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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 62h

Gold-Silver Ore (Cracow Mine, Queensland, Australia)

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 62h.

Constituent	Certified Value†	95% Expanded Uncertainty		95% Tolerance	
		Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppm)	10.54	10.44	10.65	10.51*	10.58*

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ 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

Table 2. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 62h.

Constituent	Certified	95% Expanded Uncertainty		95% Tolerance Limits	
	Value	Low	High	Low	High
Aqua Regia Digestion (sample weights 10-50g)					
Au, Gold (ppm)	10.31	10.07	10.55	10.27	10.35
4-Acid Digestion					
Ag, Silver (ppm)	7.69	7.43	7.94	7.55	7.83
Al, Aluminium (wt.%)	6.97	6.83	7.11	6.87	7.06
As, Arsenic (ppm)	109	104	114	107	112
Ba, Barium (ppm)	393	384	402	386	400
Be, Beryllium (ppm)	0.95	0.88	1.01	0.91	0.98
Bi, Bismuth (ppm)	0.13	0.11	0.15	IND	IND
Ca, Calcium (wt.%)	3.84	3.76	3.92	3.79	3.90
Cd, Cadmium (ppm)	0.45	0.41	0.48	0.42	0.48
Ce, Cerium (ppm)	30.6	29.0	32.1	29.5	31.6
Co, Cobalt (ppm)	14.7	14.0	15.4	14.4	15.1
Cr, Chromium (ppm)	31.8	29.4	34.2	30.3	33.3
Cs, Caesium (ppm)	3.88	3.68	4.09	3.79	3.97
Cu, Copper (ppm)	144	140	147	141	146
Dy, Dysprosium (ppm)	2.49	2.28	2.71	2.39	2.60
Er, Erbium (ppm)	1.47	1.34	1.60	1.38	1.56
Eu, Europium (ppm)	0.87	0.80	0.95	0.84	0.91
Fe, Iron (wt.%)	3.94	3.86	4.02	3.88	4.01
Ga, Gallium (ppm)	14.9	14.4	15.4	14.5	15.2
Gd, Gadolinium (ppm)	2.93	2.68	3.17	2.77	3.08
Hf, Hafnium (ppm)	2.58	2.44	2.72	2.47	2.69
Ho, Holmium (ppm)	0.52	0.46	0.58	IND	IND
In, Indium (ppm)	0.049	0.043	0.056	0.046	0.052
K, Potassium (wt.%)	1.80	1.76	1.84	1.77	1.83
La, Lanthanum (ppm)	14.1	13.4	14.9	13.7	14.5
Li, Lithium (ppm)	34.9	33.9	35.9	34.0	35.7
Lu, Lutetium (ppm)	0.22	0.21	0.24	0.20	0.24
Mg, Magnesium (wt.%)	1.48	1.44	1.52	1.45	1.50
Mn, Manganese (wt.%)	0.089	0.088	0.091	0.088	0.091
Mo, Molybdenum (ppm)	2.98	2.81	3.16	2.90	3.07
Na, Sodium (wt.%)	1.86	1.82	1.89	1.82	1.89
Nb, Niobium (ppm)	3.24	3.09	3.39	3.12	3.36
Nd, Neodymium (ppm)	15.4	14.3	16.6	14.8	16.0
Ni, Nickel (ppm)	18.7	17.9	19.6	18.2	19.2
P, Phosphorus (wt.%)	0.088	0.085	0.090	0.086	0.089

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

*Gold Tolerance Limits for a typical 25g aqua regia digestion are 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					
Pb, Lead (ppm)	52	50	54	50	54
Pr, Praseodymium (ppm)	3.72	3.36	4.09	3.61	3.84
Rb, Rubidium (ppm)	65	61	68	63	66
Re, Rhenium (ppm)	< 0.002	IND	IND	IND	IND
S, Sulphur (wt.%)	0.589	0.573	0.605	0.579	0.599
Sb, Antimony (ppm)	10.6	10.0	11.1	10.2	11.0
Sc, Scandium (ppm)	15.3	14.6	16.0	14.9	15.6
Sm, Samarium (ppm)	3.32	3.05	3.59	3.14	3.49
Sn, Tin (ppm)	0.98	0.88	1.08	IND	IND
Sr, Strontium (ppm)	421	411	431	415	426
Ta, Tantalum (ppm)	0.21	0.19	0.22	0.19	0.22
Tb, Terbium (ppm)	0.43	0.37	0.48	0.41	0.45
Te, Tellurium (ppm)	1.17	1.09	1.24	1.08	1.25
Th, Thorium (ppm)	3.00	2.82	3.17	2.90	3.10
Ti, Titanium (wt.%)	0.369	0.361	0.377	0.362	0.375
Tl, Thallium (ppm)	0.54	0.51	0.58	0.53	0.56
Tm, Thulium (ppm)	0.21	0.20	0.22	IND	IND
U, Uranium (ppm)	0.79	0.72	0.86	0.75	0.83
V, Vanadium (ppm)	126	122	129	123	128
W, Tungsten (ppm)	3.14	2.95	3.32	2.99	3.28
Y, Yttrium (ppm)	13.8	13.1	14.5	13.4	14.2
Yb, Ytterbium (ppm)	1.44	1.37	1.52	1.38	1.51
Zn, Zinc (ppm)	168	162	174	164	171
Zr, Zirconium (ppm)	98	94	101	95	100
Aqua Regia Digestion					
Ag, Silver (ppm)	7.70	7.45	7.96	7.54	7.87
Al, Aluminium (wt.%)	2.19	2.11	2.26	2.15	2.22
As, Arsenic (ppm)	104	100	108	101	107
B, Boron (ppm)	11.3	9.6	13.0	IND	IND
Ba, Barium (ppm)	62	59	65	60	63
Be, Beryllium (ppm)	0.48	0.46	0.49	0.46	0.49
Bi, Bismuth (ppm)	0.12	0.11	0.13	IND	IND
Ca, Calcium (wt.%)	2.94	2.88	3.01	2.88	3.00
Cd, Cadmium (ppm)	0.43	0.40	0.46	0.40	0.46
Ce, Cerium (ppm)	27.6	26.5	28.7	26.9	28.3
Co, Cobalt (ppm)	13.3	12.9	13.8	13.0	13.7
Cr, Chromium (ppm)	29.7	28.5	30.8	28.7	30.6

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
Aqua Regia Digestion continued					
Cs, Caesium (ppm)	2.18	2.07	2.30	2.12	2.24
Cu, Copper (ppm)	141	137	145	138	143
Dy, Dysprosium (ppm)	1.99	1.79	2.19	1.95	2.03
Er, Erbium (ppm)	1.01	0.91	1.11	0.97	1.05
Eu, Europium (ppm)	0.64	0.59	0.68	0.62	0.65
Fe, Iron (wt.%)	3.56	3.46	3.66	3.50	3.62
Ga, Gallium (ppm)	8.08	7.78	8.38	7.88	8.28
Gd, Gadolinium (ppm)	2.74	2.58	2.90	2.65	2.82
Ge, Germanium (ppm)	0.090	0.065	0.115	IND	IND
Hf, Hafnium (ppm)	0.69	0.63	0.75	0.66	0.72
Hg, Mercury (ppm)	0.076	0.059	0.093	IND	IND
Ho, Holmium (ppm)	0.37	0.31	0.43	0.36	0.38
In, Indium (ppm)	0.037	0.031	0.042	0.032	0.041
K, Potassium (wt.%)	0.205	0.197	0.213	0.199	0.211
La, Lanthanum (ppm)	12.5	12.1	12.9	12.2	12.8
Li, Lithium (ppm)	20.0	19.2	20.7	19.4	20.6
Lu, Lutetium (ppm)	0.13	0.12	0.14	IND	IND
Mg, Magnesium (wt.%)	1.28	1.25	1.32	1.26	1.30
Mn, Manganese (wt.%)	0.083	0.081	0.086	0.082	0.084
Mo, Molybdenum (ppm)	2.76	2.61	2.91	2.63	2.88
Na, Sodium (wt.%)	0.202	0.188	0.216	0.194	0.210
Nb, Niobium (ppm)	0.10	0.07	0.13	IND	IND
Nd, Neodymium (ppm)	13.6	12.4	14.9	13.3	13.9
Ni, Nickel (ppm)	16.2	15.3	17.2	15.8	16.7
P, Phosphorus (wt.%)	0.082	0.080	0.085	0.081	0.084
Pb, Lead (ppm)	48.6	46.9	50.3	47.5	49.7
Pr, Praseodymium (ppm)	3.36	3.08	3.65	3.28	3.45
Rb, Rubidium (ppm)	9.00	8.49	9.52	8.76	9.25
Re, Rhenium (ppm)	0.001	0.000	0.001	IND	IND
S, Sulphur (wt.%)	0.583	0.563	0.603	0.570	0.595
Sb, Antimony (ppm)	7.00	6.34	7.66	6.66	7.33
Sc, Scandium (ppm)	10.2	9.7	10.7	9.9	10.5
Se, Selenium (ppm)	0.51	0.32	0.69	IND	IND
Sm, Samarium (ppm)	2.88	2.53	3.24	2.77	3.00
Sn, Tin (ppm)	0.69	0.61	0.76	0.65	0.72
Sr, Strontium (ppm)	146	141	152	144	148
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
Aqua Regia Digestion continued					
Tb, Terbium (ppm)	0.38	0.34	0.42	0.36	0.40
Te, Tellurium (ppm)	1.06	0.98	1.14	1.00	1.12
Th, Thorium (ppm)	1.72	1.62	1.81	1.66	1.77
Ti, Titanium (wt.%)	0.145	0.138	0.153	0.142	0.149
Tl, Thallium (ppm)	0.090	0.081	0.099	IND	IND
U, Uranium (ppm)	0.40	0.38	0.42	0.37	0.43
V, Vanadium (ppm)	91	88	94	90	93
W, Tungsten (ppm)	0.92	0.79	1.05	0.85	0.99
Y, Yttrium (ppm)	10.2	9.8	10.6	10.0	10.5
Yb, Ytterbium (ppm)	0.87	0.76	0.98	0.80	0.93
Zn, Zinc (ppm)	156	153	160	154	159
Zr, Zirconium (ppm)	21.8	20.5	23.1	21.2	22.4

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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 62h.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
B	ppm	*20.2*	Hg	ppm	*< 1*			
Ge	ppm	*0.13*	Se	ppm	*1.03*			
Aqua Regia Digestion								
Pd	ppb	*< 10*	Pt	ppb	*< 5*	Tm	ppm	*0.12*
Borate Fusion XRF								
Al_2O_3	wt.%	*13.65*	MgO	wt.%	*2.59*	SiO_2	wt.%	*61.63*
CaO	wt.%	*5.44*	MnO	wt.%	*0.120*	SO_3	wt.%	*1.51*
Fe_2O_3	wt.%	*5.77*	Na_2O	wt.%	*2.55*	TiO_2	wt.%	*0.645*
K_2O	wt.%	*2.21*	P_2O_5	wt.%	*0.206*			
Thermogravimetry								
LOI^{1000}	wt.%	*4.82*						
Laser Ablation ICP-MS								
Ag	ppm	*8.45*	Hf	ppm	*3.01*	Sm	ppm	*3.72*
As	ppm	*109*	Ho	ppm	*0.58*	Sn	ppm	*1.50*
Ba	ppm	*406*	In	ppm	*< 0.05*	Sr	ppm	*418*
Be	ppm	*1.20*	La	ppm	*15.5*	Ta	ppm	*0.24*
Bi	ppm	*0.15*	Lu	ppm	*0.23*	Tb	ppm	*0.50*
Cd	ppm	*0.40*	Mn	wt.%	*0.094*	Te	ppm	*1.30*
Ce	ppm	*31.8*	Mo	ppm	*3.00*	Th	ppm	*3.16*
Co	ppm	*15.6*	Nb	ppm	*3.49*	Ti	wt.%	*0.384*

SI unit equivalents: ppb (parts per billion; 1 x 10^{-9}) ≡ µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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								
Cr	ppm	*38.5*	Nd	ppm	*16.6*	Tl	ppm	*0.40*
Cs	ppm	*4.12*	Ni	ppm	*20.0*	Tm	ppm	*0.24*
Cu	ppm	*147*	Pb	ppm	*55*	U	ppm	*0.85*
Dy	ppm	*2.80*	Pr	ppm	*4.18*	V	ppm	*136*
Er	ppm	*1.61*	Rb	ppm	*66*	W	ppm	*3.00*
Eu	ppm	*0.94*	Re	ppm	*0.013*	Y	ppm	*15.9*
Ga	ppm	*15.1*	Sb	ppm	*10.9*	Yb	ppm	*1.57*
Gd	ppm	*3.22*	Sc	ppm	*16.1*	Zn	ppm	*170*
Ge	ppm	*1.08*	Se	ppm	*< 5*	Zr	ppm	*109*
Infrared Combustion								
C	wt.%	*0.700*	S	wt.%	*0.565*			

SI unit equivalents: µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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.

Tables 1 and 2 provide the certified values and their associated 95% expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties, Table 5 provides indicative mineralogy based on semi-quantitative XRD analysis and Table 6 presents the performance gate intervals for all certified values. Gold homogeneity (via INAA) is shown in Table 7 and is also demonstrated by a nested ANOVA program using fire assay (see ‘**nested ANOVA**’ section).

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 (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 62h-DataPack.1.0.230515_151024.xlsx**).

Results are also presented in scatter plots for Gold by fire assay, Gold by aqua regia digestion, Silver by 4-acid digestion and Silver by aqua regia digestion (Figures 1 to 4, respectively) together with ±3SD (magenta) and ±5% (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

SOURCE MATERIAL

OREAS 62h was prepared from coarse reject splits of gold-silver ore samples blended with barren andesite. The gold-silver ore was sourced from the Cracow mine located 500km northwest of Brisbane in Queensland, Australia. The barren andesite was sourced from the Carboniferous Blair Duguid Hypersthene Andesite intrusive, located 70kms northwest of Newcastle, Australia. Cracow is a low sulphidation epithermal deposit hosted by meta-andesitic volcanics. High grade gold mineralisation occurs within fissure quartz veins and is associated with zones of silicification, present as quartz lode breccia and as quartz vein breccia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 62h was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of the barren andesite to >98% minus 75 microns;

- Crushing and multi stage milling of the ore materials to 100% passing 30 microns;
- Blending in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 60g units sealed in laminated foil pouches and 500g units in plastic jars.

PHYSICAL PROPERTIES

OREAS 62h 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 62h.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
621	1.17	N7	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.

MINERALOGY

The semi-quantitative XRD results shown in Table 5 below have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. Some amorphous material is likely present.

Table 5. Indicative mineralogy of OREAS 62h based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Zeolite	2
Clay minerals	10
Kaolinite	5
Chlorite	3
Muscovite	1
Plagioclase	26
K-feldspar	4
Quartz	40
Calcite	5
Pyrite	3

ANALYTICAL PROGRAM

Twenty-nine commercial analytical laboratories participated in the program to certify the 110 elements reported in Table 1. The following methods were employed:

- Au by lead collection fire assay with AAS (23 laboratories) or ICP-OES (6 laboratories) finish;
- Gold by aqua regia digestion on 10-50g sample weights with ICP-OES and/or ICP-MS (12 laboratories) and AAS (6 laboratories);

- 4-acid ($HF-HNO_3-HClO_4-HCl$) digestion for full ICP-OES and ICP-MS elemental suites (up to 26 laboratories depending on the element);
- Aqua regia digestion for full ICP-OES and ICP-MS elemental suites (up to 26 laboratories depending on the element).

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

Table 3 shows indicative values including major and trace element characterisation by 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;
- Total Carbon and Sulphur by infrared combustion furnace;
- Trace elements by laser ablation (on the fused bead) with ICP-MS finish.

For the round robin program twenty 3kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 110g pulp samples were submitted to each laboratory for analysis received by each laboratory were obtained by taking two 110g samples from each of three separate 3kg test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e., to ascertain whether between-unit variance is greater than within-unit variance.

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 7) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation (see 'Homogeneity Evaluation' section below).

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 [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.

Standard Deviation intervals (see 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 'Intended Use' section for more detail).

The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e., after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e., the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. ***The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.***

PERFORMANCE GATES

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) ± 10%.

I.e., Certified Value ± 10% ± 2DL [1].

Table 6. Performance Gates for OREAS 62h.

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	10.54	0.291	9.96	11.12	9.67	11.41	2.76%	5.51%	8.27%	10.01	11.07
Aqua Regia Digestion (sample weights 10-50g)											
Au, ppm	10.31	0.479	9.35	11.27	8.87	11.75	4.64%	9.28%	13.92%	9.79	10.83
4-Acid Digestion											
Ag, ppm	7.69	0.347	6.99	8.38	6.65	8.73	4.51%	9.02%	13.53%	7.30	8.07
Al, wt.%	6.97	0.216	6.54	7.40	6.32	7.62	3.11%	6.21%	9.32%	6.62	7.32
As, ppm	109	6	98	121	92	126	5.20%	10.40%	15.60%	104	115
Ba, ppm	393	11	372	414	361	425	2.70%	5.40%	8.10%	373	413
Be, ppm	0.95	0.11	0.73	1.16	0.63	1.26	11.22%	22.44%	33.66%	0.90	0.99
Bi, ppm	0.13	0.013	0.10	0.15	0.09	0.17	9.89%	19.78%	29.67%	0.12	0.13
Ca, wt.%	3.84	0.104	3.63	4.05	3.53	4.15	2.71%	5.43%	8.14%	3.65	4.03
Cd, ppm	0.45	0.028	0.39	0.50	0.37	0.53	6.17%	12.34%	18.51%	0.43	0.47
Ce, ppm	30.6	2.40	25.8	35.4	23.4	37.8	7.86%	15.71%	23.57%	29.0	32.1
Co, ppm	14.7	1.08	12.6	16.9	11.5	17.9	7.33%	14.65%	21.98%	14.0	15.5
Cr, ppm	31.8	3.4	25.1	38.6	21.7	41.9	10.62%	21.23%	31.85%	30.2	33.4
Cs, ppm	3.88	0.183	3.52	4.25	3.34	4.43	4.71%	9.41%	14.12%	3.69	4.08
Cu, ppm	144	3	137	151	133	154	2.41%	4.83%	7.24%	137	151
Dy, ppm	2.49	0.200	2.09	2.89	1.89	3.09	8.01%	16.03%	24.04%	2.37	2.62
Er, ppm	1.47	0.120	1.23	1.71	1.11	1.83	8.15%	16.29%	24.44%	1.40	1.55
Eu, ppm	0.87	0.041	0.79	0.95	0.75	1.00	4.72%	9.44%	14.15%	0.83	0.92
Fe, wt.%	3.94	0.131	3.68	4.20	3.55	4.33	3.32%	6.64%	9.95%	3.74	4.14
Ga, ppm	14.9	0.53	13.8	15.9	13.3	16.5	3.53%	7.05%	10.58%	14.1	15.6
Gd, ppm	2.93	0.190	2.55	3.31	2.36	3.50	6.48%	12.95%	19.43%	2.78	3.08
Hf, ppm	2.58	0.162	2.25	2.90	2.09	3.06	6.29%	12.57%	18.86%	2.45	2.71
Ho, ppm	0.52	0.030	0.46	0.58	0.43	0.61	5.73%	11.45%	17.18%	0.49	0.55
In, ppm	0.049	0.004	0.041	0.057	0.038	0.061	7.86%	15.73%	23.59%	0.047	0.052
K, wt.%	1.80	0.054	1.69	1.91	1.64	1.96	3.02%	6.03%	9.05%	1.71	1.89
La, ppm	14.1	0.97	12.2	16.1	11.2	17.0	6.84%	13.67%	20.51%	13.4	14.8
Li, ppm	34.9	1.52	31.9	37.9	30.3	39.4	4.35%	8.70%	13.06%	33.1	36.6
Lu, ppm	0.22	0.010	0.20	0.24	0.19	0.25	4.67%	9.35%	14.02%	0.21	0.23
Mg, wt.%	1.48	0.063	1.35	1.60	1.29	1.66	4.26%	8.53%	12.79%	1.40	1.55
Mn, wt.%	0.089	0.002	0.085	0.094	0.083	0.096	2.40%	4.81%	7.21%	0.085	0.094
Mo, ppm	2.98	0.223	2.54	3.43	2.31	3.65	7.49%	14.98%	22.47%	2.83	3.13
Na, wt.%	1.86	0.067	1.72	1.99	1.65	2.06	3.64%	7.28%	10.91%	1.76	1.95
Nb, ppm	3.24	0.217	2.81	3.67	2.59	3.89	6.69%	13.38%	20.07%	3.08	3.40
Nd, ppm	15.4	1.07	13.3	17.6	12.2	18.6	6.91%	13.83%	20.74%	14.7	16.2
Ni, ppm	18.7	1.05	16.6	20.8	15.6	21.9	5.58%	11.17%	16.75%	17.8	19.7
P, wt.%	0.088	0.005	0.078	0.097	0.074	0.102	5.29%	10.59%	15.88%	0.083	0.092
Pb, ppm	52	2.8	46	58	44	60	5.37%	10.75%	16.12%	49	55
Pr, ppm	3.72	0.39	2.95	4.49	2.56	4.88	10.37%	20.73%	31.10%	3.54	3.91
Rb, ppm	65	4.4	56	73	51	78	6.78%	13.55%	20.33%	61	68
Re, ppm	< 0.002	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.589	0.027	0.535	0.643	0.508	0.670	4.57%	9.14%	13.71%	0.560	0.618

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.%.

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											
Sb, ppm	10.6	0.68	9.2	11.9	8.5	12.6	6.42%	12.83%	19.25%	10.1	11.1
Sc, ppm	15.3	0.77	13.7	16.8	13.0	17.6	5.06%	10.12%	15.18%	14.5	16.1
Sm, ppm	3.32	0.221	2.88	3.76	2.66	3.98	6.65%	13.31%	19.96%	3.15	3.49
Sn, ppm	0.98	0.11	0.75	1.21	0.64	1.32	11.58%	23.16%	34.75%	0.93	1.03
Sr, ppm	421	17	387	455	370	472	4.06%	8.11%	12.17%	400	442
Ta, ppm	0.21	0.011	0.18	0.23	0.17	0.24	5.39%	10.77%	16.16%	0.20	0.22
Tb, ppm	0.43	0.04	0.34	0.52	0.30	0.56	10.20%	20.40%	30.60%	0.41	0.45
Te, ppm	1.17	0.092	0.98	1.35	0.89	1.44	7.92%	15.85%	23.77%	1.11	1.22
Th, ppm	3.00	0.193	2.61	3.38	2.42	3.58	6.43%	12.86%	19.28%	2.85	3.15
Ti, wt.%	0.369	0.012	0.344	0.393	0.332	0.406	3.34%	6.68%	10.02%	0.350	0.387
Tl, ppm	0.54	0.036	0.47	0.62	0.44	0.65	6.63%	13.26%	19.88%	0.52	0.57
Tm, ppm	0.21	0.011	0.19	0.23	0.17	0.24	5.09%	10.18%	15.27%	0.20	0.22
U, ppm	0.79	0.070	0.65	0.93	0.58	1.00	8.81%	17.62%	26.43%	0.75	0.83
V, ppm	126	5	116	135	111	140	3.88%	7.75%	11.63%	119	132
W, ppm	3.14	0.258	2.62	3.65	2.36	3.91	8.22%	16.44%	24.66%	2.98	3.29
Y, ppm	13.8	0.80	12.2	15.4	11.4	16.2	5.79%	11.58%	17.37%	13.1	14.5
Yb, ppm	1.44	0.058	1.33	1.56	1.27	1.62	4.02%	8.04%	12.07%	1.37	1.52
Zn, ppm	168	9	150	186	141	195	5.40%	10.79%	16.19%	159	176
Zr, ppm	98	5.8	86	109	80	115	5.93%	11.87%	17.80%	93	103
Aqua Regia Digestion											
Ag, ppm	7.70	0.303	7.10	8.31	6.79	8.61	3.94%	7.87%	11.81%	7.32	8.09
Al, wt.%	2.19	0.129	1.93	2.44	1.80	2.57	5.88%	11.77%	17.65%	2.08	2.29
As, ppm	104	5	93	114	88	120	5.08%	10.16%	15.23%	99	109
B, ppm	11.3	2.2	6.9	15.7	4.7	17.9	19.45%	38.89%	58.34%	10.8	11.9
Ba, ppm	62	4.7	52	71	48	76	7.60%	15.20%	22.80%	59	65
Be, ppm	0.48	0.025	0.42	0.53	0.40	0.55	5.36%	10.71%	16.07%	0.45	0.50
Bi, ppm	0.12	0.007	0.11	0.14	0.10	0.14	5.84%	11.68%	17.51%	0.12	0.13
Ca, wt.%	2.94	0.099	2.74	3.14	2.64	3.24	3.38%	6.76%	10.15%	2.79	3.09
Cd, ppm	0.43	0.021	0.39	0.47	0.37	0.49	4.81%	9.61%	14.42%	0.41	0.45
Ce, ppm	27.6	1.71	24.2	31.0	22.5	32.7	6.19%	12.37%	18.56%	26.2	29.0
Co, ppm	13.3	0.66	12.0	14.7	11.4	15.3	4.92%	9.83%	14.75%	12.7	14.0
Cr, ppm	29.7	1.52	26.6	32.7	25.1	34.2	5.12%	10.24%	15.36%	28.2	31.2
Cs, ppm	2.18	0.174	1.83	2.53	1.66	2.70	7.96%	15.91%	23.87%	2.07	2.29
Cu, ppm	141	5	131	151	126	155	3.50%	7.01%	10.51%	134	148
Dy, ppm	1.99	0.181	1.63	2.35	1.45	2.53	9.08%	18.16%	27.24%	1.89	2.09
Er, ppm	1.01	0.087	0.84	1.19	0.75	1.27	8.65%	17.29%	25.94%	0.96	1.06
Eu, ppm	0.64	0.030	0.58	0.69	0.55	0.72	4.64%	9.29%	13.93%	0.60	0.67
Fe, wt.%	3.56	0.140	3.28	3.84	3.14	3.98	3.95%	7.89%	11.84%	3.38	3.74
Ga, ppm	8.08	0.436	7.21	8.95	6.77	9.39	5.40%	10.79%	16.19%	7.68	8.49
Gd, ppm	2.74	0.105	2.53	2.95	2.42	3.05	3.84%	7.68%	11.52%	2.60	2.87
Ge, ppm	0.090	0.026	0.037	0.143	0.011	0.169	29.20%	58.39%	87.59%	0.086	0.095
Hf, ppm	0.69	0.07	0.54	0.84	0.46	0.91	10.90%	21.79%	32.69%	0.65	0.72
Hg, ppm	0.076	0.016	0.043	0.109	0.027	0.125	21.61%	43.22%	64.83%	0.072	0.080

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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
Aqua Regia Digestion continued											
Ho, ppm	0.37	0.04	0.28	0.46	0.24	0.50	11.94%	23.87%	35.81%	0.35	0.39
In, ppm	0.037	0.004	0.030	0.044	0.026	0.047	9.68%	19.36%	29.04%	0.035	0.038
K, wt.%	0.205	0.014	0.177	0.233	0.164	0.247	6.78%	13.57%	20.35%	0.195	0.216
La, ppm	12.5	0.52	11.5	13.5	10.9	14.0	4.13%	8.27%	12.40%	11.9	13.1
Li, ppm	20.0	1.24	17.5	22.4	16.3	23.7	6.19%	12.38%	18.57%	19.0	21.0
Lu, ppm	0.13	0.006	0.12	0.14	0.11	0.15	4.55%	9.10%	13.66%	0.13	0.14
Mg, wt.%	1.28	0.042	1.20	1.37	1.15	1.41	3.31%	6.62%	9.94%	1.22	1.35
Mn, wt.%	0.083	0.004	0.076	0.090	0.072	0.094	4.26%	8.53%	12.79%	0.079	0.087
Mo, ppm	2.76	0.216	2.32	3.19	2.11	3.41	7.85%	15.70%	23.55%	2.62	2.89
Na, wt.%	0.202	0.030	0.141	0.263	0.111	0.293	14.99%	29.97%	44.96%	0.192	0.212
Nb, ppm	0.10	0.04	0.02	0.18	0.00	0.22	38.22%	76.44%	114.67%	0.10	0.11
Nd, ppm	13.6	0.87	11.9	15.4	11.0	16.2	6.37%	12.74%	19.10%	13.0	14.3
Ni, ppm	16.2	1.05	14.1	18.3	13.1	19.4	6.47%	12.94%	19.41%	15.4	17.0
P, wt.%	0.082	0.004	0.075	0.089	0.072	0.093	4.29%	8.58%	12.87%	0.078	0.086
Pb, ppm	48.6	2.27	44.1	53.1	41.8	55.4	4.67%	9.33%	14.00%	46.2	51.0
Pr, ppm	3.36	0.208	2.95	3.78	2.74	3.99	6.20%	12.39%	18.59%	3.20	3.53
Rb, ppm	9.00	0.826	7.35	10.66	6.53	11.48	9.17%	18.35%	27.52%	8.55	9.46
Re, ppm	0.001	0.000	0.000	0.001	0.000	0.002	25.81%	51.63%	77.44%	0.001	0.001
S, wt.%	0.583	0.029	0.524	0.642	0.494	0.671	5.06%	10.12%	15.18%	0.553	0.612
Sb, ppm	7.00	1.18	4.64	9.36	3.45	10.55	16.88%	33.77%	50.65%	6.65	7.35
Sc, ppm	10.2	0.91	8.4	12.0	7.5	12.9	8.90%	17.81%	26.71%	9.7	10.7
Se, ppm	0.51	0.15	0.21	0.80	0.07	0.94	28.85%	57.69%	86.54%	0.48	0.53
Sm, ppm	2.88	0.32	2.24	3.53	1.91	3.85	11.22%	22.44%	33.67%	2.74	3.03
Sn, ppm	0.69	0.048	0.59	0.78	0.54	0.83	7.00%	14.00%	20.99%	0.65	0.72
Sr, ppm	146	10	126	166	116	177	6.91%	13.82%	20.73%	139	154
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.38	0.029	0.32	0.44	0.29	0.47	7.70%	15.40%	23.11%	0.36	0.40
Te, ppm	1.06	0.088	0.89	1.24	0.80	1.33	8.30%	16.59%	24.89%	1.01	1.11
Th, ppm	1.72	0.133	1.45	1.98	1.32	2.12	7.76%	15.51%	23.27%	1.63	1.80
Ti, wt.%	0.145	0.015	0.115	0.176	0.100	0.191	10.51%	21.02%	31.53%	0.138	0.153
Tl, ppm	0.090	0.005	0.079	0.101	0.074	0.106	6.05%	12.10%	18.15%	0.085	0.094
U, ppm	0.40	0.024	0.35	0.45	0.33	0.47	5.97%	11.93%	17.90%	0.38	0.42
V, ppm	91	4.6	82	100	78	105	4.99%	9.99%	14.98%	87	96
W, ppm	0.92	0.21	0.51	1.33	0.30	1.53	22.36%	44.71%	67.07%	0.87	0.96
Y, ppm	10.2	0.61	9.0	11.4	8.4	12.1	5.99%	11.98%	17.96%	9.7	10.7
Yb, ppm	0.87	0.09	0.69	1.04	0.60	1.13	10.19%	20.39%	30.58%	0.82	0.91
Zn, ppm	156	5	146	167	141	172	3.29%	6.58%	9.87%	149	164
Zr, ppm	21.8	2.04	17.7	25.9	15.7	27.9	9.36%	18.72%	28.08%	20.7	22.9

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (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.

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 141 and 146 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35).

Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.

Table 7 below shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 62h. 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 0.11% calculated for a 30g fire assay sample (1.97% at 85mg weights) confirms the high level of gold homogeneity in OREAS 62h.

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 gold in OREAS 62h has also been evaluated in a nested Analysis of Variance (**ANOVA**) of the round robin program. Each participating laboratory received six samples made up of paired samples from three different, non-adjacent sampling intervals. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between units to that of the variance within units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 62h. The test was performed using the following parameters:

- Gold fire assay – 174 samples (29 laboratories each providing analyses on 3 pairs of samples);
- 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.

P-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the *p*-value.

This process derived a *p*-value of 0.99 for Au by fire assay, an insignificant result and the Null Hypothesis is retained. Additionally, none of the other certified values showed significant *p*-values.

Please note that only results for constituents present in concentrations well above the detection levels (i.e., >20 x Lower Limit of Detection) for the various methods undertaken were considered for the objective of evaluating homogeneity

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 62h 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 62h is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

Table 7. 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	10.97	10.915
2	10.95	10.914
3	10.96	10.915
4	10.69	10.900
5	10.73	10.902
6	10.75	10.903
7	10.76	10.904
8	10.73	10.902
9	11.10	10.922
10	10.88	10.910
11	10.86	10.909
12	11.02	10.917
13	10.76	10.904
14	10.82	10.907
15	10.79	10.905
16	10.56	10.893
17	11.09	10.921
18	11.40	10.938
19	11.07	10.920
20	11.35	10.935
Mean	10.912	10.912
Median	10.867	10.909
Std Dev.	0.215	0.011
Rel.Std.Dev.	**1.97%**	**0.11%**

*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

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6. ALS, Johannesburg, South Africa
7. ALS, Lima, Peru
8. ALS, Loughrea, Galway, Ireland
9. ALS, Malaga, WA, Australia
10. ALS, Vancouver, BC, Canada
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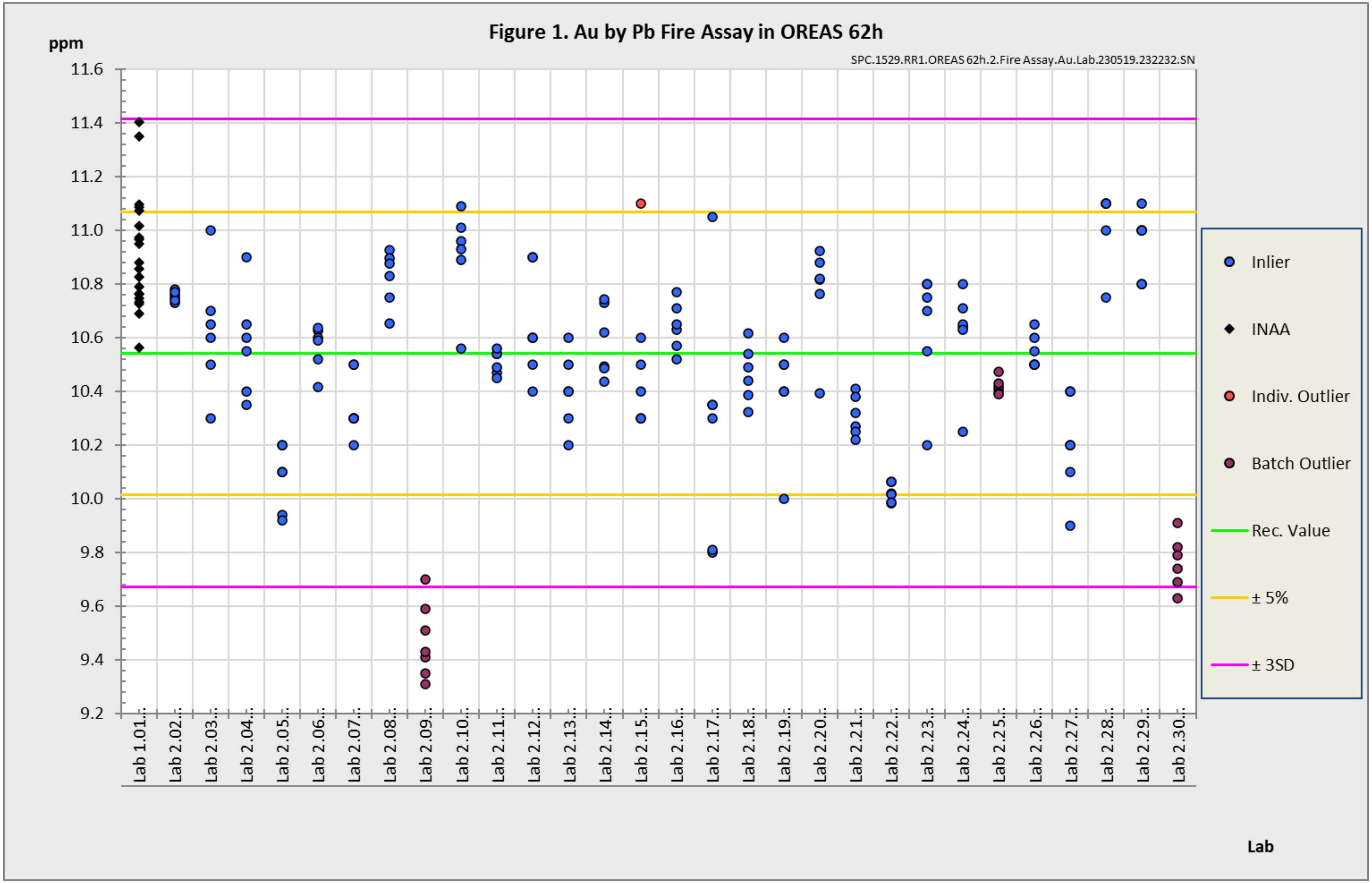
26. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
27. SGS, Ankara, Anatolia, Turkey
28. SGS del Peru, Lima, Peru
29. SGS Tarkwa, Tarkwa, Western Region, Ghana
30. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
31. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

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



ORE RESEARCH & EXPLORATION

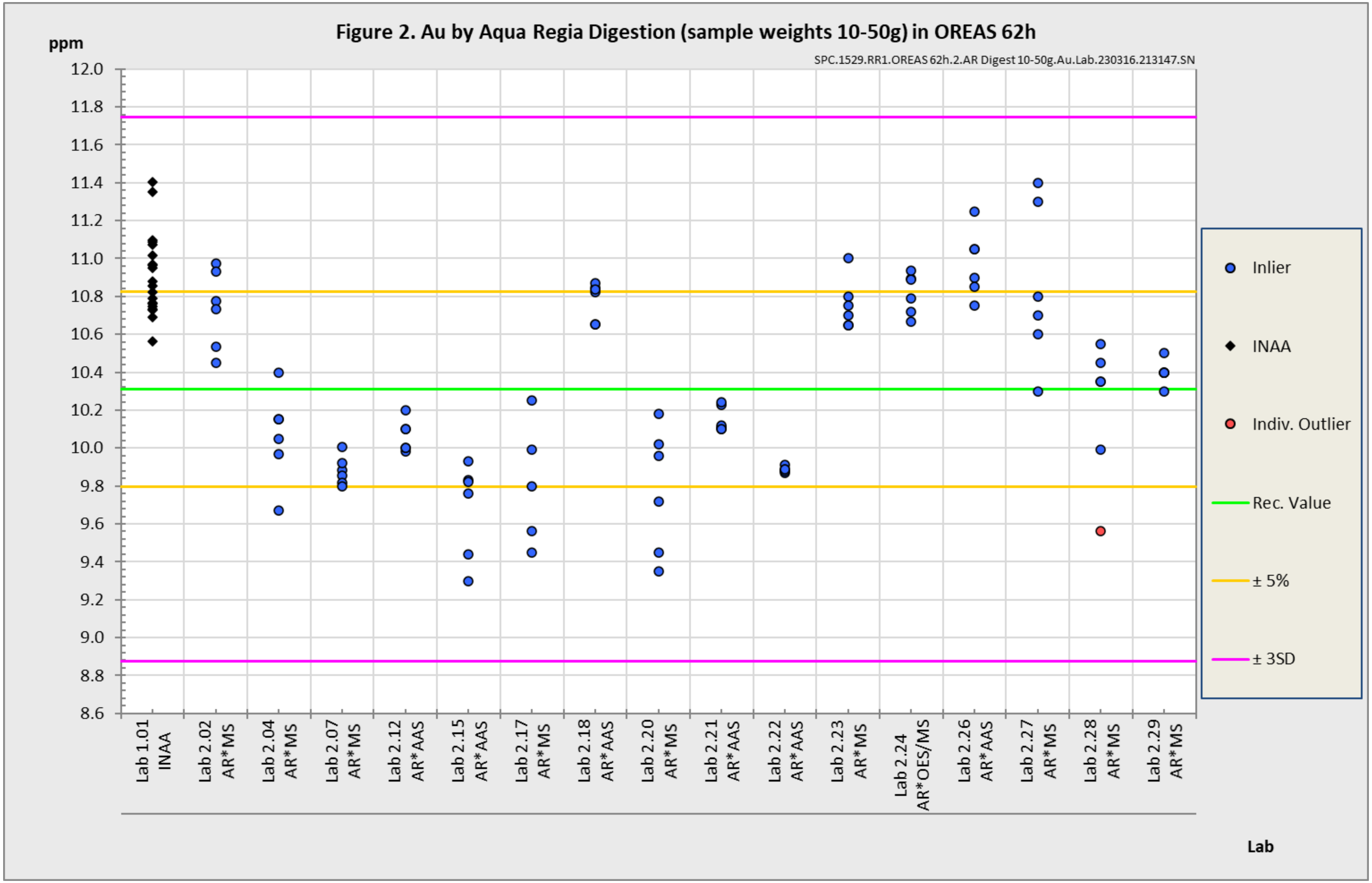


Figure 2. Au by Aqua Regia Digestion (sample weights 10-50g) in OREAS 62h
SPC.1529.RR1.OREAS 62h.2.AR Digest 10-50g.Au.Lab.230316.213147.SN
ppm
12.0
11.8
11.6
11.4
11.2
11.0
10.8
10.6
10.4
10.2
10.0
9.8
9.6
9.4
9.2
9.0
8.8
8.6
Lab 1.01 INAA
Lab 2.02 AR*MS
Lab 2.04 AR*MS
Lab 2.07 AR*MS
Lab 2.12 AR*AAS
Lab 2.15 AR*AAS
Lab 2.17 AR*MS
Lab 2.18 AR*AAS
Lab 2.20 AR*MS
Lab 2.21 AR*AAS
Lab 2.22 AR*AAS
Lab 2.23 AR*MS
Lab 2.24 AR*OES/MS
Lab 2.26 AR*AAS
Lab 2.27 AR*MS
Lab 2.28 AR*MS
Lab 2.29 AR*MS
Lab
Inlier
INAA
Indiv. Outlier
Rec. Value
± 5%
± 3SD

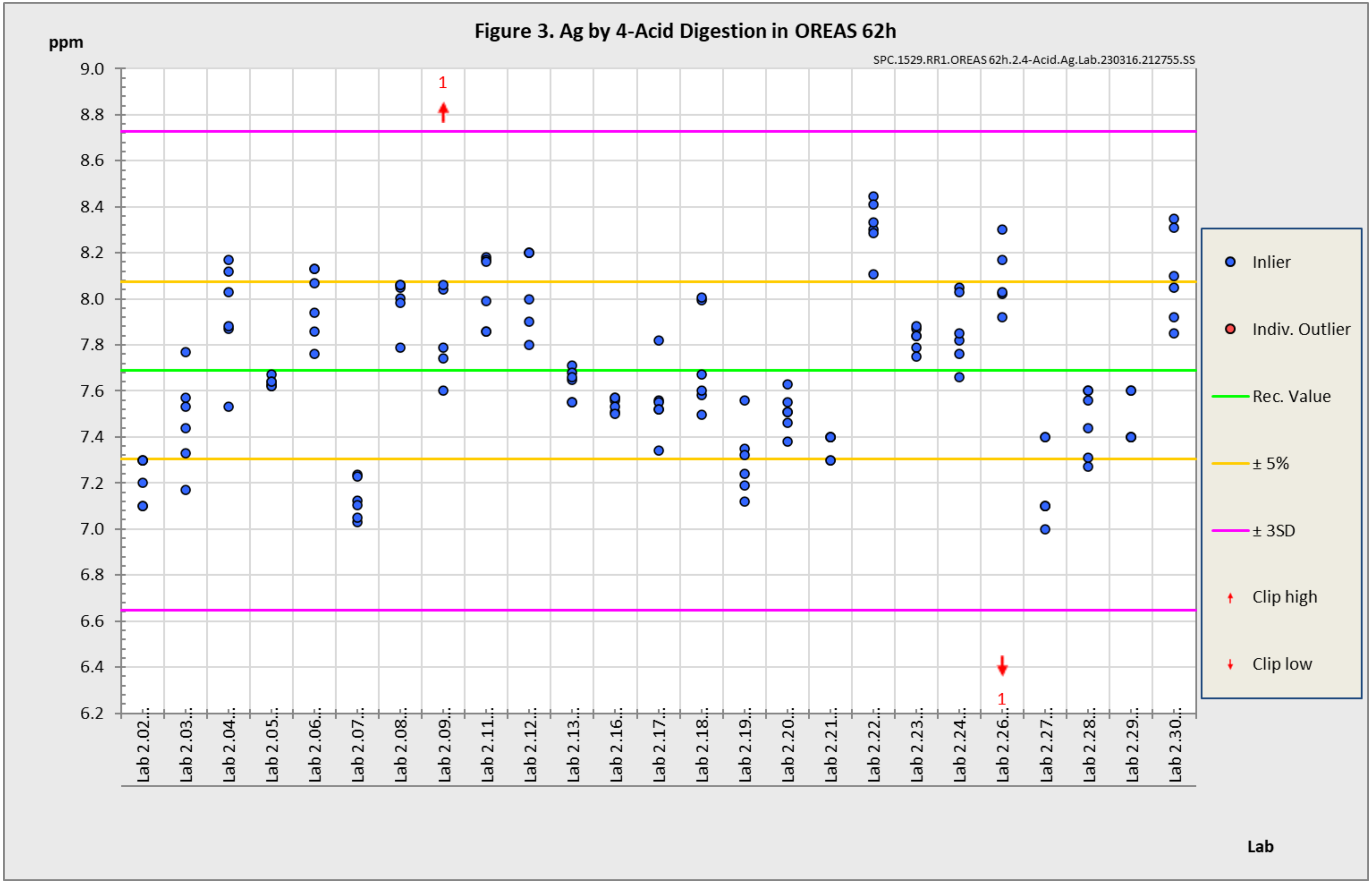


Figure 3. Ag by 4-Acid Digestion in OREAS 62h
ppm
SPC.1529.RR1.OREAS 62h.2.4-Acid.Ag.Lab.230316.212755.SS
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
1
1
Lab 2.02...
Lab 2.03...
Lab 2.04...
Lab 2.05...
Lab 2.06...
Lab 2.07...
Lab 2.08...
Lab 2.09...
Lab 2.11...
Lab 2.12...
Lab 2.13...
Lab 2.16...
Lab 2.17...
Lab 2.18...
Lab 2.19...
Lab 2.20...
Lab 2.21...
Lab 2.22...
Lab 2.23...
Lab 2.24...
Lab 2.26...
Lab 2.27...
Lab 2.28...
Lab 2.29...
Lab 2.30...
Inlier
Indiv. Outlier
Rec. Value
± 5%
± 3SD
Clip high
Clip low
Lab

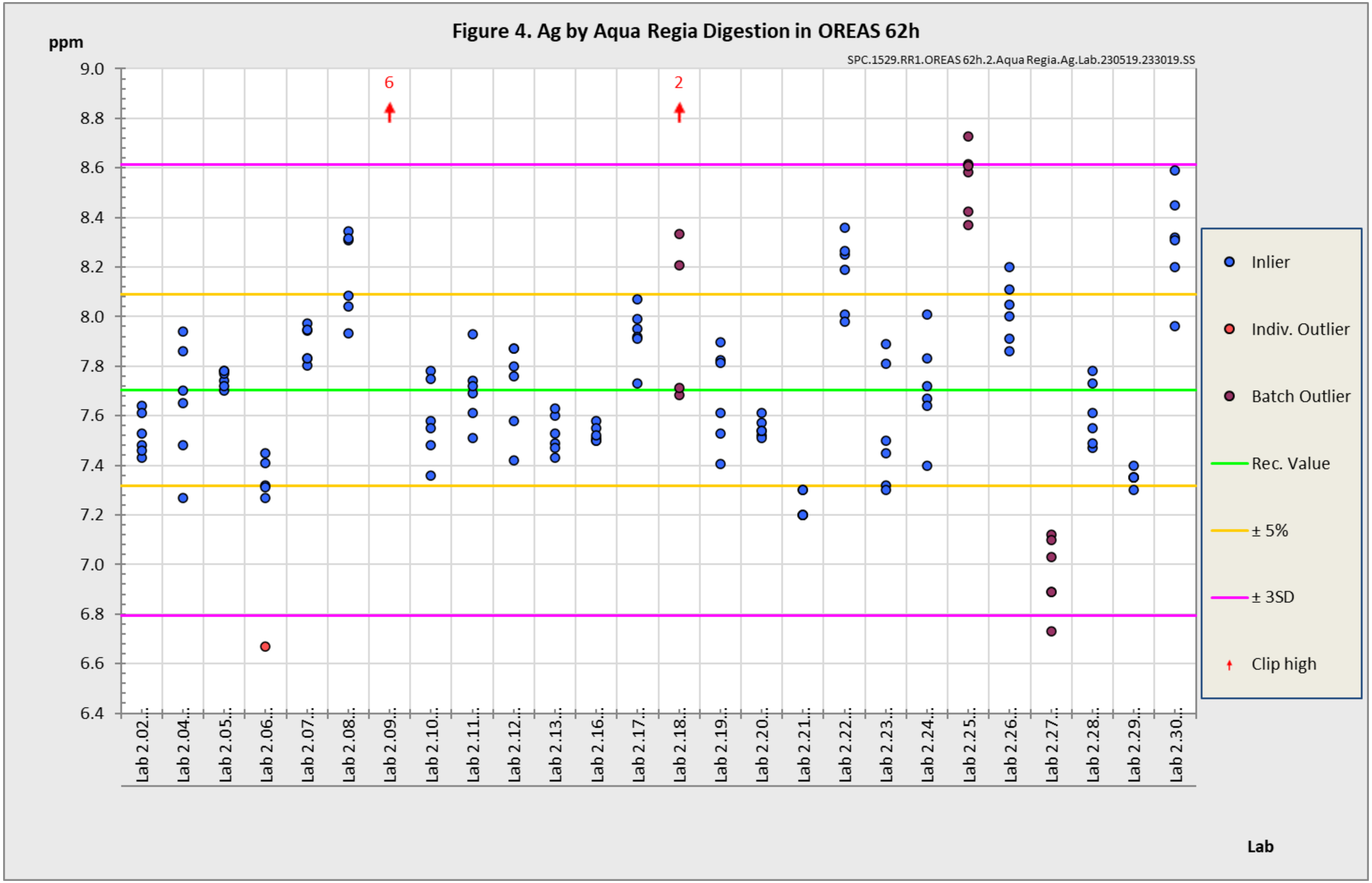


Figure 4. Ag by Aqua Regia Digestion in OREAS 62h
ppm
SPC.1529.RR1.OREAS 62h.2.Aqua Regia.Ag.Lab.230519.233019.SS
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6
2
Lab 2.02...
Lab 2.04...
Lab 2.05...
Lab 2.06...
Lab 2.07...
Lab 2.08...
Lab 2.09...
Lab 2.10...
Lab 2.11...
Lab 2.12...
Lab 2.13...
Lab 2.16...
Lab 2.17...
Lab 2.18...
Lab 2.19...
Lab 2.20...
Lab 2.21...
Lab 2.22...
Lab 2.23...
Lab 2.24...
Lab 2.25...
Lab 2.26...
Lab 2.27...
Lab 2.28...
Lab 2.29...
Lab 2.30...
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Clip high
Lab

PREPARER AND SUPPLIER

Certified reference material OREAS 62h is prepared, certified and supplied by:

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METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified value are metrologically traceable to the international measurement scale (SI) of mass (milligrams per kilogram (mg/kg)) and are expressed as the mass fraction 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. The other operationally defined measurands characterised in this certificate 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 62h 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 62h may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 62h 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: ≥10g;
- Au by aqua regia digestion ICP finish: ≥1g;
- 4-acid digestion with ICP-OES and/or MS finish: ≥0.25g;
- Aqua regia digestion with ICP-OES and/or MS finish: ≥0.5g.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 62h remains valid, within the specified measurement uncertainties, until March 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

Following analysis, it is the manufacturer's expectation that any remaining material is discarded unless the sachet is promptly resealed. It is the user's responsibility to prevent contamination and minimise exposure to the atmosphere.

Repeat-use packaging (e.g., 500g unit)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 62h 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 stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (0.59 wt.% S).

*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 adsorped moisture (weakly held H_2O- 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 (~2mm 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 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.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	23rd May, 2023	First publication.

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034.

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

23rd May, 2023

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

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