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## CERTIFICATE OF ANALYSIS FOR

### Gold Ore

(Ventersdorp Contact Reef, Mponeng (West Wits) Mine,

Witwatersrand Basin, South Africa

CERTIFIED REFERENCE MATERIAL

# OREAS 299

Table 1. Certified Values and Performance Gates for OREAS 299.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Pb Fire Assay											
Au, ppm	89.97	2.232	85.51	94.43	83.27	96.67	2.48%	4.96%	7.44%	85.47	94.47
Borate Fusion XRF											
Al <sub>2</sub> O <sub>3</sub> , wt. %	5.55	0.040	5.47	5.63	5.43	5.67	0.72%	1.44%	2.16%	5.27	5.83
As, ppm	62	12	38	86	26	98	19.13%	38.26%	57.39%	59	65
BaO, ppm	382	70	243	521	173	591	18.23%	36.46%	54.69%	363	401
CaO, wt. %	0.586	0.009	0.567	0.605	0.558	0.614	1.61%	3.23%	4.84%	0.557	0.615
Cr <sub>2</sub> O <sub>3</sub> , ppm	240	40	160	321	120	361	16.74%	33.49%	50.23%	228	253
Cu, ppm	484	31	422	546	391	577	6.41%	12.82%	19.22%	460	508
Fe <sub>2</sub> O <sub>3</sub> , wt. %	3.44	0.030	3.38	3.50	3.35	3.53	0.88%	1.75%	2.63%	3.26	3.61
K <sub>2</sub> O, wt. %	1.30	0.014	1.27	1.32	1.25	1.34	1.07%	2.14%	3.21%	1.23	1.36
MgO, wt. %	0.844	0.019	0.805	0.882	0.785	0.902	2.30%	4.60%	6.91%	0.801	0.886
MnO, wt. %	0.030	0.001	0.028	0.031	0.027	0.032	2.81%	5.62%	8.42%	0.028	0.031
Na <sub>2</sub> O, wt. %	0.499	0.014	0.470	0.528	0.456	0.542	2.87%	5.73%	8.60%	0.474	0.524
P <sub>2</sub> O <sub>5</sub> , wt. %	0.056	0.003	0.050	0.062	0.047	0.065	5.34%	10.68%	16.02%	0.054	0.059
Pb, ppm	110	27	55	165	28	192	24.92%	49.84%	74.77%	104	115

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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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
Borate Fusion XRF continued											
S, wt. %	0.606	0.021	0.564	0.648	0.543	0.669	3.47%	6.93%	10.40%	0.576	0.637
SiO <sub>2</sub> , wt. %	85.16	0.643	83.87	86.45	83.23	87.09	0.76%	1.51%	2.27%	80.90	89.42
TiO <sub>2</sub> , wt. %	0.523	0.006	0.510	0.536	0.504	0.542	1.21%	2.42%	3.63%	0.497	0.549
V <sub>2</sub> O <sub>5</sub> , ppm	65	10	45	86	34	96	15.79%	31.58%	47.37%	62	69
Zn, ppm	79	5.4	68	90	63	96	6.86%	13.72%	20.58%	75	83
Zr, ppm	344	40	264	423	224	463	11.55%	23.10%	34.65%	326	361
Borate / Peroxide Fusion ICP											
U, ppm	52	1.9	48	56	46	58	3.62%	7.23%	10.85%	49	55
Thermogravimetry											
LOI <sup>1000</sup> , wt. %	1.38	0.19	1.01	1.76	0.82	1.94	13.49%	26.99%	40.48%	1.31	1.45
4-Acid Digestion											
Ag, ppm	6.35	0.291	5.76	6.93	5.47	7.22	4.59%	9.17%	13.76%	6.03	6.66
Al, wt. %	2.90	0.115	2.67	3.14	2.56	3.25	3.98%	7.95%	11.93%	2.76	3.05
As, ppm	55	2.4	50	60	48	62	4.42%	8.84%	13.26%	52	58
Ba, ppm	327	13	300	354	287	367	4.07%	8.15%	12.22%	311	343
Be, ppm	0.97	0.062	0.85	1.09	0.79	1.16	6.36%	12.71%	19.07%	0.92	1.02
Bi, ppm	1.43	0.098	1.23	1.62	1.14	1.72	6.83%	13.66%	20.49%	1.36	1.50
Ca, wt. %	0.423	0.011	0.401	0.445	0.390	0.456	2.62%	5.24%	7.85%	0.402	0.444
Cd, ppm	0.20	0.017	0.17	0.24	0.15	0.26	8.48%	16.97%	25.45%	0.19	0.21
Ce, ppm	49.3	1.49	46.3	52.2	44.8	53.7	3.02%	6.04%	9.06%	46.8	51.7
Co, ppm	18.8	1.59	15.6	22.0	14.0	23.6	8.43%	16.86%	25.29%	17.9	19.7
Cr, ppm	134	26	81	187	54	213	19.79%	39.58%	59.36%	127	140
Cs, ppm	3.76	0.204	3.35	4.17	3.15	4.37	5.43%	10.86%	16.29%	3.57	3.95
Cu, ppm	496	18	461	532	443	550	3.58%	7.16%	10.74%	472	521
Dy, ppm	2.58	0.125	2.33	2.83	2.20	2.95	4.83%	9.67%	14.50%	2.45	2.71
Er, ppm	1.27	0.120	1.03	1.50	0.91	1.62	9.44%	18.89%	28.33%	1.20	1.33
Eu, ppm	0.70	0.043	0.61	0.78	0.57	0.83	6.10%	12.21%	18.31%	0.66	0.73
Fe, wt. %	2.40	0.089	2.22	2.58	2.13	2.67	3.72%	7.44%	11.16%	2.28	2.52
Ga, ppm	7.87	0.285	7.30	8.44	7.02	8.73	3.62%	7.24%	10.86%	7.48	8.27
Gd, ppm	3.35	0.192	2.97	3.74	2.78	3.93	5.71%	11.42%	17.14%	3.19	3.52
Hf, ppm	3.28	0.35	2.57	3.98	2.22	4.34	10.77%	21.53%	32.30%	3.11	3.44
Ho, ppm	0.47	0.018	0.43	0.50	0.41	0.52	3.88%	7.76%	11.64%	0.44	0.49
In, ppm	0.092	0.006	0.079	0.105	0.072	0.111	7.05%	14.10%	21.15%	0.087	0.096
K, wt. %	1.07	0.042	0.99	1.16	0.95	1.20	3.91%	7.83%	11.74%	1.02	1.12
La, ppm	24.5	0.80	22.9	26.1	22.1	26.9	3.26%	6.52%	9.77%	23.3	25.7
Li, ppm	22.3	1.40	19.5	25.1	18.1	26.5	6.26%	12.52%	18.78%	21.2	23.4
Lu, ppm	0.19	0.016	0.16	0.22	0.14	0.24	8.52%	17.04%	25.56%	0.18	0.20
Mg, wt. %	0.505	0.022	0.461	0.548	0.440	0.570	4.30%	8.60%	12.90%	0.480	0.530
Mn, wt. %	0.020	0.001	0.019	0.022	0.018	0.023	4.70%	9.39%	14.09%	0.019	0.022
Mo, ppm	8.07	0.435	7.20	8.94	6.77	9.38	5.39%	10.78%	16.17%	7.67	8.47
Na, wt. %	0.368	0.019	0.329	0.407	0.310	0.426	5.26%	10.52%	15.78%	0.349	0.386
Nb, ppm	9.69	0.554	8.58	10.79	8.02	11.35	5.72%	11.44%	17.16%	9.20	10.17

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
4-Acid Digestion continued											
Nd, ppm	20.7	0.43	19.9	21.6	19.4	22.0	2.09%	4.19%	6.28%	19.7	21.8
Ni, ppm	53	3.4	47	60	43	63	6.34%	12.68%	19.03%	51	56
P, wt. %	0.025	0.001	0.023	0.027	0.021	0.028	4.27%	8.54%	12.81%	0.023	0.026
Pb, ppm	106	6	94	118	88	124	5.60%	11.21%	16.81%	101	111
Pr, ppm	5.63	0.177	5.28	5.98	5.10	6.16	3.14%	6.27%	9.41%	5.35	5.91
Rb, ppm	65	4.0	57	73	53	77	6.13%	12.27%	18.40%	62	68
S, wt. %	0.602	0.039	0.525	0.679	0.487	0.718	6.40%	12.80%	19.20%	0.572	0.632
Sb, ppm	18.3	1.09	16.2	20.5	15.1	21.6	5.94%	11.88%	17.81%	17.4	19.3
Sc, ppm	5.59	0.450	4.69	6.49	4.24	6.94	8.05%	16.10%	24.15%	5.31	5.87
Sm, ppm	3.97	0.126	3.72	4.22	3.59	4.35	3.17%	6.34%	9.52%	3.77	4.17
Sn, ppm	2.05	0.185	1.68	2.42	1.49	2.60	9.04%	18.09%	27.13%	1.95	2.15
Sr, ppm	45.2	1.62	41.9	48.4	40.3	50.0	3.59%	7.17%	10.76%	42.9	47.4
Ta, ppm	1.03	0.080	0.87	1.19	0.79	1.27	7.77%	15.54%	23.31%	0.98	1.08
Tb, ppm	0.47	0.040	0.39	0.55	0.35	0.59	8.44%	16.89%	25.33%	0.45	0.49
Te, ppm	0.36	0.05	0.26	0.46	0.21	0.51	13.72%	27.44%	41.16%	0.34	0.38
Th, ppm	13.4	0.55	12.3	14.5	11.8	15.1	4.07%	8.15%	12.22%	12.8	14.1
Ti, wt. %	0.302	0.015	0.272	0.331	0.258	0.345	4.85%	9.70%	14.56%	0.286	0.317
Tl, ppm	0.38	0.030	0.32	0.44	0.29	0.47	8.02%	16.04%	24.06%	0.36	0.40
Tm, ppm	0.18	0.02	0.13	0.23	0.11	0.25	13.10%	26.20%	39.31%	0.17	0.19
U, ppm	51	2.8	45	56	42	59	5.61%	11.22%	16.82%	48	53
V, ppm	42.5	1.85	38.8	46.2	37.0	48.1	4.35%	8.70%	13.05%	40.4	44.7
W, ppm	5.93	0.334	5.26	6.60	4.93	6.93	5.63%	11.27%	16.90%	5.63	6.23
Y, ppm	10.9	0.92	9.0	12.7	8.1	13.6	8.51%	17.02%	25.53%	10.3	11.4
Yb, ppm	1.24	0.14	0.96	1.51	0.83	1.65	11.03%	22.07%	33.10%	1.18	1.30
Zn, ppm	78	2.9	72	84	69	87	3.78%	7.57%	11.35%	74	82
Zr, ppm	113	9	95	131	87	140	7.80%	15.60%	23.41%	107	119
Infrared Combustion											
S, wt. %	0.603	0.027	0.550	0.657	0.523	0.684	4.44%	8.88%	13.32%	0.573	0.634
Gas / Liquid Pycnometry											
SG, Unity	2.72	0.072	2.58	2.87	2.51	2.94	2.63%	5.26%	7.89%	2.59	2.86

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 299 is one of a suite of seven Witwatersrand ore CRMs covering the gold range 0.07ppm to 90ppm Au. 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 299 DataPack-1.2.200205\_160847.xlsx**).

## SOURCE MATERIAL

OREAS 299 has been prepared from underground sample material from the Ventersdorp Contact Reef (VCR). The material was provided by AngloGold Ashanti from the Mponeng Mine which is located 80 km west of Johannesburg in the West Wits mining district. The VCR is the youngest of the Witwatersrand palaeoplacers and comprises a gold bearing quartz pebble conglomerate preserved on a terraced unconformity surface and buried by the 2.7 Ga Ventersdorp Lava. The VCR and the footwall Witwatersrand sediments were modified (cooked) post burial by lower greenschist level hydrothermal metamorphism. This overprinting event remobilised some of the gold and pyrite within the conglomerate matrix; and deposited minor authigenic pyrrhotite, chalcopyrite, sphalerite and galena. These Reef samples were taken underground for grade control purposes and assayed. The pulp reject material was then sorted into different grade bins for the purposes of CRM manufacture. Minor barren quartz, hornfels and granodiorite have been added to the pulps to achieve targeted CRM grades.

## 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 299.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppb	14.2	Pt	ppb	< 5			
<b>Borate Fusion XRF</b>								
Bi	ppm	< 27	Lu <sub>2</sub> O <sub>3</sub>	ppm	< 1	SrO	ppm	86
CeO <sub>2</sub>	ppm	172	Mo	ppm	69	Ta	ppm	39.4
Cl	ppm	34.8	Nb <sub>2</sub> O <sub>5</sub>	ppm	< 3	Tb <sub>4</sub> O <sub>7</sub>	ppm	51
Co	ppm	19.3	Nd <sub>2</sub> O <sub>3</sub>	ppm	< 50	Th	ppm	55
Cs	ppm	< 47	Ni	ppm	55	Tm <sub>2</sub> O <sub>3</sub>	ppm	< 2
Dy <sub>2</sub> O <sub>3</sub>	ppm	< 35	Pr <sub>6</sub> O <sub>11</sub>	ppm	< 20	U <sub>3</sub> O <sub>8</sub>	ppm	54
Er <sub>2</sub> O <sub>3</sub>	ppm	18.0	Rb	ppm	< 18	W	ppm	32.6
Eu <sub>2</sub> O <sub>3</sub>	ppm	< 15	Sb	ppm	640	Y <sub>2</sub> O <sub>3</sub>	ppm	50
Gd <sub>2</sub> O <sub>3</sub>	ppm	< 2	Sc	ppm	< 1	Yb <sub>2</sub> O <sub>3</sub>	ppm	< 2
Ho <sub>2</sub> O <sub>3</sub>	ppm	< 2	Sm <sub>2</sub> O <sub>3</sub>	ppm	< 10			
La <sub>2</sub> O <sub>3</sub>	ppm	< 40	Sn	ppm	38.2			
<b>Borate / Peroxide Fusion ICP</b>								
Al <sub>2</sub> O <sub>3</sub>	wt. %	5.88	Ge	ppm	< 10	Sb	ppm	37.0
As	ppm	66	Hf	ppm	8.87	Sc	ppm	5.00
B	ppm	91	Ho	ppm	0.61	Se	ppm	< 40
Ba	ppm	354	K <sub>2</sub> O	wt. %	1.33	SiO <sub>2</sub>	wt. %	83.13
Be	ppm	< 0.2	La	ppm	25.7	Sm	ppm	4.22
Bi	ppm	29.0	Li	ppm	< 15	Sn	ppm	2.39
CaO	wt. %	0.652	Lu	ppm	0.27	Sr	ppm	46.8
Cd	ppm	< 10	MgO	wt. %	0.803	Ta	ppm	1.17
Ce	ppm	51	MnO	wt. %	0.030	Tb	ppm	0.54
Co	ppm	25.2	Mo	ppm	< 8	Th	ppm	14.1
Cr	ppm	158	Na <sub>2</sub> O	wt. %	0.492	TiO <sub>2</sub>	wt. %	0.514
Cs	ppm	3.75	Nb	ppm	9.48	Tm	ppm	0.28
Cu	ppm	408	Nd	ppm	21.7	V	ppm	49.4
Dy	ppm	3.13	Ni	ppm	56	W	ppm	5.87
Er	ppm	1.79	P <sub>2</sub> O <sub>5</sub>	wt. %	0.030	Y	ppm	16.4
Eu	ppm	0.74	Pb	ppm	123	Yb	ppm	1.89
Fe <sub>2</sub> O <sub>3</sub>	wt. %	3.66	Pr	ppm	5.99	Zr	ppm	352
Ga	ppm	8.49	Rb	ppm	65			
Gd	ppm	3.72	S	wt. %	0.580			
<b>4-Acid Digestion</b>								
B	ppm	0.11	Hg	ppm	0.28	Se	ppm	0.87
Ge	ppm	0.22	Re	ppm	0.008			
<b>Infrared Combustion</b>								
C	wt. %	0.058						

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.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 299 was prepared in the following manner:

- Drying to constant mass at 105°C;
- Crushing and milling of ore materials to 100% minus 30 microns;
- Crushing and milling of barren materials to 98% minus 75 microns;
- Blending ores and barren materials 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 299 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 299.**

Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
618	0.55	N7	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

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

- Gold by fire assay (25-50g charge weight) with gravimetric (23 laboratories), AAS (7 laboratories) or ICP-OES (3 laboratories) finish;
- Major and trace elements by borate fusion with XRF (up to 17 laboratories depending on the element);
- Uranium by fusion with ICP-MS (5 laboratories);
- Full ICP-OES and ICP-MS elemental suites by borate or peroxide fusion (up to 4 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by 4-acid (HNO<sub>3</sub>-HF-HClO<sub>4</sub>-HCl) digestion (up to 27 laboratories depending on the element);
- Specific gravity by gas (17 laboratories) or liquid (2 laboratories) pycnometry;
- Total Sulphur by infrared combustion furnace (28 laboratories).

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 1.5kg test units were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. Six 120g pulp samples were submitted to each laboratory for analysis. The samples received by each laboratory were obtained by taking two samples from each of three separate 1.5kg 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 81 certified values based on their pooled 1SD's. Table 2 shows 98 indicative values and Table 3 provides some indicative physical properties. Table 4 presents 95% confidence and tolerance limits and gold homogeneity (via INAA) is shown in Table 5. Gold homogeneity is also demonstrated by a nested ANOVA program using the fire assay data (see 'nested ANOVA' section).

Results for gold by fire assay are also presented in a scatter plot (Figure 1) 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 299 (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 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 299.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppm)	89.97	89.24	90.70	89.76	90.19
Borate Fusion XRF					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	5.55	5.53	5.57	5.52	5.58
As, Arsenic (ppm)	62	47	77	IND	IND
BaO, Barium oxide (ppm)	382	343	421	340	423
CaO, Calcium oxide (wt.%)	0.586	0.581	0.591	0.578	0.594
Cr <sub>2</sub> O <sub>3</sub> , Chromium(III) oxide (ppm)	240	221	260	217	264
Cu, Copper (ppm)	484	455	512	461	506
Fe <sub>2</sub> O <sub>3</sub> , Iron(III) oxide (wt.%)	3.44	3.42	3.45	3.41	3.47
K <sub>2</sub> O, Potassium oxide (wt.%)	1.30	1.29	1.30	1.28	1.31
MgO, Magnesium oxide (wt.%)	0.844	0.834	0.853	0.831	0.856
MnO, Manganese oxide (wt.%)	0.030	0.029	0.030	0.028	0.031
Na <sub>2</sub> O, Sodium oxide (wt.%)	0.499	0.492	0.505	0.486	0.512
P <sub>2</sub> O <sub>5</sub> , Phosphorus(V) oxide (wt.%)	0.056	0.055	0.057	0.055	0.058
Pb, Lead (ppm)	110	90	130	IND	IND
S, Sulphur (wt.%)	0.606	0.590	0.623	0.593	0.620
SiO <sub>2</sub> , Silicon dioxide (wt.%)	85.16	84.87	85.46	84.88	85.44

SI unit equivalents: ppm (parts per million)  $\equiv$  mg/kg  $\equiv$   $\mu$ 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
Borate Fusion XRF continued					
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.523	0.520	0.526	0.512	0.534
V <sub>2</sub> O <sub>5</sub> , Vanadium(V) oxide (ppm)	65	58	72	IND	IND
Zn, Zinc (ppm)	79	76	83	IND	IND
Zr, Zirconium (ppm)	344	307	380	326	361
Borate / Peroxide Fusion ICP					
U, Uranium (ppm)	52	50	54	51	53
Thermogravimetry					
LOI <sup>1000</sup> , Loss on ignition @1000°C (wt.%)	1.38	1.28	1.48	1.34	1.43
4-Acid Digestion					
Ag, Silver (ppm)	6.35	6.22	6.47	6.20	6.50
Al, Aluminium (wt.%)	2.90	2.86	2.95	2.85	2.96
As, Arsenic (ppm)	55	54	56	54	56
Ba, Barium (ppm)	327	322	332	321	333
Be, Beryllium (ppm)	0.97	0.95	0.99	0.92	1.02
Bi, Bismuth (ppm)	1.43	1.39	1.47	1.36	1.50
Ca, Calcium (wt.%)	0.423	0.419	0.428	0.414	0.433
Cd, Cadmium (ppm)	0.20	0.19	0.21	0.19	0.22
Ce, Cerium (ppm)	49.3	48.7	49.8	48.1	50.4
Co, Cobalt (ppm)	18.8	18.2	19.4	18.2	19.4
Cr, Chromium (ppm)	134	123	144	128	139
Cs, Caesium (ppm)	3.76	3.68	3.84	3.62	3.89
Cu, Copper (ppm)	496	490	503	488	505
Dy, Dysprosium (ppm)	2.58	2.50	2.66	2.48	2.68
Er, Erbium (ppm)	1.27	1.18	1.35	1.20	1.33
Eu, Europium (ppm)	0.70	0.66	0.74	0.66	0.74
Fe, Iron (wt.%)	2.40	2.37	2.43	2.36	2.44
Ga, Gallium (ppm)	7.87	7.74	8.00	7.65	8.09
Gd, Gadolinium (ppm)	3.35	3.24	3.46	3.20	3.51
Hf, Hafnium (ppm)	3.28	3.11	3.44	3.15	3.40
Ho, Holmium (ppm)	0.47	0.45	0.48	0.44	0.50
In, Indium (ppm)	0.092	0.090	0.094	0.086	0.098
K, Potassium (wt.%)	1.07	1.05	1.09	1.05	1.09
La, Lanthanum (ppm)	24.5	24.2	24.8	23.8	25.2
Li, Lithium (ppm)	22.3	21.7	22.9	21.6	23.0
Lu, Lutetium (ppm)	0.19	0.17	0.21	IND	IND
Mg, Magnesium (wt.%)	0.505	0.496	0.514	0.496	0.514
Mn, Manganese (wt.%)	0.020	0.020	0.021	0.020	0.021
Mo, Molybdenum (ppm)	8.07	7.91	8.24	7.87	8.28

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
4-Acid Digestion continued					
Na, Sodium (wt.%)	0.368	0.360	0.376	0.357	0.379
Nb, Niobium (ppm)	9.69	9.42	9.95	9.44	9.93
Nd, Neodymium (ppm)	20.7	20.5	20.9	20.2	21.3
Ni, Nickel (ppm)	53	52	55	52	55
P, Phosphorus (wt.%)	0.025	0.024	0.025	0.024	0.025
Pb, Lead (ppm)	106	104	108	104	108
Pr, Praseodymium (ppm)	5.63	5.49	5.77	5.47	5.79
Rb, Rubidium (ppm)	65	63	67	64	67
S, Sulphur (wt.%)	0.602	0.584	0.620	0.587	0.617
Sb, Antimony (ppm)	18.3	17.9	18.8	17.8	18.8
Sc, Scandium (ppm)	5.59	5.36	5.82	5.39	5.80
Sm, Samarium (ppm)	3.97	3.91	4.03	3.84	4.10
Sn, Tin (ppm)	2.05	1.96	2.14	1.95	2.15
Sr, Strontium (ppm)	45.2	44.6	45.8	44.2	46.1
Ta, Tantalum (ppm)	1.03	0.99	1.07	0.99	1.07
Tb, Terbium (ppm)	0.47	0.44	0.50	0.45	0.49
Te, Tellurium (ppm)	0.36	0.33	0.39	0.32	0.40
Th, Thorium (ppm)	13.4	13.2	13.7	13.1	13.8
Ti, Titanium (wt.%)	0.302	0.295	0.308	0.295	0.308
Tl, Thallium (ppm)	0.38	0.37	0.39	0.36	0.40
Tm, Thulium (ppm)	0.18	0.16	0.20	IND	IND
U, Uranium (ppm)	51	50	52	49	52
V, Vanadium (ppm)	42.5	41.8	43.2	41.4	43.7
W, Tungsten (ppm)	5.93	5.80	6.06	5.69	6.17
Y, Yttrium (ppm)	10.9	10.4	11.3	10.6	11.1
Yb, Ytterbium (ppm)	1.24	1.15	1.32	1.16	1.32
Zn, Zinc (ppm)	78	77	79	75	81
Zr, Zirconium (ppm)	113	109	118	109	118
Infrared Combustion					
S, Sulphur (wt.%)	0.603	0.592	0.615	0.593	0.614
Gas / Liquid Pycnometry					
SG, Specific Gravity (Unity)	2.72	2.69	2.75	2.69	2.76

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 zinc by 4-acid digestion, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of

subsamples ( $p=0.95$ ) will have concentrations lying between 75 and 81ppm. 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. 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	92.84	94.47
2	93.45	94.51
3	94.56	94.56
4	94.36	94.55
5	91.41	94.40
6	95.53	94.62
7	96.38	94.66
8	94.45	94.56
9	96.55	94.67
10	94.55	94.56
11	94.86	94.58
12	94.95	94.58
13	94.06	94.54
14	93.84	94.53
15	94.70	94.57
16	93.35	94.50
17	95.75	94.63
18	95.60	94.62
19	96.87	94.69
20	93.23	94.49
Mean	94.56	94.56
Median	94.56	94.56
Std Dev.	1.35	0.07
Rel.Std.Dev.	1.42%	0.08%

\*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

Table 5 above shows the gold INAA data determined on 20 x 85mg subsamples of OREAS 299. 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 (1.42% at 85mg weights) and confirms the high level of gold homogeneity in OREAS 299.

The homogeneity of OREAS 299 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the thirty-four 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 299. The test was performed using the following parameters:

- Gold fire assay – 198 samples (33 laboratories each providing analyses on 3 pairs of samples);
- Null Hypothesis,  $H_0$ : Between-unit variance is no greater than within-unit variance (reject  $H_0$  if  $p$ -value  $< 0.05$ );
- Alternative Hypothesis,  $H_1$ : Between-unit variance is greater than within-unit variance.

$P$ -values are a measure of probability where values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the  $p$ -value. This process derived a  $p$ -value of 0.90 for Au by fire assay 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.  $>20 \times$  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 299 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 299 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, Brisbane, QLD, Australia
5. ALS, Lima, Peru

6. ALS, Loughrea, Galway, Ireland
7. ALS, Perth, WA, Australia
8. ALS, Vancouver, BC, Canada
9. American Assay Laboratories, Sparks, Nevada, USA
10. ANSTO, Lucas Heights, NSW, Australia
11. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
12. Bureau Veritas, Abidjan, Côte d'Ivoire
13. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
14. Bureau Veritas Geoanalytical, Perth, WA, Australia
15. Inspectorate (BV), Lima, Peru
16. Inspectorate America Corporation (BV), Sparks, Nevada, USA
17. Intertek Genalysis, Adelaide, SA, Australia
18. Intertek Genalysis, Perth, WA, Australia
19. Intertek Tarkwa, Tarkwa, Ghana
20. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
21. MinAnalytical Services, Perth, WA, Australia
22. Nagrom, Perth, WA, Australia
23. Ontario Geological Survey, Sudbury, Ontario, Canada
24. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
25. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
26. Quality Laboratory Services, Dar es Salaam, Chunya, United Republic of Tanzania
27. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
28. Saskatchewan Research Council, Saskatoon, Saskatchewan, Canada
29. SGS, Randfontein, Gauteng, South Africa
30. SGS Canada Inc., Vancouver, BC, Canada
31. SGS del Peru, Lima, Peru
32. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
33. SGS Tarkwa, Tarkwa, Western Region, Ghana
34. Skyline Assayers & Laboratories, Tucson, Arizona, USA
35. 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 plot below.***





## PREPARER AND SUPPLIER

Certified reference material OREAS 299 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 299 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 299 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 299 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.

### 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 299 has been prepared from primary gold ore blended with barren quartz, barren hornfels and barren granodiorite. It is low in reactive sulphide (0.60 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 by lithium borate fusion XRF and for LOI at 1000° C are on a dry sample basis while the certified values by other methods (fire assay, infrared combustion furnace, fusion ICP, 4-acid digestion and pycnometry) are reported on a 'sample as received' basis.

## 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
2	24 <sup>th</sup> February, 2020	Edited description of gold fire assay analytical program section.
1	5 <sup>th</sup> February, 2020	Minor edits to the 'Source Material' section.
0	3 <sup>rd</sup> February, 2020	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

24<sup>th</sup> February, 2020

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

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