0.860
Sb, Antimony (ppm)	32.2	30.3	34.2	31.4	33.1
Sc, Scandium (ppm)	1.22	1.09	1.35	1.16	1.27
Se, Selenium (ppm)	3.70	3.38	4.01	3.51	3.88
Sm, Samarium (ppm)	3.13	2.72	3.55	2.99	3.28
Sn, Tin (ppm)	2.27	2.14	2.40	2.16	2.38
Sr, Strontium (ppm)	40.0	37.4	42.7	38.9	41.1
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.33	0.29	0.36	0.31	0.34
Te, Tellurium (ppm)	4.49	4.25	4.72	4.28	4.69

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					
Th, Thorium (ppm)	6.34	5.94	6.75	6.04	6.65
Ti, Titanium (wt.%)	0.011	0.010	0.012	0.011	0.012
Tl, Thallium (ppm)	1.10	1.06	1.14	1.07	1.13
U, Uranium (ppm)	1.90	1.80	2.01	1.85	1.96
V, Vanadium (ppm)	6.46	5.92	7.01	6.23	6.69
W, Tungsten (ppm)	0.92	0.81	1.02	0.87	0.96
Y, Yttrium (ppm)	5.88	5.56	6.20	5.72	6.04
Yb, Ytterbium (ppm)	0.34	0.30	0.37	0.31	0.36
Zn, Zinc (ppm)	940	917	964	928	953
Zr, Zirconium (ppm)	32.8	31.2	34.5	31.8	33.9
Infrared Combustion					
S, Sulphur (wt.%)	0.979	0.955	1.003	0.965	0.993
Alkaline Leach					
S-(Sulphate), Sulphur as SO ₄ ²⁻ (wt.%)	0.294	0.246	0.342	0.277	0.311
S-(Sulphide), Sulphur as S ²⁻ (wt.%)	0.641	0.564	0.718	0.615	0.667

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 601d.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Aqua Regia Digestion (sample weights 10-50 g)								
Pt	ppb	20.0	Ru	ppm	< 0.005	Pt	ppb	20.0
4-Acid Digestion								
B	ppm	8.67	Hg	ppm	0.15			
Aqua Regia Digestion								
Ge	ppm	0.082	Pr	ppm	4.53	Tm	ppm	0.058
Nb	ppm	0.36	Pt	ppb	20.0			
Pd	ppb	14.7	Ru	ppm	< 0.005			
Infrared Combustion								
C	wt.%	0.120						
Borate Fusion XRF								
Al ₂ O ₃	wt.%	14.07	MgO	wt.%	0.310	S	wt.%	1.02
CaO	wt.%	1.05	MnO	wt.%	0.030	SiO ₂	wt.%	69.67
Fe ₂ O ₃	wt.%	3.54	Na ₂ O	wt.%	2.25	TiO ₂	wt.%	0.320
K ₂ O	wt.%	5.43	P ₂ O ₅	wt.%	0.089			
Thermogravimetry								
LOI ¹⁰⁰⁰	wt.%	2.51						

SI unit equivalents: ppb (parts per billion; 1×10^{-9}) \equiv µg/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
Laser Ablation ICP-MS								
Ag	ppm	60.1	Hf	ppm	6.21	Sn	ppm	5.00
As	ppm	678	Ho	ppm	0.52	Sr	ppm	226
Ba	ppm	2080	In	ppm	0.50	Ta	ppm	1.09
Be	ppm	2.70	La	ppm	36.9	Tb	ppm	0.68
Bi	ppm	24.8	Lu	ppm	0.15	Te	ppm	5.30
Cd	ppm	10.6	Mn	wt. %	0.025	Th	ppm	12.9
Ce	ppm	72	Mo	ppm	6.60	Ti	wt. %	0.197
Co	ppm	4.15	Nb	ppm	13.9	Tl	ppm	4.70
Cr	ppm	12.5	Nd	ppm	32.8	Tm	ppm	0.16
Cs	ppm	5.82	Ni	ppm	7.00	U	ppm	4.39
Cu	ppm	1120	Pb	ppm	1050	V	ppm	24.1
Dy	ppm	3.28	Pr	ppm	8.77	W	ppm	3.25
Er	ppm	1.23	Rb	ppm	198	Y	ppm	13.8
Eu	ppm	1.22	Re	ppm	0.008	Yb	ppm	0.95
Ga	ppm	19.6	Sb	ppm	42.9	Zn	ppm	990
Gd	ppm	4.87	Sc	ppm	5.45	Zr	ppm	224
Ge	ppm	1.83	Sm	ppm	6.49			

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.

TABLE OF CONTENTS

INTRODUCTION	8
SOURCE MATERIAL	8
COMMUNITION AND HOMOGENISATION PROCEDURES	8
PHYSICAL PROPERTIES.....	9
MINERALOGY	9
ANALYTICAL PROGRAM	9
STATISTICAL ANALYSIS	10
Homogeneity Evaluation.....	11
PERFORMANCE GATES	14
PREPARER	17
PARTICIPATING LABORATORIES.....	18
METROLOGICAL TRACEABILITY	22
COMMUTABILITY	22
INTENDED USE.....	23
MINIMUM SAMPLE SIZE.....	23
PERIOD OF VALIDITY & STORAGE INSTRUCTIONS	23
INSTRUCTIONS FOR HANDLING & CORRECT USE	24
LEGAL NOTICE.....	25
DOCUMENT HISTORY.....	25
QMS CERTIFICATION.....	25
CERTIFYING OFFICER.....	26
REFERENCES	26

LIST OF TABLES

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by fire assay in OREAS 601d.....	1
Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 601d.....	2
Table 3. Indicative Values for OREAS 601d.....	5
Table 4. Physical properties of OREAS 601d.....	9
Table 5. Indicative mineralogy of OREAS 601d based on semi-quantitative XRD analysis.....	9
Table 6. Neutron Activation Analysis of Au on 20 x 1 g subsamples.....	12
Table 7. Performance Gates for OREAS 601d.....	14

LIST OF FIGURES

Figure 1. Au by fire assay in OREAS 601d	19
Figure 2. Ag by 4-acid digestion in OREAS 601d.....	20
Figure 3. Cu by 4-acid digestion in OREAS 601d.....	21

INTRODUCTION

Reference materials are intended to provide a method of evaluating and improving the quality of analysis of geological and downstream metallurgical samples. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS prepared 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.

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 601d-DataPack.1.0.250217_010802.xlsx**).

Results are also presented in scatter plots for Au by fire assay and Ag and Cu by 4-acid digestion method in Figures 1 to 3 respectively, together with $\pm 3SD$ (magenta) and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

SOURCE MATERIAL

OREAS 601d was prepared from a blend of silver-copper-gold ores, high-grade silver ore rhyodacite and a minor addition of copper-gold concentrate. The gold-silver-copper ores and concentrate were sourced from Evolution Mining's Mount Carlton Operation in Queensland, Australia. The mineralisation assemblage at Mount Carlton consists of pyrite, enargite/tennantite, tetrahedrite, digenite, covellite, sphalerite, galena, alunite, dickite, kaolinite and vuggy silica, hosted in advanced argillic altered rhyodacite containing sulphur-salts.

The high-grade silver ore was sourced from the Bowdens Silver Project that hosts low- to intermediate-sulphidation epithermal mineralisation within Silurian volcanic-sedimentary rocks, featuring silver, lead, zinc, and minor gold in veins, breccias, and disseminations, structurally controlled within altered rhyolites and sediments.

The rhyodacite was sourced from the Mt Dandenong Igneous Complex located approximately 34km east of Melbourne (Victoria), Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 601d was prepared in the following manner:

- Drying of ores and barren rhyodacite to constant mass at 105 °C;
- Drying of concentrate to constant mass at 85 °C;
- Multi-stage milling of ores and concentrate to 100 % minus 30 µm;
- Milling of barren rhyodacite to > 98 % minus 75 µm;
- Combining materials in appropriate proportions to achieve target grades;
- Homogenisation using OREAS' novel processing technologies;

- Packaging into 10 and 60 g units in laminated foil pouches and 500 g units in plastic jars.

PHYSICAL PROPERTIES

OREAS 601d 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 601d.

Bulk Density (kg/m ³)	Moisture (wt.%)	Munsell Notation [‡]	Munsell Colour [‡]
795	0.37	N8	Very Light Gray

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

MINERALOGY

The semi-quantitative XRD results shown in Table 5 below were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. Calcite and/or chalcopryrite and molybdenite and/or boehmite are reported together due to pattern overlaps. Trace amounts of gypsum and amorphous material might be present.

Table 5. Indicative mineralogy of OREAS 601d based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Chlorite	3
Kandite group	3
Annite - biotite - phlogopite	2
Muscovite and/or illite	5
Ca amphibole	-
Plagioclase	16
K-feldspar	10
Quartz	54
Pyrite	4
Hematite	1
Molybdenite and/or boehmite	-
Calcite and/or chalcopryrite	2
Alunite	-

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 (10-50 g charge weight) with AAS (21 laboratories), ICP-OES (6 laboratories) finish and ICP-MS (2 laboratories) finish;

- Gold via 15-50 g aqua regia digestion with ICP-MS finish (14 laboratories) and AAS (8 laboratories) finish;
- Full ICP-OES and MS elemental suites by 4-acid digestion (up to 29 laboratories depending on the element);
- Full ICP-OES and MS elemental suites by aqua regia digestion (up to 29 laboratories depending on the element);
- Total S by infrared combustion furnace (28 laboratories);
- Sulphate S by Na_2CO_3 leach of sulphates, precipitation as barium sulphate with gravimetric finish or by difference using the Total S value minus the Sulphide S (21 laboratories);
- Sulphide S by Na_2CO_3 leach of sulphates with IR combustion furnace (Leco) or by difference using the Total S value minus the Sulphate S (21 laboratories).

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:

- Whole Rock analysis by borate fusion XRF method;
- Laser Ablation Package by fused bead Laser Ablation with ICP-MS finish method;
- Infrared combustion furnace for C.

To evaluate homogeneity, Actlabs Ancaster in Canada were sent 20 x 10 g pulp samples for Au determination using instrumental neutron activation analysis (INAA) on 1.0 g subsamples. The 20 samples were comprised of paired samples from 10 of the 12 sampling intervals and were randomised prior to assigning sample numbers. The paired samples enabled an Analysis of Variance (ANOVA) by comparison of within- and between-unit variances across the 10 pairs (see 'Homogeneity Evaluation' below).

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Table 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.

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 [6] and [16]. 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.

Standard Deviation intervals (see Table 7) 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. 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*** (see 'Instructions for handling and correct use' section for more detail).

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 ($p=0.95$) will have concentrations lying between 0.105 and 0.109 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 33405:2024). ***Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.***

The homogeneity of gold has been determined by INAA at Actlabs 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. Table 6 below shows the gold INAA data determined on 20 x 1 g subsamples of OREAS 601d. 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 0.53 % calculated for a 30g fire assay sample (2.84% at 1 g weights) confirms the high level of gold homogeneity in OREAS 601d.

The homogeneity of OREAS 601d 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 601d. 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 0.56, a statistically insignificant result so the Null Hypothesis is accepted.

Table 6. Neutron Activation Analysis of Au (in ppm) on 20 x 1 g subsamples and showing the equivalent results scaled to a 30 g sample mass typical of fire assay determination.

Replicate No	Au 1 g actual	Au 30 g equivalent*
1	0.826	0.833
2	0.812	0.830
3	0.808	0.829
4	0.832	0.834
5	0.793	0.827
6	0.837	0.835
7	0.823	0.832
8	0.815	0.831
9	0.801	0.828
10	0.815	0.831
11	0.810	0.830
12	0.860	0.839
13	0.866	0.840
14	0.853	0.838
15	0.850	0.837
16	0.858	0.839
17	0.870	0.841
18	0.842	0.836
19	0.858	0.839
20	0.854	0.838
Mean	0.834	0.834
Median	0.835	0.834
Std Dev.	0.024	0.004
Rel.Std.Dev.	2.84%	0.53%

*Results calculated for a 30 g equivalent sample mass using the formula: $x^{30g Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@1g} + \bar{X}$

where $x^{30g Eq}$ = equivalent result calculated for a 30 g sample mass

(x^{INAA}) = raw INAA result at 1 g

\bar{X} = mean of 1 g INAA results

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 601d 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 601d is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PERFORMANCE GATES

Table 7 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 7. Performance Gates for OREAS 601d.

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	0.817	0.028	0.760	0.874	0.732	0.902	3.47%	6.95%	10.42%	0.776	0.858
Aqua Regia Digestion (sample weights 10-50g)											
Au, ppm	0.815	0.042	0.731	0.899	0.689	0.941	5.16%	10.31%	15.47%	0.774	0.856
4-Acid Digestion											
Ag, ppm	50.4	2.22	46.0	54.9	43.8	57.1	4.40%	8.81%	13.21%	47.9	52.9
Al, wt.%	7.13	0.317	6.50	7.77	6.18	8.08	4.44%	8.89%	13.33%	6.78	7.49
As, ppm	683	29	626	740	598	769	4.18%	8.35%	12.53%	649	718
Ba, wt.%	0.205	0.013	0.179	0.230	0.166	0.243	6.32%	12.65%	18.97%	0.194	0.215
Be, ppm	2.38	0.141	2.10	2.66	1.96	2.80	5.92%	11.83%	17.75%	2.26	2.50
Bi, ppm	23.0	1.14	20.7	25.3	19.6	26.4	4.94%	9.88%	14.83%	21.9	24.2
Ca, wt.%	0.738	0.037	0.665	0.811	0.628	0.848	4.96%	9.92%	14.88%	0.701	0.775
Cd, ppm	9.70	0.444	8.82	10.59	8.37	11.04	4.57%	9.15%	13.72%	9.22	10.19
Ce, ppm	74	5.9	62	86	57	92	7.90%	15.81%	23.71%	70	78
Co, ppm	4.02	0.309	3.41	4.64	3.10	4.95	7.67%	15.34%	23.01%	3.82	4.23
Cr, ppm	18.2	2.3	13.6	22.7	11.3	25.0	12.57%	25.13%	37.70%	17.3	19.1
Cs, ppm	5.81	0.234	5.34	6.28	5.11	6.51	4.03%	8.05%	12.08%	5.52	6.10
Cu, wt.%	0.107	0.003	0.101	0.112	0.098	0.115	2.62%	5.23%	7.85%	0.101	0.112
Dy, ppm	3.10	0.226	2.65	3.55	2.42	3.77	7.28%	14.57%	21.85%	2.94	3.25
Er, ppm	1.14	0.035	1.07	1.21	1.03	1.24	3.07%	6.13%	9.20%	1.08	1.19
Eu, ppm	1.30	0.13	1.04	1.57	0.91	1.70	10.14%	20.27%	30.41%	1.24	1.37
Fe, wt.%	2.39	0.068	2.25	2.53	2.19	2.59	2.85%	5.71%	8.56%	2.27	2.51

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

IND = indeterminate. 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 7 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											
Ga, ppm	20.1	0.74	18.7	21.6	17.9	22.4	3.70%	7.39%	11.09%	19.1	21.1
Gd, ppm	4.93	0.226	4.48	5.38	4.25	5.61	4.58%	9.16%	13.74%	4.69	5.18
Ge, ppm	0.16	0.06	0.04	0.27	0.00	0.32	36.50%	73.01%	109.5%	0.15	0.16
Hf, ppm	4.71	0.226	4.26	5.16	4.03	5.39	4.80%	9.61%	14.41%	4.48	4.95
Ho, ppm	0.45	0.036	0.38	0.52	0.34	0.56	8.00%	16.01%	24.01%	0.43	0.47
In, ppm	0.52	0.025	0.47	0.57	0.45	0.60	4.83%	9.66%	14.49%	0.50	0.55
K, wt. %	4.30	0.172	3.96	4.65	3.79	4.82	3.99%	7.98%	11.97%	4.09	4.52
La, ppm	37.6	2.39	32.8	42.4	30.5	44.8	6.36%	12.72%	19.07%	35.8	39.5
Li, ppm	26.8	1.17	24.4	29.1	23.2	30.3	4.38%	8.76%	13.14%	25.4	28.1
Lu, ppm	0.12	0.01	0.10	0.15	0.08	0.16	10.49%	20.98%	31.47%	0.11	0.13
Mg, wt. %	0.168	0.009	0.150	0.186	0.142	0.195	5.29%	10.59%	15.88%	0.160	0.177
Mn, wt. %	0.025	0.001	0.023	0.027	0.022	0.028	3.79%	7.58%	11.36%	0.024	0.026
Mo, ppm	7.32	0.488	6.34	8.29	5.85	8.78	6.67%	13.34%	20.00%	6.95	7.68
Na, wt. %	1.63	0.069	1.49	1.77	1.42	1.84	4.21%	8.42%	12.63%	1.55	1.71
Nb, ppm	14.1	0.65	12.8	15.4	12.2	16.1	4.57%	9.14%	13.71%	13.4	14.8
Nd, ppm	31.9	1.55	28.8	35.0	27.2	36.5	4.86%	9.73%	14.59%	30.3	33.5
Ni, ppm	7.22	0.531	6.15	8.28	5.62	8.81	7.36%	14.72%	22.08%	6.86	7.58
P, wt. %	0.039	0.002	0.036	0.042	0.034	0.043	3.92%	7.84%	11.76%	0.037	0.041
Pb, ppm	986	37	913	1060	876	1097	3.74%	7.48%	11.21%	937	1036
Pr, ppm	8.52	0.541	7.44	9.60	6.90	10.14	6.34%	12.69%	19.03%	8.10	8.95
Rb, ppm	201	10	180	222	170	232	5.22%	10.44%	15.66%	191	211
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt. %	0.991	0.035	0.921	1.060	0.887	1.095	3.51%	7.02%	10.53%	0.941	1.040
Sb, ppm	40.7	1.60	37.5	43.9	35.9	45.5	3.94%	7.88%	11.82%	38.7	42.8
Sc, ppm	4.88	0.424	4.04	5.73	3.61	6.16	8.69%	17.38%	26.07%	4.64	5.13
Se, ppm	4.05	0.41	3.23	4.88	2.81	5.30	10.22%	20.43%	30.65%	3.85	4.26
Sm, ppm	6.13	0.251	5.63	6.64	5.38	6.89	4.09%	8.18%	12.27%	5.83	6.44
Sn, ppm	4.86	0.252	4.35	5.36	4.10	5.61	5.19%	10.37%	15.56%	4.61	5.10
Sr, ppm	224	12	201	248	189	259	5.22%	10.43%	15.65%	213	236
Ta, ppm	1.06	0.086	0.89	1.24	0.81	1.32	8.09%	16.18%	24.26%	1.01	1.12
Tb, ppm	0.62	0.06	0.49	0.75	0.43	0.81	10.34%	20.67%	31.01%	0.59	0.65
Te, ppm	4.58	0.321	3.94	5.23	3.62	5.55	7.01%	14.03%	21.04%	4.35	4.81
Th, ppm	12.3	0.84	10.7	14.0	9.8	14.9	6.77%	13.55%	20.32%	11.7	13.0
Ti, wt. %	0.181	0.006	0.169	0.192	0.164	0.198	3.13%	6.26%	9.39%	0.172	0.190
Tl, ppm	3.96	0.223	3.52	4.41	3.29	4.63	5.63%	11.25%	16.88%	3.77	4.16
Tm, ppm	0.15	0.02	0.11	0.18	0.09	0.20	11.83%	23.65%	35.48%	0.14	0.15
U, ppm	4.24	0.148	3.94	4.53	3.79	4.68	3.50%	7.00%	10.50%	4.02	4.45
V, ppm	23.5	0.99	21.5	25.5	20.5	26.5	4.22%	8.45%	12.67%	22.3	24.7
W, ppm	2.77	0.206	2.36	3.18	2.15	3.39	7.44%	14.88%	22.32%	2.63	2.91
Y, ppm	13.4	0.78	11.8	15.0	11.1	15.8	5.85%	11.70%	17.55%	12.7	14.1

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

IND = indeterminate. 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 7 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											
Yb, ppm	0.90	0.073	0.76	1.05	0.68	1.12	8.10%	16.19%	24.29%	0.86	0.95
Zn, ppm	973	33	906	1039	873	1073	3.42%	6.83%	10.25%	924	1021
Zr, ppm	164	7	150	177	143	184	4.19%	8.38%	12.58%	156	172
Aqua Regia Digestion											
Ag, ppm	50.0	1.75	46.5	53.5	44.8	55.3	3.49%	6.98%	10.47%	47.5	52.5
Al, wt. %	0.633	0.048	0.538	0.728	0.491	0.776	7.50%	15.01%	22.51%	0.602	0.665
As, ppm	678	23	633	724	610	747	3.36%	6.72%	10.09%	644	712
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	193	24	146	241	122	264	12.25%	24.49%	36.74%	184	203
Be, ppm	0.58	0.036	0.50	0.65	0.47	0.68	6.29%	12.59%	18.88%	0.55	0.60
Bi, ppm	23.6	1.13	21.3	25.9	20.2	27.0	4.80%	9.60%	14.40%	22.4	24.8
Ca, wt. %	0.575	0.025	0.525	0.624	0.501	0.648	4.28%	8.55%	12.83%	0.546	0.603
Cd, ppm	9.59	0.595	8.40	10.78	7.81	11.38	6.20%	12.39%	18.59%	9.11	10.07
Ce, ppm	38.3	5.0	28.3	48.3	23.3	53.3	13.05%	26.09%	39.14%	36.4	40.2
Co, ppm	3.60	0.300	3.00	4.20	2.70	4.50	8.34%	16.67%	25.01%	3.42	3.78
Cr, ppm	17.0	0.97	15.0	18.9	14.1	19.9	5.72%	11.44%	17.16%	16.1	17.8
Cs, ppm	1.48	0.102	1.27	1.68	1.17	1.78	6.93%	13.87%	20.80%	1.40	1.55
Cu, wt. %	0.107	0.003	0.101	0.113	0.099	0.116	2.74%	5.48%	8.22%	0.102	0.113
Dy, ppm	1.51	0.22	1.07	1.94	0.85	2.16	14.47%	28.95%	43.42%	1.43	1.58
Er, ppm	0.48	0.05	0.38	0.57	0.33	0.62	10.36%	20.73%	31.09%	0.45	0.50
Eu, ppm	0.55	0.11	0.33	0.77	0.22	0.88	19.97%	39.94%	59.91%	0.52	0.58
Fe, wt. %	1.92	0.092	1.74	2.11	1.65	2.20	4.79%	9.58%	14.37%	1.83	2.02
Ga, ppm	3.17	0.39	2.39	3.95	1.99	4.34	12.34%	24.69%	37.03%	3.01	3.33
Gd, ppm	2.55	0.37	1.81	3.29	1.44	3.66	14.52%	29.03%	43.55%	2.43	2.68
Hf, ppm	0.88	0.087	0.71	1.06	0.62	1.15	9.87%	19.75%	29.62%	0.84	0.93
Hg, ppm	0.16	0.014	0.13	0.19	0.12	0.20	8.93%	17.85%	26.78%	0.15	0.17
Ho, ppm	0.22	0.02	0.17	0.27	0.14	0.29	11.15%	22.30%	33.45%	0.21	0.23
In, ppm	0.46	0.026	0.41	0.51	0.38	0.54	5.62%	11.23%	16.85%	0.44	0.48
K, wt. %	0.352	0.024	0.304	0.401	0.280	0.425	6.88%	13.76%	20.64%	0.335	0.370
La, ppm	19.1	2.6	14.0	24.3	11.4	26.9	13.45%	26.91%	40.36%	18.2	20.1
Li, ppm	6.07	0.72	4.64	7.51	3.92	8.23	11.82%	23.64%	35.45%	5.77	6.38
Lu, ppm	0.044	0.006	0.033	0.055	0.028	0.061	12.53%	25.05%	37.58%	0.042	0.046
Mg, wt. %	0.086	0.005	0.076	0.095	0.071	0.100	5.70%	11.41%	17.11%	0.081	0.090
Mn, wt. %	0.020	0.001	0.019	0.022	0.018	0.023	4.69%	9.38%	14.08%	0.019	0.021
Mo, ppm	6.72	0.451	5.82	7.62	5.37	8.07	6.71%	13.42%	20.13%	6.38	7.06
Na, wt. %	0.046	0.006	0.033	0.058	0.027	0.064	13.53%	27.06%	40.59%	0.043	0.048
Nd, ppm	16.3	3.2	9.9	22.7	6.7	25.9	19.65%	39.30%	58.94%	15.5	17.1
Ni, ppm	6.91	0.428	6.05	7.76	5.62	8.19	6.20%	12.39%	18.59%	6.56	7.25
P, wt. %	0.027	0.002	0.024	0.031	0.022	0.033	6.44%	12.87%	19.31%	0.026	0.029
Pb, ppm	930	30	870	991	840	1021	3.24%	6.49%	9.73%	884	977

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

IND = indeterminate. 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 7 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											
Rb, ppm	17.6	2.1	13.4	21.8	11.3	24.0	11.99%	23.98%	35.98%	16.7	18.5
Re, ppm	0.001	0.001	0.000	0.002	0.000	0.003	49.97%	99.95%	149.9%	0.001	0.001
S, wt. %	0.848	0.036	0.776	0.919	0.740	0.955	4.21%	8.43%	12.64%	0.805	0.890
Sb, ppm	32.2	3.5	25.2	39.2	21.7	42.7	10.85%	21.71%	32.56%	30.6	33.9
Sc, ppm	1.22	0.18	0.86	1.58	0.67	1.76	14.90%	29.80%	44.70%	1.16	1.28
Se, ppm	3.70	0.283	3.13	4.26	2.85	4.55	7.67%	15.33%	23.00%	3.51	3.88
Sm, ppm	3.13	0.45	2.23	4.04	1.78	4.49	14.40%	28.80%	43.20%	2.98	3.29
Sn, ppm	2.27	0.167	1.93	2.60	1.77	2.77	7.35%	14.70%	22.05%	2.15	2.38
Sr, ppm	40.0	4.9	30.1	49.9	25.2	54.9	12.36%	24.71%	37.07%	38.0	42.0
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.33	0.04	0.24	0.41	0.20	0.45	13.13%	26.25%	39.38%	0.31	0.34
Te, ppm	4.49	0.281	3.92	5.05	3.64	5.33	6.26%	12.53%	18.79%	4.26	4.71
Th, ppm	6.34	0.73	4.88	7.81	4.15	8.54	11.53%	23.07%	34.60%	6.03	6.66
Ti, wt. %	0.011	0.002	0.007	0.015	0.005	0.017	18.02%	36.04%	54.06%	0.011	0.012
Tl, ppm	1.10	0.051	1.00	1.21	0.95	1.26	4.67%	9.34%	14.01%	1.05	1.16
U, ppm	1.90	0.166	1.57	2.24	1.40	2.40	8.74%	17.49%	26.23%	1.81	2.00
V, ppm	6.46	0.80	4.86	8.06	4.07	8.86	12.36%	24.73%	37.09%	6.14	6.79
W, ppm	0.92	0.18	0.56	1.27	0.38	1.45	19.63%	39.26%	58.89%	0.87	0.96
Y, ppm	5.88	0.456	4.97	6.79	4.51	7.25	7.76%	15.53%	23.29%	5.59	6.17
Yb, ppm	0.34	0.05	0.24	0.43	0.20	0.48	14.00%	28.01%	42.01%	0.32	0.36
Zn, ppm	940	36	869	1012	833	1047	3.80%	7.59%	11.39%	893	987
Zr, ppm	32.8	2.76	27.3	38.4	24.6	41.1	8.40%	16.81%	25.21%	31.2	34.5
Infrared Combustion (wt.%)											
S	0.979	0.029	0.920	1.038	0.891	1.067	3.00%	6.00%	8.99%	0.930	1.028
Alkaline Leach (wt.%)											
S-(Sulphate)	0.294	0.091	0.112	0.476	0.021	0.566	30.90%	61.81%	92.71%	0.279	0.309
S-(Sulphide)	0.641	0.134	0.373	0.909	0.238	1.043	20.93%	41.86%	62.79%	0.609	0.673

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

IND = indeterminate. 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.

PREPARER

Certified reference material OREAS 601d is prepared and certified by:

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

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

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. Alex Stewart International, Mendoza, Argentina
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Malaga, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
11. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
12. Bureau Veritas Geoanalytical, Perth, WA, Australia
13. CERTIMIN, Lima, Peru
14. ESAN Istanbul, Istanbul, Turkey
15. Inspectorate (BV), Lima, Peru
16. Intertek, Cupang, Muntinlupa, Philippines
17. Intertek, Perth, WA, Australia
18. Intertek Genalysis, Adelaide, SA, Australia
19. Intertek Minerals Ltd, Tarkwa, Western Region, Ghana
20. Paragon Geochemical Laboratories, Sparks, Nevada, USA
21. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
22. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
23. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
24. SGS Australia Mineral Services, Perth, WA, Australia
25. SGS Canada Inc., Vancouver, BC, Canada
26. SGS del Peru, Lima, Peru
27. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
28. SGS Mineral Services, Townsville, QLD, Australia
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 numbered alphabetical list of participating laboratories above does not correspond to the Lab ID numbering used in the scatter plots below or in the DataPack.

Figure 1. Au by Pb Fire Assay in OREAS 601d

SPC.1659.RR1.OREAS 601d.2.Fire Assay.Au.Lab.250216.211829.SN

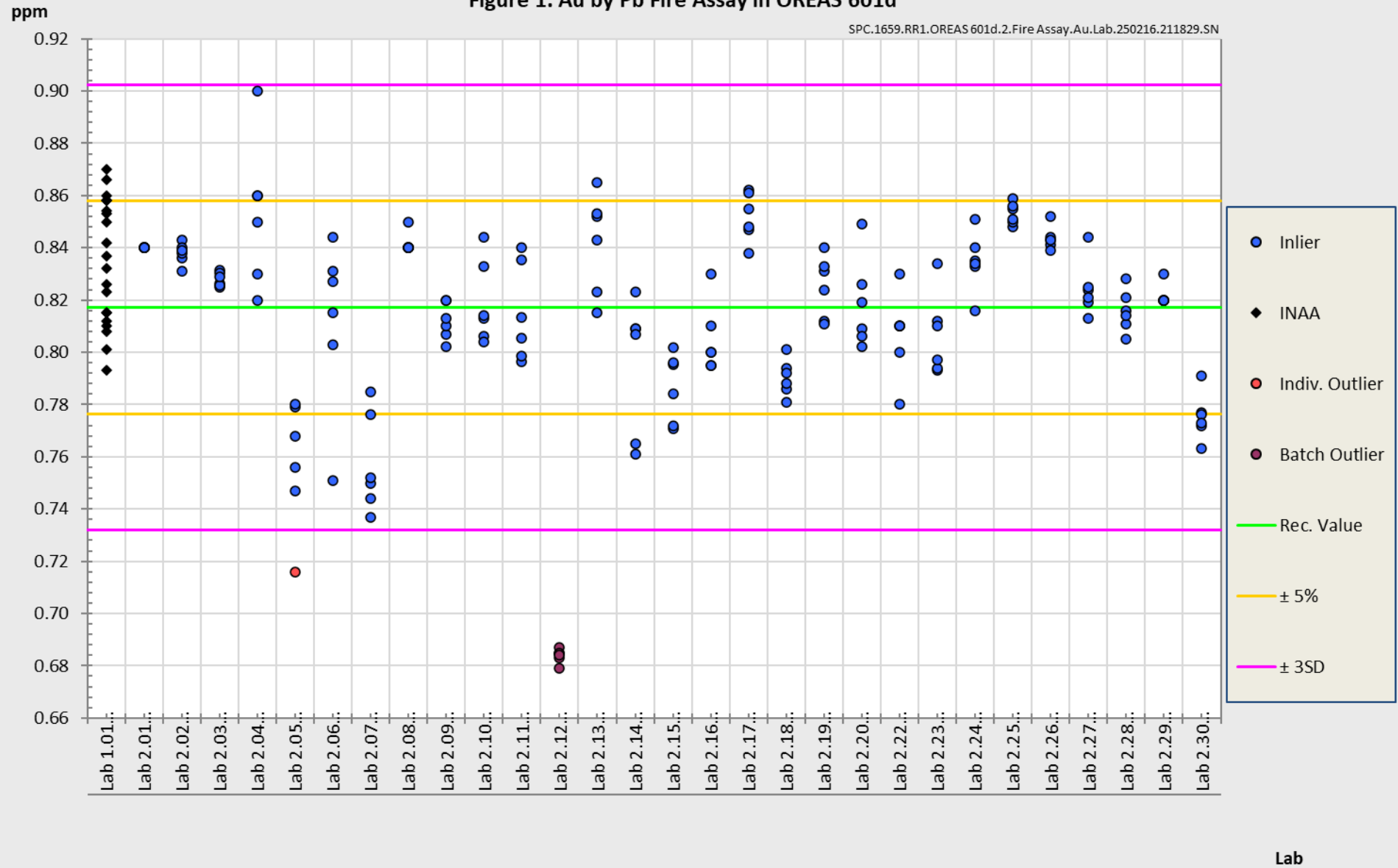
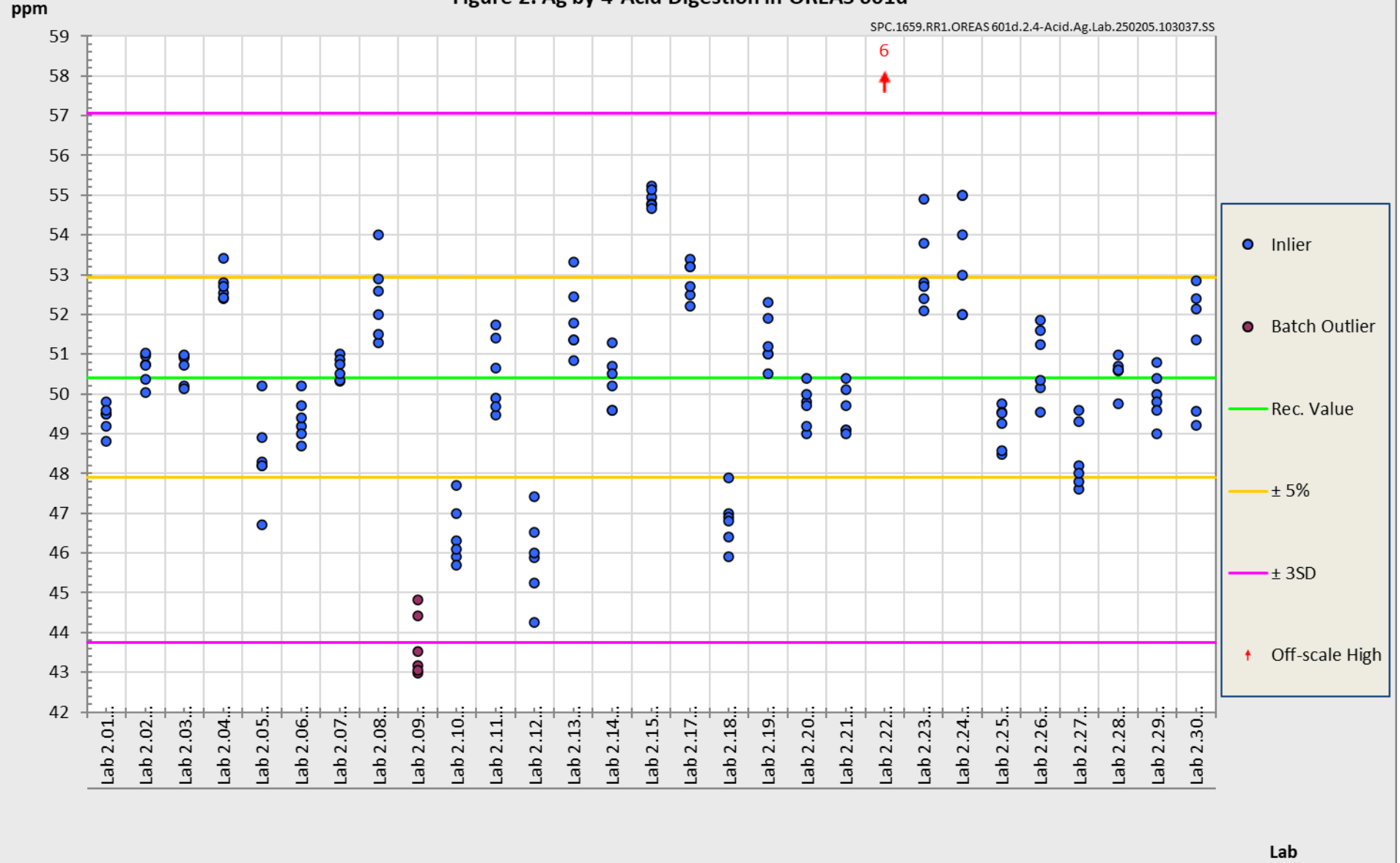


Figure 2. Ag by 4-Acid Digestion in OREAS 601d



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)) [15]. 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 representativeness 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 [8], 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) [5].”* 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 601d 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 601d may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 601d 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 validation/ 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: ≥ 25 g;
- Au by aqua regia digestion with ICP-OES and/or MS finish: ≥ 10 g;
- 4-acid digestion with ICP-OES and/or MS finish: ≥ 0.25 g;
- Aqua regia digestion with ICP-OES and/or MS finish: ≥ 0.5 g;
- Total Sulphur by infrared combustion furnace/CS analyser: ≥ 0.1 g;
- Sulphate Sulphur via various leaching methods: ≥ 0.1 g;
- Sulphide Sulphur via various leaching methods: ≥ 0.1 g.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 601d remains valid, within the specified measurement uncertainties, until June 2039, 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

OREAS 601d is relatively low in Sulphur (0.98 wt.% S) and is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

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

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 601d contains a non-hygroscopic* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate. The risk to stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration.

*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorbed moisture (weakly held H₂O molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2 mm thick) layer and left exposed for a period of 2 hours.

INSTRUCTIONS FOR HANDLING & CORRECT USE

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

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

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 7 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 interval, then generally there is no cause for concern in regard to bias.

For use with the aqua regia digestion method

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

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

LEGAL NOTICE

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

© COPYRIGHT Ore Research & Exploration Pty Ltd.
Unauthorised copying, reproduction, storage or dissemination is prohibited.

DOCUMENT HISTORY

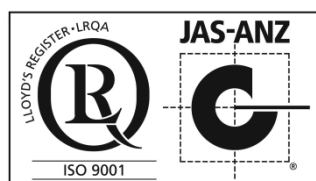
Revision No.	Date	Changes applied
1	9 th May, 2025	Changed Table 1 heading in Table of Contents to 'Au by Pb Fire Assay'
0	11 th April, 2025	First publication.

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016 (Accreditation number: 20483).



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



CERTIFYING OFFICER

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

REFERENCES

- [1] Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).
- [2] Ingamells, C. O. and Switzer, P. (1973). A Proposed Sampling Constant for Use in Geochemical Analysis, Talanta 20, 547-568.
- [3] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.
- [4] ISO 33401:2024. Reference materials – Contents of certificates, labels and accompanying documentation.
- [5] ISO 33405:2024. Reference materials – Approaches for characterization and assessment of homogeneity and stability.
- [6] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).
- [7] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.
- [8] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.
- [9] ISO 17025:2017. General requirements for the competence of testing and calibration laboratories.
- [10] ISO 17034:2016. General requirements for the competence of reference material producers.
- [11] ISO 9599:2015. Cu, Pb, Zn and Ni sulfide concentrates - Determination of hygroscopic moisture content
- [12] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.
- [13] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.
- [14] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.

- [15] Thompson, A.; Taylor, B.N. (2008), Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: <https://physics.nist.gov/cuu/pdf/sp811.pdf> (accessed Nov 2021).
- [16] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, *Accred Qual Assur* 6: 290-294.