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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 49

Anomalous Glacial Till

(District of Rainy River in western Ontario, Canada)

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Au by FA in OREAS 49.

Constituent	Certified Value†	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Pb Fire Assay					
Au, Gold (ppb)	61.3	59.4	63.1	60.2*	62.4*

SI unit equivalents: ppb (parts per billion; 1 x 10^{-9}) ≡ µg/kg.

†The operationally defined measurand meets the requirements of ISO 17034 [10] and all participating laboratories comply with the requirements of ISO 17025 [9].

*Gold Tolerance Limits for typical 30g fire assay are determined from 20 x 85 mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973) [2].

Table 2. Certified Value, Uncertainty & Tolerance Intervals for other measurands in OREAS 49.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion (sample weights 10-50g)					
Au, Gold (ppb)	61.6	60.3	62.8	60.3*	62.8*
Borate / Peroxide Fusion ICP					
Ag, Silver (ppm)	< 5	IND	IND	IND	IND
Al, Aluminium (wt.%)	7.95	7.71	8.20	7.81	8.10
As, Arsenic (ppm)	18.2	15.5	20.8	14.3	22.0
Ba, Barium (ppm)	431	414	449	418	445
Be, Beryllium (ppm)	0.89	0.61	1.17	0.73	1.05
Bi, Bismuth (ppm)	0.34	0.23	0.45	IND	IND
Ca, Calcium (wt.%)	3.80	3.65	3.94	3.71	3.88
Ce, Cerium (ppm)	66	62	70	64	68
Co, Cobalt (ppm)	67	63	70	65	68
Cr, Chromium (ppm)	301	279	323	289	313
Cs, Caesium (ppm)	3.12	2.76	3.48	2.86	3.37
Cu, Copper (ppm)	167	158	175	161	173
Dy, Dysprosium (ppm)	2.14	1.96	2.32	2.02	2.25
Er, Erbium (ppm)	1.17	1.04	1.30	1.09	1.25
Eu, Europium (ppm)	1.09	0.99	1.18	1.03	1.15
Fe, Iron (wt.%)	4.43	4.30	4.55	4.33	4.53
Ga, Gallium (ppm)	20.7	19.3	22.1	19.8	21.7
Gd, Gadolinium (ppm)	2.95	2.64	3.27	2.81	3.10
Ge, Germanium (ppm)	1.13	0.91	1.35	0.92	1.34
Ho, Holmium (ppm)	0.40	0.35	0.46	0.38	0.43
In, Indium (ppm)	< 0.3	IND	IND	IND	IND
K, Potassium (wt.%)	1.16	1.12	1.20	1.13	1.19
La, Lanthanum (ppm)	36.5	34.0	38.9	34.6	38.3
Li, Lithium (ppm)	50	47	54	49	52

SI unit equivalents: ppb (parts per billion; 1 x 10^{-9}) ≡ µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: 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).

*Gold Tolerance Limits for typical 25g aqua regia digestion are determined from 20 x 85 mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973) [2].

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Borate / Peroxide Fusion ICP continued					
Lu, Lutetium (ppm)	0.16	0.13	0.19	IND	IND
Mg, Magnesium (wt.%)	1.87	1.83	1.91	1.83	1.91
Mn, Manganese (wt.%)	0.073	0.070	0.075	0.071	0.074
Mo, Molybdenum (ppm)	15.2	12.9	17.5	14.2	16.2
Nb, Niobium (ppm)	7.02	5.94	8.10	6.13	7.91
Nd, Neodymium (ppm)	25.5	23.7	27.3	24.4	26.6
Ni, Nickel (ppm)	119	109	129	113	125
P, Phosphorus (wt.%)	0.056	0.051	0.061	0.055	0.057
Pb, Lead (ppm)	262	245	278	251	272
Pr, Praseodymium (ppm)	7.16	6.75	7.58	6.89	7.44
Rb, Rubidium (ppm)	59	56	62	57	61
Re, Rhenium (ppm)	< 0.01	IND	IND	IND	IND
S, Sulphur (wt.%)	0.110	0.100	0.120	IND	IND
Sb, Antimony (ppm)	0.57	0.44	0.71	IND	IND
Sc, Scandium (ppm)	15.0	13.8	16.1	14.4	15.5
Si, Silicon (wt.%)	29.44	28.16	30.72	28.77	30.11
Sm, Samarium (ppm)	3.96	3.66	4.25	3.70	4.21
Sn, Tin (ppm)	17.1	15.1	19.1	15.4	18.8
Sr, Strontium (ppm)	464	447	481	452	475
Ta, Tantalum (ppm)	1.15	0.92	1.39	0.98	1.33
Tb, Terbium (ppm)	0.39	0.35	0.42	0.36	0.41
Te, Tellurium (ppm)	< 0.5	IND	IND	IND	IND
Th, Thorium (ppm)	3.08	2.83	3.33	2.88	3.28
Ti, Titanium (wt.%)	0.325	0.314	0.336	0.317	0.333
Tl, Thallium (ppm)	0.38	0.31	0.45	0.32	0.44
Tm, Thulium (ppm)	0.16	0.13	0.18	0.13	0.18
U, Uranium (ppm)	0.53	0.45	0.61	0.47	0.58
V, Vanadium (ppm)	115	109	120	111	118
W, Tungsten (ppm)	0.93	0.60	1.27	IND	IND
Y, Yttrium (ppm)	11.0	10.0	11.9	10.6	11.3
Yb, Ytterbium (ppm)	1.03	0.88	1.18	0.97	1.08
Zn, Zinc (ppm)	257	243	272	249	266
Zr, Zirconium (ppm)	95	84	106	89	102
4-Acid Digestion					
Ag, Silver (ppm)	0.422	0.391	0.452	0.396	0.448
Al, Aluminium (wt.%)	7.71	7.41	8.01	7.53	7.89
As, Arsenic (ppm)	16.2	15.3	17.1	15.5	17.0
Ba, Barium (ppm)	419	403	435	405	433

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

Note: intervals may appear asymmetric due to rounding.

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

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Be, Beryllium (ppm)	0.79	0.75	0.83	0.77	0.82
Bi, Bismuth (ppm)	0.31	0.29	0.33	0.29	0.33
Ca, Calcium (wt.%)	3.64	3.54	3.74	3.57	3.71
Cd, Cadmium (ppm)	0.55	0.51	0.59	0.52	0.57
Ce, Cerium (ppm)	64	60	68	62	66
Co, Cobalt (ppm)	66	63	68	65	67
Cr, Chromium (ppm)	209	184	234	200	218
Cs, Caesium (ppm)	2.97	2.82	3.13	2.87	3.08
Cu, Copper (ppm)	165	161	169	161	169
Dy, Dysprosium (ppm)	2.08	1.96	2.20	2.01	2.16
Er, Erbium (ppm)	1.09	1.02	1.16	1.05	1.13
Eu, Europium (ppm)	1.05	0.98	1.12	1.00	1.10
Fe, Iron (wt.%)	4.32	4.21	4.43	4.23	4.41
Ga, Gallium (ppm)	19.5	18.7	20.3	18.9	20.1
Gd, Gadolinium (ppm)	2.87	2.66	3.09	2.77	2.97
Hf, Hafnium (ppm)	1.54	1.42	1.66	1.47	1.61
Ho, Holmium (ppm)	0.39	0.36	0.42	0.37	0.40
In, Indium (ppm)	0.061	0.053	0.069	0.057	0.065
K, Potassium (wt.%)	1.13	1.09	1.16	1.10	1.16
La, Lanthanum (ppm)	32.8	31.0	34.7	31.5	34.2
Li, Lithium (ppm)	49.8	47.9	51.6	48.6	50.9
Lu, Lutetium (ppm)	0.15	0.13	0.16	0.13	0.16
Mg, Magnesium (wt.%)	1.81	1.76	1.86	1.76	1.85
Mn, Manganese (wt.%)	0.070	0.068	0.073	0.069	0.072
Mo, Molybdenum (ppm)	14.4	13.8	15.0	13.9	14.8
Na, Sodium (wt.%)	2.98	2.89	3.06	2.92	3.04
Nb, Niobium (ppm)	6.69	6.26	7.11	6.39	6.98
Nd, Neodymium (ppm)	24.9	23.1	26.6	24.0	25.7
Ni, Nickel (ppm)	114	109	118	111	116
P, Phosphorus (wt.%)	0.056	0.054	0.058	0.055	0.057
Pb, Lead (ppm)	258	249	266	251	264
Pr, Praseodymium (ppm)	6.79	6.35	7.24	6.54	7.05
Rb, Rubidium (ppm)	55	51	60	53	57
S, Sulphur (wt.%)	0.109	0.104	0.113	0.106	0.111
Sb, Antimony (ppm)	0.48	0.45	0.52	0.47	0.50
Sc, Scandium (ppm)	14.9	14.2	15.6	14.5	15.4
Sm, Samarium (ppm)	3.83	3.55	4.12	3.71	3.96
Sn, Tin (ppm)	1.90	1.70	2.11	1.78	2.02
Sr, Strontium (ppm)	459	443	475	447	470

SI unit equivalents: ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).
Note: intervals may appear asymmetric due to rounding.
IND = indeterminate (due to limited reading resolution of the methods employed).

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
4-Acid Digestion continued					
Ta, Tantalum (ppm)	0.97	0.90	1.03	0.91	1.03
Tb, Terbium (ppm)	0.37	0.35	0.39	0.35	0.38
Th, Thorium (ppm)	3.01	2.78	3.24	2.87	3.15
Ti, Titanium (wt.%)	0.316	0.306	0.325	0.307	0.325
Tl, Thallium (ppm)	0.38	0.35	0.40	0.36	0.39
Tm, Thulium (ppm)	0.15	0.14	0.16	0.15	0.16
U, Uranium (ppm)	0.50	0.47	0.53	0.46	0.54
V, Vanadium (ppm)	111	107	115	108	114
W, Tungsten (ppm)	0.69	0.58	0.81	0.65	0.74
Y, Yttrium (ppm)	10.3	9.8	10.9	10.0	10.6
Yb, Ytterbium (ppm)	0.95	0.88	1.03	0.91	0.99
Zn, Zinc (ppm)	251	240	261	245	256
Zr, Zirconium (ppm)	49.4	45.7	53.1	47.8	50.9
Aqua Regia Digestion					
Ag, Silver (ppm)	0.403	0.378	0.428	0.384	0.422
Al, Aluminium (wt.%)	1.36	1.28	1.43	1.31	1.41
As, Arsenic (ppm)	16.0	15.3	16.6	15.5	16.4
B, Boron (ppm)	< 10	IND	IND	IND	IND
Ba, Barium (ppm)	69	66	72	67	71
Be, Beryllium (ppm)	0.14	0.12	0.16	IND	IND
Bi, Bismuth (ppm)	0.26	0.24	0.29	0.25	0.28
Ca, Calcium (wt.%)	0.946	0.866	1.025	0.923	0.968
Cd, Cadmium (ppm)	0.53	0.49	0.56	0.51	0.55
Ce, Cerium (ppm)	54	52	56	52	55
Co, Cobalt (ppm)	59	56	62	57	61
Cr, Chromium (ppm)	41.7	40.0	43.4	40.5	43.0
Cs, Caesium (ppm)	1.88	1.77	1.99	1.82	1.94
Cu, Copper (ppm)	168	164	172	165	171
Dy, Dysprosium (ppm)	1.06	0.86	1.26	0.98	1.15
Eu, Europium (ppm)	0.57	0.47	0.67	0.54	0.61
Fe, Iron (wt.%)	2.13	2.01	2.25	2.08	2.18
Ga, Gallium (ppm)	4.71	4.35	5.06	4.56	4.86
Gd, Gadolinium (ppm)	1.99	1.40	2.58	1.91	2.06
Ge, Germanium (ppm)	0.050	0.035	0.066	0.045	0.056
Hf, Hafnium (ppm)	0.19	0.17	0.21	0.18	0.20
Ho, Holmium (ppm)	0.18	0.16	0.21	IND	IND
In, Indium (ppm)	0.035	0.030	0.040	0.032	0.038
K, Potassium (wt.%)	0.261	0.249	0.273	0.252	0.270

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

Note: intervals may appear asymmetric due to rounding.

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

Table 2 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Aqua Regia Digestion continued					
La, Lanthanum (ppm)	28.8	27.5	30.2	27.8	29.8
Li, Lithium (ppm)	23.1	21.6	24.6	22.3	24.0
Lu, Lutetium (ppm)	0.053	0.047	0.059	0.050	0.055
Mg, Magnesium (wt.%)	0.839	0.803	0.876	0.817	0.862
Mn, Manganese (wt.%)	0.031	0.029	0.032	0.030	0.032
Mo, Molybdenum (ppm)	13.7	13.1	14.3	13.3	14.1
Na, Sodium (wt.%)	0.101	0.094	0.108	0.098	0.104
Nb, Niobium (ppm)	0.28	0.22	0.35	0.26	0.31
Nd, Neodymium (ppm)	19.2	17.3	21.0	18.7	19.6
Ni, Nickel (ppm)	94	90	98	91	96
P, Phosphorus (wt.%)	0.053	0.050	0.055	0.052	0.054
Pb, Lead (ppm)	254	244	264	248	260
Pd, Palladium (ppb)	23.1	19.7	26.5	20.7	25.5
Pr, Praseodymium (ppm)	5.59	5.16	6.02	5.44	5.74
Pt, Platinum (ppb)	36.9	28.5	45.2	31.0	42.7
Rb, Rubidium (ppm)	25.7	24.2	27.3	25.1	26.4
Re, Rhenium (ppm)	0.001	0.001	0.001	IND	IND
S, Sulphur (wt.%)	0.110	0.108	0.113	IND	IND
Sb, Antimony (ppm)	0.35	0.30	0.40	0.33	0.37
Sc, Scandium (ppm)	3.17	2.86	3.48	3.02	3.32
Sm, Samarium (ppm)	2.61	2.43	2.80	2.50	2.73
Sn, Tin (ppm)	0.65	0.59	0.71	0.61	0.69
Sr, Strontium (ppm)	48.3	44.1	52.5	47.4	49.3
Ta, Tantalum (ppm)	< 0.01	IND	IND	IND	IND
Te, Tellurium (ppm)	0.049	0.041	0.058	0.048	0.051
Th, Thorium (ppm)	2.55	2.40	2.69	2.43	2.67
Ti, Titanium (wt.%)	0.124	0.110	0.138	0.120	0.128
Tl, Thallium (ppm)	0.21	0.20	0.23	0.21	0.22
U, Uranium (ppm)	0.26	0.24	0.28	0.25	0.27
V, Vanadium (ppm)	42.8	40.3	45.4	41.3	44.3
W, Tungsten (ppm)	0.28	0.24	0.33	0.25	0.31
Y, Yttrium (ppm)	4.23	3.84	4.63	4.06	4.41
Yb, Ytterbium (ppm)	0.40	0.34	0.46	0.36	0.44
Zn, Zinc (ppm)	239	231	246	234	244
Zr, Zirconium (ppm)	5.06	4.61	5.52	4.78	5.35

SI unit equivalents: ppb (parts per billion; 1×10^{-9}) ≡ µg/kg; ppm (parts per million; 1×10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note: intervals may appear asymmetric due to rounding.

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

Table 3. Indicative Values for OREAS 49.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate / Peroxide Fusion ICP								
B	ppm	*19.0*	Hf	ppm	*2.70*	Se	ppm	*< 3*
Cd	ppm	*0.60*	Na	wt.%	*2.73*			
4-Acid Digestion								
Au	ppm	*0.066*	Hg	ppm	*0.018*	Re	ppm	*0.001*
B	ppm	*7.27*	Pd	ppb	*8.17*	Se	ppm	*0.67*
Ge	ppm	*0.070*	Pt	ppb	*45.7*	Te	ppm	*0.061*
Aqua Regia Digestion								
Er	ppm	*0.46*	Rh	ppm	*0.009*	Tm	ppm	*0.069*
Hg	ppm	*0.011*	Se	ppm	*0.26*			
Ir	ppm	*0.001*	Tb	ppm	*0.20*			
Borate Fusion XRF								
Al_2O_3	wt.%	*14.79*	K_2O	wt.%	*1.36*	SO_3	wt.%	*0.289*
BaO	ppm	*562*	MgO	wt.%	*3.15*	SrO	ppm	*423*
CaO	wt.%	*5.19*	MnO	wt.%	*0.099*	TiO_2	wt.%	*0.522*
Cr_2O_3	ppm	*445*	Na_2O	wt.%	*4.17*	V_2O_5	ppm	*200*
CuO	ppm	*217*	NiO	ppm	*138*	ZnO	ppm	*343*
Fe_2O_3	wt.%	*6.36*	P_2O_5	wt.%	*0.129*	ZrO_2	ppm	*158*
HfO_2	ppm	*17.5*	SiO_2	wt.%	*61.02*			

SI unit equivalents: ppb (parts per billion; 1 x 10^{-9}) ≡ μg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction). Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE’s in-house CRM-specific LIMS.

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INTRODUCTION

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

Table 1 (generated from data supplied by laboratories all accredited to ISO 17025 for Au by fire assay) and Table 2 (generated from data supplied by laboratories mostly 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 and Table 5 provides indicative mineralogy based on semi-quantitative XRD analysis. Gold homogeneity (via INAA) is shown in Table 6 and is also demonstrated by a nested ANOVA (see 'Homogeneity Evaluation' section) and Table 7 presents the performance gate intervals for all certified values.

Tabulated results of all analytes together with uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM[3]) are presented in the detailed certification data for this CRM (**OREAS 49-DataPack.1.0.250505_185655.xlsx**).

SOURCE MATERIAL

Certified Reference Material (CRM) OREAS 49 is sourced from glacial till from the Rainy River District, with minor additions of ore materials (BM, PGE, REE, and Li) to achieve anomalous concentration levels. A pure glacial till is also available (see OREAS 48). This till, predominantly of Late Wisconsinan age, is typically classified as an unsorted diamicton. Its composition reflects both the local Precambrian Shield bedrock including granitoid rocks, metavolcanics, and metasedimentary rocks, and Paleozoic sedimentary formations. The material contains a mix of felsic to mafic lithologies with mineral constituents such as quartz, feldspar, amphibole, and biotite, along with occasional sulphide minerals. The till's combined local and long-range glacial provenance makes it a reliable matrix for geochemical exploration, particularly for QAQC applications targeting anomalous concentrations of gold, PGE, REE, lithium, and base metals.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 49 was prepared in the following manner:

- 72-hour sterilising heat treatment by drying the till at 110 °C;
- Drying of ores to constant mass at 85 °C;
- Multi-stage milling of the till to 98% minus 75 microns;

- Milling of ore materials to 100 % minus 30 microns;
- Preliminary homogenisation and check assaying of source materials;
- Blending the till and ore materials in specific ratios to achieve target grades;
- Homogenisation using OREAS' novel processing technologies;
- Packaging in 10g and 60g units in laminated foil pouches and 1kg units in plastic wide-mouth jars.

PHYSICAL PROPERTIES

OREAS 49 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 49.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
1003	0.15	N7	Light Gray

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

MINERALOGY

The semi-quantitative XRD results shown in Table 5 below were undertaken by ALS Metallurgy in Balcatta, Western Australia. The results have been normalised to 100 % and represent the relative proportion of crystalline material. Totals greater or less than 100 % are due to rounding errors. Clay mineral is mainly vermiculite, illite, and smectite. A trace of Iowaite and/or Zunyite might be present in the samples.

Table 5. Indicative mineralogy of OREAS 49 based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Clay mineral	1
Chlorite	9
Kandite group	< 1
Annite - biotite - phlogopite	6
Muscovite	6
Ca-Na amphibole	25
Clinopyroxene	1
Plagioclase	27
K-feldspar	5
Epidote	2
Quartz	17
Calcite	1
Siderite	< 1
Pyrite	< 1
Magnetite	< 1
Goethite	< 1

ANALYTICAL PROGRAM

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

- Gold by 15-50g fire assay with ICP-OES (10 laboratories), ICP-MS (5 laboratories) and AAS (3 laboratories) finish;
- Instrumental neutron activation analysis for Au on 20 x 85mg subsamples to confirm homogeneity (1 laboratory);
- Gold by aqua regia digestion (10-50g sample weight) with ICP-OES and/or ICP-MS (9 laboratories) finish and AAS (5 laboratories) finish;
- Lithium borate fusion or sodium peroxide fusion for full ICP-OES and ICP-MS elemental suites (up to 17 laboratories depending on the element; Only two laboratories used sodium peroxide fusion);
- Full ICP-OES and ICP-MS elemental suites by 4-acid (HNO_3-HF-$HClO_4$-HCl) digestion (up to 20 laboratories depending on the element);
- Full ICP-OES and ICP-MS elemental suites by aqua regia digestion (up to 18 laboratories depending on the element).

For the round robin program twelve 1.8 kg test units 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 120 g subsample from either the *odd* or *even* sampling (lot) intervals to maximise representation. To confirm homogeneity, gold by instrumental neutron activation analysis (INAA) was undertaken on 20 x 85mg subsamples (see Table 6 in the 'Homogeneity Evaluation' section).

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 6) 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 [6] and [16]. 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. This data is intended for 'informational purposes' only.

Homogeneity Evaluation

The tolerance limits (ISO 16269:2014 [7]) 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 161 and 169 ppm. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99 % of the tolerance intervals so constructed would cover at least 95 % of the total population, and 1% of the tolerance intervals would cover less than 95 % of the total population. ***Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.***

The homogeneity of gold has been determined by INAA at ANSTO, Lucas Heights 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. Table 6 below shows the gold INAA data determined on 20 x 85 mg subsamples of OREAS 49. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 30 g fire assays were undertaken without the normal measurement error associated with this methodology. In this instance, the 1RSD of 0.57 % calculated for a 30 g fire assay sample (10.72 % at 85 mg weights) confirms the high level of gold homogeneity in OREAS 49.

The homogeneity of OREAS 49 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 different 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 49. The test was performed using the following parameters:

- Gold INAA – 20 results (1 laboratory providing duplicate analyses across 10 different sampling lots where each lot can be viewed as a 'unit' in the context of ANOVA);
- 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 Au data by INAA was not filtered for outliers prior to the calculation of the p-value. This process derived a p-value of 0.064, 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 49 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 49 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

Table 6. Neutron Activation Analysis of Au (in ppb) on 20 x 85 mg subsamples and showing the equivalent results scaled to a 30 g sample mass typical of fire assay determination.

Replicate No	Au 85 mg actual	Au 30 g equivalent*
1	58.79	65.382
2	64.81	65.703
3	62.90	65.601
4	57.02	65.287
5	63.92	65.656
6	65.58	65.744
7	62.67	65.589
8	74.88	66.240
9	58.10	65.345
10	71.09	66.038
11	68.81	65.916
12	56.91	65.281
13	72.33	66.104
14	80.96	66.564
15	62.11	65.559
16	63.05	65.609
17	68.35	65.892
18	58.14	65.347
19	65.47	65.738
20	79.17	66.469
Mean	65.753	65.753
Median	64.369	65.679
Std Dev.	7.050	0.376
Rel.Std.Dev.	**10.72%**	**0.57%**

Results calculated for a 30g equivalent sample mass using the formula: $x^{30g\ Eq} = \frac{(x^{INAA}) - RSD@30g}{RSD@85mg} + \bar{X}$

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

PERFORMANCE GATES

The standard deviations (SD's) intervals reported in Table 7 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 7 below shows intervals calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned (also see 'Intended Use' section below). Westgard Rules extend the basics of single-rule QC monitoring using multi-rules (for more information visit www.westgard.com/mltirule.htm). A second method utilises a 5% window calculated directly from the certified value. Standard deviation is also shown in relative percent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. Caution should be exercised when concentration levels approach lower limits of detection of the analytical methods employed as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow. One approach used at commercial laboratories is to set the acceptance criteria at twice the detection level (DL) ±10 %.

i.e., Certified Value ±10 % ±2DL [1].

Table 7. Performance Gates for OREAS 49.

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, ppb	61.3	3.57	54.1	68.4	50.6	72.0	5.83%	11.66%	17.49%	58.2	64.3
Aqua Regia Digestion (sample weights 10-50g)											
Au, ppb	61.6	2.12	57.3	65.8	55.2	67.9	3.44%	6.88%	10.32%	58.5	64.6
Borate / Peroxide Fusion ICP											
Ag, ppm	< 5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Al, wt.%	7.95	0.164	7.63	8.28	7.46	8.45	2.06%	4.13%	6.19%	7.56	8.35
As, ppm	18.2	3.2	11.8	24.5	8.6	27.7	17.55%	35.10%	52.65%	17.2	19.1
Ba, ppm	431	17	398	465	381	482	3.92%	7.83%	11.75%	410	453
Be, ppm	0.89	0.14	0.60	1.18	0.46	1.33	16.24%	32.49%	48.73%	0.85	0.94
Bi, ppm	0.34	0.07	0.21	0.48	0.14	0.54	19.62%	39.24%	58.86%	0.33	0.36
Ca, wt.%	3.80	0.121	3.55	4.04	3.43	4.16	3.18%	6.35%	9.53%	3.61	3.99
Ce, ppm	66	5.5	55	77	50	82	8.26%	16.51%	24.77%	63	69

SI unit equivalents: ppb (parts per billion; 1 x 10-9) ≡ µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 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 7 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate / Peroxide Fusion ICP continued											
Co, ppm	67	3.4	60	74	57	77	5.02%	10.04%	15.05%	63	70
Cr, ppm	301	23	255	347	232	370	7.67%	15.35%	23.02%	286	316
Cs, ppm	3.12	0.34	2.43	3.80	2.09	4.15	11.01%	22.02%	33.03%	2.96	3.27
Cu, ppm	167	7	153	181	146	188	4.23%	8.46%	12.70%	158	175
Dy, ppm	2.14	0.159	1.82	2.46	1.66	2.61	7.46%	14.92%	22.38%	2.03	2.24
Er, ppm	1.17	0.070	1.03	1.31	0.96	1.38	5.96%	11.92%	17.88%	1.11	1.23
Eu, ppm	1.09	0.072	0.94	1.23	0.87	1.30	6.59%	13.18%	19.77%	1.03	1.14
Fe, wt.%	4.43	0.105	4.22	4.64	4.11	4.74	2.37%	4.74%	7.11%	4.21	4.65
Ga, ppm	20.7	1.22	18.3	23.2	17.1	24.4	5.87%	11.73%	17.60%	19.7	21.8
Gd, ppm	2.95	0.203	2.55	3.36	2.35	3.56	6.87%	13.74%	20.60%	2.81	3.10
Ge, ppm	1.13	0.17	0.80	1.47	0.63	1.63	14.77%	29.55%	44.32%	1.08	1.19
Ho, ppm	0.40	0.039	0.33	0.48	0.29	0.52	9.72%	19.43%	29.15%	0.38	0.42
In, ppm	< 0.3	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
K, wt.%	1.16	0.049	1.06	1.26	1.01	1.31	4.23%	8.47%	12.70%	1.10	1.22
La, ppm	36.5	2.98	30.5	42.4	27.5	45.4	8.17%	16.34%	24.51%	34.6	38.3
Li, ppm	50	3.0	44	56	41	59	5.95%	11.91%	17.86%	48	53
Lu, ppm	0.16	0.02	0.11	0.21	0.09	0.23	15.06%	30.13%	45.19%	0.15	0.17
Mg, wt.%	1.87	0.033	1.80	1.94	1.77	1.97	1.79%	3.58%	5.37%	1.78	1.96
Mn, wt.%	0.073	0.003	0.067	0.078	0.065	0.080	3.57%	7.15%	10.72%	0.069	0.076
Mo, ppm	15.2	1.8	11.5	18.9	9.7	20.7	12.01%	24.02%	36.02%	14.4	16.0
Nb, ppm	7.02	0.592	5.84	8.20	5.24	8.79	8.43%	16.87%	25.30%	6.67	7.37
Nd, ppm	25.5	2.04	21.4	29.6	19.4	31.6	8.02%	16.04%	24.07%	24.2	26.8
Ni, ppm	119	12	95	143	83	154	9.97%	19.95%	29.92%	113	125
P, wt.%	0.056	0.004	0.048	0.064	0.044	0.068	7.01%	14.02%	21.03%	0.053	0.059
Pb, ppm	262	15	231	292	216	308	5.86%	11.73%	17.59%	249	275
Pr, ppm	7.16	0.425	6.31	8.01	5.89	8.44	5.93%	11.86%	17.79%	6.81	7.52
Rb, ppm	59	3.1	53	66	50	69	5.29%	10.58%	15.88%	56	62
Re, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
S, wt.%	0.110	0.005	0.100	0.120	0.095	0.125	4.61%	9.23%	13.84%	0.104	0.115
Sb, ppm	0.57	0.09	0.40	0.75	0.31	0.83	15.22%	30.44%	45.67%	0.54	0.60
Sc, ppm	15.0	0.79	13.4	16.5	12.6	17.3	5.26%	10.52%	15.78%	14.2	15.7
Si, wt.%	29.44	1.006	27.43	31.45	26.42	32.46	3.42%	6.83%	10.25%	27.97	30.91
Sm, ppm	3.96	0.302	3.35	4.56	3.05	4.86	7.64%	15.27%	22.91%	3.76	4.15
Sn, ppm	17.1	2.1	12.8	21.3	10.7	23.4	12.41%	24.83%	37.24%	16.2	17.9
Sr, ppm	464	22	419	508	397	530	4.78%	9.57%	14.35%	441	487
Ta, ppm	1.15	0.19	0.77	1.54	0.57	1.74	16.83%	33.66%	50.50%	1.10	1.21
Tb, ppm	0.39	0.020	0.35	0.43	0.33	0.45	5.18%	10.35%	15.53%	0.37	0.41
Te, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Th, ppm	3.08	0.137	2.80	3.35	2.67	3.49	4.44%	8.88%	13.32%	2.92	3.23
Ti, wt.%	0.325	0.008	0.309	0.341	0.301	0.350	2.51%	5.02%	7.53%	0.309	0.341
Tl, ppm	0.38	0.034	0.31	0.45	0.28	0.48	8.95%	17.90%	26.85%	0.36	0.40
Tm, ppm	0.16	0.014	0.13	0.18	0.11	0.20	8.80%	17.60%	26.40%	0.15	0.16
U, ppm	0.53	0.06	0.40	0.65	0.34	0.71	11.76%	23.52%	35.28%	0.50	0.55

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

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

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

Table 7 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Borate / Peroxide Fusion ICP continued											
V, ppm	115	7	101	128	95	134	5.74%	11.47%	17.21%	109	120
W, ppm	0.93	0.21	0.52	1.35	0.31	1.56	22.23%	44.45%	66.68%	0.89	0.98
Y, ppm	11.0	1.2	8.7	13.3	7.5	14.4	10.53%	21.07%	31.60%	10.4	11.5
Yb, ppm	1.03	0.100	0.83	1.23	0.73	1.33	9.75%	19.49%	29.24%	0.98	1.08
Zn, ppm	257	18	222	293	204	311	6.89%	13.78%	20.68%	245	270
Zr, ppm	95	8.3	79	112	71	120	8.72%	17.44%	26.15%	91	100
4-Acid Digestion											
Ag, ppm	0.422	0.036	0.350	0.493	0.315	0.528	8.43%	16.87%	25.30%	0.400	0.443
Al, wt.%	7.71	0.274	7.16	8.26	6.89	8.53	3.55%	7.11%	10.66%	7.33	8.10
As, ppm	16.2	0.63	15.0	17.5	14.3	18.1	3.86%	7.73%	11.59%	15.4	17.0
Ba, ppm	419	19	381	457	362	476	4.53%	9.06%	13.59%	398	440
Be, ppm	0.79	0.040	0.71	0.87	0.67	0.91	5.08%	10.17%	15.25%	0.75	0.83
Bi, ppm	0.31	0.019	0.27	0.35	0.25	0.37	6.24%	12.48%	18.71%	0.30	0.33
Ca, wt.%	3.64	0.121	3.40	3.88	3.28	4.00	3.33%	6.66%	9.99%	3.46	3.82
Cd, ppm	0.55	0.041	0.47	0.63	0.42	0.67	7.51%	15.02%	22.53%	0.52	0.57
Ce, ppm	64	3.0	58	70	55	73	4.68%	9.36%	14.04%	61	67
Co, ppm	66	1.6	63	69	61	71	2.39%	4.78%	7.17%	63	69
Cr, ppm	209	42	125	293	83	335	20.07%	40.15%	60.22%	199	220
Cs, ppm	2.97	0.130	2.71	3.23	2.58	3.36	4.37%	8.73%	13.10%	2.83	3.12
Cu, ppm	165	5	155	175	150	180	3.08%	6.16%	9.24%	157	173
Dy, ppm	2.08	0.142	1.80	2.37	1.66	2.51	6.80%	13.60%	20.39%	1.98	2.19
Er, ppm	1.09	0.072	0.95	1.23	0.87	1.31	6.60%	13.20%	19.79%	1.04	1.15
Eu, ppm	1.05	0.054	0.94	1.16	0.88	1.21	5.20%	10.40%	15.59%	1.00	1.10
Fe, wt.%	4.32	0.107	4.11	4.54	4.00	4.64	2.48%	4.95%	7.43%	4.11	4.54
Ga, ppm	19.5	0.85	17.8	21.2	16.9	22.1	4.38%	8.75%	13.13%	18.5	20.5
Gd, ppm	2.87	0.277	2.32	3.43	2.04	3.70	9.63%	19.26%	28.88%	2.73	3.02
Hf, ppm	1.54	0.082	1.38	1.70	1.30	1.78	5.30%	10.60%	15.90%	1.46	1.62
Ho, ppm	0.39	0.022	0.34	0.43	0.32	0.45	5.72%	11.44%	17.16%	0.37	0.41
In, ppm	0.061	0.005	0.051	0.071	0.046	0.076	8.25%	16.49%	24.74%	0.058	0.064
K, wt.%	1.13	0.022	1.09	1.17	1.06	1.19	1.92%	3.84%	5.77%	1.07	1.19
La, ppm	32.8	1.92	29.0	36.7	27.1	38.6	5.84%	11.68%	17.52%	31.2	34.5
Li, ppm	49.8	2.73	44.3	55.2	41.6	58.0	5.49%	10.98%	16.47%	47.3	52.2
Lu, ppm	0.15	0.011	0.12	0.17	0.11	0.18	7.29%	14.58%	21.86%	0.14	0.15
Mg, wt.%	1.81	0.042	1.73	1.89	1.68	1.93	2.31%	4.62%	6.93%	1.72	1.90
Mn, wt.%	0.070	0.002	0.067	0.074	0.066	0.075	2.33%	4.66%	6.99%	0.067	0.074
Mo, ppm	14.4	0.64	13.1	15.6	12.4	16.3	4.46%	8.91%	13.37%	13.6	15.1
Na, wt.%	2.98	0.083	2.81	3.14	2.73	3.23	2.77%	5.55%	8.32%	2.83	3.13
Nb, ppm	6.69	0.320	6.05	7.32	5.73	7.64	4.78%	9.56%	14.34%	6.35	7.02
Nd, ppm	24.9	1.13	22.6	27.1	21.5	28.3	4.55%	9.10%	13.66%	23.6	26.1
Ni, ppm	114	4	105	122	101	126	3.66%	7.31%	10.97%	108	119
P, wt.%	0.056	0.003	0.050	0.062	0.047	0.065	5.52%	11.04%	16.55%	0.053	0.059
Pb, ppm	258	10	237	279	226	289	4.07%	8.13%	12.20%	245	271
Pr, ppm	6.79	0.456	5.88	7.71	5.42	8.16	6.72%	13.44%	20.15%	6.45	7.13

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

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

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

Table 7 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											
Rb, ppm	55	4.9	46	65	41	70	8.80%	17.60%	26.40%	53	58
S, wt.%	0.109	0.003	0.103	0.115	0.100	0.118	2.77%	5.55%	8.32%	0.103	0.114
Sb, ppm	0.48	0.031	0.42	0.55	0.39	0.58	6.35%	12.70%	19.06%	0.46	0.51
Sc, ppm	14.9	0.47	14.0	15.9	13.5	16.4	3.17%	6.33%	9.50%	14.2	15.7
Sm, ppm	3.83	0.310	3.21	4.45	2.90	4.76	8.08%	16.16%	24.25%	3.64	4.02
Sn, ppm	1.90	0.35	1.21	2.60	0.86	2.95	18.30%	36.60%	54.90%	1.81	2.00
Sr, ppm	459	20	419	499	399	519	4.36%	8.71%	13.07%	436	482
Ta, ppm	0.97	0.055	0.86	1.08	0.80	1.13	5.65%	11.30%	16.95%	0.92	1.01
Tb, ppm	0.37	0.016	0.33	0.40	0.32	0.41	4.25%	8.50%	12.76%	0.35	0.38
Th, ppm	3.01	0.236	2.54	3.48	2.30	3.72	7.84%	15.68%	23.52%	2.86	3.16
Ti, wt.%	0.316	0.010	0.297	0.335	0.287	0.345	3.05%	6.10%	9.15%	0.300	0.332
Tl, ppm	0.38	0.028	0.32	0.43	0.29	0.46	7.59%	15.18%	22.76%	0.36	0.39
Tm, ppm	0.15	0.008	0.14	0.17	0.13	0.18	5.03%	10.07%	15.10%	0.15	0.16
U, ppm	0.50	0.034	0.43	0.57	0.40	0.60	6.81%	13.62%	20.43%	0.47	0.52
V, ppm	111	3	106	116	103	119	2.42%	4.84%	7.25%	105	116
W, ppm	0.69	0.11	0.48	0.91	0.37	1.02	15.41%	30.82%	46.24%	0.66	0.73
Y, ppm	10.3	0.62	9.1	11.6	8.5	12.2	6.02%	12.05%	18.07%	9.8	10.8
Yb, ppm	0.95	0.073	0.81	1.10	0.73	1.17	7.62%	15.25%	22.87%	0.90	1.00
Zn, ppm	251	16	218	283	202	300	6.51%	13.01%	19.52%	238	263
Zr, ppm	49.4	4.30	40.8	58.0	36.5	62.3	8.71%	17.42%	26.13%	46.9	51.8
Aqua Regia Digestion											
Ag, ppm	0.403	0.021	0.360	0.446	0.339	0.467	5.30%	10.61%	15.91%	0.383	0.423
Al, wt.%	1.36	0.112	1.13	1.58	1.02	1.70	8.27%	16.54%	24.81%	1.29	1.43
As, ppm	16.0	0.54	14.9	17.0	14.3	17.6	3.41%	6.82%	10.22%	15.2	16.8
B, ppm	< 10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Ba, ppm	69	4.1	61	77	57	81	5.88%	11.77%	17.65%	65	72
Be, ppm	0.14	0.02	0.11	0.17	0.09	0.19	11.74%	23.47%	35.21%	0.13	0.15
Bi, ppm	0.26	0.018	0.23	0.30	0.21	0.32	6.69%	13.38%	20.07%	0.25	0.28
Ca, wt.%	0.946	0.112	0.722	1.169	0.610	1.281	11.83%	23.66%	35.49%	0.898	0.993
Cd, ppm	0.53	0.025	0.48	0.58	0.46	0.60	4.65%	9.31%	13.96%	0.50	0.56
Ce, ppm	54	3.5	47	61	43	64	6.56%	13.13%	19.69%	51	57
Co, ppm	59	2.0	55	63	53	65	3.44%	6.87%	10.31%	56	62
Cr, ppm	41.7	2.16	37.4	46.0	35.3	48.2	5.17%	10.34%	15.51%	39.6	43.8
Cs, ppm	1.88	0.147	1.59	2.18	1.44	2.32	7.84%	15.68%	23.52%	1.79	1.97
Cu, ppm	168	5	158	179	152	184	3.13%	6.25%	9.38%	160	177
Dy, ppm	1.06	0.15	0.76	1.36	0.62	1.51	14.01%	28.02%	42.02%	1.01	1.12
Eu, ppm	0.57	0.07	0.43	0.71	0.36	0.79	12.54%	25.08%	37.62%	0.54	0.60
Fe, wt.%	2.13	0.176	1.78	2.48	1.61	2.66	8.24%	16.48%	24.72%	2.03	2.24
Ga, ppm	4.71	0.60	3.51	5.90	2.91	6.50	12.74%	25.47%	38.21%	4.47	4.94
Gd, ppm	1.99	0.43	1.12	2.85	0.69	3.29	21.74%	43.48%	65.22%	1.89	2.09
Ge, ppm	0.050	0.011	0.028	0.073	0.017	0.084	22.34%	44.69%	67.03%	0.048	0.053
Hf, ppm	0.19	0.02	0.14	0.24	0.12	0.26	12.22%	24.43%	36.65%	0.18	0.20
Ho, ppm	0.18	0.017	0.15	0.22	0.13	0.23	9.57%	19.14%	28.71%	0.17	0.19

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

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

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

Table 7 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5 % window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Aqua Regia Digestion continued											
In, ppm	0.035	0.004	0.027	0.043	0.024	0.046	10.78%	21.57%	32.35%	0.033	0.037
K, wt.%	0.261	0.013	0.235	0.287	0.222	0.300	4.93%	9.86%	14.79%	0.248	0.274
La, ppm	28.8	0.86	27.1	30.6	26.3	31.4	2.98%	5.95%	8.93%	27.4	30.3
Li, ppm	23.1	1.69	19.7	26.5	18.0	28.2	7.32%	14.63%	21.95%	22.0	24.3
Lu, ppm	0.053	0.004	0.044	0.061	0.039	0.066	8.49%	16.97%	25.46%	0.050	0.055
Mg, wt.%	0.839	0.047	0.746	0.932	0.700	0.979	5.54%	11.08%	16.62%	0.797	0.881
Mn, wt.%	0.031	0.002	0.027	0.035	0.025	0.037	6.21%	12.41%	18.62%	0.029	0.032
Mo, ppm	13.7	0.66	12.4	15.0	11.7	15.7	4.84%	9.69%	14.53%	13.0	14.4
Na, wt.%	0.101	0.006	0.090	0.112	0.084	0.118	5.54%	11.08%	16.61%	0.096	0.106
Nb, ppm	0.28	0.08	0.13	0.44	0.05	0.51	27.33%	54.67%	82.00%	0.27	0.30
Nd, ppm	19.2	1.58	16.0	22.3	14.4	23.9	8.25%	16.50%	24.75%	18.2	20.1
Ni, ppm	94	4.9	84	104	79	108	5.26%	10.52%	15.78%	89	98
P, wt.%	0.053	0.003	0.047	0.059	0.044	0.062	5.78%	11.57%	17.35%	0.050	0.055
Pb, ppm	254	10	233	274	223	285	4.06%	8.12%	12.17%	241	266
Pd, ppb	23.1	2.1	18.9	27.3	16.8	29.5	9.17%	18.34%	27.51%	22.0	24.3
Pr, ppm	5.59	0.310	4.97	6.21	4.66	6.52	5.55%	11.10%	16.65%	5.31	5.87
Pt, ppb	36.9	4.5	28.0	45.8	23.5	50.3	12.10%	24.21%	36.31%	35.0	38.7
Rb, ppm	25.7	2.00	21.7	29.7	19.8	31.7	7.76%	15.51%	23.27%	24.5	27.0
Re, ppm	0.001	0.000	0.000	0.001	0.000	0.001	22.18%	44.36%	66.54%	0.001	0.001
S, wt.%	0.110	0.003	0.104	0.117	0.100	0.120	3.01%	6.02%	9.03%	0.105	0.116
Sb, ppm	0.35	0.04	0.27	0.44	0.23	0.48	11.88%	23.75%	35.63%	0.33	0.37
Sc, ppm	3.17	0.41	2.35	3.99	1.94	4.40	12.93%	25.86%	38.80%	3.01	3.33
Sm, ppm	2.61	0.216	2.18	3.05	1.96	3.26	8.28%	16.57%	24.85%	2.48	2.74
Sn, ppm	0.65	0.07	0.52	0.78	0.45	0.85	10.29%	20.57%	30.86%	0.62	0.68
Sr, ppm	48.3	5.9	36.6	60.1	30.7	65.9	12.16%	24.31%	36.47%	45.9	50.7
Ta, ppm	< 0.01	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Te, ppm	0.049	0.008	0.034	0.065	0.027	0.072	15.23%	30.46%	45.70%	0.047	0.052
Th, ppm	2.55	0.158	2.23	2.86	2.07	3.02	6.21%	12.41%	18.62%	2.42	2.68
Ti, wt.%	0.124	0.022	0.080	0.168	0.058	0.191	17.85%	35.70%	53.55%	0.118	0.130
Tl, ppm	0.21	0.02	0.17	0.26	0.14	0.28	10.87%	21.75%	32.62%	0.20	0.23
U, ppm	0.26	0.021	0.22	0.30	0.20	0.32	8.13%	16.26%	24.38%	0.25	0.27
V, ppm	42.8	2.60	37.6	48.0	35.0	50.6	6.07%	12.14%	18.20%	40.7	45.0
W, ppm	0.28	0.03	0.22	0.35	0.19	0.38	11.56%	23.11%	34.67%	0.27	0.30
Y, ppm	4.23	0.62	2.99	5.47	2.37	6.09	14.64%	29.28%	43.92%	4.02	4.45
Yb, ppm	0.40	0.06	0.29	0.52	0.23	0.58	14.48%	28.97%	43.45%	0.38	0.42
Zn, ppm	239	8	222	256	213	264	3.54%	7.08%	10.62%	227	251
Zr, ppm	5.06	0.71	3.65	6.48	2.94	7.19	14.00%	27.99%	41.99%	4.81	5.32

SI unit equivalents: ppb (parts per billion; 1 x 10-9) ≡ µg/kg; ppm (parts per million; 1 x 10^{-6}) ≡ mg/kg; wt.% (weight per cent) ≡ % (mass fraction).

Note 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. AGAT Laboratories, Calgary, Alberta, Canada
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru
5. ALS, Loughrea, Galway, Ireland
6. ALS, Malaga, WA, Australia
7. ALS, Vancouver, BC, Canada
8. American Assay Laboratories, Sparks, Nevada, USA
9. ANSTO, Lucas Heights, NSW, Australia
10. ESAN Istanbul, Istanbul, Turkey
11. Intertek, Perth, WA, Australia
12. MSALABS, Vancouver, BC, Canada
13. Ontario Geological Survey, Sudbury, Ontario, Canada
14. Paragon Geochemical Laboratories, Sparks, Nevada, USA
15. PT Geoservices Ltd, Cikarang, Jakarta Raya, Indonesia
16. PT Intertek Utama Services, Jakarta Timur, DKI Jakarta, Indonesia
17. Reminex Centre de Recherche, Marrakesh, Marrakesh-Safi, Morocco
18. SGS Canada Inc., Vancouver, BC, Canada
19. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
20. Shiva Analyticals Ltd, Bangalore North, Karnataka, India
21. Stewart Assay & Environmental Laboratories LLC, Kara-Balta, Chüy, Kyrgyzstan

PREPARER AND SUPPLIER

Certified reference material OREAS 49 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)) [15]. 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) or parts per billion (ppb).

The analytical samples sent to participating laboratories were selected in a manner to be representative of the entire prepared batch of CRM. This representativeness was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the analytical results. The systematic sampling method was chosen due to the low risk of overlooking repetitive effects or trends in the batch due to the way the CRM was processed. In line with ISO 17025 [9], 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 [8], 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 33405:2024-05, 9.2.4c) [5]."* 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 49 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 49 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 49 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/ validation 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:

- Gold by fire assay: ≥ 25 g;
- Gold by aqua regia digestion: ≥10 g;
- Borate/Peroxide fusion with ICP-OES and/or MS finish: ≥0.1g;
- Multi-elements by 4-acid digestion with ICP-OES and/or MS finish: ≥ 0.25 g;
- Multi-elements by aqua regia digestion with ICP-OES and/or MS finish: ≥ 0.5 g.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

The certification of OREAS 49 remains valid, within the specified measurement uncertainties, until at least June 2039, provided the CRM is handled and stored in accordance with the instructions given below. This certification is nullified if the CRM is any way changed or contaminated.

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

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

Single-use sachets

OREAS 49 is packaged in single-use laminated foil sachets. Following analysis, it is the manufacturer's expectation that any remaining material is discarded. It is the user's responsibility to prevent contamination and avoid prolonged exposure of the sample to the atmosphere prior to analysis.

Repeat-use packaging (e.g., 1 kg plastic jars)

After taking a subsample, users should replace the lid of the jar promptly and securely to prevent accidental spills and airborne contamination. OREAS 49 contains a non-hygroscopic* matrix with an indicative value for moisture provided to enable users to check for changes to stored material by determining moisture in the user's laboratory and comparing the result to the value in Table 4 in this certificate.

The stability of the CRM in regard to oxidation from the breakdown of sulphide minerals to sulphates is negligible given its low sulphur concentration (~0.1 wt.% S).

*A non-hygroscopic matrix means exposure to atmospheres significantly different, in terms of temperature and humidity, from the climate during manufacturing should have negligible impact on the precision of results. Hygroscopic moisture is the amount of adsorped moisture (weakly held H_2O- molecules on the surface of exposed material) following exposure to the local atmosphere. Usually, equilibration of material to the local atmosphere will only occur if the material is spread into a thin (~2mm thick) layer and left exposed for a period of 2 hours.

INSTRUCTIONS FOR HANDLING & CORRECT USE

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

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

QC monitoring using multiples of the Standard Deviation (SD)

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

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

The performance gates shown in Table 7 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, particularly 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.

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034:2016 (Accreditation number 20483).

ORE Pty Ltd is ISO 9001:2015 certified by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	27th May, 2025	First publication.

CERTIFYING OFFICER

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

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