



ORE RESEARCH & EXPLORATION P/L ABN 28 006 859 856  
 37A Hosie Street · Bayswater North · VIC 3153 · AUSTRALIA  
 ☎ 61 3 9729 0333 📠 61 3 9729 8338  
 ⓘ info@ore.com.au 🌐 www.ore.com.au

**CERTIFICATE OF ANALYSIS FOR**

**COPPER-GOLD-SILVER CONCENTRATE**

**(Newcrest Cadia Valley Mine Site, Orange, NSW, Australia)**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 993**

**Table 1. Certified Values and Performance Gates for OREAS 993.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Umpire Labs (dry sample basis)</b>											
<b>Pb Fire Assay with full corrections for silver, slag and cupel</b>											
Au, ppm	56.04	0.296	55.45	56.64	55.16	56.93	0.53%	1.06%	1.59%	53.24	58.85
<b>Classical Wet Chemistry</b>											
Cu, wt. %	23.90	0.061	23.78	24.02	23.72	24.08	0.25%	0.51%	0.76%	22.70	25.09
<b>Acid Digestion (no HF)</b>											
Ag, ppm	41.6	1.12	39.3	43.8	38.2	44.9	2.69%	5.38%	8.07%	39.5	43.6

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

Note 1: SD's are based on the <sup>1</sup>Pooled Repeatability Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

Note 3: 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.



Document: COA-1416-OREAS993-R0	22-May-2020
(Template: BUP-70-10-01 Rev:2.0)	

Table 1 continued.

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>Geoanalytical Labs ('as received' sample basis)</b>											
<b>Pb Fire Assay</b>											
Au, ppm	54.85	1.324	52.20	57.49	50.87	58.82	2.41%	4.83%	7.24%	52.10	57.59
Pd, ppb	529	32	465	592	434	624	6.00%	12.01%	18.01%	502	555
Pt, ppb	43.8	5.9	32.0	55.5	26.2	61.4	13.41%	26.82%	40.23%	41.6	46.0
<b>Oxidising Fusion XRF</b>											
Al <sub>2</sub> O <sub>3</sub> , wt. %	3.03	0.073	2.88	3.17	2.81	3.25	2.42%	4.84%	7.25%	2.87	3.18
CaO, wt. %	0.982	0.012	0.959	1.006	0.947	1.018	1.20%	2.40%	3.60%	0.933	1.031
Cu, wt. %	23.36	0.364	22.63	24.09	22.27	24.45	1.56%	3.11%	4.67%	22.19	24.53
Fe, wt. %	25.41	0.291	24.82	25.99	24.53	26.28	1.15%	2.29%	3.44%	24.14	26.68
K, wt. %	0.640	0.021	0.598	0.683	0.576	0.704	3.34%	6.67%	10.01%	0.608	0.672
MgO, wt. %	0.512	0.067	0.377	0.647	0.310	0.715	13.16%	26.33%	39.49%	0.487	0.538
Mo, ppm	2512	119	2274	2751	2154	2871	4.75%	9.51%	14.26%	2387	2638
S, wt. %	30.06	0.733	28.60	31.53	27.86	32.26	2.44%	4.88%	7.31%	28.56	31.57
SiO <sub>2</sub> , wt. %	10.56	0.187	10.19	10.93	10.00	11.12	1.77%	3.53%	5.30%	10.03	11.09
TiO <sub>2</sub> , wt. %	0.198	0.013	0.171	0.225	0.157	0.238	6.82%	13.63%	20.45%	0.188	0.208
<b>Infrared Combustion</b>											
S, wt. %	29.92	0.784	28.35	31.49	27.57	32.27	2.62%	5.24%	7.86%	28.42	31.42
<b>Aqua Regia Digestion</b>											
Hg, ppm	1.92	0.143	1.64	2.21	1.49	2.35	7.44%	14.88%	22.33%	1.83	2.02
<b>Miscellaneous Assay Methods</b>											
F, ppm	511	46	418	604	372	650	9.06%	18.12%	27.18%	485	536
<b>4-Acid Digestion</b>											
Ag, ppm	39.7	1.24	37.2	42.2	36.0	43.4	3.12%	6.24%	9.36%	37.7	41.7
Al, wt. %	1.60	0.044	1.51	1.69	1.47	1.73	2.77%	5.54%	8.31%	1.52	1.68
As, ppm	143	7	129	157	122	164	4.83%	9.65%	14.48%	136	150
Ba, ppm	122	7	108	136	101	143	5.72%	11.44%	17.16%	116	128
Be, ppm	< 0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Bi, ppm	23.8	5.0	13.8	33.8	8.8	38.8	21.03%	42.06%	63.10%	22.6	25.0
Ca, wt. %	0.702	0.023	0.657	0.747	0.634	0.770	3.23%	6.45%	9.68%	0.667	0.737
Cd, ppm	6.41	1.58	3.24	9.57	1.66	11.16	24.69%	49.38%	74.08%	6.09	6.73
Ce, ppm	18.2	1.38	15.4	21.0	14.0	22.3	7.60%	15.20%	22.79%	17.3	19.1
Co, ppm	95	5.4	84	106	79	112	5.70%	11.41%	17.11%	91	100
Cr, ppm	39.5	4.2	31.2	47.8	27.0	51.9	10.53%	21.05%	31.58%	37.5	41.4
Dy, ppm	1.02	0.070	0.88	1.16	0.81	1.23	6.88%	13.76%	20.64%	0.97	1.07
Er, ppm	0.54	0.06	0.41	0.67	0.34	0.73	12.03%	24.06%	36.09%	0.51	0.57
Eu, ppm	0.43	0.07	0.30	0.57	0.23	0.64	15.94%	31.87%	47.81%	0.41	0.46
Fe, wt. %	25.75	0.735	24.28	27.22	23.55	27.96	2.85%	5.71%	8.56%	24.46	27.04
Ga, ppm	3.93	0.286	3.36	4.50	3.07	4.79	7.29%	14.58%	21.87%	3.73	4.13
Gd, ppm	1.32	0.21	0.91	1.74	0.70	1.94	15.70%	31.40%	47.09%	1.25	1.39

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

Note 1: SD's are based on the pooled Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

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

**Table 1 continued.**

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
<b>4-Acid Digestion continued</b>											
Hf, ppm	0.71	0.09	0.53	0.90	0.43	0.99	13.05%	26.10%	39.16%	0.68	0.75
Ho, ppm	0.19	0.018	0.15	0.23	0.14	0.24	9.44%	18.87%	28.31%	0.18	0.20
In, ppm	1.54	0.115	1.31	1.77	1.19	1.89	7.50%	14.99%	22.49%	1.46	1.62
K, wt. %	0.634	0.023	0.587	0.680	0.564	0.703	3.66%	7.31%	10.97%	0.602	0.665
La, ppm	10.3	0.45	9.4	11.3	9.0	11.7	4.38%	8.76%	13.14%	9.8	10.9
Lu, ppm	0.083	0.012	0.060	0.107	0.048	0.119	14.07%	28.14%	42.21%	0.079	0.088
Mg, wt. %	0.301	0.017	0.267	0.334	0.251	0.351	5.56%	11.12%	16.68%	0.286	0.316
Mn, wt. %	0.008	0.001	0.005	0.010	0.004	0.012	17.79%	35.58%	53.37%	0.007	0.008
Mo, ppm	2535	196	2144	2926	1949	3122	7.71%	15.43%	23.14%	2409	2662
Na, wt. %	0.357	0.009	0.339	0.375	0.330	0.384	2.56%	5.12%	7.68%	0.339	0.375
Nb, ppm	2.10	0.178	1.75	2.46	1.57	2.64	8.45%	16.89%	25.34%	2.00	2.21
Nd, ppm	7.55	0.325	6.90	8.20	6.57	8.52	4.31%	8.62%	12.93%	7.17	7.93
Ni, ppm	52	6	40	65	33	71	12.07%	24.15%	36.22%	50	55
P, wt. %	0.014	0.003	0.008	0.020	0.004	0.024	22.73%	45.46%	68.19%	0.013	0.015
Pb, ppm	230	17	195	265	178	282	7.58%	15.15%	22.73%	219	242
Pr, ppm	1.98	0.081	1.81	2.14	1.73	2.22	4.12%	8.23%	12.35%	1.88	2.07
Rb, ppm	20.0	1.50	17.0	23.0	15.5	24.5	7.48%	14.95%	22.43%	19.0	21.0
Re, ppm	6.15	0.65	4.85	7.45	4.20	8.10	10.57%	21.15%	31.72%	5.84	6.46
S, wt. %	30.18	1.309	27.56	32.80	26.26	34.11	4.34%	8.68%	13.01%	28.67	31.69
Sb, ppm	55	8	39	70	32	77	14.02%	28.03%	42.05%	52	57
Sc, ppm	4.94	0.369	4.21	5.68	3.84	6.05	7.46%	14.92%	22.39%	4.70	5.19
Se, ppm	194	17	161	228	144	245	8.62%	17.23%	25.85%	185	204
Sm, ppm	1.52	0.126	1.27	1.77	1.14	1.90	8.32%	16.63%	24.95%	1.44	1.59
Sn, ppm	1.64	0.35	0.95	2.33	0.60	2.68	21.06%	42.13%	63.19%	1.56	1.72
Sr, ppm	149	7	134	164	127	172	4.98%	9.96%	14.94%	142	157
Ta, ppm	0.099	0.019	0.060	0.138	0.041	0.157	19.62%	39.23%	58.85%	0.094	0.104
Tb, ppm	0.17	0.02	0.13	0.20	0.11	0.22	11.49%	22.98%	34.48%	0.16	0.17
Te, ppm	12.7	1.7	9.3	16.1	7.6	17.8	13.44%	26.88%	40.33%	12.1	13.3
Th, ppm	2.44	0.208	2.02	2.86	1.82	3.07	8.53%	17.06%	25.59%	2.32	2.56
Ti, wt. %	0.105	0.005	0.095	0.115	0.090	0.120	4.66%	9.33%	13.99%	0.100	0.110
Tl, ppm	0.52	0.13	0.26	0.79	0.13	0.92	24.88%	49.76%	74.63%	0.50	0.55
Tm, ppm	0.075	0.019	0.037	0.113	0.019	0.131	25.05%	50.11%	75.16%	0.071	0.079
U, ppm	0.86	0.059	0.74	0.98	0.68	1.04	6.89%	13.78%	20.67%	0.81	0.90
V, ppm	45.8	4.6	36.6	55.1	31.9	59.7	10.10%	20.21%	30.31%	43.5	48.1
W, ppm	2.95	0.42	2.12	3.79	1.70	4.21	14.14%	28.28%	42.42%	2.81	3.10
Y, ppm	5.41	0.332	4.75	6.08	4.42	6.41	6.14%	12.27%	18.41%	5.14	5.68
Yb, ppm	0.53	0.11	0.32	0.74	0.21	0.84	19.99%	39.98%	59.98%	0.50	0.55
Zn, ppm	885	43	799	971	756	1014	4.86%	9.72%	14.58%	841	930
Zr, ppm	24.9	1.12	22.6	27.1	21.5	28.2	4.50%	8.99%	13.49%	23.6	26.1

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

Note 1: SD's are based on the pooled Standard Deviation.

Note 2: intervals may appear asymmetric due to rounding.

Note 3: 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 OF CONTENTS

INTRODUCTION .....	5
SOURCE MATERIAL.....	5
PERFORMANCE GATES .....	5
COMMINUTION AND HOMOGENISATION PROCEDURES .....	6
ANALYTICAL PROGRAM.....	6
PHYSICAL PROPERTIES .....	8
STATISTICAL ANALYSIS.....	8
Homogeneity Evaluation .....	12
PARTICIPATING LABORATORIES.....	13
PREPARER AND SUPPLIER.....	16
METROLOGICAL TRACEABILITY .....	16
COMMUTABILITY .....	16
INTENDED USE .....	17
HANDLING INSTRUCTIONS.....	17
STABILITY AND STORAGE INSTRUCTIONS .....	17
INSTRUCTIONS FOR CORRECT USE.....	17
LEGAL NOTICE.....	18
DOCUMENT HISTORY .....	18
QMS CERTIFICATION .....	18
CERTIFYING OFFICER.....	18
REFERENCES .....	19

## LIST OF TABLES

Table 1. Certified Values and Performance Gates for OREAS 993.....	1
Table 2. Indicative Values for OREAS 993.....	7
Table 3. Physical properties of OREAS 993.....	8
Table 4. 95% Confidence & Tolerance Limits for OREAS 993.....	9
Table 5. Neutron Activation Analysis of Au.....	12

## LIST OF FIGURES

Figure 1. Au by fire assay (full corrections) in OREAS 993.....	14
Figure 2. Cu by classical wet chemistry in OREAS 993.....	15

## INTRODUCTION

OREAS reference materials are intended to provide a low-cost method of evaluating and improving the quality of analysis of geological and sulphide concentrate 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.

## SOURCE MATERIAL

OREAS 993 is a certified reference material (CRM) prepared from copper-gold-silver concentrate samples supplied by Newcrest Mining Limited's Cadia Valley Operations near Orange, New South Wales, Australia.

## PERFORMANCE GATES

The standard deviations (SD's) reported in Table 1 provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors (see 'Homogeneity Evaluation' for verification of OREAS 993's high level of homogeneity).

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

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

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

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 993 was prepared in the following manner:

- Drying of ore materials to constant mass at 85°C;
- Multi-stage milling to 100% minus 30 microns;
- Homogenisation;
- Packaging under nitrogen in 10g and 50g units in laminated foil pouches.

## ANALYTICAL PROGRAM

For the interlaboratory 'round robin' certification program, samples were taken at 10 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire prepared batch of OREAS 993.

### Umpire Laboratories

Ten 'umpire' laboratories were used to determine the key elements: Au, Cu and Ag. The term 'umpire' here refers to the routine analysis of these laboratories using classical methodologies for precious and base metals. To permit an appraisal of batch to batch variation at individual laboratories, samples were submitted in three batches at weekly intervals. Each batch consisted of a single 120g sample to be assayed for Au, Cu and Ag in triplicate.

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 Au, Cu and Ag 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:

- Gold (3 trials on undried sample) by reduced charge weight (10-20g) fire assay with gravimetric finish and full corrections for slag, cupel and silver losses;
- Copper (3 trials on undried sample) by classical wet chemistry (predominantly by short iodide titration with two laboratories employing electrogravimetry);
- Silver (3 trials on undried sample) by the laboratory's preferred method. This resulted in aqua regia digestion with AAS finish (3 laboratories), 3-acid digestion with AAS finish (4 laboratories) and ICP-MS finish (1 laboratory), 4-acid digestion with ICP-OES finish (1 laboratory) and fire assay with gravimetric finish (1 laboratory).

## Geoanalytical Laboratories

Twenty-three geoanalytical laboratories also participated in the analytical program for OREAS 993. Each laboratory was sent 6 x 110g samples to undertake the following:

- Au, Pt and Pd by fire assay with ICP-OES and MS finish (13 laboratories);
- Cu, Al<sub>2</sub>O<sub>3</sub>, MgO, SiO<sub>2</sub>, Fe, S, CaO, TiO<sub>2</sub>, K and Mo by oxidising fusion with XRF finish (up to 10 laboratories depending on the element);
- Total Sulphur by infrared combustion furnace (14 laboratories);
- Hg by low level, aqua regia digestion with ICP-OES or MS finish (15 laboratories);
- Fluorine by ion selective electrode (ISE), (14 laboratories);
- Chlorine by various methods (6 laboratories);
- Ag, As, Bi, Cd, Co, Cr, Ni, Pb, Re, Sb, Se, Te, Th, U and Zn by 4-acid digestion with ICP-OES or MS finish (up to 19 laboratories depending on the element although some laboratories used a 3-acid or 2-acid digestion for Ag, As, Co, Ni, Pb and Zn);
- Gold by instrumental neutron activation analysis (INAA) on 20 x 85mg subsamples to confirm homogeneity (undertaken by ANSTO, Lucas Heights).

Table 1 provides performance gate intervals for the certified values and Table 2 shows indicative values. Table 3 provides some indicative physical properties and Table 4 presents the 95% confidence and tolerance limits for all certified values.

**Table 2. Indicative Values for OREAS 993.**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Oxidising Fusion XRF</b>								
As	ppm	167	Nb	ppm	167	Ta	ppm	133
Ba	ppm	< 100	Ni	ppm	183	V	ppm	83
Bi	ppm	< 100	P	wt.%	0.030	WO <sub>3</sub>	ppm	67
Co	ppm	117	Pb	ppm	417	Zn	ppm	900
Cr	ppm	100	Sb	ppm	233	Zr	ppm	433
Mn	wt.%	0.013	Sn	ppm	< 100			
<b>Thermogravimetry</b>								
LOI <sup>1000</sup>	wt.%	16.49						
<b>Miscellaneous Assay Methods</b>								
Cl	ppm	76						
<b>4-Acid Digestion</b>								
B	ppm	124	Cu	wt.%	23.45	Li	ppm	3.90
Cs	ppm	0.70	Ge	ppm	< 5			

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

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

Comparisons of inter-laboratory bias and precision are graphically presented in scatter plots for gold and copper by the umpire laboratories (Figures 1 and 2) 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.

Gold homogeneity has been evaluated and confirmed by INAA on 20 x 85 milligram subsamples. Tabulated results of all elements (including Au INAA analyses) together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of

means (PDM<sup>3</sup>) are presented in an Excel-compatible file for this CRM (**OREAS 993 DataPack-1.0.200515\_130447.xlsx**).

## PHYSICAL PROPERTIES

OREAS 993 was tested at ORE Research & Exploration Pty Ltd's onsite laboratory for various physical properties. Table 3 presents these findings which should be used for informational purposes only.

**Table 3. Physical properties of OREAS 993.**

CRM Name	Bulk Density (g/L)	Moisture%	Munsell Notation <sup>‡</sup>	Munsell Color <sup>‡</sup>
OREAS 993	817	0.92	N2	Grayish Black

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

## STATISTICAL ANALYSIS

**Standard Deviation** intervals (see Table 1) provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. Two types of laboratory performance gates are presented on the basis of the round robin format.

### Umpire Laboratory Performance Gates

These performance gates include four sources of measurement uncertainty:

- Within-laboratory within-batch variance or analytical precision (repeatability);
- Within-laboratory between-batch variance (reproducibility);
- Between-laboratory variance and
- CRM variability.

The SD's have been calculated from the same filtered data set used to determine the certified value. These outliers can only be removed after the absolute homogeneity of the CRM has been independently established (see 'Homogeneity Evaluation' below), i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM.

For routine submissions (assessing the data quality of a sample batch at one laboratory) the Within-Lab SD can be used as a guide to QC monitoring. Within-Lab SD's include precision errors and batch-to-batch variance but exclude between-laboratory variance. It is calculated from the square root of the average variance for  $p$  laboratories and is known as the pooled repeatability standard deviation (NIST/SEMATECH e-Handbook of Statistical Methods, 2012).

### Geoanalytical Laboratory Performance Gates

These performance gates include three sources of measurement uncertainty:

- Between-laboratory variance;
- Within-laboratory within-batch variance or analytical precision (repeatability);
- CRM variability.



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

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

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

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

**Certified Values** are the means of accepted laboratory means after outlier filtering. The INAA data (see Table 5) is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 993 (see 'Homogeneity Evaluation' section below).

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

**Indicative (uncertified) values** (Table 2) are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where interlaboratory consensus is poor.

**Table 4. 95% Confidence & Tolerance Limits for OREAS 993.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Umpire Labs (dry sample basis)</b>					
<b>Pb Fire Assay with full corrections for silver, slag and cupel</b>					
Au, Gold (ppm)	56.04	55.87	56.22	55.84*	56.25*
<b>Classical Wet Chemistry</b>					
Cu, Copper (wt.%)	23.90	23.85	23.95	23.87	23.93
<b>Acid Digestion (no HF)</b>					
Ag, Silver (ppm)	41.6	40.7	42.5	41.2	41.9

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

\*Gold Tolerance Limits for typical 15g fire assay methods are determined from 20 x 85mg INAA results and the Sampling Constant (Ingamells & Switzer, 1973).

Note 1: intervals may appear asymmetric due to rounding.

**Table 4 continued.**

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>Geoanalytical Labs ('as received' sample basis)</b>					
<b>Pb Fire Assay</b>					
Au, Gold (ppm)	54.85	53.60	56.09	54.70**	54.99**
Pd, Palladium (ppb)	529	513	545	512	546
Pt, Platinum (ppb)	43.8	40.4	47.1	40.7	46.8
<b>Oxidising Fusion XRF</b>					
Al <sub>2</sub> O <sub>3</sub> , Aluminium(III) oxide (wt.%)	3.03	2.97	3.08	2.98	3.07
CaO, Calcium oxide (wt.%)	0.982	0.974	0.990	0.964	1.000
Cu, Copper (wt.%)	23.36	23.08	23.64	23.25	23.47
Fe, Iron (wt.%)	25.41	25.19	25.62	25.23	25.58
K, Potassium (wt.%)	0.640	0.623	0.658	0.630	0.651
MgO, Magnesium oxide (wt.%)	0.512	0.448	0.576	0.493	0.531
Mo, Molybdenum (ppm)	2512	2414	2611	2327	2697
S, Sulphur (wt.%)	30.06	29.49	30.64	29.82	30.31
SiO <sub>2</sub> , Silicon dioxide (wt.%)	10.56	10.42	10.70	10.45	10.67
TiO <sub>2</sub> , Titanium dioxide (wt.%)	0.198	0.189	0.206	0.181	0.214
<b>Infrared Combustion</b>					
S, Sulphur (wt.%)	29.92	29.48	30.37	29.58	30.26
<b>Aqua Regia Digestion</b>					
Hg, Mercury (ppm)	1.92	1.82	2.03	1.87	1.98
<b>Miscellaneous Assay Methods</b>					
F, Fluorine (ppm)	511	484	538	493	529
<b>4-Acid Digestion</b>					
Ag, Silver (ppm)	39.7	39.1	40.3	38.7	40.7
Al, Aluminium (wt.%)	1.60	1.57	1.63	1.57	1.63
As, Arsenic (ppm)	143	140	146	140	147
Ba, Barium (ppm)	122	116	127	115	128
Be, Beryllium (ppm)	< 0.5	IND	IND	IND	IND
Bi, Bismuth (ppm)	23.8	20.4	27.1	22.4	25.1
Ca, Calcium (wt.%)	0.702	0.687	0.717	0.688	0.716
Cd, Cadmium (ppm)	6.41	5.08	7.73	6.06	6.75
Ce, Cerium (ppm)	18.2	16.7	19.7	17.5	18.9
Co, Cobalt (ppm)	95	93	98	94	97
Cr, Chromium (ppm)	39.5	35.9	43.0	36.9	42.1
Dy, Dysprosium (ppm)	1.02	0.97	1.07	IND	IND
Er, Erbium (ppm)	0.54	0.48	0.59	IND	IND
Eu, Europium (ppm)	0.43	0.37	0.50	IND	IND
Fe, Iron (wt.%)	25.75	25.35	26.15	25.27	26.23
Ga, Gallium (ppm)	3.93	3.56	4.30	3.59	4.27
Gd, Gadolinium (ppm)	1.32	1.12	1.53	IND	IND
Hf, Hafnium (ppm)	0.71	0.63	0.79	IND	IND

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

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

Note 1: intervals may appear asymmetric due to rounding.

Table 4 continued.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
<b>4-Acid Digestion continued</b>					
Ho, Holmium (ppm)	0.19	0.17	0.21	IND	IND
In, Indium (ppm)	1.54	1.42	1.66	1.43	1.65
K, Potassium (wt.%)	0.634	0.618	0.650	0.619	0.649
La, Lanthanum (ppm)	10.3	10.0	10.7	9.6	11.1
Lu, Lutetium (ppm)	0.083	0.069	0.098	IND	IND
Mg, Magnesium (wt.%)	0.301	0.289	0.313	0.293	0.309
Mn, Manganese (wt.%)	0.008	0.007	0.009	0.007	0.008
Mo, Molybdenum (ppm)	2535	2406	2665	2475	2595
Na, Sodium (wt.%)	0.357	0.352	0.362	0.340	0.373
Nb, Niobium (ppm)	2.10	1.98	2.22	IND	IND
Nd, Neodymium (ppm)	7.55	7.17	7.93	7.22	7.88
Ni, Nickel (ppm)	52	49	55	50	54
P, Phosphorus (wt.%)	0.014	0.012	0.016	IND	IND
Pb, Lead (ppm)	230	221	239	224	236
Pr, Praseodymium (ppm)	1.98	1.88	2.07	1.84	2.11
Rb, Rubidium (ppm)	20.0	18.6	21.5	19.3	20.8
Re, Rhenium (ppm)	6.15	5.49	6.81	5.79	6.51
S, Sulphur (wt.%)	30.18	28.80	31.56	29.44	30.93
Sb, Antimony (ppm)	55	49	60	51	58
Sc, Scandium (ppm)	4.94	4.75	5.13	IND	IND
Se, Selenium (ppm)	194	182	207	188	201
Sm, Samarium (ppm)	1.52	1.38	1.66	IND	IND
Sn, Tin (ppm)	1.64	1.53	1.75	IND	IND
Sr, Strontium (ppm)	149	144	155	146	152
Ta, Tantalum (ppm)	0.099	0.077	0.121	IND	IND
Tb, Terbium (ppm)	0.17	0.15	0.18	IND	IND
Te, Tellurium (ppm)	12.7	10.9	14.5	11.8	13.6
Th, Thorium (ppm)	2.44	2.22	2.66	2.30	2.58
Ti, Titanium (wt.%)	0.105	0.102	0.108	0.102	0.108
Tl, Thallium (ppm)	0.52	0.44	0.61	IND	IND
Tm, Thulium (ppm)	0.075	0.060	0.090	IND	IND
U, Uranium (ppm)	0.86	0.81	0.91	IND	IND
V, Vanadium (ppm)	45.8	42.0	49.7	42.9	48.8
W, Tungsten (ppm)	2.95	2.44	3.47	IND	IND
Y, Yttrium (ppm)	5.41	5.13	5.69	5.19	5.64
Yb, Ytterbium (ppm)	0.53	0.46	0.59	IND	IND
Zn, Zinc (ppm)	885	865	906	873	898
Zr, Zirconium (ppm)	24.9	24.4	25.3	23.0	26.8

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

Note 1: intervals may appear asymmetric due to rounding.

## Homogeneity Evaluation

The tolerance limits (ISO 16269:2014) shown in Table 1 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 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 23.87 and 23.93 wt.%. Put more precisely, this means that if the same number of subsamples were taken and analysed in the same manner repeatedly, 99% of the tolerance intervals so constructed would cover at least 95% of the total population, and 1% of the tolerance intervals would cover less than 95% of the total population (ISO Guide 35). **Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.**

Table 5 below shows the INAA data determined on 20 x 85mg subsamples of OREAS 993. An equivalent scaled version of the results is also provided to demonstrate an appreciation of what this data means if 15g fire assay determinations were undertaken without the normal measurement error associated with this methodology.

**Table 5. Neutron Activation Analysis of Au (in ppm) on 20 x 85mg subsamples and showing the equivalent results scaled to a 15g sample mass typical of fire assay determination.**

Replicate No	Au 85mg actual	Au 15g equivalent*
1	54.985	55.406
2	54.803	55.392
3	54.684	55.383
4	55.031	55.409
5	55.476	55.443
6	55.345	55.433
7	55.543	55.448
8	54.492	55.369
9	55.484	55.444
10	54.524	55.371
11	54.859	55.396
12	54.199	55.347
13	55.661	55.457
14	56.223	55.500
15	56.986	55.557
16	55.633	55.455
17	56.497	55.520
18	54.674	55.382
19	56.809	55.544
20	56.900	55.551
Mean	55.440	55.440
Median	55.410	55.438
Std Dev.	0.852	0.064
<b>Rel.Std.Dev.</b>	<b>1.54%</b>	<b>0.116%</b>

\*Results calculated for a 15g equivalent sample mass using the formula:  $x^{15g Eq} = \frac{(x^{INAA} - \bar{X}) \times RSD@15g}{RSD@85mg} + \bar{X}$

where  $x^{15g Eq}$  = equivalent result calculated for a 15g sample mass  
 $(x^{INAA})$  = raw INAA result at 85mg  
 $\bar{X}$  = mean of 85mg INAA results

The homogeneity of gold has been determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the sample aliquot is substantially reduced to a point where most of the variability in replicate assays should be due to inhomogeneity of the reference material and measurement error becomes negligible. In this instance a subsample weight of 85 milligrams was employed and the 1RSD of 0.116% calculated for a 15g fire assay sample (1.54% at 85mg weights) confirms the high level of gold homogeneity in OREAS 993.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 993 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PARTICIPATING LABORATORIES

1. ♦\*AH Knight, St Helens, Merseyside, UK
2. ♦\*AH Knight, Tianjin, China
3. \*ALS, Brisbane, QLD, Australia
4. \*ALS, Lima, Peru
5. \*ALS, Loughrea, Galway, Ireland
6. \*ALS, Perth, WA, Australia
7. \*ALS, Ulaanbaatar, Khan-Uul District, Mongolia
8. \*ALS, Vancouver, BC, Canada
9. \*ANSTO, Lucas Heights, NSW, Australia
10. \*Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
11. \*Bureau Veritas Geoanalytical, Adelaide, SA, Australia
12. \*Bureau Veritas Geoanalytical, Perth, WA, Australia
13. ♦Independent, Perth, WA, Australia
14. ♦\*Inspectorate (BV), Shanghai, Bao Shan District, China
15. ♦\*Inspectorate (BV), Witham, Essex, UK
16. \*Intertek Genalysis, Perth, WA, Australia
17. ♦\*Intertek LSI, Rotterdam, Zuid-Holland, Netherlands
18. \*Nagrom, Perth, WA, Australia
19. ♦\*New Gold, Kamloops, BC, Canada
20. ♦\*Newcrest Laboratory Services, Orange, NSW, Australia
21. \*SGS Australia Mineral Services, Perth, WA, Australia
22. \*SGS del Peru, Lima, Peru
23. \*SGS Mineral Services, Townsville, QLD, Australia
24. ♦\*SGS Nederland B.V., Spijkenisse, Zuid-Holland, Netherlands
25. ♦SRL, Perth, WA, Australia

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

**Please note: Above numbered alphabetical list of participating laboratories does not reflect the Lab ID numbering on the scatter plots below.**

Figure 1. Au by Fire Assay (Umpire) in OREAS 993

SPC.1416.CVOCuCON6 CRM.OREAS 993.5.Fire Assay Umpire.Au.Lab.200514.194427.SN

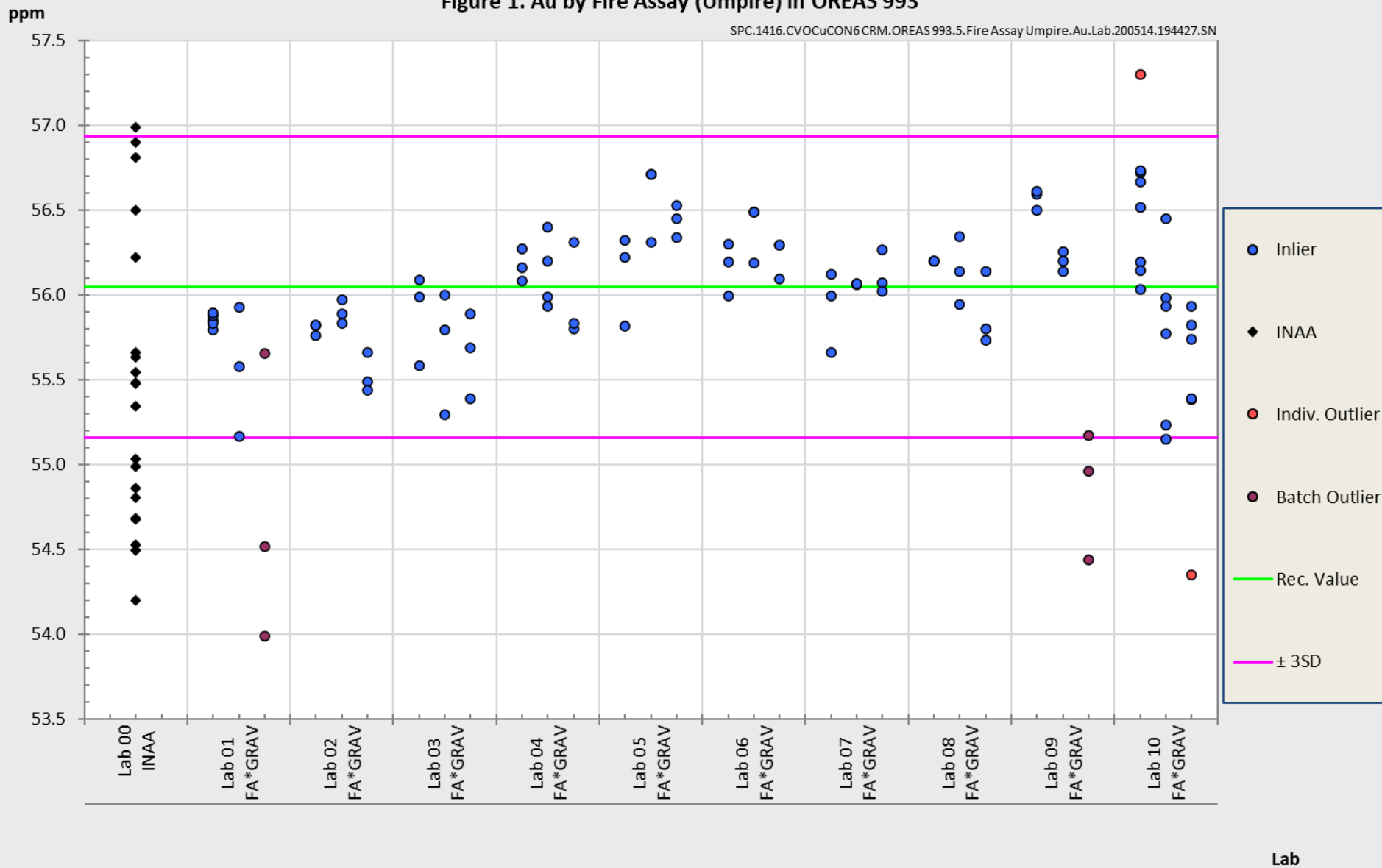
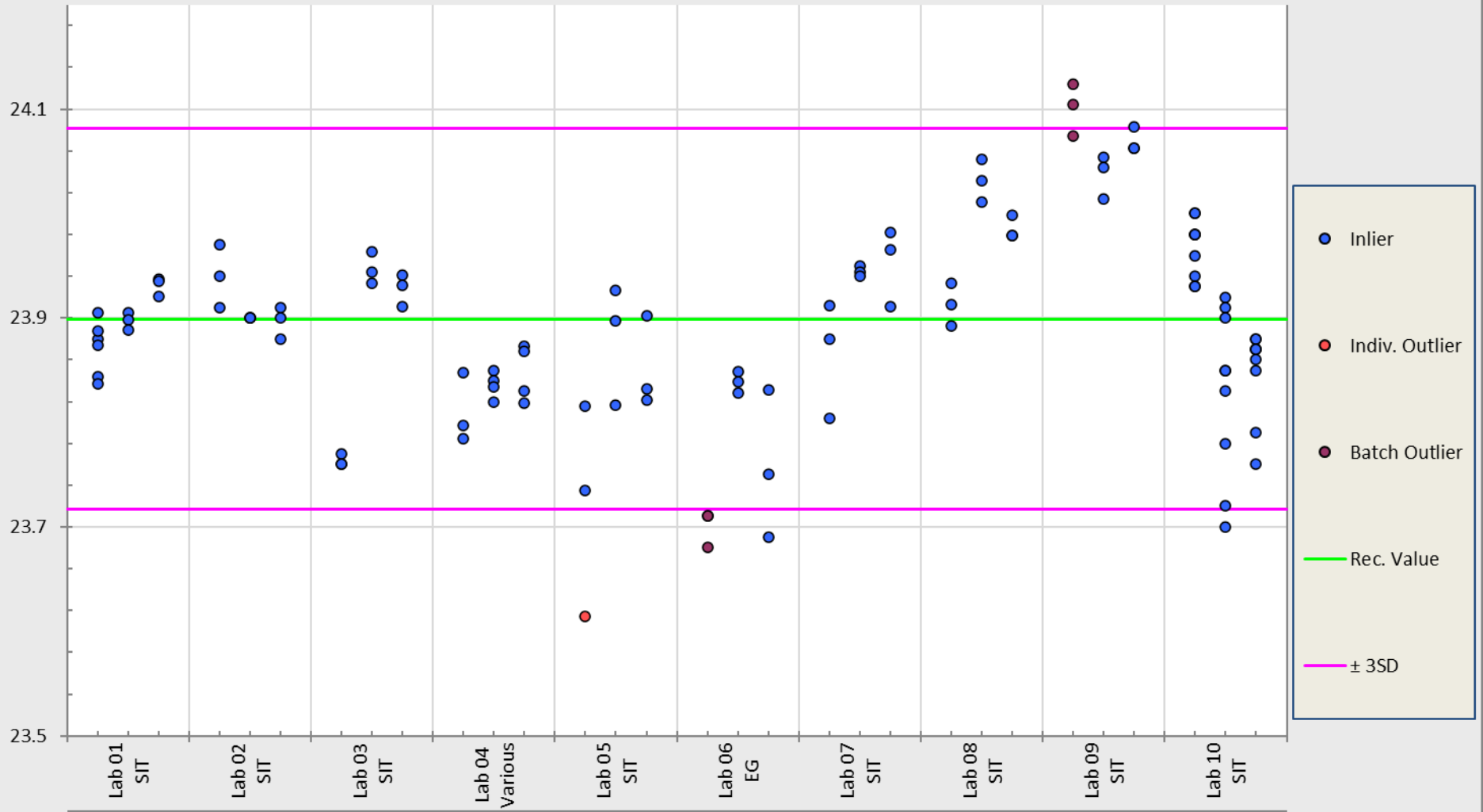


Figure 2. Cu by Classical in OREAS 993

SPC.1416.CVOCuCON6 CRM. OREAS 993.5.Classical.Cu.Lab.200514.194700.SN

wt. %



Lab

## PREPARER AND SUPPLIER

Certified reference material OREAS 993 was prepared, certified and supplied by:



ORE Research & Exploration Pty Ltd  
37A Hosie Street  
Bayswater North VIC 3153  
AUSTRALIA

Tel: +613-9729 0333  
Fax: +613-9729 8338  
Web: [www.ore.com.au](http://www.ore.com.au)  
Email: [info@ore.com.au](mailto:info@ore.com.au)

## METROLOGICAL TRACEABILITY

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

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

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

## COMMUTABILITY

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



## INTENDED USE

OREAS 993 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 993 may be used to calibrate the entire procedure by producing a pure substance CRM transformed into a calibration solution.

OREAS 993 is intended for the following uses:

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

## HANDLING INSTRUCTIONS

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

## STABILITY AND STORAGE INSTRUCTIONS

OREAS 993 was prepared from concentrate samples supplied by Newcrest Mining Limited's Cadia Valley Operations near Orange, New South Wales. To ensure a long shelf life it has been sealed under nitrogen in robust laminated foil pouches. In its unopened state under normal conditions of storage it has a shelf life beyond five years.

## INSTRUCTIONS FOR CORRECT USE

The 'umpire lab' Cu, Au and Ag certified values for OREAS 993 refer to the concentration levels on a dry sample basis. All analyses were performed on the samples after equilibration with the laboratory atmosphere for a minimum of 2 hours and 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, Au and Ag as per ISO 9599. The results were then corrected to dry basis using the moisture value. Moisture content varied amongst the laboratories from 0.24-0.34% with an average of 0.31% (excluding one lab which reported a mean of 0.63%).

The 'geoanalytical lab' certified values for OREAS 993 are on a 'sample as received' basis. The CRM should not be dried prior to analysis.

### Hg by aqua regia digestion

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

The 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.

### 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. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

## 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	22 <sup>nd</sup> May, 2020	First publication.

## QMS CERTIFICATION

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



## CERTIFYING OFFICER

22<sup>nd</sup> May, 2020

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

## REFERENCES

- Govett, G.J.S. (1983). Handbook of Exploration Geochemistry, Volume 2: Statistics and Data Analysis in Geochemical Prospecting (Variations of accuracy and precision).
- Ingamells, C. O. and Switzer, P. (1973). Talanta 20, 547-568.
- ISO Guide 30:2015. Terms and definitions used in connection with reference materials.
- ISO Guide 31:2015. Reference materials – Contents of certificates and labels.
- ISO Guide 35:2017. Certification of reference materials - General and statistical principals.
- ISO 16269:2014. Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals.
- ISO/TR 16476:2016, Reference Materials – Establishing and expressing metrological traceability of quantity values assigned to reference materials.
- Munsell Rock Color Book (2014). Rock-Color Chart Committee, Geological Society of America (GSA), 4300 44th Street SE, Grand Rapids, MI 49512.
- <sup>1</sup>NIST/SEMATECH Engineering Statistics Handbook (2005), Chapter 2.3.5.1. Control of Precision [online] <http://atomic.phys.uni-sofia.bg/local/nist-e-handbook/e-handbook/tool aids/pff/mpc.pdf> (page 227 of 483, accessed 22<sup>nd</sup> May, 2020).