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**CERTIFICATE OF ANALYSIS FOR**

**Carlin Ore (Leeville Mine, Nevada, USA)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 278**

**Table 1. Certified Values and Performance Gates for OREAS 278.**

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
<b>Pb Fire Assay</b>											
Au, ppm	4.99	0.173	4.64	5.34	4.47	5.51	3.47%	6.95%	10.42%	4.74	5.24
Au, opt	0.146	0.005	0.135	0.156	0.130	0.161	3.47%	6.95%	10.42%	0.138	0.153
<b>Infrared Combustion</b>											
C, wt. %	1.40	0.047	1.31	1.50	1.26	1.55	3.38%	6.75%	10.13%	1.33	1.47
C-(Carbonate), wt. %	1.21	0.032	1.15	1.28	1.12	1.31	2.60%	5.20%	7.80%	1.15	1.27
C-(non.Carbonate), wt. %	0.210	0.037	0.136	0.284	0.098	0.321	17.71%	35.42%	53.14%	0.199	0.220
C-(Graphite), wt. %	0.124	0.060	0.003	0.244	0.000	0.305	48.87%	97.74%	146.6%	0.117	0.130
C-(Organic), wt. %	0.106	0.057	0.000	0.221	0.000	0.279	53.90%	107.8%	161.7%	0.101	0.112
S, wt. %	0.942	0.039	0.863	1.021	0.824	1.060	4.18%	8.36%	12.54%	0.895	0.989
S-(Sulphate), wt. %	0.244	0.106	0.032	0.456	0.000	0.561	43.34%	86.68%	130.0%	0.232	0.256
S-(Sulphide), wt. %	0.669	0.098	0.472	0.865	0.374	0.964	14.71%	29.42%	44.13%	0.635	0.702
<b>Aqua Regia Digestion</b>											
Ag, ppm	0.136	0.014	0.109	0.164	0.095	0.178	10.16%	20.31%	30.47%	0.129	0.143
Al, wt. %	0.859	0.049	0.762	0.957	0.713	1.006	5.67%	11.35%	17.02%	0.816	0.902
As, ppm	703	31	641	765	610	796	4.39%	8.78%	13.16%	668	738
B, ppm	< 20	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND

SI unit equivalents: ppm (parts per million) ≡ mg/kg ≡ µg/g ≡ 0.0001 wt. % ≡ 1000 ppb (parts per billion).

1opt (troy ounce per short ton) ≡ 34.2857ppm.

Note 1: intervals may appear asymmetric due to rounding.

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



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**Table 1 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
<b>Aqua Regia Digestion continued</b>											
Be, ppm	0.31	0.03	0.24	0.38	0.21	0.41	11.21%	22.42%	33.63%	0.29	0.32
Bi, ppm	0.46	0.023	0.41	0.51	0.39	0.53	5.09%	10.18%	15.27%	0.44	0.48
Ca, wt. %	3.19	0.175	2.84	3.54	2.66	3.71	5.49%	10.98%	16.48%	3.03	3.35
Cd, ppm	0.54	0.040	0.46	0.62	0.42	0.66	7.49%	14.97%	22.46%	0.51	0.56
Ce, ppm	28.7	1.28	26.2	31.3	24.9	32.6	4.46%	8.93%	13.39%	27.3	30.2
Co, ppm	5.64	0.242	5.16	6.12	4.92	6.37	4.29%	8.58%	12.86%	5.36	5.92
Cr, ppm	29.3	1.71	25.9	32.7	24.2	34.4	5.84%	11.67%	17.51%	27.8	30.8
Cs, ppm	1.77	0.165	1.44	2.10	1.28	2.27	9.30%	18.60%	27.91%	1.68	1.86
Cu, ppm	43.9	1.98	39.9	47.8	37.9	49.8	4.51%	9.02%	13.53%	41.7	46.1
Dy, ppm	1.63	0.059	1.52	1.75	1.46	1.81	3.62%	7.25%	10.87%	1.55	1.72
Er, ppm	0.71	0.048	0.62	0.81	0.57	0.86	6.73%	13.46%	20.19%	0.68	0.75
Eu, ppm	0.56	0.09	0.39	0.74	0.30	0.82	15.33%	30.67%	46.00%	0.54	0.59
Fe, wt. %	1.77	0.061	1.65	1.90	1.59	1.96	3.44%	6.87%	10.31%	1.69	1.86
Ga, ppm	2.76	0.30	2.17	3.36	1.87	3.65	10.75%	21.51%	32.26%	2.62	2.90
Gd, ppm	2.41	0.153	2.11	2.72	1.95	2.87	6.34%	12.68%	19.02%	2.29	2.53
Ge, ppm	0.052	0.013	0.026	0.079	0.012	0.093	25.56%	51.11%	76.67%	0.050	0.055
Hf, ppm	0.39	0.04	0.31	0.47	0.27	0.52	10.34%	20.68%	31.02%	0.37	0.41
Hg, ppm	6.87	0.641	5.59	8.15	4.95	8.79	9.33%	18.65%	27.98%	6.53	7.22
Ho, ppm	0.27	0.019	0.24	0.31	0.22	0.33	6.76%	13.53%	20.29%	0.26	0.29
In, ppm	0.030	0.002	0.027	0.034	0.026	0.035	5.39%	10.78%	16.18%	0.029	0.032
K, wt. %	0.311	0.026	0.259	0.363	0.233	0.389	8.36%	16.72%	25.07%	0.295	0.327
La, ppm	15.5	0.76	14.0	17.0	13.2	17.8	4.89%	9.79%	14.68%	14.7	16.3
Li, ppm	10.9	1.08	8.8	13.1	7.7	14.2	9.88%	19.75%	29.63%	10.4	11.5
Lu, ppm	0.077	0.006	0.065	0.088	0.060	0.093	7.33%	14.65%	21.98%	0.073	0.080
Mg, wt. %	1.04	0.033	0.97	1.11	0.94	1.14	3.17%	6.34%	9.51%	0.99	1.09
Mn, wt. %	0.021	0.001	0.019	0.023	0.018	0.023	4.71%	9.42%	14.13%	0.020	0.022
Mo, ppm	6.51	0.344	5.82	7.20	5.48	7.54	5.29%	10.58%	15.87%	6.18	6.83
Na, wt. %	0.014	0.004	0.005	0.023	0.001	0.028	30.99%	61.99%	92.98%	0.014	0.015
Nb, ppm	0.097	0.023	0.052	0.142	0.029	0.165	23.41%	46.81%	70.22%	0.092	0.102
Nd, ppm	14.2	0.84	12.5	15.8	11.6	16.7	5.97%	11.93%	17.90%	13.4	14.9
Ni, ppm	23.2	1.89	19.4	27.0	17.5	28.9	8.16%	16.33%	24.49%	22.0	24.4
P, wt. %	0.052	0.002	0.048	0.056	0.046	0.058	3.85%	7.70%	11.54%	0.050	0.055
Pb, ppm	6.86	0.425	6.01	7.71	5.59	8.14	6.19%	12.38%	18.57%	6.52	7.21
Pd, ppb	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Pr, ppm	3.57	0.234	3.10	4.04	2.87	4.27	6.55%	13.09%	19.64%	3.39	3.75
Pt, ppb	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Rb, ppm	21.6	1.31	19.0	24.2	17.7	25.6	6.04%	12.08%	18.12%	20.6	22.7
Re, ppm	0.004	0.000	0.003	0.005	0.003	0.005	11.01%	22.02%	33.03%	0.004	0.004
S, wt. %	0.948	0.044	0.859	1.036	0.815	1.081	4.68%	9.36%	14.05%	0.900	0.995
Sb, ppm	47.1	4.8	37.4	56.7	32.6	61.5	10.24%	20.48%	30.72%	44.7	49.4
Sc, ppm	2.89	0.30	2.29	3.49	1.99	3.78	10.36%	20.72%	31.08%	2.74	3.03
Se, ppm	0.96	0.13	0.69	1.22	0.56	1.35	13.86%	27.71%	41.57%	0.91	1.00
Sm, ppm	2.61	0.187	2.24	2.99	2.05	3.17	7.14%	14.28%	21.42%	2.48	2.74

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

Note 1: intervals may appear asymmetric due to rounding.

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

**Table 1 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
<b>Aqua Regia Digestion continued</b>											
Sn, ppm	1.20	0.110	0.98	1.42	0.87	1.53	9.16%	18.31%	27.47%	1.14	1.26
Sr, ppm	40.2	2.21	35.8	44.7	33.6	46.9	5.49%	10.98%	16.47%	38.2	42.3
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.31	0.04	0.24	0.38	0.20	0.42	11.78%	23.57%	35.35%	0.29	0.32
Te, ppm	0.19	0.02	0.15	0.24	0.12	0.26	11.87%	23.75%	35.62%	0.18	0.20
Th, ppm	4.86	0.355	4.15	5.57	3.80	5.93	7.30%	14.60%	21.90%	4.62	5.11
Ti, wt.%	0.025	0.003	0.018	0.032	0.015	0.035	13.70%	27.39%	41.09%	0.024	0.026
Tl, ppm	10.3	0.77	8.7	11.8	8.0	12.6	7.52%	15.04%	22.56%	9.8	10.8
Tm, ppm	0.090	0.007	0.077	0.103	0.070	0.109	7.30%	14.60%	21.90%	0.085	0.094
U, ppm	2.00	0.115	1.77	2.22	1.65	2.34	5.76%	11.52%	17.28%	1.90	2.09
V, ppm	38.7	3.64	31.4	46.0	27.8	49.6	9.41%	18.82%	28.23%	36.8	40.6
W, ppm	3.77	0.53	2.71	4.84	2.18	5.37	14.07%	28.13%	42.20%	3.59	3.96
Y, ppm	7.67	0.423	6.82	8.52	6.40	8.94	5.51%	11.02%	16.53%	7.29	8.05
Yb, ppm	0.55	0.06	0.44	0.66	0.38	0.72	10.04%	20.08%	30.13%	0.52	0.58
Zn, ppm	63	2.6	58	68	55	71	4.08%	8.16%	12.23%	60	66
Zr, ppm	13.4	1.4	10.7	16.1	9.4	17.5	10.12%	20.23%	30.35%	12.8	14.1

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

Note 1: intervals may appear asymmetric due to rounding.

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

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## INTRODUCTION

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

OREAS 278 is one of a suite of three Carlin ore CRMs available: OREAS 277: 3.39ppm (0.1opt); OREAS 278: 4.99ppm (0.15opt) and OREAS 279: 6.54ppm (0.19opt). OREAS 278 contains 72 certified values including C and S speciation and full ICP-OES and MS suites by aqua regia digestion. Furthermore, 100 indicative values are provided including Au by cyanide leach, Au by aqua regia digestion and major and trace element composition (see Table 2 below). Au by the aforementioned leach methods was uncertifiable due to a lack of inter-laboratory consensus. The refractory nature of the gold ore and presence of carbon caused a wide variation in results. However, it is important to note that a high level of repeatability (precision) was displayed within each laboratory's data set for both cyanide leach and aqua regia digestion.

Tabulated results of all elements together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 278-DataPack.2.0.200825\_122302.xlsx**).

## SOURCE MATERIAL

OREAS 278 was prepared from a blend of high grade gold-bearing ore and barren sediments (shale, quartz and limestone). The ore was sourced from the Leeville Mine, located near the western crest of the Tuscarora Mountains, about 20 miles northwest of Carlin in Nevada, USA. Leeville is an underground high-grade refractory gold deposit located on the Carlin Trend. Gold mineralisation is hosted within decalcified and weakly to moderately silicified rocks composed of 60 to 70% quartz, 10 to 30% dolomite, 5 to 12% kaolinite, 2 to 4% illite and 2 to 4% pyrite (Jackson et al, 1997).

## PERFORMANCE GATES

Table 1 above shows intervals calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit [www.westgard.com/mltirule.htm](http://www.westgard.com/mltirule.htm)). A second method utilises a 5% window calculated directly from the certified value.

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

*i.e. Certified Value  $\pm$  10%  $\pm$  2DL (adapted from Govett, 1983).*

**Table 2. Indicative Values for OREAS 278.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Ag	ppm	0.282	Pd	ppb	0.507	Pt	ppb	0.395
<b>Cyanide Leach</b>								
Au	ppm	0.962						
<b>Aqua Regia Digestion</b>								
Au	ppm	2.18	Ir	ppb	0.910	Si	wt. %	0.047
Ba	ppm	494	Rh	ppb	8.91			
<b>X-ray Photon Assay</b>								
Au	ppm	4.91						
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt. %	5.25	MgO	wt. %	2.06	S	wt. %	0.941
CaO	wt. %	4.74	MnO	wt. %	0.030	SiO <sub>2</sub>	wt. %	76.04
Fe <sub>2</sub> O <sub>3</sub>	wt. %	2.66	Na <sub>2</sub> O	wt. %	0.310	TiO <sub>2</sub>	wt. %	0.325
K <sub>2</sub> O	wt. %	0.957	P <sub>2</sub> O <sub>5</sub>	wt. %	0.120			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt. %	5.72						
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	0.200	Hf	ppm	3.62	Sm	ppm	3.35
As	ppm	571	Ho	ppm	0.50	Sn	ppm	2.40
Ba	ppm	1400	In	ppm	< 0.05	Sr	ppm	51
Be	ppm	0.90	La	ppm	20.0	Ta	ppm	0.45
Bi	ppm	0.41	Lu	ppm	0.20	Tb	ppm	0.44
Cd	ppm	0.65	Mn	wt. %	0.022	Te	ppm	0.15
Ce	ppm	35.8	Mo	ppm	6.00	Th	ppm	6.04
Co	ppm	5.95	Nb	ppm	5.96	Ti	wt. %	0.194
Cr	ppm	53	Nd	ppm	17.4	Tl	ppm	13.9
Cs	ppm	3.53	Ni	ppm	26.0	Tm	ppm	0.20
Cu	ppm	41.0	Pb	ppm	7.00	U	ppm	3.26
Dy	ppm	2.57	Pr	ppm	4.58	V	ppm	88
Er	ppm	1.43	Rb	ppm	45.5	W	ppm	20.5
Eu	ppm	0.61	Re	ppm	< 0.01	Y	ppm	14.5
Ga	ppm	6.85	Sb	ppm	54	Yb	ppm	1.37
Gd	ppm	2.88	Sc	ppm	5.80	Zn	ppm	70
Ge	ppm	0.75	Se	ppm	< 5	Zr	ppm	126

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

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

**Table 2 continued.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>4-Acid Digestion</b>								
Al	wt.%	2.50	Cu	ppm	47.0	Pb	ppm	13.8
As	ppm	692	Fe	wt.%	1.72	Re	ppm	10.8
Ba	ppm	1312	K	wt.%	0.730	Sb	ppm	60
Be	ppm	1.00	Mg	wt.%	1.17	Se	ppm	< 10
Bi	ppm	< 12	Mn	wt.%	0.022	Sn	ppm	3.17
Ca	wt.%	3.29	Mo	ppm	8.17	Te	ppm	< 6
Cd	ppm	2.17	Na	wt.%	0.212	Ti	wt.%	0.172
Co	ppm	7.00	Ni	ppm	21.5	Tl	ppm	< 8
Cr	ppm	32.0	P	wt.%	0.049	Zn	ppm	65

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

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

## COMMINATION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 278 was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of the barren sediments to 98% minus 75 microns;
- Crushing and milling of the ore material to 100% minus 30 microns;
- Blending in appropriate proportions to achieve the desired grade;
- Packaging in 60g units sealed in laminated foil pouches and 500g units in plastic jars.

## PHYSICAL PROPERTIES

OREAS 278 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 278.**

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
670	0.64	N6	Medium Light Gray

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

## ANALYTICAL PROGRAM

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

- Gold by fire assay (15-50g charge weight) with AAS (31 laboratories), ICP-OES (8 laboratories) or ICP-MS (2 laboratories) finish;

- Gold by cyanide leach – A variety of cyanide leach methods were undertaken by the participating laboratories including the use of LeachWELL tablets, alkaline added sodium cyanide solution as well as sodium cyanide liquor with LeachWELL powder. The sample weights included: 5g (1 laboratory by AAS finish), 10g (1 laboratory by ICP-OES finish), 30g (8 laboratories by AAS, 1 laboratory by ICP-OES and 1 laboratory ICP-MS finish), 50g (1 laboratory by AAS and 2 laboratory by ICP-MS finish) and 200g (5 laboratories by AAS, 2 laboratories by ICP-OES and 2 laboratories by ICP-MS finish);
- Aqua regia digestion for full ICP-OES and ICP-MS elemental suites (up to 34 laboratories depending on the element);
- Total Carbon (29 laboratories) and Total Sulphur (30 laboratories) by infrared combustion furnace;
- Carbonate Carbon and Non-Carbonate Carbon by a range of methodologies including HCl or HClO<sub>4</sub> leach followed by infrared combustion furnace or coulometry, or via difference: Total C minus Non-Carbonate C (for Carbonate C determination) or, Total C minus Carbonate C (for Non-Carbonate C determination);
- Graphitic Carbon by HCl digestion to remove carbonates followed by roasting to remove organic carbon, the residue is then determined by infrared combustion furnace;
- Sulphate Sulphur by HCl leach followed by gravimetric (6 laboratories) or ICP-OES (1 laboratory) finish or roasting (at 500-550°C) followed by infrared combustion furnace (7 laboratories);
- Sulphide Sulphur by difference (Total S minus Sulphate Sulphur).

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

For the round robin program twenty 3.0kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 100-350g pulp samples were submitted to each laboratory for analysis (the weight provided depended on whether the laboratory was anticipated to undertake assays by gold cyanide leach). The samples received by each laboratory were obtained by taking two samples from each of three separate 3.0kg test units. This format enabled a nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance.

Table 1 provides performance gate intervals for the 72 certified values based on their pooled 1SD's. Table 2 shows 100 indicative values including gold by Chrysol Corporation's Photon Assay technique and major and trace element characterisation by Bureau Veritas Perth using the following methodologies:

- Major oxides by lithium borate fusion with X-ray fluorescence;
- LOI at 1000°C by thermogravimetric analyser;
- Trace element characterisation by laser ablation with ICP-MS finish.

Table 2 also presents twenty additional indicative values (Al to Zn) by 4-acid digestion with ICP-OES/MS finish performed by one of the participating laboratories.



Table 3 provides some indicative physical properties and Table 4 presents 95% confidence and tolerance limits. Gold homogeneity (via INAA) is shown in Table 5 and is also demonstrated by a nested ANOVA program using fire assay (see 'nested ANOVA' section).

Results are also presented in scatter plots for gold by fire assay, total carbon by infrared combustion furnace and total sulphur by infrared combustion furnace (Figures 1 to 3, respectively) together with  $\pm 3SD$  (magenta) and  $\pm 5\%$  (yellow) control lines and certified value (green line). Accepted individual results are coloured blue and individual and dataset outliers are identified in red and violet, respectively.

## STATISTICAL ANALYSIS

**Standard Deviation** intervals (see Table 1) provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program (see Intended Use section for more detail).

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

**Certified Values, Standard Deviations, Confidence Limits and Tolerance Limits** (Table 4) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration).

For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores  $> 2.5$  and with per cent deviations (i)  $> 3$  and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if  $> 2.5$ . After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

**Certified Values** are the means of accepted laboratory means after outlier filtering. The INAA data (see Table 5) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 278 (see 'Homogeneity Evaluation' section below).

**95% Confidence Limits** are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. **95% Confidence Limits should not be used as control limits for laboratory performance.**

**Indicative (uncertified) values** (Table 2) are provided for the major and trace elements determined by borate fusion XRF ( $\text{Al}_2\text{O}_3$  to  $\text{TiO}_2$ ), laser ablation with ICP-MS (Ag to Zr) and LOI at  $1000^\circ\text{C}$  and are the means of duplicate assays from Bureau Veritas, Perth. Au by Chryso's Photon Assay technique is also shown. Additional indicative values by other analytical methods are present where the number of laboratories reporting a particular analyte is insufficient ( $< 5$ ) to support certification or where inter-laboratory consensus is poor.

**Table 4. 95% Confidence & Tolerance Limits for OREAS 278.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	4.99	4.94	5.04	4.98*	5.00*
Au, Gold (opt)	0.146	0.144	0.147	0.145*	0.146*
<b>Infrared Combustion (wt.%)</b>					
C, Carbon	1.40	1.38	1.42	1.39	1.42
C-(Carbonate)	1.21	1.20	1.23	1.20	1.23
C-(non.Carbonate)	0.210	0.180	0.240	0.195	0.225
C-(Graphite)	0.124	0.091	0.157	0.112	0.136
C-(Organic)	0.106	0.062	0.151	0.089	0.124
S, Sulphur	0.942	0.927	0.956	0.925	0.958
S-(Sulphate), Sulphur as $\text{SO}_4^{2-}$	0.244	0.179	0.309	0.234	0.254
S-(Sulphide), Sulphur as $\text{S}^{2-}$	0.669	0.605	0.733	0.646	0.691
<b>Aqua Regia Digestion</b>					
Ag, Silver (ppm)	0.136	0.131	0.142	0.122	0.150
Al, Aluminium (wt.%)	0.859	0.841	0.878	0.841	0.878
As, Arsenic (ppm)	703	692	714	685	721
B, Boron (ppm)	< 20	IND	IND	IND	IND
Be, Beryllium (ppm)	0.31	0.29	0.33	0.29	0.33
Bi, Bismuth (ppm)	0.46	0.45	0.47	0.44	0.48
Ca, Calcium (wt.%)	3.19	3.13	3.25	3.13	3.24
Cd, Cadmium (ppm)	0.54	0.52	0.55	0.51	0.56

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

\*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.

Table 4 continued.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Ce, Cerium (ppm)	28.7	28.2	29.3	28.0	29.5
Co, Cobalt (ppm)	5.64	5.54	5.75	5.43	5.86
Cr, Chromium (ppm)	29.3	28.7	29.9	28.4	30.2
Cs, Caesium (ppm)	1.77	1.69	1.85	1.72	1.82
Cu, Copper (ppm)	43.9	43.3	44.5	42.5	45.3
Dy, Dysprosium (ppm)	1.63	1.58	1.69	1.54	1.73
Er, Erbium (ppm)	0.71	0.66	0.77	0.68	0.74
Eu, Europium (ppm)	0.56	0.47	0.66	0.53	0.59
Fe, Iron (wt.%)	1.77	1.75	1.80	1.74	1.81
Gd, Gadolinium (ppm)	2.41	2.25	2.58	2.33	2.49
Ge, Germanium (ppm)	0.052	0.036	0.068	IND	IND
Hf, Hafnium (ppm)	0.39	0.37	0.41	0.37	0.41
Hg, Mercury (ppm)	6.87	6.58	7.17	6.72	7.03
Ho, Holmium (ppm)	0.27	0.26	0.29	0.26	0.29
In, Indium (ppm)	0.030	0.030	0.031	0.028	0.033
K, Potassium (wt.%)	0.311	0.301	0.321	0.301	0.321
La, Lanthanum (ppm)	15.5	15.2	15.9	15.1	15.9
Li, Lithium (ppm)	10.9	10.5	11.4	10.6	11.3
Lu, Lutetium (ppm)	0.077	0.073	0.081	IND	IND
Mg, Magnesium (wt.%)	1.04	1.03	1.05	1.02	1.06
Mn, Manganese (wt.%)	0.021	0.020	0.021	0.020	0.021
Mo, Molybdenum (ppm)	6.51	6.37	6.65	6.29	6.73
Na, Sodium (wt.%)	0.014	0.013	0.016	0.014	0.015
Nb, Niobium (ppm)	0.097	0.083	0.110	IND	IND
Nd, Neodymium (ppm)	14.2	13.3	15.0	13.7	14.6
Ni, Nickel (ppm)	23.2	22.5	23.9	22.5	23.9
P, Phosphorus (wt.%)	0.052	0.052	0.053	0.051	0.053
Pb, Lead (ppm)	6.86	6.67	7.05	6.57	7.15
Pd, Palladium (ppb)	< 10	IND	IND	IND	IND
Pr, Praseodymium (ppm)	3.57	3.37	3.77	3.41	3.73
Pt, Platinum (ppb)	< 5	IND	IND	IND	IND
Rb, Rubidium (ppm)	21.6	21.0	22.2	20.9	22.3
Re, Rhenium (ppm)	0.004	0.004	0.004	IND	IND
S, Sulphur (wt.%)	0.948	0.931	0.964	0.930	0.965
Sb, Antimony (ppm)	47.1	45.3	48.8	45.3	48.8
Sc, Scandium (ppm)	2.89	2.76	3.01	2.77	3.00
Se, Selenium (ppm)	0.96	0.90	1.01	IND	IND
Sm, Samarium (ppm)	2.61	2.47	2.76	2.50	2.73

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

Note: intervals may appear asymmetric due to rounding.

Table 4 continued.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Sn, Tin (ppm)	1.20	1.15	1.25	1.15	1.24
Sr, Strontium (ppm)	40.2	39.5	41.0	39.4	41.1
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Tb, Terbium (ppm)	0.31	0.28	0.33	0.29	0.32
Te, Tellurium (ppm)	0.19	0.19	0.20	0.16	0.22
Th, Thorium (ppm)	4.86	4.70	5.03	4.72	5.00
Ti, Titanium (wt.%)	0.025	0.024	0.026	0.024	0.026
Tl, Thallium (ppm)	10.3	9.9	10.6	10.0	10.6
Tm, Thulium (ppm)	0.090	0.082	0.098	IND	IND
U, Uranium (ppm)	2.00	1.95	2.04	1.93	2.06
V, Vanadium (ppm)	38.7	37.3	40.1	37.7	39.7
W, Tungsten (ppm)	3.77	3.53	4.02	3.62	3.93
Y, Yttrium (ppm)	7.67	7.49	7.85	7.51	7.83
Yb, Ytterbium (ppm)	0.55	0.49	0.61	0.52	0.58
Zn, Zinc (ppm)	63	62	64	61	65
Zr, Zirconium (ppm)	13.4	12.8	14.0	13.1	13.8

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

Note: intervals may appear asymmetric due to rounding.

### Homogeneity Evaluation

For analytes other than gold the tolerance limits (ISO 16269:2014) shown in Table 4 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for arsenic by aqua-regia digestion, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 685 and 721 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 5 below shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 278. An equivalent scaled version of the results is also provided to demonstrate the level of repeatability that would be achieved if 30g fire assay determinations were undertaken without the normal measurement error associated with this methodology. The homogeneity of gold has been determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material (i.e. sampling error) and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.08% was calculated for a 30g fire assay

sample (1.58% at 85mg weights) and confirms the high level of gold homogeneity in OREAS 278.

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

Replicate No	Au 85mg actual	Au 30g equivalent*
1	5.165	5.162
2	5.026	5.154
3	5.103	5.158
4	5.151	5.161
5	5.015	5.154
6	5.357	5.172
7	5.211	5.164
8	5.163	5.162
9	5.080	5.157
10	5.055	5.156
11	5.177	5.162
12	5.184	5.163
13	5.207	5.164
14	5.278	5.168
15	5.168	5.162
16	5.156	5.161
17	5.212	5.164
18	5.133	5.160
19	5.166	5.162
20	5.227	5.165
Mean	5.162	5.162
Median	5.165	5.162
Std Dev.	0.082	0.004
<b>Rel.Std.Dev.</b>	<b>1.58%</b>	<b>0.084%</b>

\*Results calculated for a 30g equivalent sample mass using the formula:  $x^{30g Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@85mg} + \bar{X}$   
 where  $x^{30g Eq}$  = equivalent result calculated for a 30g sample mass  
 $(x^{INAA})$  = raw INAA result at 85mg  
 $\bar{X}$  = mean of 85mg INAA results

The homogeneity of OREAS 278 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the forty-two round robin laboratories received six samples per CRM and these samples were 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 278. The test was performed using the following parameters:

- Gold fire assay – 246 samples (41 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis, H<sub>0</sub>: Between-unit variance is no greater than within-unit variance (reject H<sub>0</sub> if p-value < 0.05);
- Alternative Hypothesis, H<sub>1</sub>: Between-unit variance is greater than within-unit variance.

*P*-values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the *p*-value. This process derived a *p*-value of 0.999 which is an insignificant result and the Null Hypothesis is therefore 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 278 and whether the variance between two subsamples from the same unit is statistically distinguishable from the variance of two subsamples taken from any two separate units. A reference material therefore can possess poor absolute homogeneity yet still pass a relative homogeneity (ANOVA) test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 278 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## **PARTICIPATING LABORATORIES**

1. Actlabs, Ancaster, Ontario, Canada
2. AGAT Laboratories, Mississauga, Ontario, Canada
3. Alex Stewart International, Mendoza, Argentina
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Perth, WA, Australia
7. ALS, Reno, Nevada, USA
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ANSTO, Lucas Heights, NSW, Australia
11. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
12. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
13. Bureau Veritas Geoanalytical, Perth, WA, Australia
14. CERTIMIN, Lima, Peru
15. Chrysos Corporation, Perth, WA, Australia
16. Couer Mining Rochester Laboratory, Lovelock, Nevada, USA
17. Gekko Assay Labs, Ballarat, VIC, Australia
18. Information and Research Center, Kara-Balta, Chuy Region, Kyrgyzstan
19. Inspectorate (BV), Lima, Peru
20. Inspectorate America Corporation (BV), Sparks, Nevada, USA
21. Intertek Genalysis, Perth, WA, Australia

22. Intertek Tarkwa, Tarkwa, Ghana
23. Intertek Testing Services, Townsville, QLD, Australia
24. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
25. MinAnalytical Services, Kalgoorlie, WA, Australia
26. MinAnalytical Services, Perth, WA, Australia
27. Nagrom, Perth, WA, Australia
28. Nevada Gold Mines Assay Lab, Carlin, Nevada, USA
29. On Site Laboratory Services, Bendigo, VIC, Australia
30. Ontario Geological Survey, Sudbury, Ontario, Canada
31. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
32. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
33. PT SGS Indo Assay Laboratories, Jakarta, Indonesia
34. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
35. Rio Tinto Kennecott Copper Central Laboratory, Kennecott, Utah, USA
36. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
37. SGS, Randfontein, Gauteng, South Africa
38. SGS Australia Mineral Services, Kalgoorlie, WA, Australia
39. SGS Canada Inc., Vancouver, BC, Canada
40. SGS del Peru, Lima, Peru
41. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
42. SGS Tarkwa, Tarkwa, Western Region, Ghana
43. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
44. Skyline Assayers & Laboratories, Tucson, Arizona, USA
45. UIS Analytical Services, Centurion, South Africa

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





Figure 2. C by IRC in OREAS 278

SPC.1432.RR1.OREAS 278.5.IRC.C.Lab.191022.170051.SS

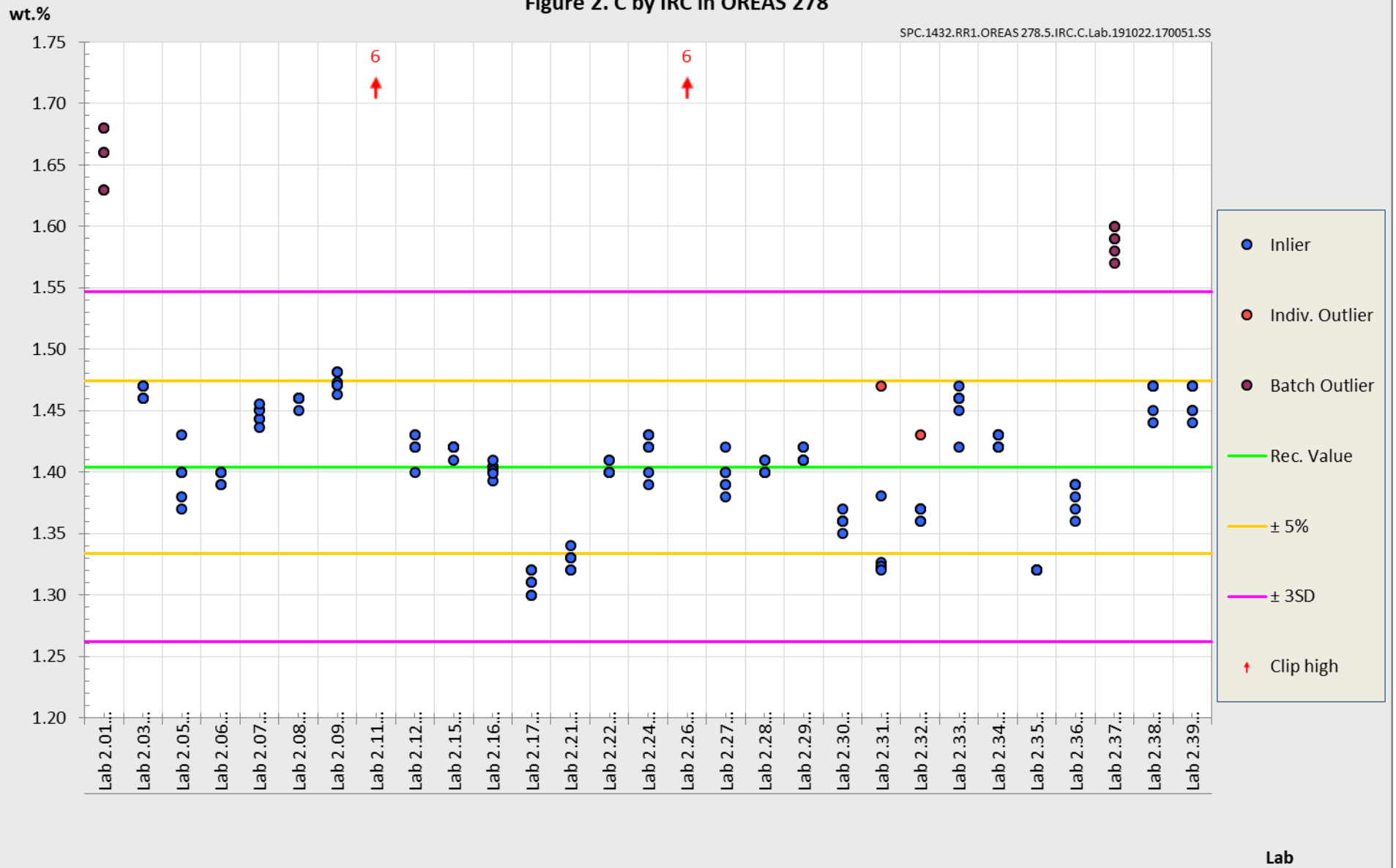
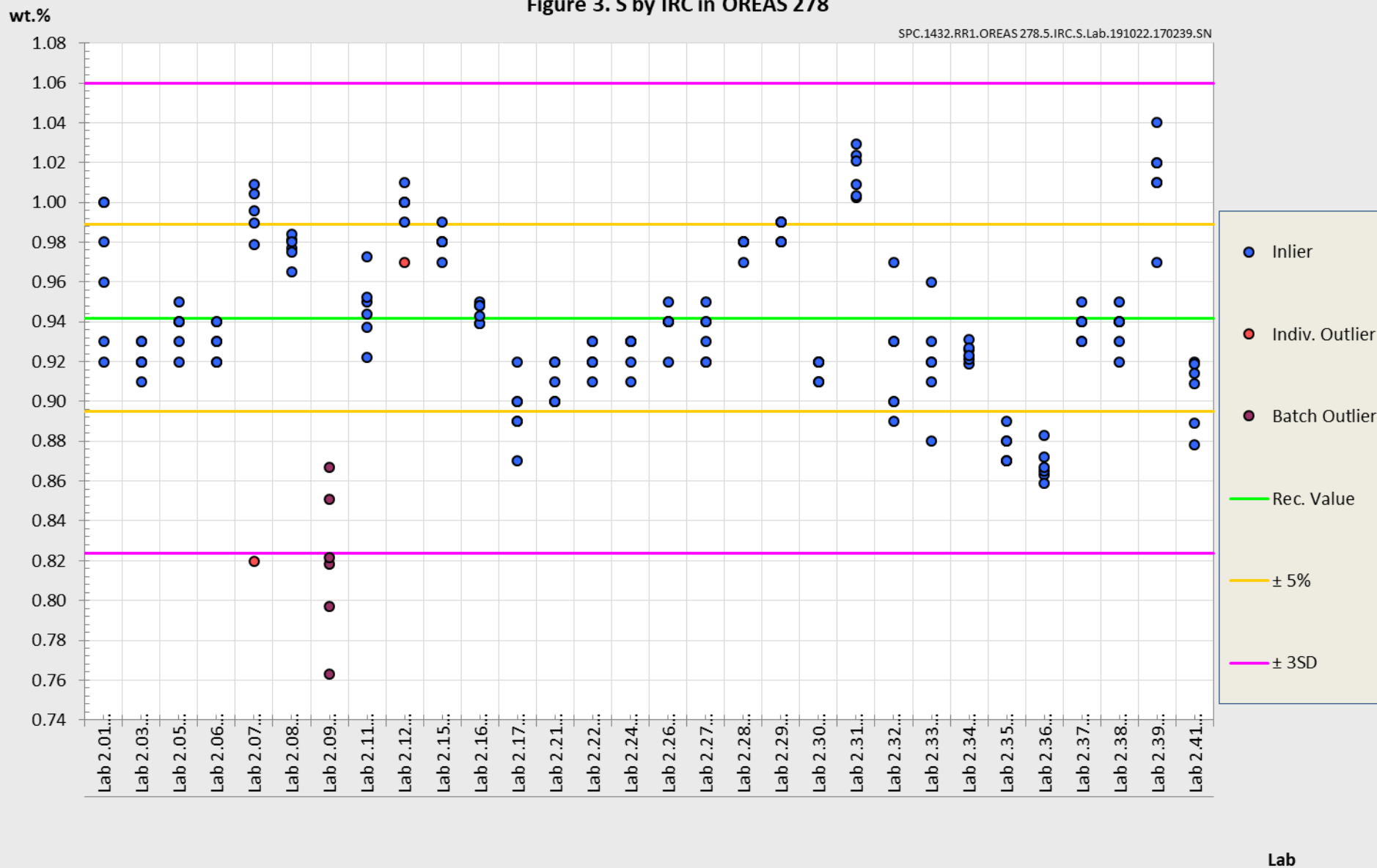


Figure 3. S by IRC in OREAS 278

SPC.1432.RR1.OREAS 278.5.IRC.S.Lab.191022.170239.SN



## PREPARER AND SUPPLIER

Certified reference material OREAS 278 was prepared, certified and supplied by:



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## METROLOGICAL TRACEABILITY

The analytical samples were selected in a manner representative of the entire batch of the prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment, as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, **only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results** (see ISO Guide 35:2006, Clause 10)."*

## COMMUTABILITY

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

## INTENDED USE

OREAS 278 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 278 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 278 is intended for the following uses:

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

### For use with the aqua regia digestion method

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

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

### QC monitoring using multiples of the Standard Deviation (SD)

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-laboratory bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 278 has been prepared from primary gold ore blended with barren sediments. It is low in reactive sulphide (0.94 wt.% S) and in its unopened state and under normal

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

## INSTRUCTIONS FOR CORRECT USE

The certified values for OREAS 278 refer to the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

## HANDLING INSTRUCTIONS

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

## LEGAL NOTICE

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

## DOCUMENT HISTORY

Revision No.	Date	Changes applied
1	25 <sup>th</sup> Aug 2020	Corrected indicative values for XRF, laser ablation and LOI <sup>1000°C</sup> .
0	23 <sup>rd</sup> Oct 2019	First publication.

## QMS CERTIFICATION

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

25<sup>th</sup> August, 2020

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

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