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**CERTIFICATE OF ANALYSIS FOR**  
**CERTIFIED REFERENCE MATERIAL**  
**OREAS 525**  
**Iron Oxide Copper-Gold Ore, Australia**

**Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 525.**

Constituent	Certified Value <sup>†</sup>	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	0.818	0.811	0.825	0.812*	0.823*

SI unit equivalents: ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg.

<sup>†</sup>This operationally defined measurand meets the requirements of ISO 17034 and all participating laboratories comply with the requirements of ISO 17025.

\*Gold Tolerance Limits for typical 30g fire assay are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.



Accredited for compliance with ISO 17034



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**Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 525.**

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion (sample weights 10-50g)</b>					
Au, Gold (ppm)	0.792	0.768	0.815	0.786*	0.798*
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	2.09	1.99	2.18	2.02	2.16
Al, Aluminium (wt.%)	5.25	5.13	5.37	5.19	5.31
As, Arsenic (ppm)	331	322	341	324	339
Be, Beryllium (ppm)	1.66	1.55	1.78	1.62	1.70
Bi, Bismuth (ppm)	8.45	8.12	8.77	8.20	8.70
Ca, Calcium (wt.%)	2.41	2.35	2.47	2.38	2.45
Cd, Cadmium (ppm)	0.21	0.16	0.25	0.19	0.22
Ce, Cerium (ppm)	154	145	164	149	160
Co, Cobalt (ppm)	379	369	389	372	385
Cr, Chromium (ppm)	22.4	20.8	23.9	21.1	23.6
Cs, Caesium (ppm)	2.39	2.29	2.49	2.31	2.47
Cu, Copper (wt.%)	1.35	1.32	1.37	1.33	1.36
Dy, Dysprosium (ppm)	4.03	3.76	4.31	3.90	4.17
Er, Erbium (ppm)	1.97	1.84	2.09	1.89	2.05
Eu, Europium (ppm)	2.16	1.90	2.42	2.12	2.21
Fe, Iron (wt.%)	16.77	16.31	17.23	16.56	16.98
Ga, Gallium (ppm)	18.1	17.3	18.8	17.5	18.6
Gd, Gadolinium (ppm)	5.85	5.49	6.21	5.63	6.07
Ge, Germanium (ppm)	0.25	0.21	0.30	0.22	0.28
Hf, Hafnium (ppm)	4.87	4.65	5.10	4.73	5.02
Ho, Holmium (ppm)	0.75	0.70	0.80	0.73	0.77
In, Indium (ppm)	0.31	0.29	0.33	0.30	0.32
K, Potassium (wt.%)	3.28	3.20	3.37	3.21	3.36
La, Lanthanum (ppm)	144	134	154	139	148
Li, Lithium (ppm)	16.2	15.6	16.8	15.7	16.7
Lu, Lutetium (ppm)	0.29	0.26	0.31	0.27	0.30
Mg, Magnesium (wt.%)	0.773	0.753	0.793	0.761	0.784
Mn, Manganese (wt.%)	0.323	0.314	0.331	0.318	0.328
Mo, Molybdenum (ppm)	190	185	196	187	194
Na, Sodium (wt.%)	1.15	1.12	1.17	1.12	1.17
Nb, Niobium (ppm)	12.1	11.1	13.1	11.6	12.7
Nd, Neodymium (ppm)	42.7	40.0	45.5	41.2	44.2
Ni, Nickel (ppm)	54	53	56	53	56
P, Phosphorus (wt.%)	0.105	0.103	0.108	0.104	0.107
Pb, Lead (ppm)	18.4	16.7	20.0	17.7	19.0

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

\*Gold Tolerance Limits are calculated for a typical 25g aqua regia digestion and determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
Pr, Praseodymium (ppm)	13.1	12.2	14.1	12.7	13.6
Rb, Rubidium (ppm)	115	110	119	111	118
Re, Rhenium (ppm)	0.11	0.10	0.12	0.10	0.11
S, Sulphur (wt.%)	2.39	2.31	2.46	2.34	2.43
Sb, Antimony (ppm)	5.59	5.37	5.81	5.43	5.75
Sc, Scandium (ppm)	8.45	8.09	8.82	8.18	8.72
Se, Selenium (ppm)	3.06	2.32	3.81	2.64	3.49
Sm, Samarium (ppm)	6.82	6.36	7.28	6.53	7.10
Sn, Tin (ppm)	6.97	6.62	7.31	6.74	7.19
Sr, Strontium (ppm)	222	210	234	217	227
Ta, Tantalum (ppm)	0.81	0.74	0.88	0.77	0.85
Tb, Terbium (ppm)	0.79	0.72	0.85	0.76	0.81
Te, Tellurium (ppm)	1.06	0.98	1.14	1.01	1.11
Th, Thorium (ppm)	12.2	11.3	13.2	11.6	12.9
Ti, Titanium (wt.%)	0.319	0.307	0.331	0.311	0.328
Tl, Thallium (ppm)	0.42	0.40	0.44	0.40	0.44
Tm, Thulium (ppm)	0.27	0.24	0.30	0.26	0.28
U, Uranium (ppm)	30.8	29.4	32.2	30.0	31.6
V, Vanadium (ppm)	107	104	110	105	109
W, Tungsten (ppm)	85	81	89	82	88
Y, Yttrium (ppm)	19.5	18.8	20.3	19.1	20.0
Yb, Ytterbium (ppm)	1.71	1.59	1.83	1.65	1.78
Zn, Zinc (ppm)	68	66	71	66	70
<b>Aqua Regia Digestion</b>					
Ag, Silver (ppm)	1.97	1.90	2.04	1.92	2.02
Al, Aluminium (wt.%)	1.10	1.06	1.14	1.08	1.13
As, Arsenic (ppm)	321	312	330	316	326
Be, Beryllium (ppm)	0.69	0.65	0.74	0.67	0.72
Bi, Bismuth (ppm)	8.44	8.05	8.84	8.22	8.67
Ca, Calcium (wt.%)	2.15	2.10	2.20	2.09	2.20
Cd, Cadmium (ppm)	0.18	0.15	0.22	0.17	0.20
Ce, Cerium (ppm)	133	126	140	129	137
Co, Cobalt (ppm)	378	367	389	372	384
Cr, Chromium (ppm)	18.7	17.6	19.7	18.1	19.3
Cs, Caesium (ppm)	0.78	0.73	0.84	0.75	0.81
Cu, Copper (wt.%)	1.35	1.32	1.38	1.33	1.37
Fe, Iron (wt.%)	16.07	15.69	16.45	15.83	16.31
Ga, Gallium (ppm)	9.03	8.43	9.63	8.79	9.26

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

Note: intervals may appear asymmetric due to rounding.

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Aqua Regia Digestion continued</b>					
Ge, Germanium (ppm)	0.20	0.16	0.24	0.18	0.22
Hf, Hafnium (ppm)	1.73	1.64	1.83	1.68	1.79
In, Indium (ppm)	0.28	0.26	0.30	0.27	0.29
K, Potassium (wt.%)	0.536	0.517	0.555	0.527	0.546
La, Lanthanum (ppm)	131	121	141	128	134
Li, Lithium (ppm)	11.2	10.7	11.6	10.8	11.5
Lu, Lutetium (ppm)	0.19	0.17	0.20	IND	IND
Mg, Magnesium (wt.%)	0.703	0.682	0.724	0.690	0.715
Mn, Manganese (wt.%)	0.303	0.294	0.312	0.299	0.307
Mo, Molybdenum (ppm)	186	179	192	182	189
Na, Sodium (wt.%)	0.090	0.085	0.094	0.088	0.092
Nb, Niobium (ppm)	1.12	0.97	1.27	1.07	1.17
Ni, Nickel (ppm)	52	50	54	51	53
P, Phosphorus (wt.%)	0.098	0.095	0.101	0.096	0.100
Pb, Lead (ppm)	14.4	13.1	15.6	13.8	14.9
Rb, Rubidium (ppm)	30.3	28.9	31.8	29.4	31.3
Re, Rhenium (ppm)	0.11	0.10	0.11	0.10	0.11
S, Sulphur (wt.%)	2.42	2.36	2.48	2.38	2.47
Sb, Antimony (ppm)	3.57	3.21	3.93	3.45	3.69
Sc, Scandium (ppm)	5.82	5.51	6.13	5.64	6.00
Se, Selenium (ppm)	2.93	2.67	3.19	2.71	3.15
Sn, Tin (ppm)	5.03	4.81	5.26	4.86	5.21
Sr, Strontium (ppm)	68	62	74	66	70
Ta, Tantalum (ppm)	< 0.05	IND	IND	IND	IND
Tb, Terbium (ppm)	0.58	0.53	0.62	0.55	0.60
Te, Tellurium (ppm)	1.01	0.90	1.11	0.96	1.05
Th, Thorium (ppm)	8.99	8.34	9.64	8.70	9.29
Ti, Titanium (wt.%)	0.118	0.110	0.127	0.116	0.121
Tl, Thallium (ppm)	0.13	0.12	0.15	IND	IND
U, Uranium (ppm)	27.3	25.9	28.6	26.5	28.0
V, Vanadium (ppm)	97	94	100	95	99
W, Tungsten (ppm)	67	63	71	65	69
Y, Yttrium (ppm)	13.9	13.3	14.5	13.5	14.3
Yb, Ytterbium (ppm)	1.18	1.09	1.28	IND	IND
Zn, Zinc (ppm)	47.7	45.5	49.8	46.5	48.9
Zr, Zirconium (ppm)	66	64	69	65	68

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

Note: intervals may appear asymmetric due to rounding.

IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

Table 2 continued.

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Peroxide Fusion ICP</b>					
Al, Aluminium (wt.%)	5.38	5.25	5.50	5.28	5.47
As, Arsenic (ppm)	329	299	358	319	338
Ba, Barium (wt.%)	1.55	1.41	1.70	1.52	1.59
Be, Beryllium (ppm)	2.16	1.78	2.54	IND	IND
Bi, Bismuth (ppm)	8.67	7.87	9.46	8.07	9.26
Ca, Calcium (wt.%)	2.45	2.35	2.54	2.40	2.49
Ce, Cerium (ppm)	175	161	189	169	182
Co, Cobalt (ppm)	385	370	401	378	393
Cs, Caesium (ppm)	2.37	2.17	2.57	2.22	2.52
Cu, Copper (wt.%)	1.35	1.32	1.38	1.33	1.38
Dy, Dysprosium (ppm)	4.31	3.84	4.79	4.13	4.50
Er, Erbium (ppm)	2.11	1.94	2.29	1.95	2.27
Fe, Iron (wt.%)	17.18	16.81	17.56	16.90	17.47
Ga, Gallium (ppm)	18.5	17.4	19.7	17.5	19.5
Gd, Gadolinium (ppm)	5.76	5.12	6.39	5.45	6.06
Ho, Holmium (ppm)	0.82	0.76	0.88	0.73	0.91
In, Indium (ppm)	0.32	0.23	0.42	IND	IND
K, Potassium (wt.%)	3.39	3.28	3.49	3.30	3.47
La, Lanthanum (ppm)	176	167	185	171	180
Li, Lithium (ppm)	16.6	14.0	19.3	15.5	17.7
Lu, Lutetium (ppm)	0.32	0.28	0.37	0.30	0.34
Mg, Magnesium (wt.%)	0.801	0.779	0.823	0.786	0.816
Mn, Manganese (wt.%)	0.336	0.324	0.347	0.328	0.343
Mo, Molybdenum (ppm)	195	180	209	190	200
Nb, Niobium (ppm)	14.9	13.2	16.5	14.4	15.3
Nd, Neodymium (ppm)	43.3	39.7	46.9	42.0	44.6
Ni, Nickel (ppm)	58	52	65	54	62
P, Phosphorus (wt.%)	0.109	0.098	0.120	IND	IND
Pr, Praseodymium (ppm)	13.6	12.9	14.4	13.3	14.0
Rb, Rubidium (ppm)	116	110	122	113	120
S, Sulphur (wt.%)	2.78	2.71	2.86	2.73	2.84
Sb, Antimony (ppm)	5.97	5.02	6.93	5.54	6.40
Si, Silicon (wt.%)	21.22	20.52	21.92	20.78	21.66
Sm, Samarium (ppm)	6.64	6.11	7.18	6.33	6.96
Sr, Strontium (ppm)	250	239	262	244	256
Ta, Tantalum (ppm)	1.08	0.94	1.22	IND	IND
Tb, Terbium (ppm)	0.79	0.75	0.83	0.70	0.89

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

Note: intervals may appear asymmetric due to rounding.

IND = indeterminate (due to limited reading resolution of the methods employed).

**Table 2 continued.**

Constituent	Certified Value	95% Expanded Uncertainty		95% Tolerance Limits	
		Low	High	Low	High
<b>Peroxide Fusion ICP continued</b>					
Th, Thorium (ppm)	13.4	12.8	14.0	12.9	13.9
Ti, Titanium (wt.%)	0.375	0.363	0.386	0.367	0.383
Tl, Thallium (ppm)	< 0.5	IND	IND	IND	IND
Tm, Thulium (ppm)	0.30	0.27	0.33	0.28	0.33
U, Uranium (ppm)	31.0	29.2	32.7	30.0	32.0
V, Vanadium (ppm)	113	103	124	110	116
W, Tungsten (ppm)	83	76	89	80	86
Y, Yttrium (ppm)	22.4	21.4	23.5	21.7	23.1
Yb, Ytterbium (ppm)	1.98	1.80	2.15	IND	IND
Zn, Zinc (ppm)	71	63	80	68	75
<b>3-Acid Digestion (no HF)</b>					
Ag, Silver (ppm)	2.19	1.94	2.44	2.01	2.37
As, Arsenic (ppm)	324	311	336	316	332
Co, Cobalt (ppm)	370	353	388	365	376
Cu, Copper (wt.%)	1.33	1.29	1.37	1.31	1.35
Fe, Iron (wt.%)	16.65	15.97	17.34	16.35	16.96
Mo, Molybdenum (ppm)	186	179	192	182	189
S, Sulphur (wt.%)	2.48	2.39	2.58	2.42	2.55
<b>Infrared Combustion</b>					
S, Sulphur (wt.%)	2.75	2.68	2.82	2.72	2.79
<b>Ion Selective Electrode</b>					
F, Fluorine (ppm)	2780	2611	2948	2697	2862

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

Note: intervals may appear asymmetric due to rounding; IND = indeterminate (due to limited reading resolution of the methods employed. For practical purposes the 95% Expanded Uncertainty can be set between zero and a two times multiple of the upper bound/non-detect limit value).

**Table 3. Indicative Values for OREAS 525.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppb	1.80	Pt	ppb	1.03			
<b>4-Acid Digestion</b>								
Ba	ppm	215	Hg	ppm	< 1			
<b>Aqua Regia Digestion</b>								
B	ppm	7.57	Gd	ppm	4.30	Pr	ppm	10.3
Ba	ppm	112	Hg	ppm	0.094	Pt	ppb	10.1
Dy	ppm	2.86	Ho	ppm	0.52	Sm	ppm	4.72
Er	ppm	1.40	Nd	ppm	30.8	Tm	ppm	0.19
Eu	ppm	1.59	Pd	ppb	< 10			

SI unit equivalents: ppb (parts per billion;  $1 \times 10^{-9}$ )  $\equiv$   $\mu$ g/kg; ppm (parts per million;  $1 \times 10^{-6}$ )  $\equiv$  mg/kg; wt.% (weight per cent)  $\equiv$  % (mass fraction).

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

Table 3 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Peroxide Fusion ICP</b>								
Ag	ppm	2.08	Hf	ppm	5.96	Se	ppm	< 20
B	ppm	54	Hg	ppm	< 5	Sn	ppm	7.45
Cd	ppm	0.40	Na	wt. %	0.087	Te	ppm	1.13
Cr	ppm	57	Pb	ppm	< 100	Zr	ppm	165
Eu	ppm	4.22	Re	ppm	0.10			
<b>Infrared Combustion</b>								
C	wt. %	0.762						
<b>Ion Selective Electrode</b>								
Cl	ppm	209						
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt. %	10.15	MnO	wt. %	0.443	Sr	ppm	219
BaO	wt. %	1.95	Na <sub>2</sub> O	wt. %	1.59	TiO <sub>2</sub>	wt. %	0.625
CaO	wt. %	3.44	NiO	ppm	76	V <sub>2</sub> O <sub>5</sub>	ppm	206
Cu	wt. %	1.34	P	wt. %	0.107	Zn	ppm	139
Fe	wt. %	17.34	Pb	ppm	105	ZrO <sub>2</sub>	ppm	329
K <sub>2</sub> O	wt. %	4.07	S	wt. %	2.67			
MgO	wt. %	1.37	SiO <sub>2</sub>	wt. %	45.00			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt. %	3.12						
<b>Laser Ablation ICP-MS</b>								
Ag	ppm	1.95	Hf	ppm	5.55	Sm	ppm	6.85
As	ppm	337	Ho	ppm	0.78	Sn	ppm	7.80
Ba	wt. %	1.58	In	ppm	0.30	Sr	ppm	237
Be	ppm	2.00	La	ppm	169	Ta	ppm	0.99
Bi	ppm	8.43	Lu	ppm	0.28	Tb	ppm	0.78
Cd	ppm	0.30	Mn	wt. %	0.326	Te	ppm	1.10
Ce	ppm	165	Mo	ppm	183	Th	ppm	13.1
Co	ppm	378	Nb	ppm	14.2	Ti	wt. %	0.366
Cr	ppm	24.5	Nd	ppm	42.8	Tl	ppm	< 0.2
Cs	ppm	2.25	Ni	ppm	56	Tm	ppm	0.29
Cu	wt. %	1.36	Pb	ppm	19.0	U	ppm	30.1
Dy	ppm	4.05	Pr	ppm	14.0	V	ppm	109
Er	ppm	2.07	Rb	ppm	110	W	ppm	85
Eu	ppm	2.09	Re	ppm	0.085	Y	ppm	20.8
Ga	ppm	17.0	Sb	ppm	5.90	Yb	ppm	1.89
Gd	ppm	5.31	Sc	ppm	8.30	Zn	ppm	75
Ge	ppm	0.78	Se	ppm	< 5	Zr	ppm	211

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

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

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## INTRODUCTION

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

Table 1 (all laboratories accredited to ISO 17025) and Table 2 (most laboratories accredited to ISO 17025) provide the certified values and their associated 95% expanded uncertainty and tolerance intervals, Table 3 shows indicative values including major and trace element characterisation, Table 4 provides some indicative physical properties. Gold homogeneity (via INAA) is shown in Table 5 and is also demonstrated by a nested ANOVA (see 'Homogeneity Evaluation' section) and Table 6 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<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 525-DataPack.1.0.231121\_172144.xlsx**). Results are also presented in scatter plots for Au by fire assay, Co and Cu by 4-acid digestion (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.

## SOURCE MATERIAL

OREAS 525 was prepared from a blend of iron oxide copper-gold (IOCG) ore, barren waste rock and barren rhyodacite. The IOCG ore and waste rock were sourced from the Ernest Henry Mine located about 38 kilometres north-east of Cloncurry in north-west Queensland. The barren rhyodacite was sourced from a quarry in the Mt Dandenong Igneous Complex located approximately 34km east of Melbourne, Australia.

IOCG mineralisation at the Ernest Henry Mine is hosted in breccia comprising strongly altered and replaced felsic volcanic fragments in a matrix largely composed of magnetite, calcite, pyrite, biotite, chalcopyrite, K feldspar titanite and quartz. Accessory minerals include garnet, barite, molybdenite, fluorite, amphibole, apatite, monazite, arsenopyrite, a LREE fluorocarbonate, galena, cobaltite, sphalerite, scheelite, uraninite and tourmaline. Copper occurs as native copper, bornite and chalcopyrite. Gold occurs mainly in the molecular framework of the chalcopyrite. Significant levels of cobalt, molybdenum, rare earth elements and low levels of uranium are also present.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 525 was prepared in the following manner:

- Drying of ore and barren materials to constant mass at 105° C;
- Crushing and milling of the ore material to 100% minus 30 microns;
- Crushing and milling of the barren materials to >98% minus 75 microns;
- Blending ore and barren materials in appropriate proportions to achieve the desired grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10g and 60g units sealed under nitrogen in laminated foil pouches.

## PHYSICAL PROPERTIES

OREAS 525 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 4 presents these findings that should be used for informational purposes only.

**Table 4. Physical properties of OREAS 525.**

Bulk Density (kg/m <sup>3</sup> )	Moisture (wt.%)	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
681	0.81	N4	Medium Dark 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

Twenty-nine commercial analytical laboratories participated in the program to certify the elements reported in Tables 1 and 2. The following methods were employed:

- Gold by fire assay (25-50g charge weight) with AAS (18 laboratories), ICP-OES (8 laboratories) finish and ICP-MS (1 laboratory) finish;
- Gold by aqua regia digestion (15-30g sample weight) with AAS (4 laboratories) finish, ICP-OES and/or ICP-MS (14 laboratories) finish;
- Full ICP-OES and ICP-MS elemental suites by 4-acid (HNO<sub>3</sub>-HF-HClO<sub>4</sub>-HCl) digestion (up to 26 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by aqua regia digestion (up to 23 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by peroxide fusion (up to 21 laboratories depending on the element);
- Ag, As, Cu, Co, Fe, Mo and S by 3-acid (HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) digestion with ICP-OES finish (10 laboratories);
- Total Sulphur by infrared combustion furnace (25 laboratories) and
- Fluorine by various methods (16 laboratories).

Instrumental neutron activation analysis for Au on 20 x 85mg subsamples was also undertaken at ANSTO, Lucas Heights to confirm homogeneity (see Table 5).

Table 3 shows indicative values including major and trace element characterisation based on two samples analysed at Bureau Veritas in Perth, Western Australia which includes:

- Major oxides by lithium borate fusion with X-ray fluorescence;
- LOI at 1000° C by thermogravimetric analyser;
- Trace elements by laser ablation (on the fused bead) with ICP-MS finish and
- Total Carbon and Sulphur by infrared combustion furnace.

For the round robin program, ten 2.5kg test units (lots) were taken at predetermined intervals during the bagging stage, immediately following homogenisation and are considered representative of the entire prepared batch. The six samples received by each laboratory were obtained by taking a 110g scoop split from six different 2.5kg lots.

The 20 individual INAA results upon which much of the homogeneity evaluation is based, included paired 10g samples taken from each of the 10 different test units. This format enabled a nested ANOVA treatment of the INAA results to evaluate homogeneity (see 'Homogeneity Evaluation' section below).

## STATISTICAL ANALYSIS

**Certified Values and their uncertainty intervals** (Tables 1 and 2) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration).

For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores  $> 2.5$  and with per cent deviations (i)  $> 3$  and (ii) more than three times the average absolute per cent deviation for the batch. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if  $> 2.5$ . After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status. However, while statistics are taken into account, the exercise of a statistician's prerogative plays a significant role in identifying outliers.

**Certified Values** are the means of accepted laboratory means after outlier filtering and are the present best estimate of the true value. The INAA data (see Table 5) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation.

**95% Expanded Uncertainty** provides a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits and is calculated according to the method outlined in ISO 98-3:2008 [6]. All known or suspected sources of bias have been investigated or taken into account.

**Indicative (uncertified) values** (Table 3) are present where the number of laboratories reporting a particular analyte is insufficient ( $< 5$ ) to support certification or where interlaboratory consensus is poor.

### Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Tables 1 and 2 were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for copper by 4-acid digestion, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have

concentrations lying between 1.33 and 1.36 wt.%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35:2017). **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 525. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.22% calculated for a 30g fire assay sample (4.09% at 85mg weights) confirms the high level of gold homogeneity in OREAS 525.

**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	0.827	0.852
2	0.811	0.851
3	0.822	0.852
4	0.840	0.853
5	0.846	0.853
6	0.837	0.852
7	0.864	0.854
8	0.829	0.852
9	0.834	0.852
10	0.868	0.854
11	0.900	0.856
12	0.901	0.856
13	0.867	0.854
14	0.917	0.857
15	0.836	0.852
16	0.808	0.851
17	0.809	0.851
18	0.921	0.857
19	0.860	0.854
20	0.869	0.854
Mean	0.853	0.853
Median	0.843	0.853
Std Dev.	0.035	0.002
<b>Rel.Std.Dev.</b>	<b>4.09%</b>	<b>0.22%</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 gold has been determined by INAA at ANSTO using the reduced analytical subsample method which utilises the known relationship between standard

deviation and analytical subsample weight (Ingamells and Switzer, 1973 [2]). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material and measurement error becomes negligible.

The homogeneity of OREAS 525 has also been evaluated in an Analysis of Variance (**ANOVA**) of the INAA data. The 20 samples were comprised of paired samples from each of 10 sampling lot intervals (representative of the prepared batch) and were randomised prior to assigning sample numbers. The duplicate samples enabled an ANOVA by comparison of within- and between-unit variances across the 10 pairs. The purpose of the ANOVA is to test that no statistically significant difference exists in the variance between units to that of the variance within units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 525. The test was performed using the following parameters:

- Gold INAA – 20 results (1 laboratory providing duplicate analyses on 10 samples where each sample can be viewed as a ‘unit’);
- Null Hypothesis,  $H_0$ : Between-unit variance is no greater than within-unit variance (reject  $H_0$  if  $p$ -value < 0.05);
- Alternative Hypothesis,  $H_1$ : Between-unit variance is greater than within-unit variance.

The data was not filtered for outliers prior to the calculation of the  $p$ -value. This process derived a  $p$ -value of 0.76, a statistically insignificant result so the Null Hypothesis is accepted.

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 525 and whether the variance between two subsamples from the same unit is statistically distinguishable from the variance of two subsamples taken from any two separate units. A reference material therefore can possess poor absolute homogeneity yet still pass a relative homogeneity (ANOVA) test if the within-unit heterogeneity is large and similar across all units.

Based on the statistical analysis of ANOVA and the results of the interlaboratory certification program, it can be concluded that OREAS 525 is fit-for-purpose as a certified reference material (see ‘Intended Use’ below).

## PERFORMANCE GATES

The standard deviations (SD’s) intervals reported in Table 6 provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability.

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

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

Table 6 below shows intervals calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit [www.westgard.com/mltirule.htm](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 [1].*

**Table 6. Performance Gates for OREAS 525.**

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	0.818	0.017	0.784	0.852	0.767	0.869	2.08%	4.15%	6.23%	0.777	0.859
<b>Aqua Regia Digestion (sample weights 10-50g)</b>											
Au, ppm	0.792	0.045	0.702	0.881	0.658	0.926	5.64%	11.29%	16.93%	0.752	0.831
<b>4-Acid Digestion</b>											
Ag, ppm	2.09	0.087	1.92	2.26	1.83	2.35	4.15%	8.31%	12.46%	1.98	2.19
Al, wt. %	5.25	0.159	4.93	5.57	4.78	5.73	3.02%	6.04%	9.07%	4.99	5.51
As, ppm	331	13	304	358	291	371	4.04%	8.09%	12.13%	315	348
Be, ppm	1.66	0.122	1.42	1.91	1.30	2.03	7.35%	14.71%	22.06%	1.58	1.75
Bi, ppm	8.45	0.426	7.60	9.30	7.17	9.73	5.05%	10.09%	15.14%	8.03	8.87
Ca, wt. %	2.41	0.093	2.23	2.60	2.13	2.69	3.85%	7.70%	11.55%	2.29	2.53
Cd, ppm	0.21	0.05	0.10	0.31	0.05	0.36	25.74%	51.47%	77.21%	0.20	0.22
Ce, ppm	154	15	124	184	109	199	9.76%	19.51%	29.27%	147	162
Co, ppm	379	15	349	408	335	423	3.86%	7.72%	11.59%	360	398
Cr, ppm	22.4	1.66	19.1	25.7	17.4	27.4	7.42%	14.84%	22.27%	21.3	23.5
Cs, ppm	2.39	0.122	2.15	2.64	2.03	2.76	5.12%	10.23%	15.35%	2.27	2.51
Cu, wt. %	1.35	0.027	1.29	1.40	1.27	1.43	2.01%	4.03%	6.04%	1.28	1.41
Dy, ppm	4.03	0.264	3.51	4.56	3.24	4.83	6.55%	13.10%	19.65%	3.83	4.24
Er, ppm	1.97	0.114	1.74	2.19	1.62	2.31	5.81%	11.62%	17.43%	1.87	2.06
Eu, ppm	2.16	0.26	1.63	2.69	1.37	2.95	12.22%	24.44%	36.67%	2.05	2.27
Fe, wt. %	16.77	0.636	15.50	18.04	14.86	18.67	3.79%	7.58%	11.37%	15.93	17.61

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

Note 1: intervals may appear asymmetric due to rounding.

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



Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>4-Acid Digestion continued</b>											
Ga, ppm	18.1	0.82	16.4	19.7	15.6	20.5	4.51%	9.03%	13.54%	17.2	19.0
Gd, ppm	5.85	0.309	5.23	6.47	4.92	6.78	5.28%	10.57%	15.85%	5.56	6.14
Ge, ppm	0.25	0.04	0.17	0.33	0.14	0.37	15.45%	30.91%	46.36%	0.24	0.26
Hf, ppm	4.87	0.200	4.47	5.27	4.27	5.47	4.10%	8.20%	12.30%	4.63	5.12
Ho, ppm	0.75	0.034	0.68	0.82	0.65	0.85	4.51%	9.02%	13.53%	0.71	0.79
In, ppm	0.31	0.018	0.27	0.35	0.26	0.37	5.93%	11.85%	17.78%	0.30	0.33
K, wt. %	3.28	0.114	3.06	3.51	2.94	3.62	3.46%	6.93%	10.39%	3.12	3.45
La, ppm	144	17	109	178	92	196	12.05%	24.10%	36.15%	137	151
Li, ppm	16.2	0.80	14.6	17.8	13.8	18.6	4.95%	9.89%	14.84%	15.4	17.0
Lu, ppm	0.29	0.023	0.24	0.33	0.22	0.35	8.16%	16.33%	24.49%	0.27	0.30
Mg, wt. %	0.773	0.032	0.709	0.836	0.678	0.867	4.09%	8.19%	12.28%	0.734	0.811
Mn, wt. %	0.323	0.011	0.300	0.346	0.289	0.357	3.53%	7.06%	10.59%	0.307	0.339
Mo, ppm	190	8	174	206	166	215	4.23%	8.45%	12.68%	181	200
Na, wt. %	1.15	0.032	1.08	1.21	1.05	1.24	2.77%	5.54%	8.31%	1.09	1.20
Nb, ppm	12.1	1.4	9.2	15.0	7.8	16.5	11.94%	23.89%	35.83%	11.5	12.7
Nd, ppm	42.7	2.32	38.1	47.4	35.8	49.7	5.42%	10.84%	16.26%	40.6	44.9
Ni, ppm	54	3.2	48	61	45	64	5.85%	11.70%	17.55%	52	57
P, wt. %	0.105	0.003	0.099	0.112	0.095	0.116	3.21%	6.41%	9.62%	0.100	0.111
Pb, ppm	18.4	2.8	12.7	24.0	9.8	26.9	15.46%	30.93%	46.39%	17.4	19.3
Pr, ppm	13.1	0.79	11.6	14.7	10.8	15.5	6.02%	12.03%	18.05%	12.5	13.8
Rb, ppm	115	5	105	124	100	129	4.30%	8.60%	12.89%	109	120
Re, ppm	0.11	0.008	0.09	0.12	0.08	0.13	7.93%	15.86%	23.79%	0.10	0.11
S, wt. %	2.39	0.077	2.23	2.54	2.16	2.62	3.22%	6.45%	9.67%	2.27	2.51
Sb, ppm	5.59	0.234	5.12	6.05	4.89	6.29	4.18%	8.37%	12.55%	5.31	5.87
Sc, ppm	8.45	0.569	7.31	9.59	6.74	10.16	6.73%	13.46%	20.20%	8.03	8.87
Se, ppm	3.06	0.54	1.99	4.14	1.45	4.67	17.51%	35.02%	52.53%	2.91	3.22
Sm, ppm	6.82	0.412	5.99	7.64	5.58	8.05	6.04%	12.08%	18.12%	6.48	7.16
Sn, ppm	6.97	0.458	6.05	7.88	5.59	8.34	6.58%	13.16%	19.75%	6.62	7.31
Sr, ppm	222	20	182	262	161	283	9.10%	18.21%	27.31%	211	233
Ta, ppm	0.81	0.064	0.68	0.94	0.62	1.00	7.94%	15.88%	23.81%	0.77	0.85
Tb, ppm	0.79	0.075	0.64	0.94	0.56	1.01	9.58%	19.16%	28.75%	0.75	0.82
Te, ppm	1.06	0.103	0.85	1.26	0.75	1.37	9.75%	19.49%	29.24%	1.00	1.11
Th, ppm	12.2	1.2	9.8	14.7	8.5	15.9	10.09%	20.17%	30.26%	11.6	12.8
Ti, wt. %	0.319	0.017	0.285	0.353	0.269	0.370	5.30%	10.60%	15.89%	0.303	0.335
Tl, ppm	0.42	0.024	0.37	0.47	0.35	0.49	5.78%	11.56%	17.34%	0.40	0.44
Tm, ppm	0.27	0.018	0.23	0.31	0.22	0.33	6.77%	13.54%	20.30%	0.26	0.28
U, ppm	30.8	1.76	27.3	34.3	25.5	36.1	5.72%	11.44%	17.16%	29.3	32.3
V, ppm	107	3	101	113	98	117	2.93%	5.87%	8.80%	102	112
W, ppm	85	3.8	77	93	73	97	4.53%	9.06%	13.59%	81	89
Y, ppm	19.5	1.04	17.5	21.6	16.4	22.7	5.30%	10.59%	15.89%	18.6	20.5
Yb, ppm	1.71	0.099	1.51	1.91	1.42	2.01	5.77%	11.54%	17.31%	1.63	1.80
Zn, ppm	68	3.2	62	75	59	78	4.75%	9.51%	14.26%	65	72
Zr, ppm	182	8	166	199	157	207	4.53%	9.05%	13.58%	173	191

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

Note 1: intervals may appear asymmetric due to rounding.

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

Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Aqua Regia Digestion</b>											
Ag, ppm	1.97	0.093	1.79	2.16	1.69	2.25	4.70%	9.40%	14.10%	1.87	2.07
Al, wt. %	1.10	0.054	0.99	1.21	0.94	1.27	4.93%	9.86%	14.79%	1.05	1.16
As, ppm	321	12	296	345	284	358	3.82%	7.63%	11.45%	305	337
Be, ppm	0.69	0.064	0.56	0.82	0.50	0.89	9.28%	18.57%	27.85%	0.66	0.73
Bi, ppm	8.44	0.448	7.55	9.34	7.10	9.79	5.30%	10.61%	15.91%	8.02	8.87
Ca, wt. %	2.15	0.083	1.98	2.31	1.90	2.40	3.86%	7.72%	11.58%	2.04	2.26
Cd, ppm	0.18	0.03	0.12	0.25	0.08	0.29	18.15%	36.29%	54.44%	0.18	0.19
Ce, ppm	133	10	114	152	104	161	7.16%	14.32%	21.47%	126	139
Co, ppm	378	13	352	404	339	417	3.42%	6.84%	10.26%	359	397
Cr, ppm	18.7	1.51	15.7	21.7	14.2	23.2	8.06%	16.12%	24.18%	17.7	19.6
Cs, ppm	0.78	0.073	0.64	0.93	0.56	1.00	9.33%	18.67%	28.00%	0.74	0.82
Cu, wt. %	1.35	0.025	1.30	1.40	1.27	1.43	1.87%	3.74%	5.61%	1.28	1.42
Fe, wt. %	16.07	0.446	15.18	16.96	14.74	17.41	2.77%	5.55%	8.32%	15.27	16.88
Ga, ppm	9.03	0.765	7.50	10.56	6.73	11.32	8.47%	16.94%	25.42%	8.58	9.48
Ge, ppm	0.20	0.03	0.14	0.25	0.12	0.28	13.91%	27.83%	41.74%	0.19	0.21
Hf, ppm	1.73	0.127	1.48	1.99	1.35	2.12	7.33%	14.66%	22.00%	1.65	1.82
In, ppm	0.28	0.015	0.25	0.31	0.24	0.33	5.24%	10.48%	15.72%	0.27	0.30
K, wt. %	0.536	0.028	0.481	0.592	0.453	0.619	5.15%	10.30%	15.46%	0.510	0.563
La, ppm	131	16	99	163	83	179	12.13%	24.25%	36.38%	124	138
Li, ppm	11.2	0.48	10.2	12.1	9.7	12.6	4.33%	8.67%	13.00%	10.6	11.7
Lu, ppm	0.19	0.008	0.17	0.20	0.16	0.21	4.36%	8.71%	13.07%	0.18	0.20
Mg, wt. %	0.703	0.042	0.619	0.786	0.578	0.827	5.92%	11.84%	17.76%	0.668	0.738
Mn, wt. %	0.303	0.017	0.269	0.337	0.252	0.354	5.59%	11.18%	16.77%	0.288	0.318
Mo, ppm	186	9	168	203	160	212	4.68%	9.36%	14.03%	176	195
Na, wt. %	0.090	0.007	0.076	0.104	0.070	0.110	7.56%	15.11%	22.67%	0.085	0.094
Nb, ppm	1.12	0.19	0.73	1.51	0.54	1.70	17.28%	34.56%	51.85%	1.06	1.18
Ni, ppm	52	3.0	46	58	43	61	5.82%	11.64%	17.46%	49	55
P, wt. %	0.098	0.005	0.088	0.108	0.083	0.113	5.19%	10.38%	15.56%	0.093	0.103
Pb, ppm	14.4	1.7	10.9	17.9	9.1	19.6	12.15%	24.30%	36.44%	13.7	15.1
Rb, ppm	30.3	2.20	25.9	34.7	23.7	36.9	7.26%	14.52%	21.78%	28.8	31.8
Re, ppm	0.11	0.004	0.10	0.11	0.09	0.12	4.00%	8.00%	12.00%	0.10	0.11
S, wt. %	2.42	0.095	2.23	2.61	2.14	2.71	3.94%	7.88%	11.83%	2.30	2.54
Sb, ppm	3.57	0.64	2.29	4.85	1.65	5.49	17.90%	35.81%	53.71%	3.39	3.75
Sc, ppm	5.82	0.455	4.91	6.73	4.45	7.18	7.82%	15.63%	23.45%	5.53	6.11
Se, ppm	2.93	0.198	2.54	3.33	2.34	3.52	6.75%	13.50%	20.25%	2.78	3.08
Sn, ppm	5.03	0.247	4.54	5.53	4.29	5.77	4.90%	9.81%	14.71%	4.78	5.29
Sr, ppm	68	12	44	92	32	104	17.78%	35.56%	53.34%	65	71
Ta, ppm	< 0.05	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tb, ppm	0.58	0.035	0.50	0.65	0.47	0.68	6.09%	12.19%	18.28%	0.55	0.60
Te, ppm	1.01	0.10	0.80	1.21	0.69	1.32	10.38%	20.76%	31.15%	0.95	1.06
Th, ppm	8.99	0.94	7.12	10.86	6.19	11.80	10.40%	20.81%	31.21%	8.54	9.44
Ti, wt. %	0.118	0.015	0.089	0.148	0.074	0.163	12.63%	25.26%	37.89%	0.112	0.124
Tl, ppm	0.13	0.011	0.11	0.16	0.10	0.17	8.08%	16.17%	24.25%	0.13	0.14
U, ppm	27.3	1.11	25.1	29.5	23.9	30.6	4.06%	8.12%	12.18%	25.9	28.6

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

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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



Table 6 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Aqua Regia Digestion continued</b>											
V, ppm	97	6.0	85	109	79	115	6.21%	12.42%	18.62%	92	102
W, ppm	67	5.0	57	77	52	82	7.53%	15.06%	22.60%	63	70
Y, ppm	13.9	0.72	12.5	15.3	11.7	16.1	5.19%	10.37%	15.56%	13.2	14.6
Yb, ppm	1.18	0.068	1.05	1.32	0.98	1.39	5.73%	11.46%	17.18%	1.12	1.24
Zn, ppm	47.7	3.24	41.2	54.2	38.0	57.4	6.79%	13.57%	20.36%	45.3	50.1
Zr, ppm	66	2.9	61	72	58	75	4.30%	8.60%	12.90%	63	70
<b>Peroxide Fusion ICP</b>											
Al, wt. %	5.38	0.121	5.13	5.62	5.01	5.74	2.26%	4.52%	6.78%	5.11	5.64
As, ppm	329	34	260	397	226	431	10.42%	20.84%	31.26%	312	345
Ba, wt. %	1.55	0.18	1.20	1.91	1.02	2.09	11.49%	22.98%	34.46%	1.48	1.63
Be, ppm	2.16	0.23	1.71	2.62	1.48	2.85	10.59%	21.18%	31.77%	2.06	2.27
Bi, ppm	8.67	0.457	7.75	9.58	7.29	10.04	5.28%	10.56%	15.83%	8.23	9.10
Ca, wt. %	2.45	0.086	2.28	2.62	2.19	2.71	3.53%	7.06%	10.58%	2.33	2.57
Ce, ppm	175	12	150	200	138	213	7.11%	14.23%	21.34%	167	184
Co, ppm	385	17	351	419	334	436	4.42%	8.84%	13.27%	366	405
Cs, ppm	2.37	0.152	2.07	2.67	1.91	2.83	6.41%	12.81%	19.22%	2.25	2.49
Cu, wt. %	1.35	0.024	1.31	1.40	1.28	1.43	1.79%	3.58%	5.36%	1.29	1.42
Dy, ppm	4.31	0.199	3.91	4.71	3.72	4.91	4.61%	9.23%	13.84%	4.10	4.53
Er, ppm	2.11	0.138	1.84	2.39	1.70	2.53	6.54%	13.08%	19.61%	2.01	2.22
Fe, wt. %	17.18	0.372	16.44	17.93	16.07	18.30	2.17%	4.33%	6.50%	16.32	18.04
Ga, ppm	18.5	0.79	16.9	20.1	16.1	20.9	4.27%	8.54%	12.80%	17.6	19.4
Gd, ppm	5.76	0.500	4.76	6.76	4.26	7.26	8.68%	17.36%	26.04%	5.47	6.05
Ho, ppm	0.82	0.036	0.75	0.89	0.71	0.93	4.39%	8.78%	13.18%	0.78	0.86
In, ppm	0.32	0.06	0.21	0.44	0.15	0.50	17.66%	35.32%	52.97%	0.31	0.34
K, wt. %	3.39	0.095	3.20	3.58	3.10	3.67	2.81%	5.61%	8.42%	3.22	3.56
La, ppm	176	11	155	197	144	208	6.01%	12.03%	18.04%	167	185
Li, ppm	16.6	2.6	11.4	21.8	8.8	24.4	15.64%	31.29%	46.93%	15.8	17.5
Lu, ppm	0.32	0.027	0.27	0.37	0.24	0.40	8.41%	16.82%	25.24%	0.30	0.34
Mg, wt. %	0.801	0.018	0.766	0.836	0.748	0.854	2.20%	4.39%	6.59%	0.761	0.841
Mn, wt. %	0.336	0.009	0.318	0.353	0.309	0.362	2.61%	5.22%	7.82%	0.319	0.352
Mo, ppm	195	18	159	231	141	248	9.15%	18.30%	27.46%	185	205
Nb, ppm	14.9	1.29	12.3	17.5	11.0	18.7	8.69%	17.37%	26.06%	14.1	15.6
Nd, ppm	43.3	1.96	39.4	47.2	37.4	49.2	4.54%	9.08%	13.61%	41.1	45.4
Ni, ppm	58	9	39	77	30	86	16.18%	32.37%	48.55%	55	61
P, wt. %	0.109	0.010	0.089	0.129	0.079	0.139	9.17%	18.33%	27.50%	0.104	0.115
Pr, ppm	13.6	0.43	12.8	14.5	12.4	14.9	3.15%	6.29%	9.44%	13.0	14.3
Rb, ppm	116	6	105	127	99	133	4.82%	9.65%	14.47%	110	122
S, wt. %	2.78	0.098	2.59	2.98	2.49	3.08	3.52%	7.04%	10.55%	2.65	2.92
Sb, ppm	5.97	0.69	4.60	7.35	3.92	8.03	11.48%	22.96%	34.43%	5.68	6.27
Si, wt. %	21.22	0.970	19.28	23.16	18.31	24.13	4.57%	9.14%	13.71%	20.16	22.28
Sm, ppm	6.64	0.307	6.03	7.26	5.72	7.56	4.62%	9.24%	13.86%	6.31	6.97
Sr, ppm	250	14	223	277	210	291	5.43%	10.86%	16.28%	238	263
Ta, ppm	1.08	0.087	0.91	1.26	0.82	1.34	8.06%	16.12%	24.17%	1.03	1.14
Tb, ppm	0.79	0.027	0.74	0.85	0.71	0.87	3.35%	6.71%	10.06%	0.75	0.83

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

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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

**Table 6 continued.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Peroxide Fusion ICP continued</b>											
Th, ppm	13.4	0.39	12.6	14.2	12.2	14.6	2.95%	5.89%	8.84%	12.7	14.1
Ti, wt. %	0.375	0.013	0.348	0.401	0.335	0.414	3.53%	7.07%	10.60%	0.356	0.393
Tl, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Tm, ppm	0.30	0.018	0.27	0.34	0.25	0.36	5.83%	11.67%	17.50%	0.29	0.32
U, ppm	31.0	1.67	27.6	34.3	26.0	36.0	5.39%	10.79%	16.18%	29.4	32.5
V, ppm	113	13	88	139	76	151	11.13%	22.26%	33.39%	108	119
W, ppm	83	6.7	69	96	63	103	8.09%	16.17%	24.26%	79	87
Y, ppm	22.4	0.91	20.6	24.3	19.7	25.2	4.07%	8.13%	12.20%	21.3	23.6
Yb, ppm	1.98	0.100	1.78	2.17	1.68	2.27	5.05%	10.11%	15.16%	1.88	2.07
Zn, ppm	71	9	54	89	45	98	12.48%	24.96%	37.44%	68	75
<b>3-Acid Digestion (no HF)</b>											
Ag, ppm	2.19	0.138	1.91	2.46	1.78	2.60	6.29%	12.58%	18.86%	2.08	2.30
As, ppm	324	14	296	351	282	365	4.26%	8.52%	12.77%	307	340
Co, ppm	370	18	334	407	316	425	4.92%	9.84%	14.76%	352	389
Cu, wt. %	1.33	0.020	1.29	1.37	1.27	1.39	1.51%	3.01%	4.52%	1.26	1.40
Fe, wt. %	16.65	0.667	15.32	17.99	14.65	18.65	4.00%	8.01%	12.01%	15.82	17.49
Mo, ppm	186	7	171	200	163	208	4.04%	8.08%	12.13%	176	195
S, wt. %	2.48	0.057	2.37	2.60	2.31	2.66	2.31%	4.62%	6.94%	2.36	2.61
<b>Infrared Combustion</b>											
S, wt. %	2.75	0.098	2.56	2.95	2.46	3.04	3.56%	7.12%	10.68%	2.61	2.89
<b>Ion Selective Electrode</b>											
F, ppm	2780	245	2290	3270	2045	3515	8.82%	17.63%	26.45%	2641	2919

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

Note 1: intervals may appear asymmetric due to rounding; IND = indeterminate.

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

## PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. Alex Stewart International, Mendoza, Argentina
3. ALS, Johannesburg, South Africa
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Malaga, WA, Australia
7. ALS, Vancouver, BC, Canada
8. ANSTO, Lucas Heights, NSW, Australia
9. ARGETEST Mineral Processing, Ankara, Central Anatolia, Turkey
10. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
11. Bureau Veritas Geoanalytical, Perth, WA, Australia
12. Bureau Veritas Minerals, Ankara, Central Anatolia, Turkey
13. CERTIMIN, Lima, Peru
14. Inspectorate (BV), Lima, Peru
15. Intertek Genalysis, Adelaide, SA, Australia
16. Intertek Genalysis, Perth, WA, Australia
17. Intertek Testing Services Philippines, Cupang, Muntinlupa, Philippines
18. MSALABS, Vancouver, BC, Canada
19. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
20. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
21. SGS, Ankara, Anatolia, Turkey
22. SGS, Randfontein, Gauteng, South Africa
23. SGS Australia Mineral Services, Perth, WA, Australia
24. SGS Canada Inc., Vancouver, BC, Canada
25. SGS de Mexico SA de CV, Cd. Industrial, Durango, Mexico
26. SGS del Peru, Lima, Peru
27. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
28. Skyline Assayers & Laboratories, Tucson, Arizona, USA
29. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

***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 1. Au by Pb Fire Assay in OREAS 525

SPC.1592.RR1.OREAS 525.2.Fire Assay.Au.Lab.231111.143755.SS

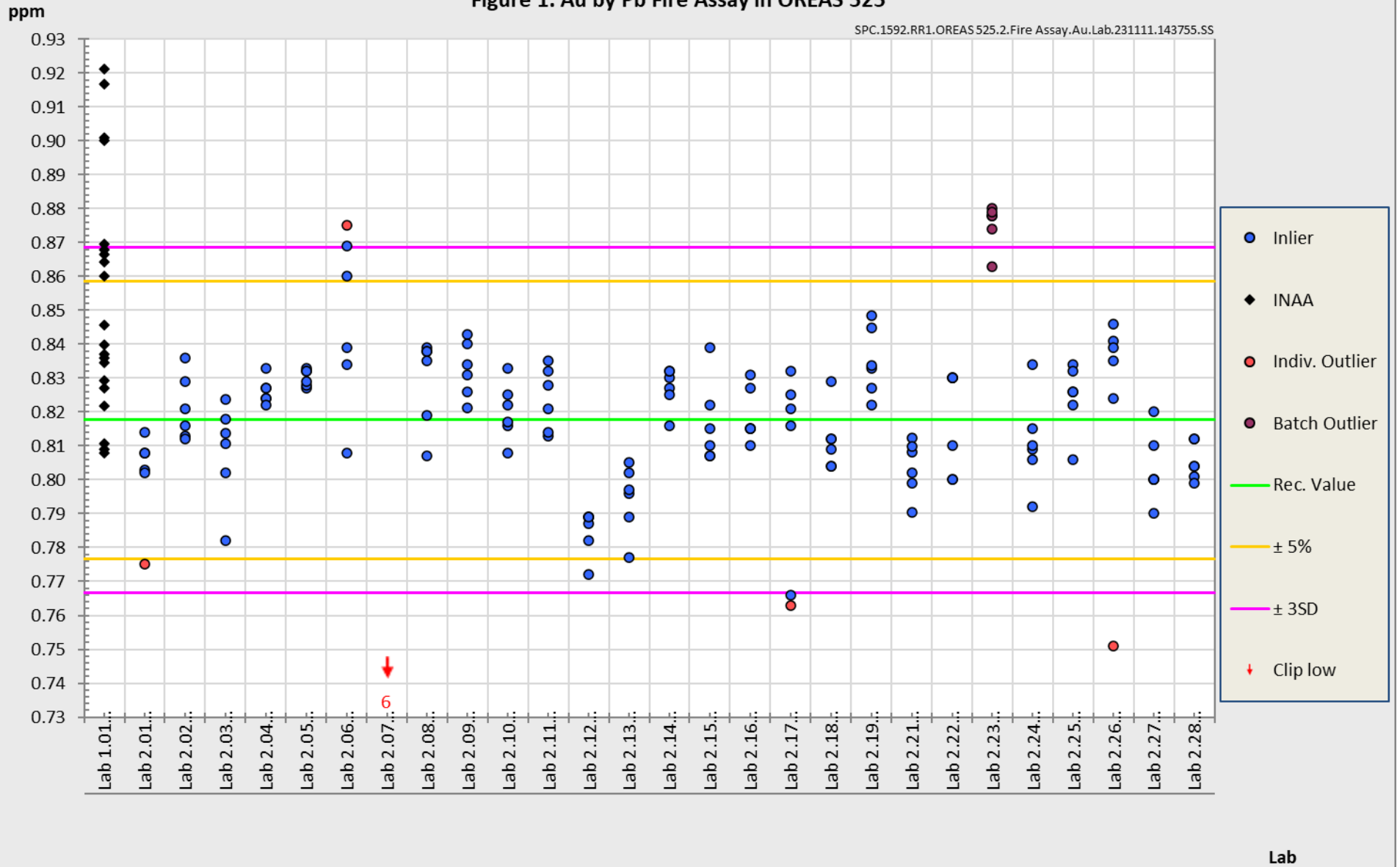


Figure 2. Co by 4-Acid Digestion in OREAS 525

SPC.1592.RR1.OREAS 525.2.4-Acid.Co.Lab.231121.165942.SN

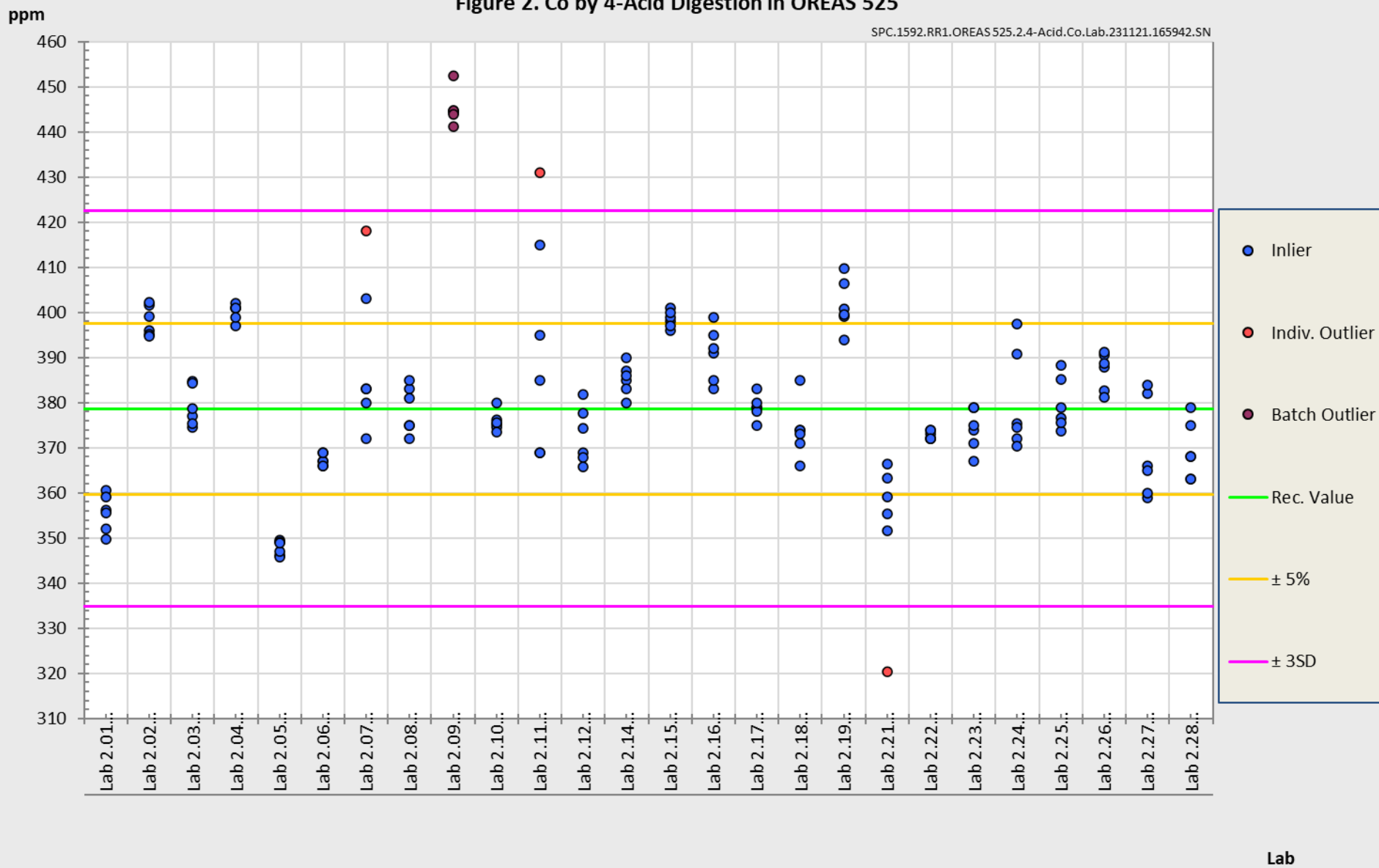
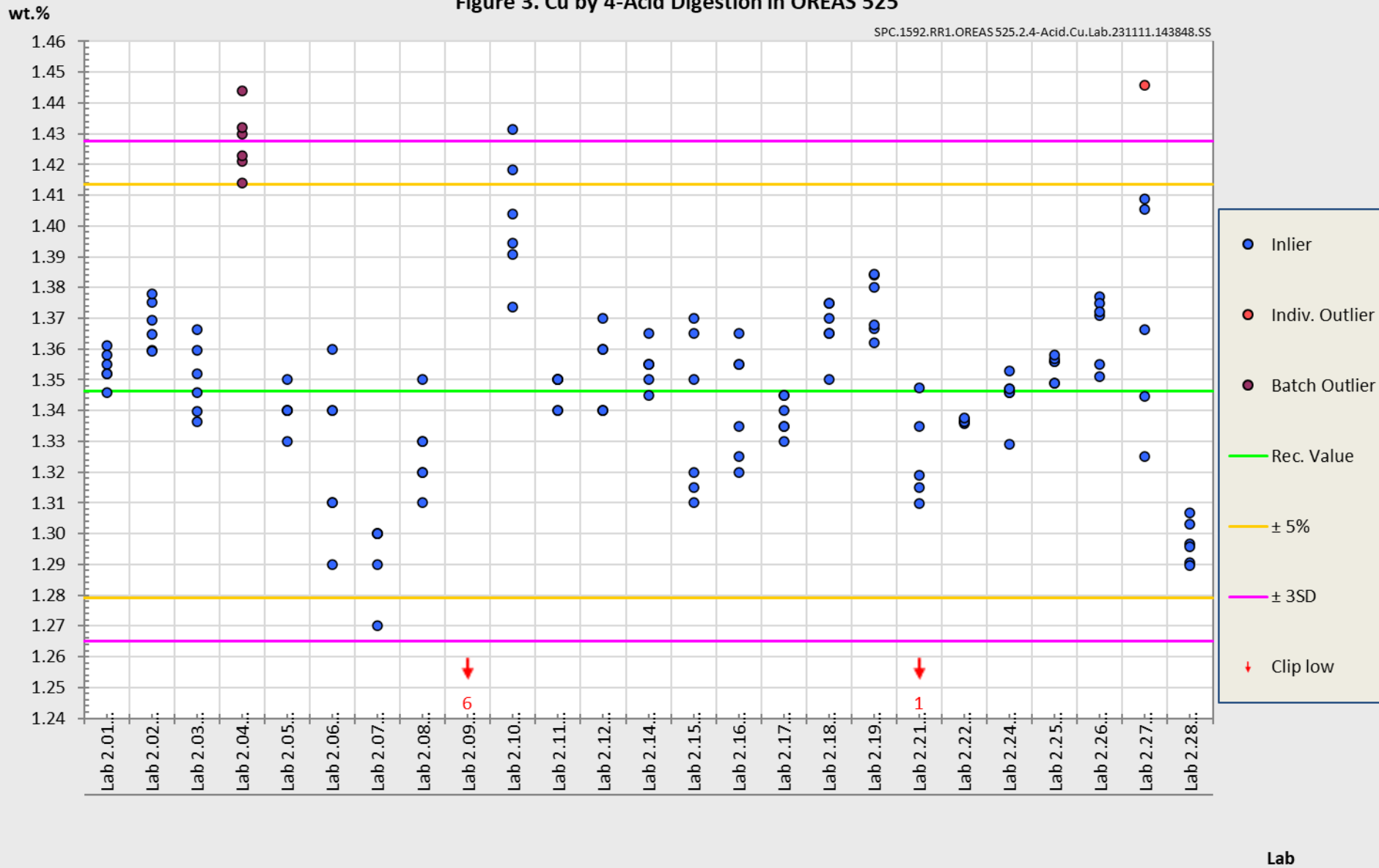


Figure 3. Cu by 4-Acid Digestion in OREAS 525

SPC.1592.RR1.OREAS525.2.4-Acid.Cu.Lab.231111.143848.SS



## PREPARER AND SUPPLIER

Certified reference material OREAS 525 is prepared, certified and supplied by:



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## 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)). 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 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [10], each analytical data set received from the participating laboratories has been validated by its assayer through the inclusion of internal reference materials and QC checks during and post analysis.

The participating laboratories were chosen on the basis of their competence (from past performance in interlaboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite and sample matrix. These laboratories are accredited to ISO 17025 for Au by fire assay (Table 1). The other operationally defined measurands characterised in this certificate (Table 2) are derived from data procured mostly from ISO 17025 accredited laboratories. The certified values presented in this report are calculated from the means of accepted data following robust technical and statistical analysis as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, only a comparison among different laboratories using the same procedure is possible. In this case, it is impossible to demonstrate absence of method bias; therefore, the result is an operationally defined measurand (ISO Guide 35:2017, 9.2.4c)."* Certification takes place on the basis of agreement among operationally defined, independent measurement results.

## COMMUTABILITY

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

## INTENDED USE

OREAS 525 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 525 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 525 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 and 2 in geological samples;
- For the verification of analytical methods for analytes reported in Tables 1 and 2;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 and 2. When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. Users can determine an approximation of the standard uncertainty by calculating one fourth of the width of the Expanded Uncertainty interval given in this certificate (Expanded Uncertainty intervals are provided in Tables 1 and 2).

## MINIMUM SAMPLE SIZE

To relate analytical determinations to the values in this certificate, the minimum mass of sample used should match the typical mass that the laboratories used in the interlaboratory (round robin) certification program. This means that different minimum sample masses should be used depending on the operationally defined methodology as follows:

- Au by fire assay:  $\geq 25\text{g}$ ;
- Au by aqua regia digestion:  $\geq 15\text{g}$ ;
- 4-acid digestion with ICP-OES and/or MS finish:  $\geq 0.25\text{g}$ ;
- Aqua regia digestion with ICP-OES and/or MS finish:  $\geq 0.5\text{g}$ ;
- Peroxide fusion with ICP-OES and/or MS finish:  $\geq 0.1\text{g}$ ;
- 3-acid digestion with ICP-OES finish:  $\geq 0.3\text{g}$ ;
- Total Sulphur by infrared combustion furnace/CS analyser:  $\geq 0.1\text{g}$ ;
- Fluorine by ion selective electrode:  $\geq 0.2\text{g}$ .



## PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 525 remains valid, within the specified measurement uncertainties, until November 2033, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

Store in a clean and cool dry place away from direct sunlight.

Long-term stability will be monitored at appropriate intervals and purchasers notified if any changes are observed. The period of validity may well be indefinite and will be reassessed prior to expiry with the aim of extending the validity if possible.

### **Single-use sachets (e.g., 10g and 60g units)**

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

## INSTRUCTIONS FOR HANDLING & CORRECT USE

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

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

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

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

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

The performance gates shown in Table 6 are intended only to be used as a preliminary guide as to what a laboratory may be able to achieve. Over a period of time monitoring your own laboratory's data for this CRM, SD's should be calculated directly from your own laboratory's process. This will enable you to establish more specific performance gates that are fit for purpose for your application as well as the ability to monitor bias. If your long-term trend analysis shows an average value that is within the 95% expanded uncertainty then generally there is no cause for concern in regard to bias.

## For use with the aqua regia digestion method

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

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

## LEGAL NOTICE

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

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## DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	12 <sup>th</sup> December, 2023	First publication.

## CERTIFYING OFFICER



12<sup>th</sup> December, 2023

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

## QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016.



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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