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CERTIFICATE OF ANALYSIS FOR

CERTIFIED REFERENCE MATERIAL

OREAS 996

Copper Sulphide Concentrate (various sources)

Table 1. Certified Value, Uncertainty & Tolerance Intervals for Cu by titration in OREAS 996.

Constituent	Certified Value†	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Umpire Labs (dry sample basis)					
Classical Wet Chemistry					
Cu, Copper (wt.%)	29.27	29.23	29.31	29.24	29.30

SI unit equivalents: wt.% (weight per cent) ≡ % (mass fraction).

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

Note: intervals may appear asymmetric due to rounding.

Table 2. Certified Values, Uncertainty & Tolerance Intervals for Au by fire assay, multi-elements by 4-acid digestion and S by infrared combustion in OREAS 996.

Constituent	Certified Value†	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Geoanalytical Labs ('as received' sample basis)					
Pb Fire Assay					
Au, Gold (ppm)	9.54	9.39	9.69	9.46*	9.63*
4-Acid Digestion					
Ag, Silver (ppm)	145	141	148	143	146
Al, Aluminium (wt.%)	0.983	0.953	1.013	0.963	1.003
As, Arsenic (ppm)	926	880	973	907	946
Ba, Barium (ppm)	61	48	74	58	64
Be, Beryllium (ppm)	< 0.5	IND	IND	IND	IND
Bi, Bismuth (ppm)	307	286	329	299	316
Ca, Calcium (wt.%)	0.732	0.688	0.776	0.710	0.754
Cd, Cadmium (ppm)	48.7	45.1	52.3	47.5	49.9
Ce, Cerium (ppm)	61	49	73	59	63
Co, Cobalt (ppm)	208	200	215	204	212
Cr, Chromium (ppm)	29.7	27.3	32.1	28.6	30.8
Cs, Caesium (ppm)	1.56	1.43	1.68	1.50	1.61
Cu, Copper (wt.%)	29.61	29.05	30.17	29.24	29.99
Dy, Dysprosium (ppm)	1.12	0.80	1.45	IND	IND
Er, Erbium (ppm)	0.56	0.47	0.65	IND	IND
Eu, Europium (ppm)	0.52	0.42	0.62	IND	IND
Fe, Iron (wt.%)	22.46	21.93	22.98	22.18	22.73
Ga, Gallium (ppm)	4.04	3.71	4.37	3.84	4.24
Gd, Gadolinium (ppm)	1.90	1.56	2.25	IND	IND
Hf, Hafnium (ppm)	0.29	0.20	0.38	0.25	0.32
Ho, Holmium (ppm)	0.20	0.19	0.21	IND	IND
In, Indium (ppm)	13.5	12.8	14.2	13.1	13.9
K, Potassium (wt.%)	0.288	0.274	0.303	0.279	0.298
Li, Lithium (ppm)	7.66	6.78	8.55	7.29	8.04
Mg, Magnesium (wt.%)	0.269	0.251	0.288	0.259	0.280
Mn, Manganese (wt.%)	0.025	0.024	0.026	0.025	0.026
Mo, Molybdenum (wt.%)	0.191	0.181	0.202	0.186	0.197
Na, Sodium (wt.%)	0.126	0.117	0.135	0.121	0.132
Nb, Niobium (ppm)	1.36	1.21	1.52	1.32	1.41
Nd, Neodymium (ppm)	18.7	15.9	21.5	17.7	19.7

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

*Gold Tolerance Limits for typical 30g fire assay methods are determined from 20 x 1 g INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note: intervals may appear asymmetric due to rounding.

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

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
Geoanalytical Labs ('as received' sample basis)					
4-Acid Digestion continued					
Ni, Nickel (ppm)	182	170	195	176	189
P, Phosphorus (wt.%)	0.025	0.024	0.027	0.024	0.026
Pb, Lead (wt.%)	0.136	0.132	0.141	0.134	0.138
Pr, Praseodymium (ppm)	5.74	4.90	6.58	5.45	6.03
Rb, Rubidium (ppm)	13.6	13.0	14.1	13.1	14.1
Re, Rhenium (ppm)	2.97	2.77	3.17	2.83	3.11
S, Sulphur (wt.%)	25.44	24.31	26.57	25.01	25.87
Sb, Antimony (ppm)	589	525	652	565	612
Sc, Scandium (ppm)	1.26	1.15	1.38	IND	IND
Se, Selenium (ppm)	168	152	185	162	175
Sm, Samarium (ppm)	2.63	2.10	3.16	2.44	2.82
Sn, Tin (ppm)	42.8	40.1	45.5	41.0	44.6
Sr, Strontium (ppm)	72	69	75	70	74
Ta, Tantalum (ppm)	0.093	0.086	0.100	IND	IND
Te, Tellurium (ppm)	21.4	18.4	24.5	20.8	22.1
Th, Thorium (ppm)	2.54	2.39	2.70	2.42	2.67
Ti, Titanium (wt.%)	0.048	0.045	0.052	0.046	0.050
Tl, Thallium (ppm)	3.41	3.16	3.65	3.27	3.54
U, Uranium (ppm)	4.31	4.07	4.55	4.16	4.45
V, Vanadium (ppm)	18.1	16.1	20.1	16.9	19.4
W, Tungsten (ppm)	23.8	22.4	25.1	23.1	24.4
Y, Yttrium (ppm)	5.57	5.01	6.12	5.29	5.84
Yb, Ytterbium (ppm)	0.48	0.37	0.60	IND	IND
Zn, Zinc (wt.%)	1.17	1.12	1.21	1.13	1.20
Zr, Zirconium (ppm)	8.75	7.48	10.01	8.33	9.16
Infrared Combustion					
S, Sulphur (wt.%)	27.66	27.07	28.25	27.40	27.92

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

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

Note: intervals may appear asymmetric due to rounding;

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

Table 3. Certified Values, Uncertainty & Tolerance Intervals for other measurands in OREAS 996.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Geoanalytical Labs ('as received' sample basis)					
Peroxide Fusion ICP					
Al, Aluminium (wt.%)	1.02	1.00	1.05	1.00	1.04
As, Arsenic (ppm)	996	933	1059	983	1008
Ba, Barium (ppm)	66	62	71	64	69
Be, Beryllium (ppm)	< 1	IND	IND	IND	IND
Bi, Bismuth (ppm)	323	290	357	314	333
Ca, Calcium (wt.%)	0.753	0.684	0.822	0.721	0.784
Cd, Cadmium (ppm)	53	45	61	51	56
Ce, Cerium (ppm)	73	64	82	71	75
Co, Cobalt (ppm)	202	186	218	196	208
Cs, Caesium (ppm)	1.56	1.17	1.95	IND	IND
Cu, Copper (wt.%)	29.39	28.66	30.12	29.11	29.67
Dy, Dysprosium (ppm)	1.42	1.21	1.63	IND	IND
Er, Erbium (ppm)	0.83	0.64	1.01	IND	IND
Eu, Europium (ppm)	0.53	0.40	0.66	IND	IND
Fe, Iron (wt.%)	23.23	22.66	23.81	22.95	23.52
Ga, Gallium (ppm)	4.17	3.00	5.35	IND	IND
Gd, Gadolinium (ppm)	1.98	1.70	2.25	IND	IND
Ho, Holmium (ppm)	0.27	0.23	0.31	IND	IND
In, Indium (ppm)	13.8	12.1	15.5	13.2	14.4
K, Potassium (wt.%)	0.296	0.269	0.323	0.279	0.313
La, Lanthanum (ppm)	44.5	42.0	46.9	43.2	45.7
Li, Lithium (ppm)	8.18	5.79	10.56	7.67	8.68
Mg, Magnesium (wt.%)	0.286	0.266	0.306	0.275	0.297
Mn, Manganese (wt.%)	0.026	0.025	0.028	0.026	0.027
Mo, Molybdenum (wt.%)	0.209	0.200	0.217	0.204	0.213
Nd, Neodymium (ppm)	23.7	21.3	26.0	22.2	25.1
Ni, Nickel (ppm)	190	169	212	179	202
Pb, Lead (wt.%)	0.141	0.131	0.150	0.138	0.143
Pr, Praseodymium (ppm)	7.15	6.04	8.26	6.74	7.56
Rb, Rubidium (ppm)	14.6	13.5	15.8	13.9	15.4
S, Sulphur (wt.%)	27.62	26.98	28.26	27.41	27.83
Sb, Antimony (ppm)	614	571	658	590	638
Si, Silicon (wt.%)	3.79	3.62	3.96	3.68	3.89
Sm, Samarium (ppm)	2.97	2.56	3.39	2.59	3.36
Sn, Tin (ppm)	44.3	37.1	51.6	IND	IND
Sr, Strontium (ppm)	78	73	83	76	80

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 3 continued.

Constituent	Certified Value	95 % Expanded Uncertainty		95 % Tolerance Limits	
		Low	High	Low	High
Geoanalytical Labs ('as received' sample basis)					
Peroxide Fusion ICP continued					
Th, Thorium (ppm)	2.51	2.22	2.80	2.33	2.69
Ti, Titanium (wt.%)	0.070	0.067	0.074	IND	IND
Tl, Thallium (ppm)	3.59	3.34	3.84	3.34	3.85
U, Uranium (ppm)	4.26	3.86	4.65	3.99	4.52
V, Vanadium (ppm)	22.3	18.8	25.8	19.8	24.7
W, Tungsten (ppm)	26.6	21.8	31.4	23.4	29.8
Y, Yttrium (ppm)	7.13	5.77	8.50	6.67	7.60
Yb, Ytterbium (ppm)	0.64	0.46	0.83	IND	IND
Zn, Zinc (wt.%)	1.18	1.13	1.23	1.15	1.20
Ion Selective Electrode					
F, Fluorine (ppm)	157	132	182	146	168

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 4. Indicative Values for OREAS 996.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Umpire Labs ('as received' sample basis)								
Thermogravimetry								
H_2O-	wt.%	*0.499*						
Geoanalytical Labs ('as received' sample basis)								
4-Acid Digestion								
B	ppm	*60*	La	ppm	*36.8*	Tm	ppm	*0.071*
Ge	ppm	*0.73*	Lu	ppm	*0.066*			
Hg	ppm	*0.78*	Tb	ppm	*0.24*			
Infrared Combustion								
C	wt.%	*0.150*						
Peroxide Fusion ICP								
Ag	ppm	*150*	Lu	ppm	*0.14*	Ta	ppm	*0.25*
B	ppm	*< 50*	Na	wt.%	*0.151*	Tb	ppm	*0.27*
Cr	ppm	*48.2*	Nb	ppm	*3.15*	Te	ppm	*22.1*
Ge	ppm	*3.64*	P	wt.%	*0.028*	Tm	ppm	*0.11*
Hf	ppm	*0.80*	Re	ppm	*3.05*	Zr	ppm	*30.5*
Hg	ppm	*< 5*	Sc	ppm	*3.98*			
LOI^{1000}	wt.%	*17.52*	Se	ppm	*179*			

SI unit equivalents: 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

Reference materials are intended to provide a method of evaluating and improving the quality of analysis of geological and downstream metallurgical samples. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. OREAS prepared 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.

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 996-DataPack.1.0.241219_124733.xlsx**).

Results are also presented in scatter plots for Cu by classical wet chemistry, Au by fire assay and Mo by 4-acid digestion method in Figures 1 to 3 respectively, together with ±3SD (magenta) 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 996 was designed to replace OREAS 994 and was prepared from a blend of copper concentrates sourced predominantly from Chilean, Philippine and Australian mine site metallurgical plants. Copper, Iron and Sulphur by mass account for approx. 77 % of the total chemical composition of OREAS 996.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 996 was prepared in the following manner:

- Drying to constant mass at 85 °C;
- Multi-stage milling to 100 % minus 30 µm;
- Homogenisation using OREAS' novel processing technologies;
- Packaging into 10 g units sealed under nitrogen in laminated foil pouches.

PHYSICAL PROPERTIES

OREAS 996 was tested at ORE Research & Exploration Pty Ltd's onsite facility for various physical properties. Table 5 presents these findings that should be used for informational purposes only.

Table 5. Physical properties of OREAS 996.

Bulk Density (kg/m^3)	Moisture (wt.%)	Munsell Notation‡	Munsell Color‡
1042	0.53	5Y 2/1	Olive Black

‡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 6 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.

The most representative minerals in the sample are chalcopyrite, followed by pyrite, quartz, covellite, antlerite, magnetite, K-feldspar and plagioclase.

A presence of some amorphous material is very likely. A trace amount of apatite and calcite might be present in the sample.

Table 6. Indicative mineralogy of OREAS 996 based on semi-quantitative XRD analysis.

Mineral / Mineral Group	% (mass ratio)
Pyrite	29
Chalcopyrite	47
Covellite	4
Digenite and/or chalcocite	1
Molybdenite	< 1
Magnetite	2
Stilpnomelane and/or Sepiolite	1
Chlorite	1
Annite - biotite - phlogopite	1
Muscovite	1
Ca amphibole	1
Fe-Mg amphibole	< 1
Plagioclase	1
K-feldspar and/or rutile	1
Quartz	5
Gypsum	< 1
Jarosite	1
Antlerite	2
Posnjakite	1
Chalcanthite	< 1

ANALYTICAL PROGRAM

For the interlaboratory ‘round robin’ certification program, a 400 g sample was taken at each of 12 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire prepared batch of OREAS 996.

Umpire Laboratories

Seventeen 'umpire' laboratories each received a single 10 g sample and undertook copper and moisture analysis on the sample as received. The term 'umpire' here refers to the routine analysis by these laboratories using classical methodologies for precious and base metals.

Strict, pre-assay instructions were provided to ensure proper handling of moisture including:

- Equilibration of sample material to laboratory atmosphere for a minimum of 2 hours;
- Hygroscopic moisture analysis at 105 °C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Cu as per ISO 9599.

The laboratories were requested to report analyte concentrations on both a dry (using the moisture value to correct the sample to dry basis) and moisture-bearing basis and include all results for moisture determinations. **The 'Umpire Lab' certified values shown in Table 1 are on a dry sample basis (see 'Instructions for correct use' section).**

The following analytical methods were undertaken:

- Copper (3 trials on undried sample) by classical wet chemistry (short iodide titration).

Geoanalytical Laboratories

Nineteen geoanalytical laboratories also participated in the program where each laboratory received 6 x 35 g samples taken from either the odd or even sampling intervals in order to maximise representation. The laboratories were instructed to undertake the following analyses:

- Gold by fire assay (14 laboratories used 30 g charge weights and 1 laboratory used 25 g charge weights) with AAS (11 laboratories) or ICP-OES (3 laboratories) finish or gravimetric finish (1 laboratory);
- 4-acid (HNO_3-HF-$HClO_4$-HCl) digestion with full suite ICP-OES and ICP-MS elemental packages (up to 16 laboratories depending on the element);
- Total S by infrared combustion furnace/CS analyser (17 laboratories)
- Lithium borate or sodium peroxide fusion with full suite ICP-OES and ICP-MS elemental packages (up to 16 laboratories depending on the element);
- Fluorine by ion selective electrode (12 laboratories).

To evaluate homogeneity, Actlabs Ancaster in Canada were sent 20 x 10 g pulp samples for Au determination using instrumental neutron activation analysis (INAA) on 1 g subsamples. The 20 samples were comprised of paired samples from 10 of the 12 sampling intervals and were randomised prior to assigning sample numbers. The paired samples enabled an Analysis of Variance (ANOVA) by comparison of within- and between-unit variances across the 10 pairs (see 'Homogeneity Evaluation' below).

STATISTICAL ANALYSIS

Certified Values and their uncertainty intervals (Table 1, 2 and 3) 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.

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 4) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor.

Standard Deviation intervals (see Table 8) provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e., after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e., the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. ***The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program*** (see 'Instructions for handling and correct use' section for more detail).

Homogeneity Evaluation

The statistical tolerance limits (ISO Guide 16269:2014) for Au were determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the latter parameter is substantially reduced to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible.

Statistical tolerance limits (as shown in Table 1, 2 and 3) are a function of repeat analysis of the CRM and may be illustrated for Cu by classical wet chemistry, where 99 % of the time ($1-\alpha=0.99$) at least 95 % of subsamples ($\rho=0.95$) will have concentrations lying between 29.24 and 29.30 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. Table 7 below shows the gold INAA data determined on 20 x 1 g subsamples of OREAS 996. 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 0.27 % calculated for a 30 g fire assay sample (1.43 % at 1 g weights) confirms a high level of gold homogeneity.

The homogeneity of OREAS 996 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 entire 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 996. 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.

Table 7. Neutron Activation Analysis of Au (in ppm) on 20 x 1 g subsamples showing the equivalent results scaled to a typical fire assay (30 g sample mass) method.

Replicate	Au	Au
No	1 g actual	30 g equivalent*
1	9.45	9.54
2	9.55	9.56
3	9.37	9.53
4	9.73	9.60
5	9.43	9.54
6	9.79	9.61
7	9.70	9.59
8	9.54	9.56
9	9.60	9.57
10	9.75	9.60
11	9.53	9.56
12	9.64	9.58
13	9.59	9.57
14	9.58	9.57
15	9.69	9.59
16	9.57	9.57
17	9.47	9.55
18	9.48	9.55
19	9.23	9.50
20	9.62	9.58
Mean	9.566	9.566
Median	9.575	9.567
Std Dev.	0.137	0.026
Rel.Std.Dev.	**1.43%**	**0.27%**

*Results calculated for a 30g equivalent sample mass using the formula: $x^{30g\ Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@30g}{RSD@1g} + \bar{X}$

where $x^{30g\ Eq}$ = equivalent result calculated for a 30g sample mass
(x^{INAA}) = raw INAA result at 1g
$\bar{X}$ = mean of 1g INAA results

The data was not filtered for outliers prior to the calculation of the *p*-value. This process derived a *p*-value of 0.13, 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 996 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 996 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PERFORMANCE GATES

Table 8 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 8. Performance Gates for OREAS 996.

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
Umpire Labs (dry sample basis)											
Classical Wet Chemistry											
Cu, wt.%	29.27	0.090	29.09	29.45	29.00	29.54	0.31%	0.61%	0.92%	27.81	30.73
Geoanalytical Labs ('as received' sample basis)											
Pb Fire Assay											
Au, ppm	9.54	0.287	8.97	10.12	8.68	10.41	3.01%	6.01%	9.02%	9.07	10.02

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

IND = indeterminate. 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 8 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
Geoanalytical Labs ('as received' sample basis)											
4-Acid Digestion											
Ag, ppm	145	3	139	150	136	153	1.90%	3.80%	5.70%	137	152
Al, wt.%	0.983	0.041	0.900	1.066	0.859	1.107	4.22%	8.43%	12.65%	0.934	1.032
As, ppm	926	55	817	1035	763	1090	5.89%	11.78%	17.67%	880	973
Ba, ppm	61	11	39	83	28	94	18.27%	36.54%	54.81%	58	64
Be, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	307	27	254	361	228	387	8.64%	17.28%	25.93%	292	323
Ca, wt.%	0.732	0.032	0.668	0.796	0.636	0.828	4.38%	8.77%	13.15%	0.696	0.769
Cd, ppm	48.7	3.63	41.4	55.9	37.8	59.6	7.46%	14.92%	22.38%	46.2	51.1
Ce, ppm	61	13	36	87	23	100	20.94%	41.88%	62.82%	58	64
Co, ppm	208	10	187	228	177	239	4.93%	9.87%	14.80%	197	218
Cr, ppm	29.7	2.60	24.5	34.9	21.9	37.5	8.75%	17.50%	26.25%	28.2	31.2
Cs, ppm	1.56	0.073	1.41	1.70	1.34	1.77	4.68%	9.36%	14.04%	1.48	1.63
Cu, wt.%	29.61	0.459	28.70	30.53	28.24	30.99	1.55%	3.10%	4.65%	28.13	31.09
Dy, ppm	1.12	0.21	0.70	1.55	0.49	1.76	18.78%	37.56%	56.34%	1.07	1.18
Er, ppm	0.56	0.042	0.47	0.64	0.43	0.68	7.51%	15.02%	22.53%	0.53	0.59
Eu, ppm	0.52	0.047	0.42	0.61	0.38	0.66	9.01%	18.01%	27.02%	0.49	0.54
Fe, wt.%	22.46	0.522	21.41	23.50	20.89	24.02	2.33%	4.65%	6.98%	21.33	23.58
Ga, ppm	4.04	0.225	3.59	4.49	3.36	4.72	5.58%	11.16%	16.74%	3.84	4.24
Gd, ppm	1.90	0.29	1.32	2.49	1.02	2.78	15.38%	30.75%	46.13%	1.81	2.00
Hf, ppm	0.29	0.05	0.18	0.39	0.13	0.44	18.14%	36.28%	54.42%	0.27	0.30
Ho, ppm	0.20	0.010	0.18	0.21	0.17	0.22	4.90%	9.81%	14.71%	0.19	0.21
In, ppm	13.5	0.64	12.3	14.8	11.6	15.4	4.70%	9.40%	14.10%	12.8	14.2
K, wt.%	0.288	0.015	0.259	0.318	0.244	0.332	5.08%	10.16%	15.24%	0.274	0.303
Li, ppm	7.66	0.77	6.13	9.20	5.36	9.97	10.02%	20.04%	30.06%	7.28	8.05
Mg, wt.%	0.269	0.021	0.228	0.311	0.207	0.332	7.70%	15.40%	23.09%	0.256	0.283
Mn, wt.%	0.025	0.001	0.024	0.027	0.023	0.027	2.99%	5.97%	8.96%	0.024	0.026
Mo, wt.%	0.191	0.012	0.166	0.216	0.154	0.229	6.51%	13.02%	19.54%	0.182	0.201
Na, wt.%	0.126	0.008	0.111	0.141	0.104	0.149	5.98%	11.97%	17.95%	0.120	0.132
Nb, ppm	1.36	0.126	1.11	1.62	0.98	1.74	9.27%	18.55%	27.82%	1.29	1.43
Nd, ppm	18.7	2.6	13.4	24.0	10.8	26.7	14.15%	28.30%	42.45%	17.8	19.7
Ni, ppm	182	7	168	197	161	204	3.99%	7.98%	11.97%	173	192
P, wt.%	0.025	0.002	0.022	0.029	0.020	0.030	7.06%	14.12%	21.18%	0.024	0.026
Pb, wt.%	0.136	0.005	0.127	0.146	0.122	0.151	3.56%	7.12%	10.67%	0.130	0.143
Pr, ppm	5.74	0.71	4.32	7.16	3.61	7.87	12.38%	24.77%	37.15%	5.45	6.03
Rb, ppm	13.6	0.47	12.6	14.5	12.2	15.0	3.48%	6.96%	10.44%	12.9	14.3
Re, ppm	2.97	0.109	2.75	3.19	2.64	3.29	3.66%	7.31%	10.97%	2.82	3.12
S, wt.%	25.44	1.552	22.34	28.54	20.78	30.09	6.10%	12.20%	18.30%	24.17	26.71
Sb, ppm	589	46	497	681	451	727	7.81%	15.63%	23.44%	559	618

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

IND = indeterminate. 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 8 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
Geoanalytical Labs ('as received' sample basis)											
4-Acid Digestion continued											
Sc, ppm	1.26	0.063	1.14	1.39	1.08	1.45	4.95%	9.89%	14.84%	1.20	1.33
Se, ppm	168	21	127	210	106	231	12.34%	24.67%	37.01%	160	177
Sm, ppm	2.63	0.28	2.07	3.19	1.78	3.48	10.72%	21.44%	32.16%	2.50	2.76
Sn, ppm	42.8	2.70	37.4	48.2	34.7	50.9	6.30%	12.59%	18.89%	40.7	45.0
Sr, ppm	72	3.5	65	79	61	83	4.88%	9.76%	14.65%	68	76
Ta, ppm	0.093	0.007	0.079	0.106	0.073	0.113	7.17%	14.34%	21.50%	0.088	0.097
Te, ppm	21.4	3.9	13.7	29.2	9.9	33.0	17.98%	35.96%	53.94%	20.4	22.5
Th, ppm	2.54	0.092	2.36	2.73	2.26	2.82	3.64%	7.28%	10.91%	2.42	2.67
Ti, wt.%	0.048	0.004	0.041	0.056	0.037	0.059	7.66%	15.33%	22.99%	0.046	0.051
Tl, ppm	3.41	0.236	2.93	3.88	2.70	4.11	6.93%	13.87%	20.80%	3.24	3.58
U, ppm	4.31	0.136	4.03	4.58	3.90	4.71	3.15%	6.30%	9.45%	4.09	4.52
V, ppm	18.1	2.6	13.0	23.3	10.4	25.9	14.21%	28.41%	42.62%	17.2	19.0
W, ppm	23.8	1.35	21.0	26.5	19.7	27.8	5.69%	11.38%	17.07%	22.6	24.9
Y, ppm	5.57	0.441	4.68	6.45	4.24	6.89	7.93%	15.85%	23.78%	5.29	5.84
Yb, ppm	0.48	0.046	0.39	0.58	0.35	0.62	9.55%	19.09%	28.64%	0.46	0.51
Zn, wt.%	1.17	0.038	1.09	1.24	1.05	1.28	3.27%	6.53%	9.80%	1.11	1.22
Zr, ppm	8.75	0.90	6.95	10.55	6.05	11.45	10.29%	20.58%	30.88%	8.31	9.19
Infrared Combustion											
S, wt.%	27.66	0.695	26.27	29.05	25.57	29.74	2.51%	5.02%	7.53%	26.28	29.04
Peroxide Fusion ICP											
Al, wt.%	1.02	0.019	0.98	1.06	0.96	1.08	1.88%	3.75%	5.63%	0.97	1.07
As, ppm	996	49	898	1094	849	1143	4.92%	9.83%	14.75%	946	1045
Ba, ppm	66	4.6	57	76	53	80	6.99%	13.98%	20.96%	63	70
Be, ppm	< 1	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	323	21	281	366	259	387	6.59%	13.18%	19.78%	307	339
Ca, wt.%	0.753	0.038	0.677	0.829	0.639	0.867	5.05%	10.11%	15.16%	0.715	0.790
Cd, ppm	53	6	40	66	34	72	12.12%	24.24%	36.36%	50	56
Ce, ppm	73	6.9	59	87	52	94	9.53%	19.07%	28.60%	69	77
Co, ppm	202	15	173	231	158	245	7.21%	14.42%	21.63%	192	212
Cs, ppm	1.56	0.28	1.00	2.13	0.72	2.41	18.07%	36.15%	54.22%	1.48	1.64
Cu, wt.%	29.39	0.562	28.27	30.51	27.71	31.08	1.91%	3.82%	5.73%	27.92	30.86
Dy, ppm	1.42	0.133	1.16	1.69	1.02	1.82	9.34%	18.69%	28.03%	1.35	1.49
Er, ppm	0.83	0.11	0.60	1.05	0.49	1.16	13.57%	27.14%	40.71%	0.78	0.87
Eu, ppm	0.53	0.05	0.42	0.64	0.37	0.69	10.00%	20.00%	30.01%	0.50	0.56
Fe, wt.%	23.23	0.500	22.23	24.23	21.73	24.73	2.15%	4.31%	6.46%	22.07	24.40
Ga, ppm	4.17	0.80	2.58	5.77	1.78	6.57	19.09%	38.19%	57.28%	3.97	4.38
Gd, ppm	1.98	0.172	1.63	2.32	1.46	2.49	8.70%	17.39%	26.09%	1.88	2.08
Ho, ppm	0.27	0.04	0.19	0.36	0.15	0.40	15.15%	30.30%	45.46%	0.26	0.29
In, ppm	13.8	1.06	11.7	15.9	10.6	17.0	7.72%	15.43%	23.15%	13.1	14.5

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

IND = indeterminate. 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 8 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
Geoanalytical Labs ('as received' sample basis)											
4-Acid Digestion continued											
K, wt.%	0.296	0.027	0.242	0.350	0.214	0.378	9.20%	18.41%	27.61%	0.281	0.311
La, ppm	44.5	1.87	40.7	48.2	38.9	50.1	4.20%	8.39%	12.59%	42.2	46.7
Li, ppm	8.18	1.14	5.90	10.45	4.77	11.59	13.90%	27.80%	41.70%	7.77	8.59
Mg, wt.%	0.286	0.013	0.259	0.313	0.246	0.326	4.70%	9.41%	14.11%	0.272	0.300
Mn, wt.%	0.026	0.003	0.020	0.032	0.017	0.036	11.74%	23.48%	35.22%	0.025	0.028
Mo, wt.%	0.209	0.007	0.196	0.222	0.189	0.229	3.13%	6.27%	9.40%	0.198	0.219
Nd, ppm	23.7	1.78	20.1	27.2	18.3	29.0	7.54%	15.09%	22.63%	22.5	24.8
Ni, ppm	190	26	138	242	112	268	13.69%	27.39%	41.08%	181	200
Pb, wt.%	0.141	0.008	0.124	0.158	0.115	0.166	6.00%	12.01%	18.01%	0.134	0.148
Pr, ppm	7.15	0.90	5.36	8.95	4.46	9.85	12.55%	25.10%	37.65%	6.80	7.51
Rb, ppm	14.6	0.80	13.1	16.2	12.3	17.0	5.43%	10.86%	16.29%	13.9	15.4
S, wt.%	27.62	0.615	26.39	28.85	25.77	29.47	2.23%	4.46%	6.68%	26.24	29.00
Sb, ppm	614	30	555	674	525	704	4.87%	9.75%	14.62%	584	645
Si, wt.%	3.79	0.173	3.44	4.13	3.27	4.31	4.58%	9.15%	13.73%	3.60	3.98
Sm, ppm	2.97	0.198	2.58	3.37	2.38	3.57	6.66%	13.32%	19.97%	2.82	3.12
Sn, ppm	44.3	5.9	32.6	56.1	26.7	62.0	13.27%	26.54%	39.80%	42.1	46.5
Sr, ppm	78	4.8	69	88	64	93	6.12%	12.23%	18.35%	74	82
Th, ppm	2.51	0.201	2.11	2.91	1.91	3.11	7.99%	15.99%	23.98%	2.38	2.64
Ti, wt.%	0.070	0.002	0.067	0.074	0.065	0.076	2.63%	5.27%	7.90%	0.067	0.074
Tl, ppm	3.59	0.135	3.32	3.86	3.19	4.00	3.77%	7.54%	11.30%	3.41	3.77
U, ppm	4.26	0.191	3.87	4.64	3.68	4.83	4.49%	8.98%	13.46%	4.04	4.47
V, ppm	22.3	2.7	16.9	27.6	14.2	30.3	12.06%	24.12%	36.18%	21.2	23.4
W, ppm	26.6	3.0	20.5	32.7	17.5	35.7	11.42%	22.84%	34.26%	25.3	27.9
Y, ppm	7.13	1.13	4.88	9.39	3.75	10.52	15.80%	31.61%	47.41%	6.78	7.49
Yb, ppm	0.64	0.11	0.43	0.86	0.32	0.97	16.71%	33.42%	50.13%	0.61	0.68
Zn, wt.%	1.18	0.035	1.11	1.25	1.07	1.28	2.98%	5.96%	8.93%	1.12	1.24
Ion Selective Electrode											
F, ppm	157	25	108	207	83	232	15.76%	31.53%	47.29%	149	165

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

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

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

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♦ *= Umpire laboratory (classical methods); * = Geoanalytical laboratory (instrumental methods).*

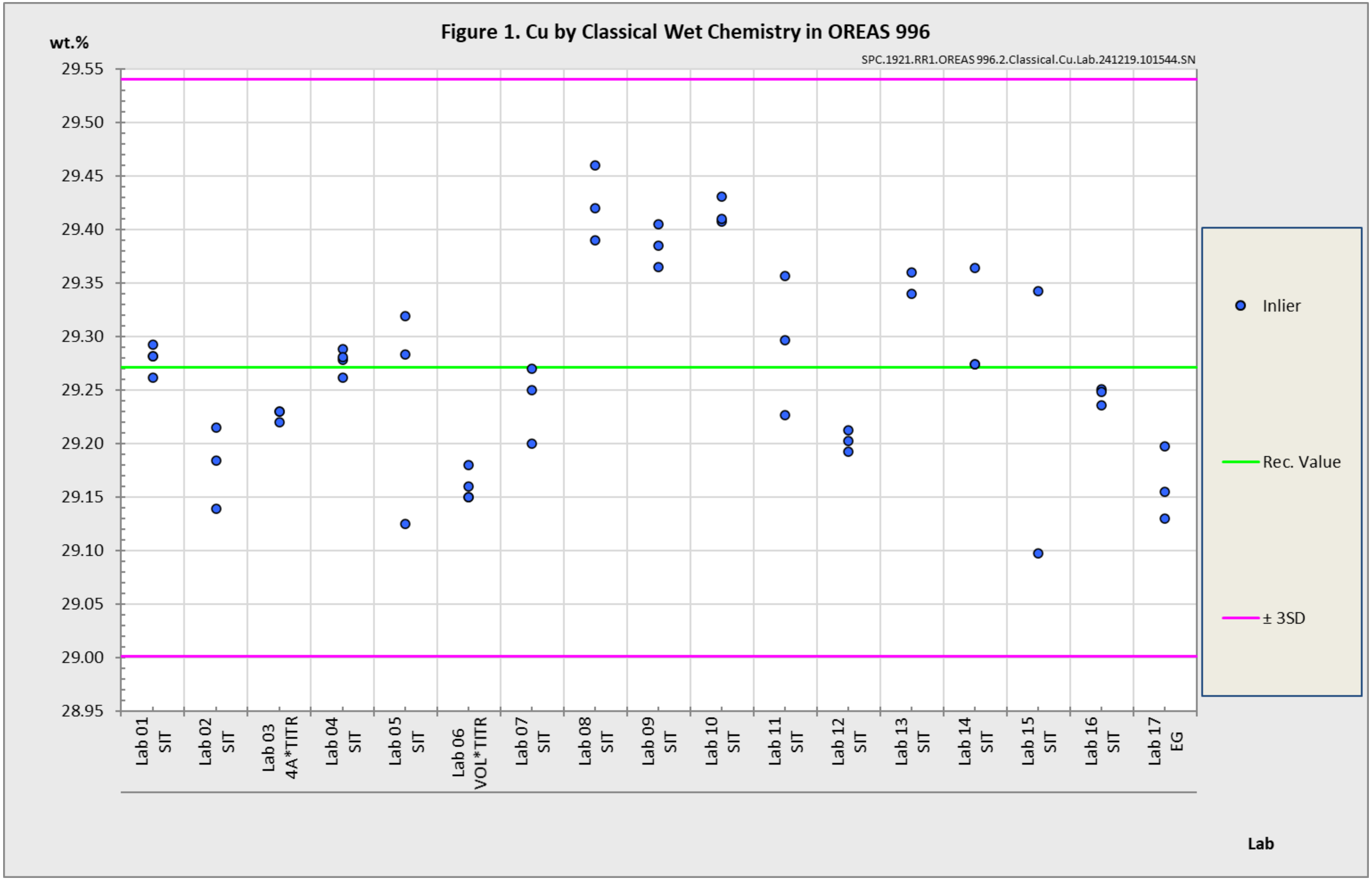
Figure 1. Cu by Classical Wet Chemistry in OREAS 996
wt.%
SPC.1921.RR1.OREAS 996.2.Classical.Cu.Lab.241219.101544.SN
29.55
29.50
29.45
29.40
29.35
29.30
29.25
29.20
29.15
29.10
29.05
29.00
28.95
Lab 01 SIT
Lab 02 SIT
Lab 03 4A*TITR
Lab 04 SIT
Lab 05 SIT
Lab 06 VOL*TITR
Lab 07 SIT
Lab 08 SIT
Lab 09 SIT
Lab 10 SIT
Lab 11 SIT
Lab 12 SIT
Lab 13 SIT
Lab 14 SIT
Lab 15 SIT
Lab 16 SIT
Lab 17 EG
Inlier
Rec. Value
± 3SD
Lab

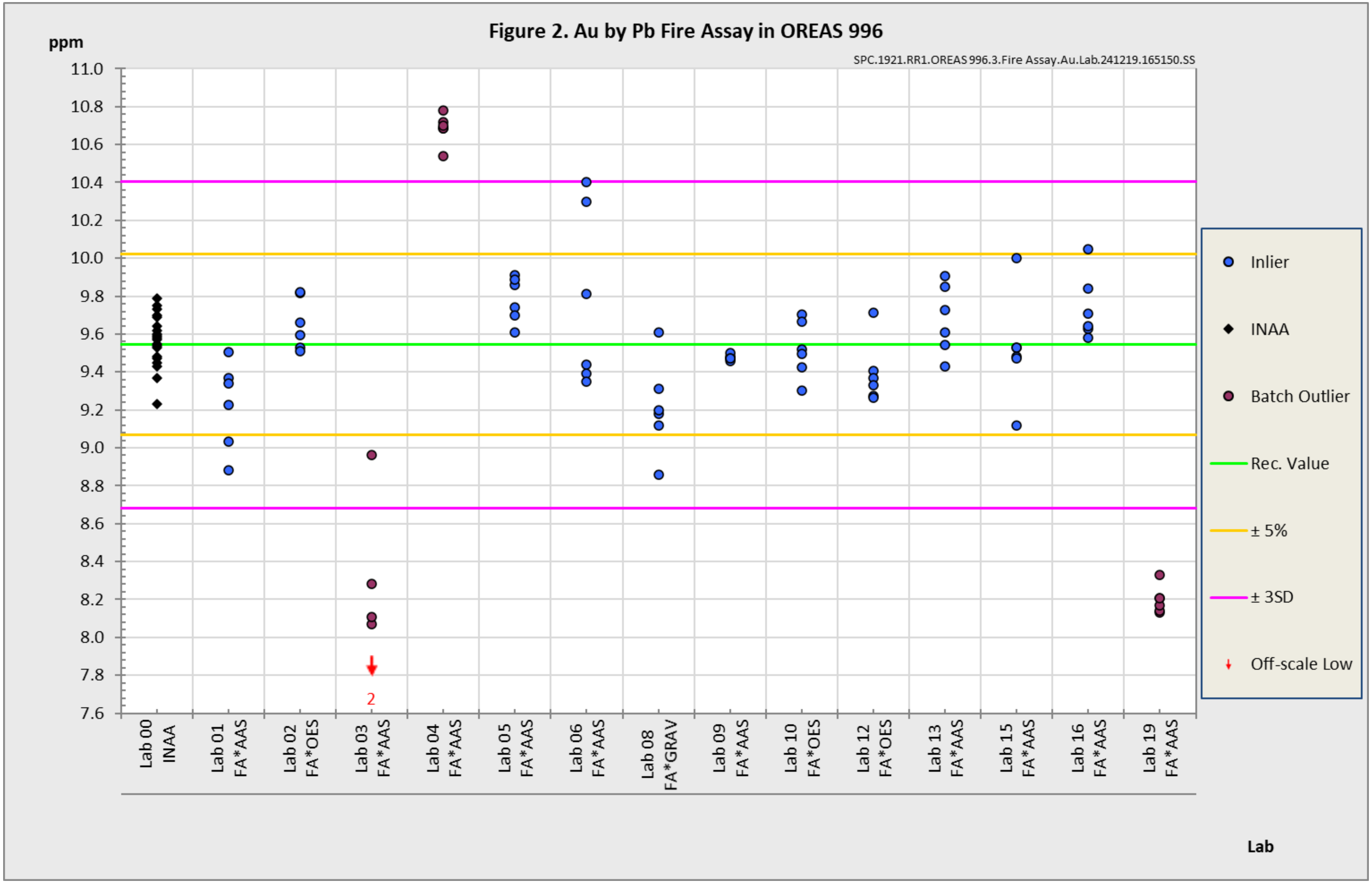
Figure 2. Au by Pb Fire Assay in OREAS 996
ppm
SPC.1921.RR1.OREAS 996.3.Fire Assay.Au.Lab.241219.165150.SS
11.0
10.8
10.6
10.4
10.2
10.0
9.8
9.6
9.4
9.2
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
2
Lab 00 INAA
Lab 01 FA*AAS
Lab 02 FA*OES
Lab 03 FA*AAS
Lab 04 FA*AAS
Lab 05 FA*AAS
Lab 06 FA*AAS
Lab 08 FA*GRAV
Lab 09 FA*AAS
Lab 10 FA*OES
Lab 12 FA*OES
Lab 13 FA*AAS
Lab 15 FA*AAS
Lab 16 FA*AAS
Lab 19 FA*AAS
Lab
Inlier
INAA
Batch Outlier
Rec. Value
± 5%
± 3SD
Off-scale Low

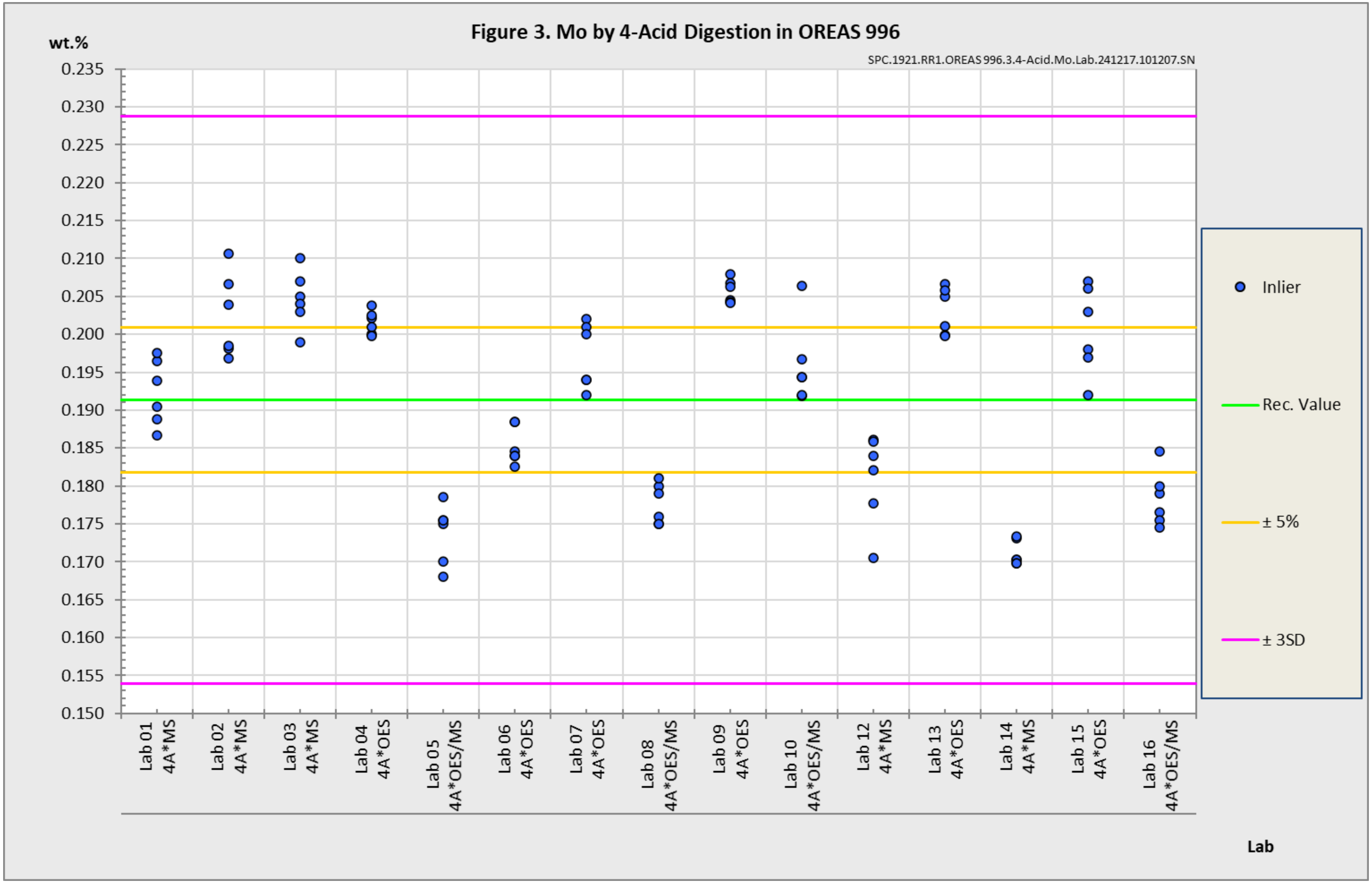

Figure 3. Mo by 4-Acid Digestion in OREAS 996
wt.%
SPC.1921.RR1.OREAS 996.3.4-Acid.Mo.Lab.241217.101207.SN
0.235
0.230
0.225
0.220
0.215
0.210
0.205
0.200
0.195
0.190
0.185
0.180
0.175
0.170
0.165
0.160
0.155
0.150
Lab 01 4A*MS
Lab 02 4A*MS
Lab 03 4A*MS
Lab 04 4A*OES
Lab 05 4A*OES/MS
Lab 06 4A*OES
Lab 07 4A*OES
Lab 08 4A*OES/MS
Lab 09 4A*OES
Lab 10 4A*OES/MS
Lab 12 4A*MS
Lab 13 4A*OES
Lab 14 4A*MS
Lab 15 4A*OES
Lab 16 4A*OES/MS
Inlier
Rec. Value
± 5%
± 3SD
Lab

METROLOGICAL TRACEABILITY

The interlaboratory results that underpin the certified values are metrologically traceable to the international measurement scale (SI) of mass (either as a % mass fraction or as milligrams per kilogram (mg/kg)) [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).

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 [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 Cu by classical wet chemistry methods, Au by fire assay, multi-elements by 4-acid digestion and S by IR combustion furnace. The other operationally defined measurands characterised in this certificate 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, 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 'metallurgical concentrate' 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 996 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 996 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 996 is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of analytes reported in Tables 1 to 3 in geological samples;
- For the verification of analytical methods for analytes reported in Tables 1 to 3;
- For the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1 to 3. 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 to 3).

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:

- Cu by classical wet chemistry: ≥ 0.5 g;
- Au by fire assay: ≥ 5 g;
- 4-acid digestion with ICP-OES and/or MS finish: ≥ 0.25 g;
- Total S by Infrared combustion furnace/CS analyser: ≥ 0.1 g;
- Peroxide fusion with ICP-OES and/or MS finish: ≥ 0.1 g;
- Fluorine by ion selective electrode: ≥ 0.2 g.

PERIOD OF VALIDITY & STORAGE INSTRUCTIONS

OREAS 996 is high in reactive sulphide content and has been packaged under a nitrogen environment in robust laminated foil pouches in single-use 10 g units. In its unopened state in the sachets (sealed under nitrogen), OREAS 996 has a shelf life of at least ten years (Dec 2036).

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

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.

Umpire laboratories using classical methods:

The umpire laboratory certified value for Cu refers to the concentration level on a dry sample basis. At each laboratory, analyses were performed on the sample as received (without drying) with the subsample for moisture analysis weighed simultaneously with the subsamples for Cu assay. The Cu data was then corrected to dry basis using the moisture value obtained at each laboratory.

Moisture content varied amongst the laboratories from 0.26-1.3 % with a best consensus value of 0.5 %. The indicative value provided for moisture (H_2O-) should be viewed as informational only. Hygroscopic moisture is a dynamic property of pulp materials and will vary in response to the local laboratory atmosphere following equilibration.

Geoanalytical laboratories using instrumental methods:

All analyses were performed on the samples as received and reported as such in line with conventional instrumental method procedures.

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 8 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 interval, then generally there is no cause for concern in regard to bias.

LEGAL NOTICE

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

DOCUMENT HISTORY

Revision No.	Date	Changes applied
0	23rd December, 2024	First publication.

QMS CERTIFICATION

ORE Pty Ltd is accredited for compliance with ISO 17034.

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

CERTIFYING OFFICER

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REFERENCES

[1] Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).

[2] Ingamells, C. O. and Switzer, P. (1973). A Proposed Sampling Constant for Use in Geochemical Analysis, Talanta 20, 547-568.

[3] ISO Guide 30:2015. Terms and definitions used in connection with reference materials.

[4] ISO Guide 33401:2024-01. Reference materials – Contents of certificates, labels and accompanying documentation.

[5] ISO Guide 33405:2024-05. Reference materials – Approaches for characterization and assessment of homogeneity and stability.

[6] ISO Guide 98-3:2008. Guide to the expression of uncertainty in measurement (GUM:1995).

[7] ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.

[8] ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.

[9] ISO 17025:2017. General requirements for the competence of testing and calibration laboratories.

[10] ISO Guide 17034:2016. General requirements for the competence of reference material producers.

[11] ISO 9599:2015. Cu, Pb, Zn and Ni sulfide concentrates - Determination of hygroscopic moisture content

[12] OREAS-BUP-70-09-11: Statistical Analysis - OREAS Evaluation Method.

[13] OREAS-TN-04-1498: Stability under transport; an experimental study of OREAS CRMs.

[14] OREAS-TN-05-1674: Long-term storage stability; an experimental study of OREAS CRMs.

[15] Thompson, A.; Taylor, B.N. (2008), Guide for the Use of the International System of Units (SI); NIST Special Publication 811; U.S. Government Printing Office: Washington, DC; available at: https://physics.nist.gov/cuu/pdf/sp811.pdf (accessed Nov 2021).

[16] Van der Veen A.M.H. et al. (2001). Uncertainty calculations in the certification of reference materials, Accred Qual Assur 6: 290-294.