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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 320

Zn–Pb–Ag Ore

(Mt Isa Region, Queensland, Australia)

Table 1. Certified Values, Uncertainty & Tolerance Intervals in OREAS 320.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
Al_2O_3, Aluminium(III) oxide (wt.%)	4.53	4.45	4.60	4.44	4.61
BaO, Barium oxide (wt.%)	0.159	0.147	0.170	0.146	0.171
CaO, Calcium oxide (wt.%)	3.01	2.96	3.06	2.97	3.04
Cu, Copper (wt.%)	0.911	0.880	0.942	0.898	0.924
Fe_2O_3, Iron(III) oxide (wt.%)	12.76	12.61	12.91	12.65	12.88
K_2O, Potassium oxide (wt.%)	1.22	1.21	1.24	1.20	1.24
MgO, Magnesium oxide (wt.%)	1.95	1.89	2.00	1.91	1.98
MnO_2, Manganese dioxide (wt.%)	0.507	0.492	0.522	0.498	0.515
P_2O_5, Phosphorus(V) oxide (wt.%)	0.061	0.046	0.076	IND	IND
Pb, Lead (wt.%)	11.97	11.65	12.30	11.83	12.12
S, Sulphur (wt.%)	16.43	15.82	17.04	16.16	16.70
SiO_2, Silicon dioxide (wt.%)	25.17	24.62	25.72	24.93	25.40
TiO_2, Titanium dioxide (wt.%)	0.187	0.173	0.202	IND	IND
Zn, Zinc (wt.%)	17.16	16.83	17.50	17.04	17.29
Thermogravimetry					
LOI^{1000}, Loss on ignition @1000 °C (wt.%)	12.32	11.65	13.00	12.17	12.48
Infrared Combustion					
C, Carbon (wt.%)	1.67	1.63	1.70	1.64	1.69
S, Sulphur (wt.%)	16.70	16.33	17.06	16.55	16.84
Borate / Peroxide Fusion ICP					
Al, Aluminium (wt.%)	2.39	2.31	2.48	2.33	2.46
As, Arsenic (ppm)	309	287	332	298	321
Ba, Barium (wt.%)	0.145	0.140	0.149	0.141	0.148
Bi, Bismuth (ppm)	20.5	18.3	22.7	19.8	21.3
Ca, Calcium (wt.%)	2.13	2.03	2.23	2.05	2.20
Cd, Cadmium (ppm)	478	454	501	463	492
Ce, Cerium (ppm)	39.4	35.1	43.7	37.4	41.4
Co, Cobalt (ppm)	91	84	97	88	93
Cr, Chromium (ppm)	69	59	79	60	78
Cs, Caesium (ppm)	1.71	1.51	1.90	IND	IND
Cu, Copper (wt.%)	0.905	0.886	0.924	0.889	0.921
Dy, Dysprosium (ppm)	1.86	1.62	2.11	1.69	2.04
Er, Erbium (ppm)	1.06	0.90	1.21	0.98	1.13
Eu, Europium (ppm)	0.59	0.47	0.70	0.50	0.67
Fe, Iron (wt.%)	9.04	8.71	9.38	8.87	9.22
Ga, Gallium (ppm)	8.63	7.65	9.62	8.31	8.96
Gd, Gadolinium (ppm)	2.37	2.11	2.63	2.19	2.54

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 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued					
Ge, Germanium (ppm)	13.1	11.8	14.5	IND	IND
Ho, Holmium (ppm)	0.39	0.38	0.40	IND	IND
In, Indium (ppm)	1.67	1.48	1.86	IND	IND
K, Potassium (wt.%)	1.07	1.00	1.14	1.03	1.11
La, Lanthanum (ppm)	21.5	19.2	23.7	20.2	22.7
Li, Lithium (ppm)	14.4	11.4	17.5	IND	IND
Lu, Lutetium (ppm)	0.16	0.12	0.20	IND	IND
Mg, Magnesium (wt.%)	1.14	1.11	1.18	1.11	1.18
Mn, Manganese (wt.%)	0.319	0.309	0.329	0.311	0.327
Mo, Molybdenum (ppm)	17.3	14.9	19.8	IND	IND
Nd, Neodymium (ppm)	15.4	14.2	16.6	14.5	16.4
P, Phosphorus (wt.%)	0.024	0.013	0.035	IND	IND
Pb, Lead (wt.%)	12.06	11.59	12.52	11.87	12.25
Pr, Praseodymium (ppm)	4.23	3.88	4.58	3.93	4.53
Rb, Rubidium (ppm)	48.2	46.1	50.2	46.7	49.7
S, Sulphur (wt.%)	16.70	16.15	17.24	16.23	17.16
Sb, Antimony (ppm)	208	183	233	204	212
Si, Silicon (wt.%)	12.12	11.73	12.50	11.70	12.54
Sm, Samarium (ppm)	2.65	2.34	2.97	2.50	2.80
Sr, Strontium (ppm)	41.5	35.9	47.1	38.1	44.9
Tb, Terbium (ppm)	0.31	0.27	0.35	0.28	0.33
Th, Thorium (ppm)	5.14	4.56	5.73	4.98	5.30
Ti, Titanium (wt.%)	0.110	0.103	0.116	0.105	0.115
Tl, Thallium (ppm)	42.6	39.3	45.8	40.5	44.7
Tm, Thulium (ppm)	0.16	0.13	0.18	IND	IND
U, Uranium (ppm)	3.77	3.40	4.13	3.59	3.95
V, Vanadium (ppm)	46.7	41.9	51.6	43.6	49.8
Y, Yttrium (ppm)	10.4	9.1	11.6	9.8	10.9
Yb, Ytterbium (ppm)	1.08	0.92	1.24	IND	IND
Zn, Zinc (wt.%)	17.34	16.89	17.80	17.02	17.67
4-Acid Digestion					
Ag, Silver (ppm)	235	229	241	231	239
Al, Aluminium (wt.%)	2.37	2.30	2.43	2.31	2.42
As, Arsenic (ppm)	299	285	313	287	311
Be, Beryllium (ppm)	1.29	1.14	1.44	1.23	1.35
Bi, Bismuth (ppm)	20.7	19.2	22.1	19.6	21.7
Ca, Calcium (wt.%)	2.13	2.07	2.20	2.09	2.18

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 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Cd, Cadmium (ppm)	460	441	479	453	468
Ce, Cerium (ppm)	32.0	27.8	36.3	30.9	33.2
Co, Cobalt (ppm)	94	91	98	92	97
Cr, Chromium (ppm)	60	55	64	57	62
Cs, Caesium (ppm)	1.50	1.39	1.62	1.42	1.59
Cu, Copper (wt.%)	0.915	0.889	0.940	0.901	0.928
Dy, Dysprosium (ppm)	1.46	1.15	1.77	1.36	1.55
Er, Erbium (ppm)	0.79	0.61	0.96	0.72	0.86
Eu, Europium (ppm)	0.52	0.38	0.66	IND	IND
Fe, Iron (wt.%)	9.06	8.81	9.31	8.85	9.27
Ga, Gallium (ppm)	7.99	7.44	8.54	7.62	8.36
Gd, Gadolinium (ppm)	2.13	1.89	2.38	1.97	2.29
Hf, Hafnium (ppm)	1.09	1.00	1.18	1.02	1.17
Ho, Holmium (ppm)	0.28	0.20	0.36	IND	IND
In, Indium (ppm)	1.61	1.45	1.77	1.53	1.70
K, Potassium (wt.%)	1.03	1.00	1.06	1.01	1.05
La, Lanthanum (ppm)	16.9	15.5	18.4	16.1	17.8
Li, Lithium (ppm)	13.1	12.1	14.1	12.6	13.5
Lu, Lutetium (ppm)	0.11	0.09	0.13	IND	IND
Mg, Magnesium (wt.%)	1.13	1.10	1.16	1.10	1.16
Mn, Manganese (wt.%)	0.314	0.306	0.323	0.308	0.321
Mo, Molybdenum (ppm)	17.7	16.7	18.7	16.8	18.5
Na, Sodium (wt.%)	0.091	0.086	0.095	0.088	0.093
Nb, Niobium (ppm)	1.79	1.52	2.06	1.69	1.90
Nd, Neodymium (ppm)	14.4	13.0	15.8	13.7	15.1
Ni, Nickel (ppm)	26.9	25.6	28.2	25.8	28.1
P, Phosphorus (wt.%)	0.023	0.021	0.025	0.022	0.024
Pb, Lead (wt.%)	12.13	11.81	12.46	11.95	12.32
Pr, Praseodymium (ppm)	4.02	3.74	4.30	3.80	4.24
Rb, Rubidium (ppm)	46.1	42.5	49.7	44.6	47.6
Re, Rhenium (ppb)	10.9	8.5	13.3	IND	IND
S, Sulphur (wt.%)	16.30	15.70	16.89	15.89	16.70
Sb, Antimony (ppm)	203	190	215	195	211
Sc, Scandium (ppm)	3.80	3.61	3.99	3.64	3.97
Se, Selenium (ppm)	5.94	4.84	7.05	5.37	6.52
Sm, Samarium (ppm)	2.54	2.09	2.99	2.37	2.70
Sn, Tin (ppm)	2.05	1.88	2.21	1.91	2.19

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 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Sr, Strontium (ppm)	33.0	30.9	35.1	31.7	34.3
Tb, Terbium (ppm)	0.28	0.21	0.35	IND	IND
Te, Tellurium (ppm)	1.53	1.33	1.73	1.45	1.61
Th, Thorium (ppm)	4.58	4.18	4.98	4.39	4.77
Ti, Titanium (wt.%)	0.075	0.070	0.081	0.073	0.078
Tl, Thallium (ppm)	40.4	36.4	44.3	39.0	41.7
U, Uranium (ppm)	3.63	3.40	3.86	3.47	3.79
V, Vanadium (ppm)	46.3	44.5	48.0	44.9	47.7
W, Tungsten (ppm)	1.52	1.36	1.67	1.39	1.64
Y, Yttrium (ppm)	7.92	7.03	8.82	7.52	8.32
Yb, Ytterbium (ppm)	0.82	0.64	1.00	IND	IND
Zn, Zinc (wt.%)	17.50	17.04	17.96	17.30	17.70
Zr, Zirconium (ppm)	37.9	35.6	40.2	36.6	39.2
Aqua Regia Digestion					
Ag, Silver (ppm)	233	229	237	228	237
Al, Aluminium (wt.%)	0.423	0.397	0.450	0.411	0.436
As, Arsenic (ppm)	303	290	315	294	312
B, Boron (ppm)	< 10	IND	IND	IND	IND
Be, Beryllium (ppm)	0.34	0.29	0.38	0.31	0.36
Bi, Bismuth (ppm)	20.5	19.0	22.0	19.5	21.5
Ca, Calcium (wt.%)	2.07	1.99	2.16	2.02	2.13
Cd, Cadmium (ppm)	444	428	461	435	454
Ce, Cerium (ppm)	16.2	14.3	18.1	15.5	16.9
Co, Cobalt (ppm)	92	87	98	91	94
Cr, Chromium (ppm)	21.0	19.8	22.2	20.1	21.9
Cs, Caesium (ppm)	0.58	0.53	0.64	0.55	0.61
Cu, Copper (wt.%)	0.897	0.853	0.940	0.879	0.915
Dy, Dysprosium (ppm)	0.80	0.57	1.04	0.74	0.86
Fe, Iron (wt.%)	8.79	8.39	9.18	8.57	9.00
Ga, Gallium (ppm)	2.42	2.21	2.63	2.33	2.51
Gd, Gadolinium (ppm)	1.08	0.70	1.46	0.98	1.19
Ge, Germanium (ppm)	0.25	0.16	0.34	0.22	0.28
Hf, Hafnium (ppm)	0.29	0.26	0.33	0.27	0.31
Hg, Mercury (ppm)	6.91	6.37	7.45	6.62	7.20
In, Indium (ppm)	1.69	1.58	1.79	1.64	1.74
K, Potassium (wt.%)	0.143	0.131	0.154	0.138	0.148
La, Lanthanum (ppm)	5.55	4.98	6.12	5.27	5.83

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. 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 1 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion continued					
Li, Lithium (ppm)	5.05	4.47	5.64	4.84	5.26
Mg, Magnesium (wt.%)	1.01	0.97	1.05	0.98	1.03
Mn, Manganese (wt.%)	0.314	0.301	0.327	0.307	0.321
Mo, Molybdenum (ppm)	18.3	17.2	19.4	17.8	18.8
Nb, Niobium (ppm)	0.21	0.17	0.25	0.20	0.22
Ni, Nickel (ppm)	25.0	23.4	26.6	24.2	25.9
P, Phosphorus (wt.%)	0.020	0.019	0.022	0.020	0.021
Pb, Lead (wt.%)	12.06	11.71	12.40	11.87	12.24
Rb, Rubidium (ppm)	7.05	6.06	8.05	6.72	7.39
Re, Rhenium (ppb)	11.0	9.2	12.8	IND	IND
S, Sulphur (wt.%)	15.68	15.12	16.24	15.28	16.08
Sb, Antimony (ppm)	183	171	195	178	189
Sc, Scandium (ppm)	1.00	0.85	1.15	IND	IND
Se, Selenium (ppm)	7.07	5.30	8.84	6.76	7.37
Sm, Samarium (ppm)	1.14	0.82	1.45	1.00	1.27
Sn, Tin (ppm)	1.16	1.06	1.27	IND	IND
Sr, Strontium (ppm)	14.0	12.3	15.6	13.4	14.5
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Te, Tellurium (ppm)	1.56	1.43	1.70	1.46	1.67
Th, Thorium (ppm)	3.11	2.73	3.49	2.95	3.28
Ti, Titanium (ppm)	62	51	72	IND	IND
Tl, Thallium (ppm)	33.9	31.8	36.1	32.8	35.1
U, Uranium (ppm)	2.99	2.81	3.18	2.91	3.08
V, Vanadium (ppm)	8.09	6.94	9.23	7.57	8.60
W, Tungsten (ppm)	0.68	0.59	0.78	0.63	0.74
Y, Yttrium (ppm)	3.82	3.34	4.29	3.66	3.97
Zn, Zinc (wt.%)	17.38	16.79	17.97	17.08	17.68
Zr, Zirconium (ppm)	9.03	7.95	10.10	8.54	9.52

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. 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. Indicative Values for OREAS 320.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
As	ppm	*272*	Na_2O	wt.%	*2.18*	Sr	ppm	*65*
Bi	ppm	*83*	Nb	ppm	*95*	Ta	ppm	*267*
CeO_2	ppm	*< 100*	Nd	ppm	*< 90*	Th	ppm	*142*
Co	ppm	*132*	Ni	ppm	*98*	V	ppm	*97*
Cr	ppm	*93*	Pr	ppm	*131*	W	ppm	*< 10*
Gd	ppm	*< 90*	Rb	ppm	*63*	Y_2O_3	ppm	*< 50*
HfO_2	ppm	*< 100*	Sb	ppm	*276*	Zr	ppm	*406*
La_2O_3	ppm	*< 100*	Sm	ppm	*< 90*			
Mo	ppm	*40.8*	Sn	ppm	*< 50*			
Borate / Peroxide Fusion ICP								
Ag	ppm	*231*	Nb	ppm	*4.93*	Sn	ppm	*2.73*
B	ppm	*72*	Ni	ppm	*31.9*	Ta	ppm	*0.38*
Be	ppm	*1.39*	Re	ppm	*< 0.1*	Te	ppm	*1.46*
Hf	ppm	*3.44*	Sc	ppm	*3.93*	W	ppm	*2.30*
Na	wt.%	*0.082*	Se	ppm	*5.25*	Zr	ppm	*49.6*
4-Acid Digestion								
B	ppm	*54*	Ge	ppm	*0.42*	Ta	ppm	*0.14*
Ba	ppm	*256*	Hg	ppm	*4.86*	Tm	ppm	*0.10*
Aqua Regia Digestion								
Au	ppm	*0.066*	Lu	ppm	*0.052*	Pt	ppb	*< 10*
Ba	ppm	*19.0*	Na	wt.%	*0.010*	Tb	ppm	*0.14*
Er	ppm	*0.39*	Nd	ppm	*6.54*	Tm	ppm	*0.048*
Eu	ppm	*0.25*	Pd	ppb	*58.3*	Yb	ppm	*0.39*
Ho	ppm	*0.15*	Pr	ppm	*1.42*			

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.

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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 handling and correct use' should be read carefully.

Table 1 provides the certified values and their associated 95 % expanded uncertainty and tolerance intervals, Table 2 shows indicative values including major and trace element characterisation, Table 3 provides some indicative physical properties, Table 4 shows indicative mineralogy by semi-quantitative XRD analysis and Table 5 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 320-DataPack.1.0.260213_113350.xlsx**). The certified values and uncertainties in this Certificate are the sole authoritative figures. Any additional significant figures in the DataPack are provided for reference only and do not affect the certified results.

Results are also presented in scatter plots for Zn, Pb and Cu by fusion XRF and Zn, Pb, Ag and Cu by 4-acid digestion in Figures 1 to 7 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 320 is a certified reference material (CRM) prepared from a blend of polymetallic zinc–lead ores and Zn, Cu, Pb and Ag concentrates together with barren black slate. The barren black slate was sourced from a quarry in Victoria. The principal ores and concentrates were obtained from the George Fisher deposit (Mount Isa district), Dugald River (Queensland) and Rosebery (Tasmania), Australia. OREAS 320 is representative of polymetallic Zn–Pb mineralisation dominated by sphalerite (ZnS) and galena (PbS), with silver occurring both within galena and as minor silver-bearing sulphosalts. Associated sulphides include pyrite and pyrrhotite with lesser chalcopyrite, hosted within a black slate matrix.

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials constituting OREAS 320 was prepared in the following manner:

- Drying to constant mass at 105 °C;
- multi-stage milling to approximately 98% less than 75 microns;
- Preliminary homogenisation of the ores and barren materials;

- Representative sampling and check assaying of ore source material;
- Blending the ore and barren black slate in appropriate proportions to achieve target grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10 g units sealed under nitrogen in laminated foil pouches.

PHYSICAL PROPERTIES

OREAS 320 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 3 presents these findings that should be used for informational purposes only.

Table 3. Physical properties of OREAS 320.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
963	0.44	N3	Dark Gray

‡The Munsell Rock Color 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 4 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. Some amorphous material may be present. 'Kandite group' appears to be mainly kaolinite. A trace of apatite, tourmaline group, serpentine, gypsum and plagioclase may be present.

Table 4. Indicative mineralogy of OREAS 320 by semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Chalcopyrite	2
Pyrrhotite	5
Pyrite	5
Sphalerite	27
Galena	11
Goethite	1
Kandite group	< 1
Chlorite	1
Annite - biotite - phlogopite	< 1
Muscovite - illite	9
K-feldspar and/or rutile	4
Quartz	24
Magnesite - siderite	2
Dolomite - ankerite	9

ANALYTICAL PROGRAM

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

- Lithium borate fusion whole rock analysis package by X-ray fluorescence (up to 16 laboratories depending on the element);
- Thermogravimetry: Loss on Ignition (LOI) at 1000 °C (4 laboratories used a thermogravimetric analyser, 4 laboratories included LOI with their fusion package and 6 laboratories used a conventional muffle furnace);
- Total C and S by IR combustion furnace (up to 21 laboratories depending on the element);
- Lithium borate or sodium peroxide fusion with full suite ICP-OES and ICP-MS elemental packages (up to 18 laboratories depending on the element);
- 4-acid (HNO_3-HF-$HClO_4$-HCl) digestion with full suite ICP-OES and ICP-MS elemental packages (up to 22 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by aqua regia digestion (up to 21 laboratories depending on the element).

For the round robin program, six 2 kg test units were collected at predetermined intervals during the bagging stage, immediately after homogenisation. These units are considered representative of the entire prepared batch. Each participating laboratory received six test portions, obtained by subsampling 15 g from each of the six distinct 2 kg units.

Homogeneity was assessed by submitting 12 pulp samples to a single laboratory for analysis. Paired 10 g samples were drawn from each of the six test units, enabling an Analysis of Variance (ANOVA) to compare within-unit and between-unit variances. This statistical method provides a relative measure of homogeneity and tests the null hypothesis that all units derive from the same population distribution (refer to the 'Homogeneity Evaluation' section below).

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). Outlier evaluation was conducted in accordance with ISO 17034:2017 and ISO 33405:2024. While formal statistical tests were applied, professional statistical judgment was also exercised in determining the validity of potential outliers. Assessment of systematic bias and performance using independent control materials (CRMs) was incorporated to ensure compliance with the referenced standards and to establish metrological traceability of the certified values.

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 [5] and [14]. All known or suspected sources of bias have been investigated or taken into account.

Indicative (uncertified) values (Table 2) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor. This data is intended for 'informational purposes' only.

Standard Deviation intervals (see Table 5, ‘Performance Gates’) 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.***

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Table 1 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for Cu by fusion XRF, where 99 % of the time (1-α=0.99) at least 95 % of subsamples (ρ=0.95) will have concentrations lying between 0.898 and 0.924 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. ***Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.***

Analysis of Variance (ANOVA) Study

Homogeneity of OREAS 320 was evaluated using an ANOVA study in addition to the precision error method. Twelve 10 g pulp samples (paired from six sampling units) were analysed for Cu and Zn by oxidising fusion with X-ray fluorescence finish (ME-XRF15b). The ANOVA compares within- and between-unit variances to test the null hypothesis that all units are drawn from the same population. A p-value < 0.05 indicates statistically significant heterogeneity between units.

Both p-values for Cu and Zn were statistically insignificant, and the null hypothesis is retained. ANOVA provides a relative measure of homogeneity, and poor absolute homogeneity could theoretically pass if within-unit variance is consistent across units. Cu and Zn are minor constituents in OREAS 320, and achieving homogeneity for minor constituents is generally more challenging than for major constituents. Therefore, the successful demonstration of homogeneity for Cu and Zn provides strong evidence that the material is also homogeneous for all certified analytes. This approach is consistent with ISO Guide 35:2017, which allows the use of representative analytes to infer homogeneity of the full suite of certified properties.

Based on this statistical analysis and the results of the interlaboratory certification program, OREAS 320 is concluded to be fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

PERFORMANCE GATES

Table 5 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 5. Performance Gates for OREAS 320.

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
Borate Fusion XRF											
Al_2O_3, wt.%	4.53	0.078	4.37	4.68	4.29	4.76	1.73%	3.47%	5.20%	4.30	4.75
BaO, wt.%	0.159	0.010	0.139	0.179	0.129	0.189	6.32%	12.65%	18.97%	0.151	0.167
CaO, wt.%	3.01	0.054	2.90	3.11	2.84	3.17	1.81%	3.62%	5.43%	2.86	3.16
Cu, wt.%	0.911	0.046	0.818	1.004	0.772	1.050	5.09%	10.18%	15.26%	0.865	0.957
Fe_2O_3, wt.%	12.76	0.187	12.39	13.14	12.20	13.32	1.46%	2.93%	4.39%	12.12	13.40
K_2O, wt.%	1.22	0.019	1.18	1.26	1.16	1.28	1.59%	3.18%	4.77%	1.16	1.28
MgO, wt.%	1.95	0.071	1.80	2.09	1.73	2.16	3.64%	7.28%	10.92%	1.85	2.04
MnO_2, wt.%	0.507	0.015	0.477	0.537	0.462	0.552	2.97%	5.95%	8.92%	0.481	0.532
P_2O_5, wt.%	0.061	0.009	0.043	0.079	0.034	0.089	14.97%	29.93%	44.90%	0.058	0.064
Pb, wt.%	11.97	0.276	11.42	12.52	11.14	12.80	2.31%	4.62%	6.92%	11.37	12.57
S, wt.%	16.43	0.893	14.64	18.22	13.75	19.11	5.44%	10.88%	16.31%	15.61	17.25
SiO_2, wt.%	25.17	0.532	24.10	26.23	23.57	26.76	2.11%	4.23%	6.34%	23.91	26.43
TiO_2, wt.%	0.187	0.011	0.166	0.208	0.155	0.219	5.65%	11.31%	16.96%	0.178	0.197
Zn, wt.%	17.16	0.469	16.22	18.10	15.76	18.57	2.73%	5.47%	8.20%	16.30	18.02
Thermogravimetry											
LOI^{1000}, wt.%	12.32	0.784	10.76	13.89	9.97	14.67	6.36%	12.72%	19.07%	11.71	12.94
Infrared Combustion											
C, wt.%	1.67	0.035	1.59	1.74	1.56	1.77	2.13%	4.26%	6.39%	1.58	1.75
S, wt.%	16.70	0.509	15.68	17.71	15.17	18.22	3.05%	6.09%	9.14%	15.86	17.53
Borate / Peroxide Fusion ICP											
Al, wt.%	2.39	0.051	2.29	2.50	2.24	2.55	2.13%	4.25%	6.38%	2.28	2.51
As, ppm	309	21	268	350	247	371	6.68%	13.37%	20.05%	294	325
Ba, wt.%	0.145	0.007	0.131	0.158	0.124	0.165	4.77%	9.54%	14.31%	0.137	0.152
Bi, ppm	20.5	1.83	16.9	24.2	15.0	26.0	8.93%	17.86%	26.79%	19.5	21.5

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

Notes: intervals may appear asymmetric due to rounding; 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 5 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
Borate / Peroxide Fusion ICP											
Ca, wt.%	2.13	0.076	1.98	2.28	1.90	2.36	3.55%	7.10%	10.65%	2.02	2.24
Cd, ppm	478	14	449	506	435	520	2.96%	5.93%	8.89%	454	502
Ce, ppm	39.4	2.46	34.5	44.3	32.0	46.8	6.25%	12.51%	18.76%	37.4	41.4
Co, ppm	91	8.2	74	107	66	115	9.02%	18.05%	27.07%	86	95
Cr, ppm	69	8	52	86	44	94	12.27%	24.53%	36.80%	65	72
Cs, ppm	1.71	0.18	1.34	2.07	1.16	2.25	10.65%	21.30%	31.96%	1.62	1.79
Cu, wt.%	0.905	0.019	0.868	0.942	0.849	0.961	2.06%	4.12%	6.17%	0.860	0.950
Dy, ppm	1.86	0.25	1.37	2.36	1.12	2.61	13.36%	26.72%	40.08%	1.77	1.96
Er, ppm	1.06	0.14	0.78	1.33	0.64	1.47	13.17%	26.33%	39.50%	1.00	1.11
Eu, ppm	0.59	0.053	0.48	0.69	0.43	0.75	9.03%	18.06%	27.09%	0.56	0.62
Fe, wt.%	9.04	0.331	8.38	9.71	8.05	10.04	3.66%	7.31%	10.97%	8.59	9.50
Ga, ppm	8.63	0.633	7.37	9.90	6.74	10.53	7.33%	14.66%	21.99%	8.20	9.07
Gd, ppm	2.37	0.148	2.07	2.67	1.92	2.81	6.25%	12.51%	18.76%	2.25	2.49
Ge, ppm	13.1	1.08	11.0	15.3	9.9	16.4	8.20%	16.41%	24.61%	12.5	13.8
Ho, ppm	0.39	0.013	0.37	0.42	0.35	0.43	3.34%	6.68%	10.03%	0.37	0.41
In, ppm	1.67	0.19	1.29	2.05	1.09	2.25	11.50%	23.01%	34.51%	1.59	1.75
K, wt.%	1.07	0.071	0.93	1.21	0.86	1.28	6.62%	13.23%	19.85%	1.02	1.12
La, ppm	21.5	2.9	15.8	27.2	12.9	30.0	13.30%	26.59%	39.89%	20.4	22.5
Li, ppm	14.4	1.8	10.8	18.1	8.9	19.9	12.71%	25.43%	38.14%	13.7	15.2
Lu, ppm	0.16	0.02	0.11	0.21	0.09	0.23	15.32%	30.63%	45.95%	0.15	0.17
Mg, wt.%	1.14	0.032	1.08	1.21	1.05	1.24	2.79%	5.58%	8.36%	1.09	1.20
Mn, wt.%	0.319	0.008	0.303	0.336	0.294	0.344	2.61%	5.22%	7.82%	0.303	0.335
Mo, ppm	17.3	2.1	13.0	21.6	10.9	23.8	12.39%	24.77%	37.16%	16.5	18.2
Nd, ppm	15.4	1.06	13.3	17.6	12.2	18.6	6.88%	13.76%	20.63%	14.7	16.2
P, wt.%	0.024	0.005	0.015	0.033	0.010	0.038	19.34%	38.68%	58.02%	0.023	0.025
Pb, wt.%	12.06	0.404	11.25	12.86	10.85	13.27	3.35%	6.69%	10.04%	11.45	12.66
Pr, ppm	4.23	0.382	3.47	4.99	3.09	5.38	9.02%	18.04%	27.06%	4.02	4.44
Rb, ppm	48.2	2.09	44.0	52.4	41.9	54.5	4.35%	8.69%	13.04%	45.8	50.6
S, wt.%	16.70	0.472	15.75	17.64	15.28	18.11	2.83%	5.66%	8.49%	15.86	17.53
Sb, ppm	208	31	147	269	116	300	14.73%	29.46%	44.19%	197	218
Si, wt.%	12.12	0.363	11.39	12.84	11.03	13.21	3.00%	5.99%	8.99%	11.51	12.72
Sm, ppm	2.65	0.220	2.21	3.09	1.99	3.31	8.28%	16.55%	24.83%	2.52	2.79
Sr, ppm	41.5	5.9	29.6	53.4	23.7	59.3	14.28%	28.57%	42.85%	39.4	43.6
Tb, ppm	0.31	0.021	0.27	0.35	0.25	0.37	6.81%	13.62%	20.44%	0.29	0.32
Th, ppm	5.14	0.57	3.99	6.29	3.42	6.86	11.16%	22.32%	33.49%	4.88	5.40
Ti, wt.%	0.110	0.007	0.096	0.123	0.089	0.130	6.15%	12.29%	18.44%	0.104	0.115
Tl, ppm	42.6	1.98	38.6	46.5	36.6	48.5	4.65%	9.31%	13.96%	40.4	44.7
Tm, ppm	0.16	0.014	0.13	0.18	0.11	0.20	9.03%	18.07%	27.10%	0.15	0.16
U, ppm	3.77	0.279	3.21	4.33	2.93	4.61	7.41%	14.83%	22.24%	3.58	3.96
V, ppm	46.7	4.36	38.0	55.4	33.7	59.8	9.32%	18.64%	27.96%	44.4	49.1
Y, ppm	10.4	1.1	8.2	12.5	7.1	13.6	10.31%	20.62%	30.93%	9.8	10.9
Yb, ppm	1.08	0.071	0.94	1.22	0.87	1.29	6.52%	13.04%	19.56%	1.03	1.14
Zn, wt.%	17.34	0.445	16.45	18.23	16.01	18.68	2.57%	5.14%	7.71%	16.48	18.21

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

Notes: intervals may appear asymmetric due to rounding; 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.

IND = indeterminate.

Table 5 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											
Ag, ppm	235	6	222	248	216	254	2.75%	5.49%	8.24%	223	247
Al, wt.%	2.37	0.089	2.19	2.54	2.10	2.63	3.77%	7.53%	11.30%	2.25	2.48
As, ppm	299	16	266	332	250	348	5.50%	11.01%	16.51%	284	314
Be, ppm	1.29	0.14	1.00	1.57	0.86	1.71	11.03%	22.06%	33.09%	1.22	1.35
Bi, ppm	20.7	2.2	16.4	25.0	14.2	27.2	10.46%	20.92%	31.37%	19.6	21.7
Ca, wt.%	2.13	0.089	1.96	2.31	1.87	2.40	4.18%	8.36%	12.53%	2.03	2.24
Cd, ppm	460	29	402	518	373	547	6.28%	12.57%	18.85%	437	483
Ce, ppm	32.0	7.5	17.1	47.0	9.6	54.5	23.38%	46.77%	70.15%	30.4	33.7
Co, ppm	94	5.4	84	105	78	111	5.69%	11.39%	17.08%	90	99
Cr, ppm	60	8	44	75	36	83	12.97%	25.95%	38.92%	57	63
Cs, ppm	1.50	0.15	1.20	1.81	1.04	1.97	10.26%	20.53%	30.79%	1.43	1.58
Cu, wt.%	0.915	0.030	0.855	0.975	0.825	1.005	3.27%	6.54%	9.82%	0.869	0.960
Dy, ppm	1.46	0.30	0.85	2.06	0.55	2.37	20.79%	41.57%	62.36%	1.39	1.53
Er, ppm	0.79	0.18	0.43	1.14	0.25	1.32	22.67%	45.35%	68.02%	0.75	0.83
Eu, ppm	0.52	0.11	0.30	0.74	0.19	0.84	20.94%	41.88%	62.83%	0.49	0.54
Fe, wt.%	9.06	0.301	8.46	9.67	8.16	9.97	3.32%	6.65%	9.97%	8.61	9.52
Ga, ppm	7.99	0.632	6.73	9.25	6.10	9.89	7.90%	15.81%	23.71%	7.59	8.39
Gd, ppm	2.13	0.194	1.74	2.52	1.55	2.71	9.09%	18.18%	27.27%	2.02	2.24
Hf, ppm	1.09	0.080	0.93	1.25	0.85	1.33	7.28%	14.56%	21.83%	1.04	1.15
Ho, ppm	0.28	0.07	0.15	0.42	0.08	0.48	23.84%	47.67%	71.51%	0.27	0.30
In, ppm	1.61	0.26	1.10	2.13	0.84	2.38	15.95%	31.90%	47.85%	1.53	1.69
K, wt.%	1.03	0.029	0.97	1.09	0.94	1.12	2.86%	5.71%	8.57%	0.98	1.08
La, ppm	16.9	2.6	11.7	22.2	9.1	24.8	15.50%	31.00%	46.49%	16.1	17.8
Li, ppm	13.1	1.24	10.6	15.6	9.4	16.8	9.44%	18.89%	28.33%	12.4	13.7
Lu, ppm	0.11	0.02	0.08	0.14	0.06	0.16	15.33%	30.66%	45.99%	0.10	0.11
Mg, wt.%	1.13	0.054	1.02	1.24	0.97	1.29	4.80%	9.61%	14.41%	1.07	1.19
Mn, wt.%	0.314	0.010	0.295	0.333	0.285	0.343	3.06%	6.12%	9.17%	0.299	0.330
Mo, ppm	17.7	1.52	14.6	20.7	13.1	22.2	8.62%	17.24%	25.86%	16.8	18.6
Na, wt.%	0.091	0.008	0.074	0.107	0.066	0.116	9.09%	18.18%	27.26%	0.086	0.095
Nb, ppm	1.79	0.41	0.97	2.61	0.56	3.02	22.91%	45.83%	68.74%	1.70	1.88
Nd, ppm	14.4	1.6	11.2	17.6	9.6	19.2	11.17%	22.34%	33.51%	13.7	15.1
Ni, ppm	26.9	1.73	23.4	30.4	21.7	32.1	6.45%	12.89%	19.34%	25.6	28.2
P, wt.%	0.023	0.002	0.019	0.027	0.017	0.029	8.39%	16.77%	25.16%	0.022	0.024
Pb, wt.%	12.13	0.225	11.68	12.58	11.46	12.81	1.85%	3.70%	5.56%	11.53	12.74
Pr, ppm	4.02	0.154	3.71	4.32	3.56	4.48	3.82%	7.65%	11.47%	3.82	4.22
Rb, ppm	46.1	5.4	35.2	56.9	29.8	62.4	11.79%	23.57%	35.36%	43.8	48.4
Re, ppb	10.9	1.6	7.7	14.1	6.1	15.7	14.61%	29.21%	43.82%	10.3	11.4
S, wt.%	16.30	0.773	14.75	17.84	13.98	18.62	4.74%	9.49%	14.23%	15.48	17.11
Sb, ppm	203	19	164	241	145	261	9.53%	19.06%	28.59%	193	213
Sc, ppm	3.80	0.211	3.38	4.23	3.17	4.44	5.56%	11.11%	16.67%	3.61	3.99
Se, ppm	5.94	1.68	2.58	9.31	0.89	10.99	28.32%	56.65%	84.97%	5.65	6.24
Sm, ppm	2.54	0.48	1.57	3.50	1.09	3.98	18.99%	37.97%	56.96%	2.41	2.66
Sn, ppm	2.05	0.166	1.71	2.38	1.55	2.55	8.12%	16.24%	24.35%	1.94	2.15
Sr, ppm	33.0	3.14	26.7	39.3	23.6	42.4	9.53%	19.06%	28.59%	31.3	34.6

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

Notes: intervals may appear asymmetric due to rounding; 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 5 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											
Tb, ppm	0.28	0.07	0.14	0.42	0.08	0.48	24.34%	48.68%	73.02%	0.27	0.29
Te, ppm	1.53	0.26	1.01	2.05	0.75	2.31	16.98%	33.96%	50.93%	1.46	1.61
Th, ppm	4.58	0.61	3.37	5.79	2.77	6.40	13.21%	26.42%	39.64%	4.35	4.81
Ti, wt.%	0.075	0.012	0.051	0.099	0.040	0.111	15.82%	31.64%	47.45%	0.071	0.079
Tl, ppm	40.4	7.1	26.1	54.6	19.0	61.8	17.67%	35.34%	53.01%	38.3	42.4
U, ppm	3.63	0.356	2.92	4.34	2.56	4.70	9.80%	19.60%	29.39%	3.45	3.81
V, ppm	46.3	2.33	41.6	50.9	39.3	53.3	5.04%	10.09%	15.13%	44.0	48.6
W, ppm	1.52	0.21	1.10	1.93	0.90	2.13	13.57%	27.14%	40.72%	1.44	1.59
Y, ppm	7.92	1.48	4.97	10.87	3.50	12.35	18.63%	37.26%	55.89%	7.53	8.32
Yb, ppm	0.82	0.20	0.43	1.21	0.23	1.41	23.96%	47.93%	71.89%	0.78	0.86
Zn, wt.%	17.50	0.587	16.33	18.67	15.74	19.26	3.35%	6.71%	10.06%	16.62	18.37
Zr, ppm	37.9	3.29	31.4	44.5	28.1	47.8	8.68%	17.35%	26.03%	36.0	39.8
Aqua Regia Digestion											
Ag, ppm	233	6	220	245	214	252	2.72%	5.44%	8.16%	221	244
Al, wt.%	0.423	0.033	0.358	0.489	0.325	0.522	7.76%	15.52%	23.28%	0.402	0.445
As, ppm	303	18	268	338	250	356	5.84%	11.67%	17.51%	288	318
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Be, ppm	0.34	0.06	0.22	0.46	0.16	0.52	17.76%	35.51%	53.27%	0.32	0.35
Bi, ppm	20.5	1.87	16.8	24.2	14.9	26.1	9.14%	18.28%	27.42%	19.5	21.5
Ca, wt.%	2.07	0.115	1.84	2.31	1.73	2.42	5.55%	11.10%	16.65%	1.97	2.18
Cd, ppm	444	19	406	483	387	502	4.29%	8.59%	12.88%	422	467
Ce, ppm	16.2	2.3	11.5	20.8	9.2	23.2	14.34%	28.68%	43.03%	15.4	17.0
Co, ppm	92	8.6	75	110	66	118	9.33%	18.66%	27.99%	88	97
Cr, ppm	21.0	1.45	18.1	23.9	16.7	25.4	6.89%	13.78%	20.67%	20.0	22.1
Cs, ppm	0.58	0.07	0.45	0.72	0.38	0.79	11.52%	23.04%	34.57%	0.55	0.61
Cu, wt.%	0.897	0.057	0.783	1.011	0.726	1.068	6.36%	12.72%	19.07%	0.852	0.942
Dy, ppm	0.80	0.16	0.47	1.13	0.31	1.29	20.46%	40.92%	61.38%	0.76	0.84
Fe, wt.%	8.79	0.466	7.85	9.72	7.39	10.18	5.31%	10.61%	15.92%	8.35	9.22
Ga, ppm	2.42	0.27	1.89	2.95	1.62	3.22	11.00%	22.01%	33.01%	2.30	2.54
Gd, ppm	1.08	0.26	0.56	1.61	0.29	1.87	24.31%	48.63%	72.94%	1.03	1.14
Ge, ppm	0.25	0.10	0.05	0.45	0.00	0.55	40.59%	81.18%	121.77%	0.24	0.26
Hf, ppm	0.29	0.03	0.23	0.35	0.20	0.38	10.31%	20.63%	30.94%	0.28	0.31
Hg, ppm	6.91	0.634	5.64	8.18	5.01	8.81	9.17%	18.33%	27.50%	6.57	7.26
In, ppm	1.69	0.104	1.48	1.90	1.37	2.00	6.18%	12.35%	18.53%	1.60	1.77
K, wt.%	0.143	0.018	0.106	0.180	0.088	0.198	12.89%	25.79%	38.68%	0.136	0.150
La, ppm	5.55	0.77	4.01	7.09	3.23	7.86	13.91%	27.81%	41.72%	5.27	5.83
Li, ppm	5.05	0.65	3.74	6.36	3.09	7.01	12.94%	25.89%	38.83%	4.80	5.30
Mg, wt.%	1.01	0.036	0.94	1.08	0.90	1.12	3.58%	7.17%	10.75%	0.96	1.06
Mn, wt.%	0.314	0.017	0.279	0.348	0.262	0.366	5.51%	11.02%	16.52%	0.298	0.329
Mo, ppm	18.3	1.02	16.3	20.3	15.2	21.4	5.56%	11.13%	16.69%	17.4	19.2
Nb, ppm	0.21	0.05	0.12	0.30	0.07	0.35	21.94%	43.89%	65.83%	0.20	0.22
Ni, ppm	25.0	1.99	21.0	29.0	19.0	31.0	7.95%	15.90%	23.85%	23.8	26.3
P, wt.%	0.020	0.001	0.018	0.023	0.017	0.024	6.02%	12.05%	18.07%	0.019	0.021

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

Notes: intervals may appear asymmetric due to rounding; 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.

IND = indeterminate.

Table 5 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											
Pb, wt.%	12.06	0.345	11.37	12.75	11.02	13.09	2.86%	5.73%	8.59%	11.45	12.66
Rb, ppm	7.05	1.35	4.35	9.76	2.99	11.12	19.19%	38.39%	57.58%	6.70	7.41
Re, ppb	11.0	1.3	8.4	13.5	7.1	14.8	11.70%	23.41%	35.11%	10.4	11.5
S, wt.%	15.68	0.477	14.72	16.63	14.25	17.11	3.04%	6.08%	9.12%	14.89	16.46
Sb, ppm	183	20	143	224	122	244	11.05%	22.10%	33.15%	174	192
Sc, ppm	1.00	0.22	0.56	1.45	0.34	1.67	22.13%	44.26%	66.38%	0.95	1.05
Se, ppm	7.07	2.70	1.66	12.47	0.00	15.17	38.24%	76.48%	114.72%	6.71	7.42
Sm, ppm	1.14	0.23	0.68	1.59	0.46	1.81	19.86%	39.72%	59.58%	1.08	1.19
Sn, ppm	1.16	0.095	0.97	1.35	0.88	1.45	8.19%	16.38%	24.57%	1.10	1.22
Sr, ppm	14.0	2.6	8.9	19.1	6.3	21.6	18.29%	36.58%	54.86%	13.3	14.7
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Te, ppm	1.56	0.18	1.20	1.93	1.01	2.12	11.77%	23.54%	35.31%	1.49	1.64
Th, ppm	3.11	0.58	1.96	4.27	1.38	4.85	18.58%	37.16%	55.75%	2.96	3.27
Ti, ppm	62	12	38	85	27	96	18.79%	37.58%	56.37%	58	65
Tl, ppm	33.9	2.36	29.2	38.7	26.9	41.0	6.95%	13.89%	20.84%	32.2	35.6
U, ppm	2.99	0.172	2.65	3.34	2.48	3.51	5.73%	11.46%	17.19%	2.84	3.14
V, ppm	8.09	1.48	5.13	11.04	3.65	12.52	18.30%	36.60%	54.90%	7.68	8.49
W, ppm	0.68	0.10	0.48	0.89	0.37	0.99	15.10%	30.19%	45.29%	0.65	0.72
Y, ppm	3.82	0.69	2.44	5.19	1.75	5.88	18.04%	36.08%	54.12%	3.63	4.01
Zn, wt.%	17.38	0.703	15.98	18.79	15.27	19.49	4.04%	8.09%	12.13%	16.51	18.25
Zr, ppm	9.03	1.16	6.71	11.35	5.54	12.51	12.86%	25.71%	38.57%	8.58	9.48

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

Notes: intervals may appear asymmetric due to rounding; 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.

IND = indeterminate.

PREPARER AND SUPPLIER

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20. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
21. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
22. UIS Analytical Services, Centurion, South Africa

Please note: To maintain anonymity of participating laboratories, the alphabetical list above does not correspond to the Lab ID numbers shown in the scatter plots below.

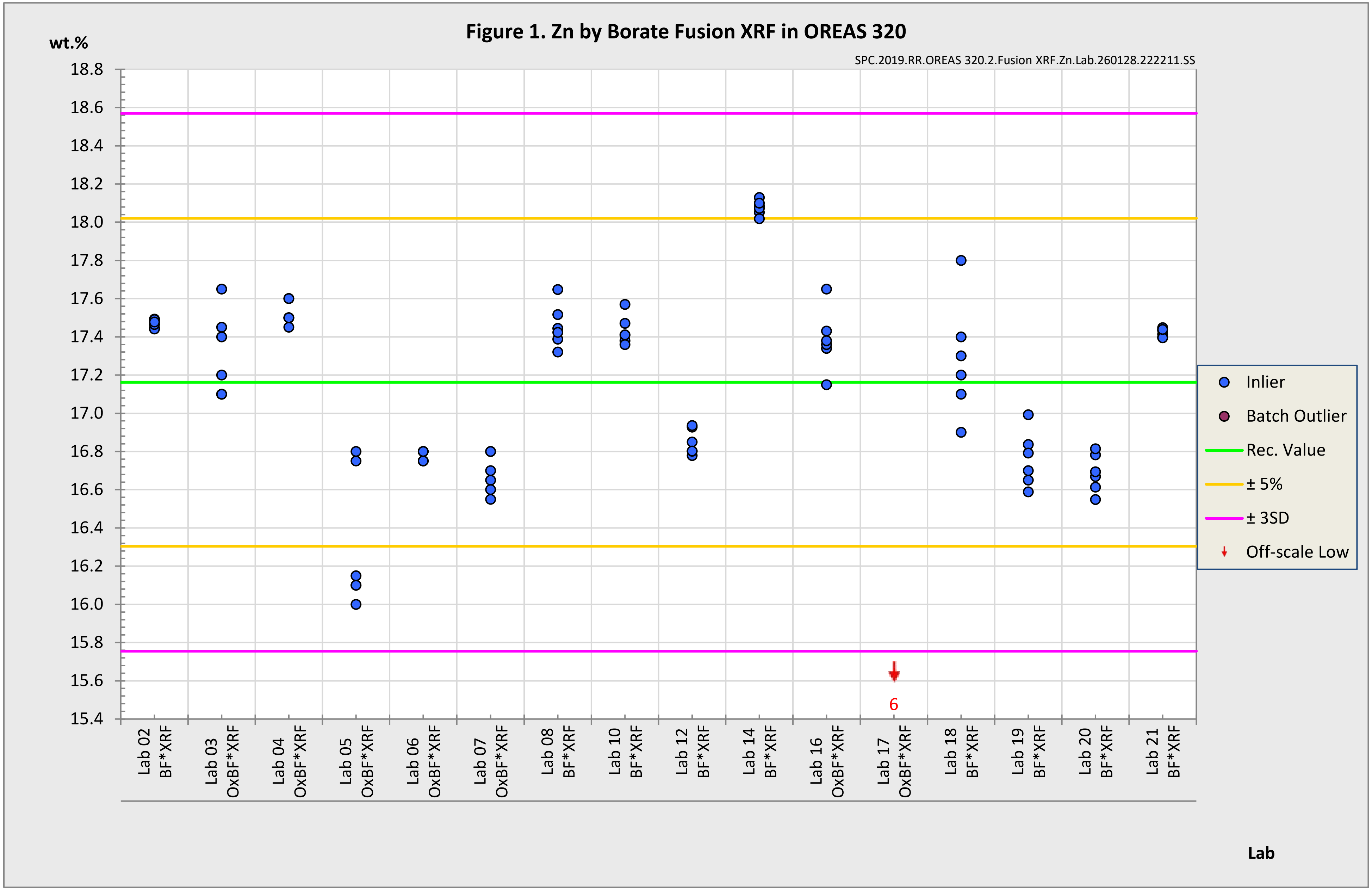

Figure 1. Zn by Borate Fusion XRF in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.Fusion XRF.Zn.Lab.260128.222211.SS
18.8
18.6
18.4
18.2
18.0
17.8
17.6
17.4
17.2
17.0
16.8
16.6
16.4
16.2
16.0
15.8
15.6
15.4
6
Lab 02 BF*XRF
Lab 03 OxBF*XRF
Lab 04 OxBF*XRF
Lab 05 OxBF*XRF
Lab 06 OxBF*XRF
Lab 07 OxBF*XRF
Lab 08 BF*XRF
Lab 10 BF*XRF
Lab 12 BF*XRF
Lab 14 BF*XRF
Lab 16 OxBF*XRF
Lab 17 OxBF*XRF
Lab 18 BF*XRF
Lab 19 BF*XRF
Lab 20 BF*XRF
Lab 21 BF*XRF
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale Low
Lab

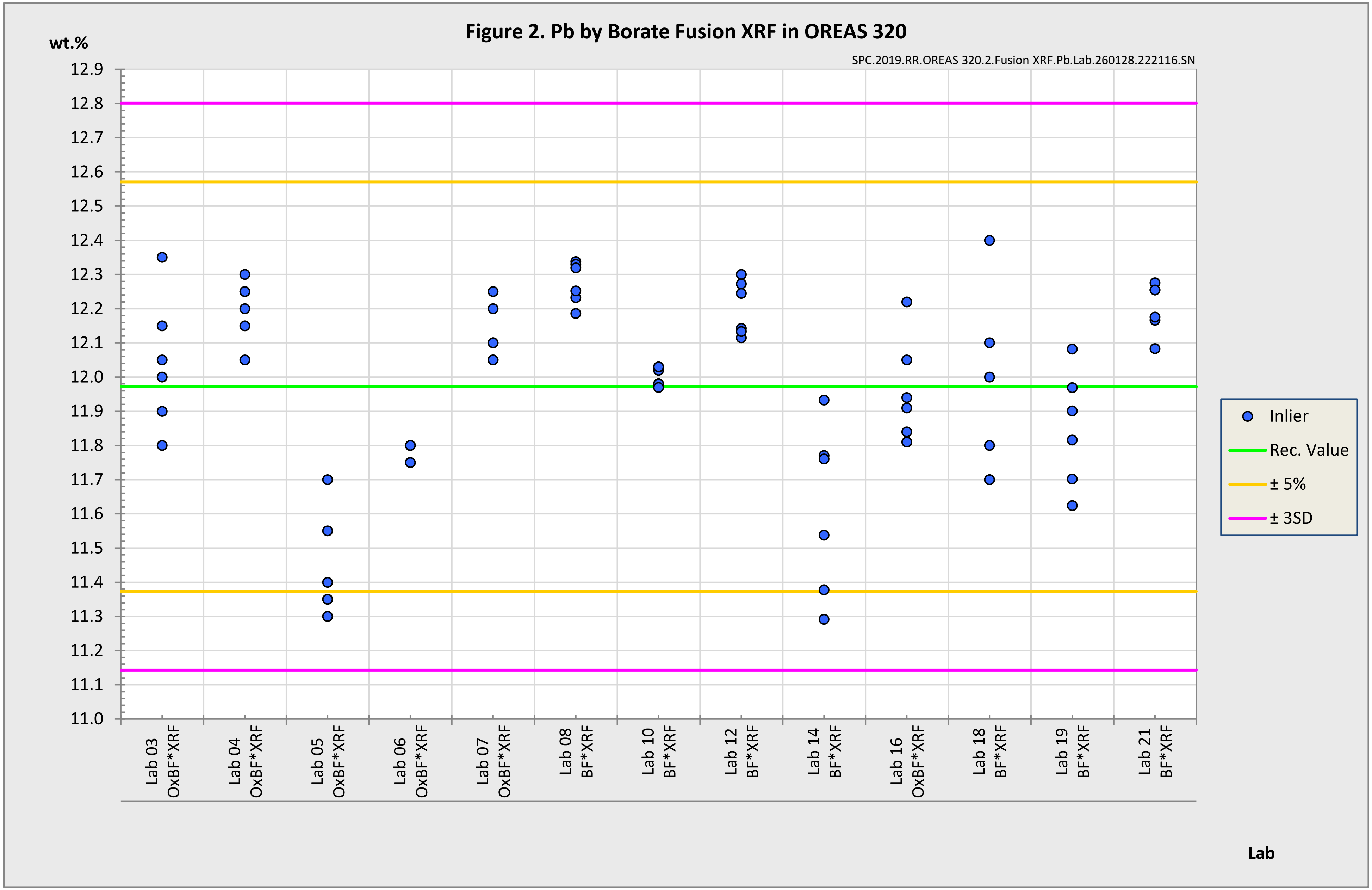
Figure 2. Pb by Borate Fusion XRF in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.Fusion XRF.Pb.Lab.260128.222116.SN
12.9
12.8
12.7
12.6
12.5
12.4
12.3
12.2
12.1
12.0
11.9
11.8
11.7
11.6
11.5
11.4
11.3
11.2
11.1
11.0
Lab 03 OxBF*XRF
Lab 04 OxBF*XRF
Lab 05 OxBF*XRF
Lab 06 OxBF*XRF
Lab 07 OxBF*XRF
Lab 08 BF*XRF
Lab 10 BF*XRF
Lab 12 BF*XRF
Lab 14 BF*XRF
Lab 16 OxBF*XRF
Lab 18 BF*XRF
Lab 19 BF*XRF
Lab 21 BF*XRF
Inlier
Rec. Value
± 5%
± 3SD
Lab

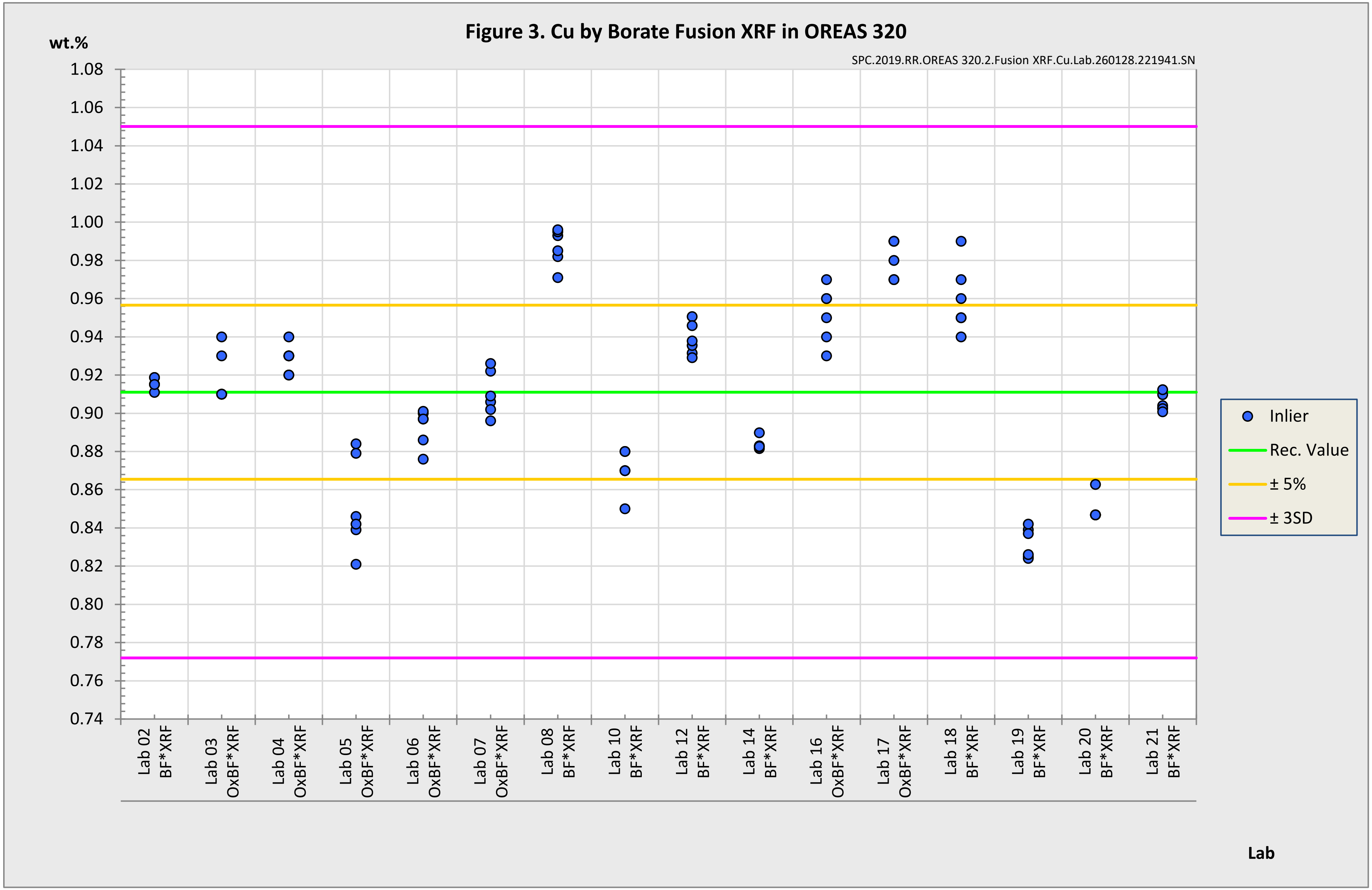
Figure 3. Cu by Borate Fusion XRF in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.Fusion XRF.Cu.Lab.260128.221941.SN
1.08
1.06
1.04
1.02
1.00
0.98
0.96
0.94
0.92
0.90
0.88
0.86
0.84
0.82
0.80
0.78
0.76
0.74
Lab 02 BF*XRF
Lab 03 OxBF*XRF
Lab 04 OxBF*XRF
Lab 05 OxBF*XRF
Lab 06 OxBF*XRF
Lab 07 OxBF*XRF
Lab 08 BF*XRF
Lab 10 BF*XRF
Lab 12 BF*XRF
Lab 14 BF*XRF
Lab 16 OxBF*XRF
Lab 17 OxBF*XRF
Lab 18 BF*XRF
Lab 19 BF*XRF
Lab 20 BF*XRF
Lab 21 BF*XRF
Inlier
Rec. Value
± 5%
± 3SD
Lab

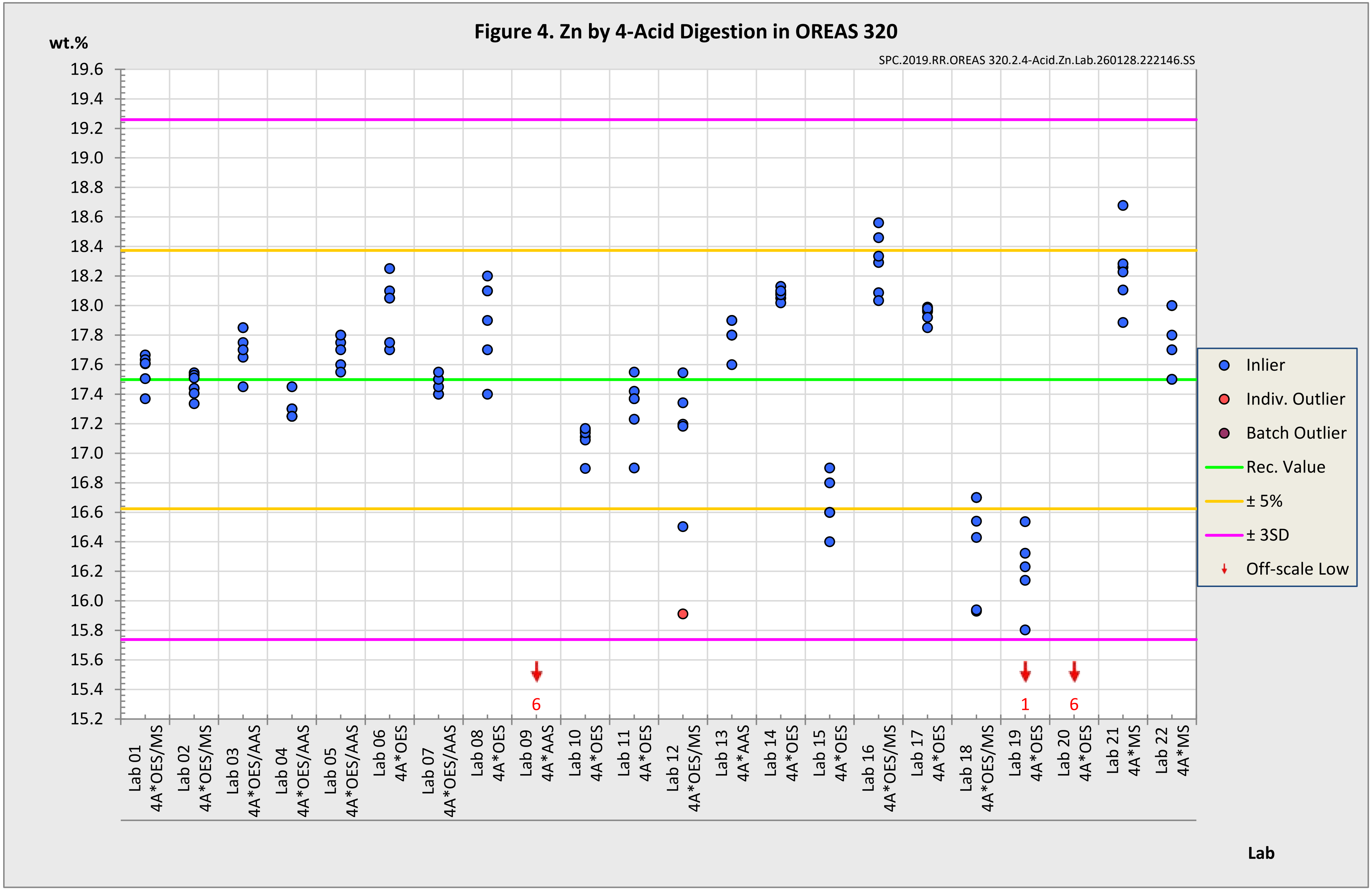
Figure 4. Zn by 4-Acid Digestion in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.4-Acid.Zn.Lab.260128.222146.SS
19.6
19.4
19.2
19.0
18.8
18.6
18.4
18.2
18.0
17.8
17.6
17.4
17.2
17.0
16.8
16.6
16.4
16.2
16.0
15.8
15.6
15.4
15.2
6
1
6
Lab 01 4A*OES/MS
Lab 02 4A*OES/MS
Lab 03 4A*OES/AAS
Lab 04 4A*OES/AAS
Lab 05 4A*OES/AAS
Lab 06 4A*OES
Lab 07 4A*OES/AAS
Lab 08 4A*OES
Lab 09 4A*AAS
Lab 10 4A*OES
Lab 11 4A*OES
Lab 12 4A*OES/MS
Lab 13 4A*AAS
Lab 14 4A*OES
Lab 15 4A*OES
Lab 16 4A*OES/MS
Lab 17 4A*OES
Lab 18 4A*OES/MS
Lab 19 4A*OES
Lab 20 4A*OES
Lab 21 4A*MS
Lab 22 4A*MS
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale Low
Lab

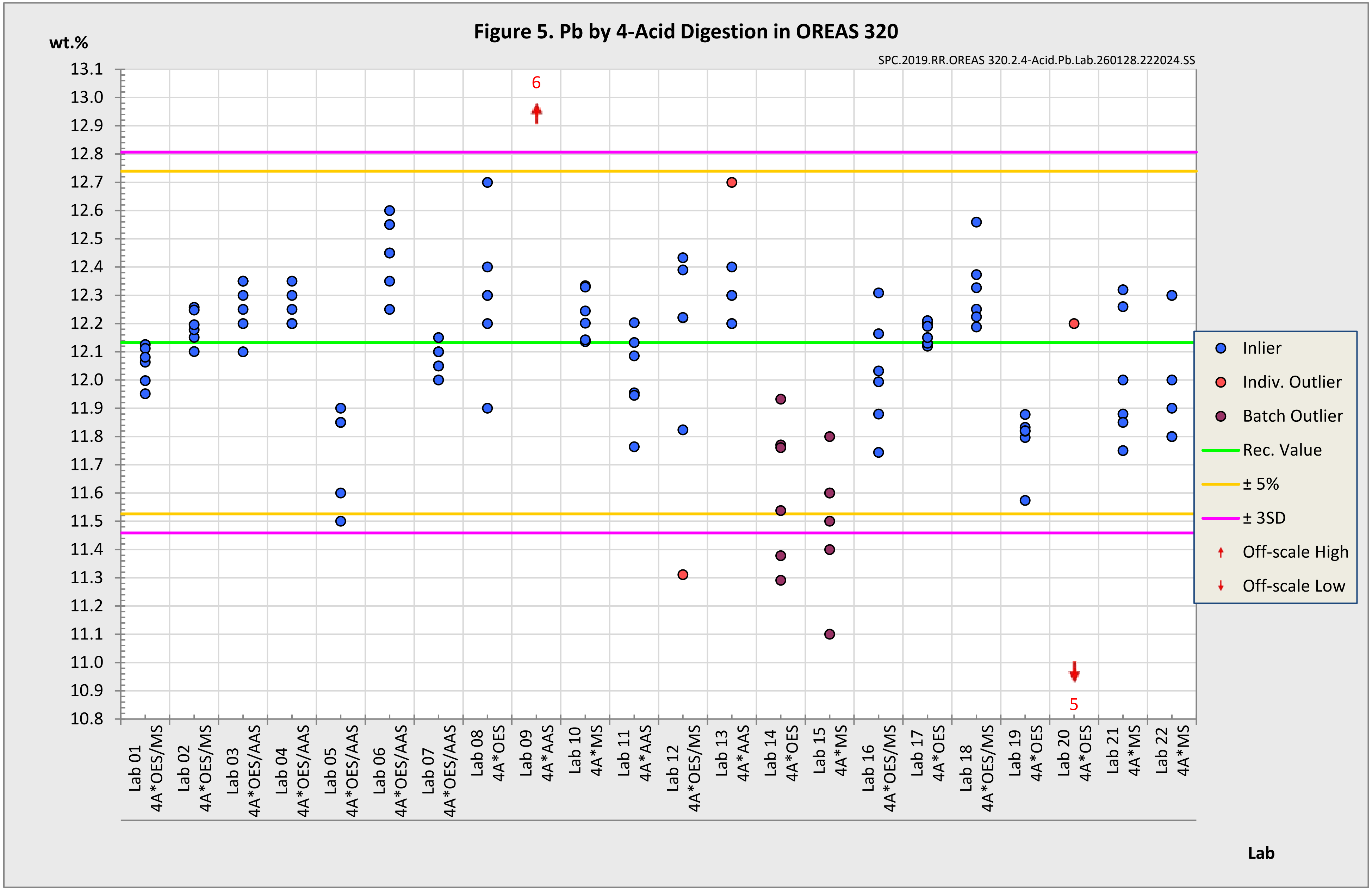
Figure 5. Pb by 4-Acid Digestion in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.4-Acid.Pb.Lab.260128.222024.SS
13.1
13.0
12.9
12.8
12.7
12.6
12.5
12.4
12.3
12.2
12.1
12.0
11.9
11.8
11.7
11.6
11.5
11.4
11.3
11.2
11.1
11.0
10.9
10.8
6
5
Lab 01 4A*OES/MS
Lab 02 4A*OES/MS
Lab 03 4A*OES/AAS
Lab 04 4A*OES/AAS
Lab 05 4A*OES/AAS
Lab 06 4A*OES/AAS
Lab 07 4A*OES/AAS
Lab 08 4A*OES
Lab 09 4A*AAS
Lab 10 4A*MS
Lab 11 4A*AAS
Lab 12 4A*OES/MS
Lab 13 4A*AAS
Lab 14 4A*OES
Lab 15 4A*MS
Lab 16 4A*OES/MS
Lab 17 4A*OES
Lab 18 4A*OES/MS
Lab 19 4A*OES
Lab 20 4A*OES
Lab 21 4A*MS
Lab 22 4A*MS
Lab
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale High
Off-scale Low

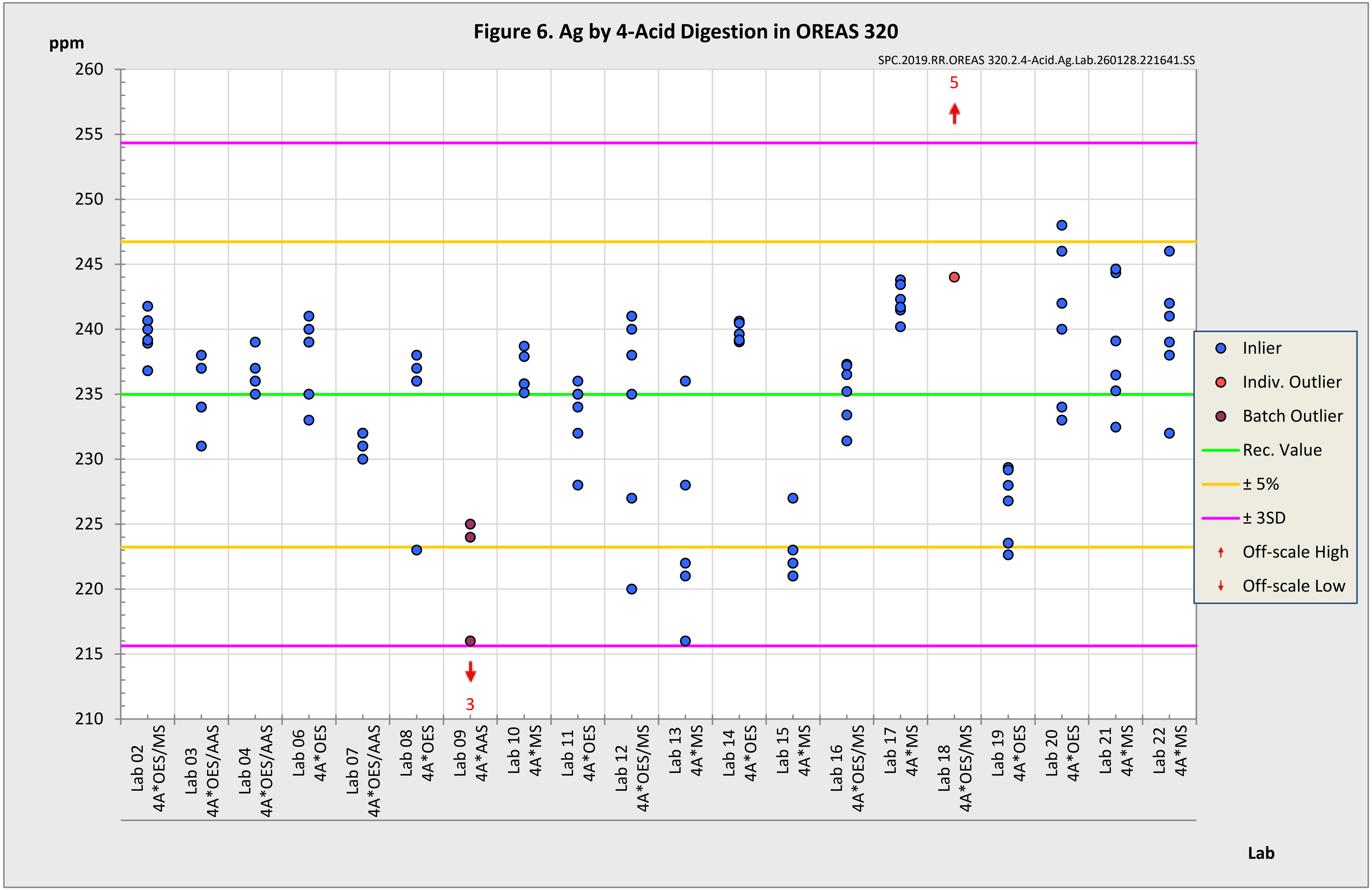

Figure 6. Ag by 4-Acid Digestion in OREAS 320
ppm
SPC.2019.RR.OREAS 320.2.4-Acid.Ag.Lab.260128.221641.SS
260
255
250
245
240
235
230
225
220
215
210
5
3
Inlier
Indiv. Outlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale High
Off-scale Low
Lab 02 4A*OES/MS
Lab 03 4A*OES/AAS
Lab 04 4A*OES/AAS
Lab 06 4A*OES
Lab 07 4A*OES/AAS
Lab 08 4A*OES
Lab 09 4A*AAS
Lab 10 4A*MS
Lab 11 4A*OES
Lab 12 4A*OES/MS
Lab 13 4A*MS
Lab 14 4A*OES
Lab 15 4A*MS
Lab 16 4A*OES/MS
Lab 17 4A*MS
Lab 18 4A*OES/MS
Lab 19 4A*OES
Lab 20 4A*OES
Lab 21 4A*MS
Lab 22 4A*MS
Lab

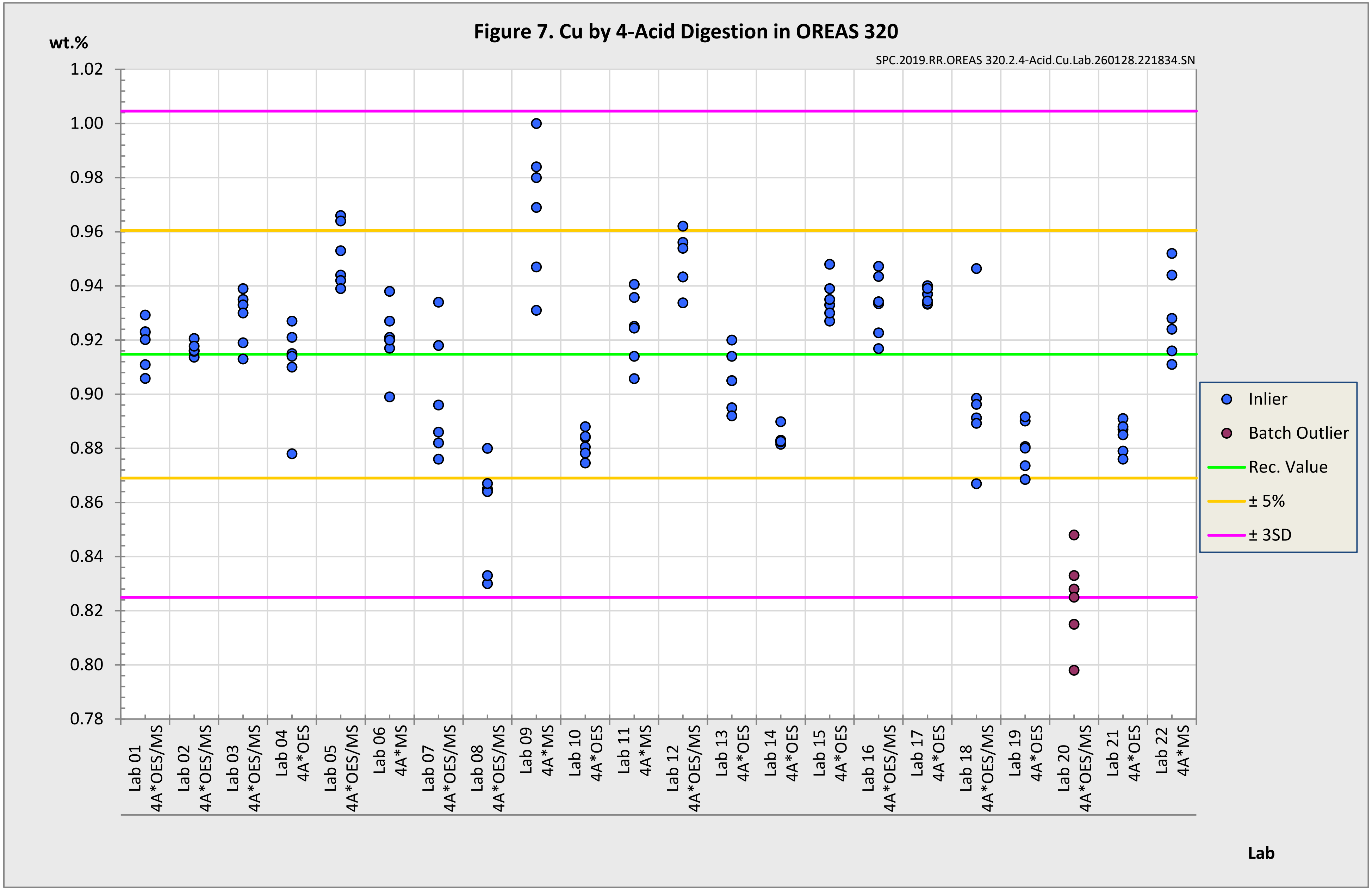
Figure 7. Cu by 4-Acid Digestion in OREAS 320
wt.%
SPC.2019.RR.OREAS 320.2.4-Acid.Cu.Lab.260128.221834.SN
1.02
1.00
0.98
0.96
0.94
0.92
0.90
0.88
0.86
0.84
0.82
0.80
0.78
Lab 01 4A*OES/MS
Lab 02 4A*OES/MS
Lab 03 4A*OES/MS
Lab 04 4A*OES
Lab 05 4A*OES/MS
Lab 06 4A*MS
Lab 07 4A*OES/MS
Lab 08 4A*OES/MS
Lab 09 4A*MS
Lab 10 4A*OES
Lab 11 4A*MS
Lab 12 4A*OES/MS
Lab 13 4A*OES
Lab 14 4A*OES
Lab 15 4A*OES
Lab 16 4A*OES/MS
Lab 17 4A*OES
Lab 18 4A*OES/MS
Lab 19 4A*OES
Lab 20 4A*OES/MS
Lab 21 4A*OES
Lab 22 4A*MS
Inlier
Batch Outlier
Rec. Value
± 5%
± 3SD
Lab

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)) [14]. 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 [7], 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.

Participating laboratories were selected based on demonstrated analytical competence, including prior performance in interlaboratory comparison programs conducted by ORE Pty Ltd, with consideration given to their expertise in relevant analytical methods, measurands, and sample matrices. For the measurands reported in this certificate (Table 1), data were sourced from laboratories accredited to ISO/IEC 17025. Where formal accreditation was not held for specific operationally defined measurands, metrological traceability was verified through the use of well-characterised, independently certified reference materials (CRMs) included as control samples in the round robin study.

In accordance with ISO 33405:2024-05 [4], clause 9.2.5, and ISO 17034:2016 [8], clause 7.12.4 b), the use of such control samples provides an acceptable means of demonstrating traceability in the absence of formal accreditation. In this certification program, traceability was further supported by the agreement of measured values for control samples with their known certified values, thereby offering additional confidence in the calibration and validity of measurement results across participating laboratories.

Operationally Defined Measurands

In accordance with ISO 33405:2024-05, Clause 9.2.4, measurands (analytes) may be certified as operationally defined. For these measurands, traceability to the SI may not be achievable because the analytical procedure involves sample transformations (e.g., leaching or extraction). While instrument calibration can be traceable to appropriate units, the transformation steps themselves are not directly traceable and can only be evaluated through reference comparisons or harmonized procedures.

Accordingly, characterisation of these measurands has been based on the concordance of results obtained from multiple laboratories using a common, well-defined procedure. This approach ensures fitness-for-purpose and fulfils the requirements for metrological traceability as specified in ISO 17034 and ISO 33405 for operationally defined measurands.

COMMUTABILITY

The certified values reported herein are derived from measurements performed using analytical methods involving sample pre-treatment steps, such as fusion or acid digestion. These processes convert the sample matrix into a chemically simplified and stable form, facilitating calibration traceable to primary standards via solution-based calibration protocols. Due to the established robustness and effectiveness of these pre-treatment methods, issues related to commutability are not expected to impact the suitability of this Certified Reference Material (CRM) for its intended use.

OREAS CRMs are prepared from natural ore materials, ensuring the presence of matrix and mineralogical characteristics representative of typical exploration and process samples. Consistent with ISO 17034:2016 and ISO Guide 30, users are advised to select CRMs with matrix and mineralisation styles closely matching those of their routine ‘field’ samples to minimize matrix effects and enhance analytical comparability. Detailed descriptions of the CRM’s source material and mineralogical characteristics are provided in the ‘Source Material’ section to guide appropriate CRM selection.

INTENDED USE

OREAS 320 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 320 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 320 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Table 1 in geological samples;
- For the verification/ validation of analytical methods for analytes reported in Table 1;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Table 1. 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 Table 1).

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:

- Borate fusion with X-ray fluorescence finish: ≥0.2 g;
- Loss on Ignition (LOI) at 1000 °C: ≥1 g;
- C and S by infrared combustion furnace/CS analyser: ≥ 0.1 g;
- Borate fusion /Sodium peroxide with ICP-OES and/or MS finish: ≥0.2 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.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 320 remains valid, within the specified measurement uncertainties, until at least June 2040, 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 320 contains sulphides (16.4 wt.% Total Sulphur) and is packaged in single-use, 10 g 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.

INSTRUCTIONS FOR HANDLING & CORRECT USE

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

Pre-homogenisation of the CRM prior to subsampling and analysis is not necessary as there is no particle segregation under transport [11].

As per routine analysis at commercial laboratories, the certified values derived by borate fusion with XRF finish are on a dry sample basis. Analytes by all other methods refer to the concentration levels in the packaged state. There is no need for drying prior to weighing and analysis for these methods.

Authoritative Source of Information

This Certificate of Analysis constitutes the primary and authoritative document for the certified values, associated expanded uncertainties, and their correct use. While the accompanying DataPack provides supporting information, including raw data and uncertainty estimates with additional significant figures, these extended figures are provided solely for transparency, convenience and statistical reference. Users must rely exclusively on the values stated in this Certificate, rounded to an appropriate number of significant figures, for all metrological and analytical purposes. Any discrepancy between values presented in the DataPack and those in this Certificate shall be resolved in favour of the information provided herein.

Notice on Certificate Updates

The version of the Certificate of Analysis (COA) available on the OREAS website is considered the official and most current version. As COAs may be revised following periodic reviews, re-evaluation of data, or the availability of new information, users are strongly advised to refer to the latest online version prior to each use.
It is the user’s responsibility to ensure that the most recent and applicable certificate is used to support the traceability, validity, and fitness-for-purpose of the certified reference material

(CRM). Any significant changes to the sections of this certificate will be clearly documented in the revised certificate.

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 5 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	16th February 2026	First publication.

CERTIFYING OFFICER

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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.

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