

#### **CERTIFICATE OF ANALYSIS FOR**

# Copper Sulphide Ore (Tritton Cu Project, NSW) CERTIFIED REFERENCE MATERIAL OREAS 110



COA-724-OREAS110-R1 Printed: 03-Sep-2018

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 110.

Constituent	Certified Value		nfidence erval	Tolerance Interval 1-α=0.99, ρ=0.95		
		Low	High	Low	High	
Peroxide Fusion						
Silver, Ag (ppm)	<10	IND	IND	IND	IND	
Arsenic, As (ppm)	103	99	108	94	112	
Cadmium, Cd (ppm)	<2	IND	IND	IND	IND	
Cobalt, Co (ppm)	32	18	47	26	38	
Copper, Cu (wt.%)	0.160	0.155	0.164	0.150	0.170	
Iron, Fe (wt.%)	24.7	23.2	26.1	24.1	25.2	
Lead, Pb (ppm)	46	28	64	41	50	
Antimony, Sb (ppm)	16	12	20	16	16	
Zinc, Zn (ppm)	95	71	119	77	113	
Acid Digest						
Silver, Ag (ppm)	0.58	0.51	0.65	0.19	0.97	
Arsenic, As (ppm)	103	99	107	99	107	
Cadmium, Cd (ppm)	<0.5	IND	IND	IND	IND	
Cobalt, Co (ppm)	24	21	27	23	25	
Copper, Cu (wt.%)	0.162	0.159	0.166	0.156	0.168	
Iron, Fe (wt.%)	25.1	23.6	26.5	24.6	25.5	
Lead, Pb (ppm)	36.2	34.4	38.0	34.9	37.5	
Antimony, Sb (ppm)	15.2	14.4	16.1	14.2	16.3	
Zinc, Zn (ppm)	72.0	70.7	73.4	68.9	75.1	

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

Note: intervals may appear asymmetric due to rounding

Table 2. Indicative Values for OREAS 110.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Oxidising Fu	sion XRI	F						
Al <sub>2</sub> O <sub>3</sub>	wt.%	1.18	Fe <sub>2</sub> O <sub>3</sub>	wt.%	36.39	SnO <sub>2</sub>	ppm	12.7
As	ppm	130	K <sub>2</sub> O	wt.%	0.040	SO₃	wt.%	19.42
BaO	ppm	44.7	MgO	wt.%	0.570	SrO	ppm	177
CaO	wt.%	2.45	MnO	wt.%	0.034	TiO <sub>2</sub>	wt.%	0.049
CI	ppm	< 10	NiO	ppm	38.2	V <sub>2</sub> O <sub>5</sub>	ppm	107
CoO	ppm	31.8	P <sub>2</sub> O <sub>5</sub>	wt.%	0.203	ZnO	ppm	81
Cr <sub>2</sub> O <sub>3</sub>	ppm	36.5	PbO	ppm	32.3	ZrO <sub>2</sub>	ppm	27.0
CuO	ppm	1984	SiO <sub>2</sub>	wt.%	52.92			
Thermograv	imetry							
LOI <sup>1000</sup>	wt.%	5.50						
Laser Ablation	on ICP-M	S						
Ag	ppm	0.500	Hf	ppb	285	Sn	ppm	4.10
As	ppm	104	Но	ppb	125	Sr	ppm	128
Ва	ppm	35.5	In	ppm	< 0.05	Та	ppb	75.0
Be	ppm	1.30	La	ppm	3.09	Tb	ppb	65.0
Bi	ppm	0.72	Lu	ppb	60.0	Te	ppb	200
Cd	ppm	0.20	Мо	ppm	14.5	Th	ppm	4.01

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

Table 2. Indicative Values for OREAS 110 continued.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Laser Ablatic	on ICP-M	S						
Ce	ppm	4.82	Nb	ppm	0.87	TI	ppm	1.20
Со	ppm	23.2	Nd	ppm	2.28	Tm	ppb	55.0
Cr	ppm	31.5	Ni	ppm	22.0	U	ppm	7.34
Cs	ppm	0.39	Pb	wt.%	0.005	V	ppm	76
Cu	ppm	1640	Pr	ppm	0.54	W	ppm	25.7
Dy	ppm	0.53	Rb	ppm	1.58	Y	ppm	4.37
Er	ppm	0.38	Re	ppb	< 10	Yb	ppb	370
Eu	ppb	165	Sb	ppm	16.9	Zn	ppm	65
Ga	ppm	6.70	Sc	ppm	0.50	Zr	ppm	11.8
Gd	ppm	0.50	Se	ppm	< 5			
Ge	ppb	2925	Sm	ppm	0.66			
Infrared Con	nbustion							
С	wt.%	0.775	S	wt.%	7.59			

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

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

#### 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.

#### SOURCE MATERIAL

OREAS 110 is a low grade Cu ore certified reference material (CRM) prepared from material sourced from the Tritton Copper Project near Nyngan, New South Wales. The deposit consists of sulphide ore bodies (massive pyrite and chalcopyrite breccias) underlying oxide ores. The mineralisation is interpreted as stratiform "Besshi style" volcanic-hosted massive sulphide, within Ordovician turbidite metasediments and mafic volcanics. OREAS 110 is one of a suite of five CRMs and was prepared from jasper rock material containing about 10% pyrite. All five CRMs have been characterised for Ag, As, Cd, Co, Cu, Fe, Pb, Sb and Zn by 4-acid ICP and sodium peroxide fusion ICP methods.

#### COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- Drying at 65°C to constant mass;
- Crushing and screening;
- Multi-stage milling to 100% minus 35 microns;
- Final homogenisation;
- Packaging into 10g units sealed under nitrogen in laminated foil pouches.

#### ANALYITICAL PROGRAM

Ten commercial laboratories participated in the analytical program to characterise Aq. As. Cd, Co, Cu, Fe, Pb, Sb and Zn. Their results together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM3) are presented in Tables A2 and A19 (Appendix). The parameter PDM<sup>3</sup> is a measure of laboratory accuracy while the relative standard deviation is an effective measure of analytical precision where homogeneity of the test material has been confirmed.

The approximate major and trace element composition of OREAS 110 is provided in Table 2. The non-certified values contained in this table are the means of duplicate assays from one laboratory.

The analytical methods employed by each laboratory are explained, together with other abbreviations used, in Table A1 (Appendix).

Each participating laboratory received 5 samples of 30g each. Each set of subsamples submitted to each laboratory was taken at regular intervals during packaging of the standard in order to maximise their representation. All ten laboratories reported 4-acid data for the requested elements while eight included sodium peroxide fusion results. Laboratories were instructed to assay samples as received.

#### STATISTICAL EVALUATION

#### **Certified Value and Confidence Intervals**

The certified value is the mean of means of accepted replicate values of accepted participating laboratories computed according to the formulae

$$\overline{x}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} x_{ij} \qquad \qquad \dot{x} = \frac{1}{p} \sum_{i=1}^{p} \overline{x}_i$$

where,

 $x_{ii}$  is the jth result reported by laboratory i; p is the number of participating laboratories;  $n_i$  is the number of results reported by laboratory i;

 $\bar{x}_i$  is the mean for laboratory i;

 $\ddot{x}$  is the mean of means.



The confidence intervals are obtained by calculation of the variance ( $\hat{V}$ ) of the consensus value ( $\hat{x}$ ) (mean of means) and reference to Student's-*t* distribution with degrees of freedom (*p*-1).

$$\hat{V}(\ddot{x}) = \frac{1}{p(p-1)} \sum_{i=1}^{p} (\overline{x}_i - \ddot{x})^2$$

Confidence Interval = 
$$\ddot{x} \pm t_{1-x/2}(p-1)(\hat{V}(\ddot{x}))^{1/2}$$

where,

 $t_{1-x/2}(p-1)$  is the 1-x/2 fractile of the t-distribution with (p-1) degrees of freedom.

The distribution of the values is assumed to be symmetrical about the mean in the calculation of the confidence interval. The test for rejection of individual outliers from each laboratory data set is based on z scores (rejected if  $|z_i| > 2.5$ ) computed from the robust estimators of location and scale, T and S, respectively, according to the formulae

$$S = 1.483 \text{ median } / x_j - \text{ median } (x_i) / i = 1....n$$

$$z_i = \frac{x_i - T}{S}$$

where,

*T is the median value in a data set;* 

S is the median of all absolute deviations from the sample median multiplied by 1.483, a correction factor to make the estimator consistent with the usual parameter of a normal distribution.

The z-score test is used in combination with a second method of individual outlier detection that determines the percent deviation of the individual value from the median. Outliers in general are selected on the basis of z-scores > 2.5 and with percent deviations > 1.5%. In certain instances statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set is tested for outlying status based on z-score discrimination and rejected if  $|z_i| > 2.5$ . After individual and lab data set 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.

Individual outliers and, more rarely, laboratory means deemed to be outlying are shown left justified and in bold in the tabulated results (see Appendix) and have been omitted in the determination of certified values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It is a measure of the reliability of the certified value, i.e. the narrower the confidence interval the greater the certainty in the certified value. A 95% confidence interval indicates a 95% probability that the interval includes the true value of the analyte under consideration.

#### Indicative (uncertified) values

The indicative (uncertified) values (Table 2) are provided for the major and trace elements determined by oxidising fusion XRF (Al<sub>2</sub>O<sub>3</sub> to ZrO<sub>2</sub>), LOI at 1000°C and laser ablation with ICP-MS (Ag to Zr) and are the means of duplicate assays from Bureau Veritas, Perth.

Additional indicative values by other analytical methods are present where the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification or where inter-laboratory consensus is poor.

#### Statement of Homogeneity

The standard deviation of each laboratory data set includes error due to both the imprecision of the analytical method employed and to possible inhomogeneity of the material analysed. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material analysed and, in particular, to deficiencies in accuracy of each analytical method.

In determining tolerance intervals that component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \overline{x}_i + \frac{\sum_{i=1}^{p} \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^{p} n_i}$$

where,

 $x_{ij}$  is the jth raw result reported by laboratory i;  $x'_{ij}$  is the jth transformed result reported by laboratory i;  $n_i$  is the number of results reported by laboratory i; p is the number of participating laboratories;  $\overline{x}_i$  is the raw mean for laboratory i.

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

Lower limit is 
$$\ddot{x} - k'_2(n, p, l - \alpha)s''_g$$
  
Upper limit is  $\ddot{x} + k'_2(n, p, l - \alpha)s''_g$ 

where,

n is the number of results;  $1-\alpha$  is the confidence level; p is the proportion of results expected within the tolerance limits;  $k'_2$  is the factor for two – sided tolerance limits  $(m, \alpha \ unknown)$ ;  $s''_g$  is the corrected grand s  $\tan \alpha$  deviation.

The meaning of these tolerance limits may be illustrated for Cu by 4-acid digest, where 99% of the time at least 95% of subsamples will have concentrations lying between 0.156 and 0.168 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).

The corrected grand standard deviation,  $s_g$ , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula:

$$s''_{g} = \frac{\sum_{i=1}^{p} (s_{i}(1 - \frac{S_{i}}{S'_{g}}))}{\sum_{i=1}^{p} (1 - \frac{S_{i}}{S'_{g}})}$$

where,

$$1 - (\frac{s_i}{2s'_g})$$
 is the weighting factor for laboratory  $i$ ;

 $s'_{e}$  is the grand standard deviation computed from the transformed (i.e. means -adjusted) results

according to the formula

$$s'_{g} = \left[ \frac{\sum_{i=j}^{p} \sum_{j=i}^{n_{i}} (x'_{ij} - \overline{x}'_{i})^{2}}{\sum_{i=1}^{p} n_{i} - I} \right]^{1/2}$$

where  $\overline{x}'_i$  is the transformed mean for laboratorty i

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error.

#### **Performance Gates**

Performance gates 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 and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. Sources of measurement error include inter-lab bias, analytical precision (repeatability) and inter-batch bias (reproducibility).

Two methods have been employed to calculate performance gates. The first method uses the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers. 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 individual analyses generated from the certification program. Table 3 shows performance gates 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. 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.

Table 3. Performance Gates for OREAS 110.

Constituent	Certified	1SD	2SD v	vindow	3SD v	vindow	Relative	Standard D	eviations	5% w	indow
Constituent	Value	130	Low	High	Low	High	1RSD	2RSD	3RSD	Low	High
Peroxide Fusion											
Silver, Ag (ppm)	<10	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Arsenic, As (ppm)	103	10	83	124	73	134	9.93%	19.9%	29.8%	98	108
Cadmium, Cd (ppm)	<2	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cobalt, Co (ppm)	32	8	16	49	7	57	25.6%	51.2%	76.7%	31	34
Copper, Cu (wt.%)	0.160	0.005	0.149	0.170	0.144	0.175	3.20%	6.39%	9.59%	0.152	0.168
Iron, Fe (wt.%)	24.7	1.7	21.2	28.2	19.4	29.9	7.08%	14.2%	21.3%	23.4	25.9
Lead, Pb (ppm)	46	11	23	68	12	79	24.5%	49.1%	73.6%	43	48
Antimony, Sb (ppm)	16	2	11	21	9	23	14.5%	29.1%	43.6%	15	17
Zinc, Zn (ppm)	95	23	48	142	24	165	24.7%	49.5%	74.2%	90	100
Acid Digest											
Silver, Ag (ppm)	0.58	0.09	0.39	0.76	0.30	0.86	15.9%	31.9%	47.8%	0.55	0.61
Arsenic, As (ppm)	103	6	91	115	86	120	5.62%	11.2%	16.8%	98	108
Cadmium, Cd (ppm)	<0.5	IND	IND	IND	IND	IND	IND	IND	IND	IND	IND
Cobalt, Co (ppm)	24	4	15	32	11	36	17.6%	35.1%	52.7%	23	25
Copper, Cu (wt.%)	0.162	0.006	0.151	0.174	0.145	0.180	3.52%	7.05%	10.6%	0.154	0.171
Iron, Fe (wt.%)	25.1	1.9	21.3	28.9	19.3	30.8	7.61%	15.2%	22.8%	23.8	26.3
Lead, Pb (ppm)	36.2	2.4	31.5	40.9	29.2	43.3	6.49%	13.0%	19.5%	34.4	38.0
Antimony, Sb (ppm)	15.2	1.0	13.2	17.3	12.2	18.3	6.76%	13.5%	20.3%	14.5	16.0
Zinc, Zn (ppm)	72.0	3.2	65.7	78.3	62.6	81.5	4.38%	8.76%	13.1%	68.4	75.6

SI unit equivalents: ppm, parts per million  $\equiv$  mg/kg  $\equiv$   $\mu$ g/g  $\equiv$  0.0001 wt.%  $\equiv$  1000 ppb, parts per billion. Note: intervals may appear asymmetric due to rounding

#### PARTICIPATING LABORATORIES

- 1. Acme Analytical Laboratories Ltd, Vancouver, BC, Canada
- 2. Activation Laboratories, Ancaster, ONtario, Canada
- 3. ALS Chemex, Brisbane, QLD, Australia
- 4. ALS Chemex, Vancouver, BC, Canada
- 5. Amdel Laboratories, Perth, WA, Australia
- 6. Bureau Veritas (Ultra Trace) Geoanalytical, Perth, WA, Australia
- 7. Genalysis Laboratory Services Pty Ltd, Perth, WA, Australia
- 8. Intertek Testing Services, Jakarta, Indonesia
- 9. OMAC Laboratories Ltd, Loughrea, County Galway, Ireland
- 10. SGS Australia, Perth, WA, Australia

#### PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 110 has been prepared and certified by:



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OREAS 110 available in 10g units sealed under a nitrogen environment in laminated foil pouches.

#### **INTENDED USE**

OREAS 110 is a reference material intended for the following:

- i) For the monitoring of laboratory performance in the analysis of Ag, As, Cd, Co, Cu, Fe, Pb, Sb and Zn in geological samples;
- ii) For the calibration of instruments used in the determination of the concentration of Ag, As, Cd, Co, Cu, Fe, Pb, Sb and Zn;
- iii) For the verification of analytical methods for Ag, As, Cd, Co, Cu, Fe, Pb, Sb and Zn.

#### STABILITY AND STORAGE INSTRUCTIONS

OREAS 110 is a reference material made from medium grade copper sulphide ore from the Tritton Copper Mine. In its unopened state in the nitrogen-purged laminated foil pouches and under normal conditions of storage it has a shelf life beyond five years.

#### INSTRUCTIONS FOR THE CORRECT USE

The certified values for OREAS 110 refer to the concentration level of Ag, As, Cd, Co, Cu, Fe, Pb, Sb and Zn in its packaged state. The CRM should not be dried prior to weighing and analysis.

#### HANDLING INSTRUCTIONS

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

### **METROLOGICAL TRACEABILITY**

The analytical samples were selected in a manner to represent the entire batch of 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.

#### **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
1	3 <sup>rd</sup> Sep, 2018	Added major and trace element characterisation
0	7 <sup>th</sup> Aug, 2012	First publication

## **QMS ACCREDITED**

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





#### **CERTIFYING OFFICER**

8/

3<sup>rd</sup> Sep, 2018

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

#### **REFERENCES**

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

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.

# APPENDIX Analytical Data for OREAS 110

Table A1. Explanation of abbreviations used in Tables A2 – A11.

Abbreviation	Explanation
Std.Dev.	one standard deviation
Rel.Std.Dev.	one relative standard deviation (%)
PDM <sup>3</sup>	percent deviation of lab mean from corrected mean of means
NR	not reported
4A	four acid digest (HF-HNO <sub>3</sub> -HClO <sub>4</sub> -HCl)
MAR	modified aqua regia digest
PF	sodium peroxide fusion
AAS	atomic absorption spectrometry
OES	inductively coupled plasma optical emission spectrometry
MS	inductively coupled plasma mass spectrometry

Table A2. Fusion results for Ag in OREAS 110 (abbreviations as in Table A1; values in ppm).

				,			,		1 /	
Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	PF*MS	PF*MS	PF*MS	-	•	-	-	PF*MS	-
1	< 10	<5	NR	7.0	NR	NR	NR	NR	<10	NR
2	< 10	<5	NR	7.0	NR	NR	NR	NR	<10	NR
3	< 10	<5	NR	6.0	NR	NR	NR	NR	<10	NR
4	< 10	<5	NR	6.0	NR	NR	NR	NR	<10	NR
5	< 10	<5	NR	7.0	NR	NR	NR	NR	<10	NR
Mean				6.6						
Median				7.0						
Std.Dev.				0.5						
Rel.Std.Dev.				8.30%						
PDM <sup>3</sup>										

Table A3. Fusion results for As in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Ε	F	G	Н	1	J
	PF*OES	PF*MS	PF*MS	PF*MS	-	-	PF*OES		PF*MS	-
1	105	105	106	100	NR	NR	100	NR	53	NR
2	82	100	98	90	NR	NR	100	NR	59	NR
3	134	115	104	100	NR	NR	100	NR	55	NR
4	156	110	97	100	NR	NR	100	NR	51	NR
5	95	115	102	120	NR	NR	100	NR	53	NR
Mean	114	109	101	102			100		54	
Median	105	110	102	100			100		53	
Std.Dev.	30	7	4	11			0		3	
Rel.Std.Dev.	26.3%	5.98%	3.79%	10.7%			0.00%		5.60%	
PDM <sup>3</sup>	10.8%	5.54%	-1.82%	-1.24%			-3.18%		-47.5%	

Table A4. Fusion results for Cd in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	PF*MS	PF*MS	PF*MS	-	-	-	-	PF*MS	-
1	< 2	<10	NR	NR	NR	NR	NR	NR	<50	NR
2	< 2	<10	NR	0.50	NR	NR	NR	NR	<50	NR
3	< 2	<10	NR	1.20	NR	NR	NR	NR	<50	NR
4	< 2	<10	NR	NR	NR	NR	NR	NR	<50	NR
5	< 2	<10	NR	NR	NR	NR	NR	NR	<50	NR
Mean				0.85						
Median				0.85						
Std.Dev.				0.49						
Rel.Std.Dev.				58.23%						
PDM <sup>3</sup>										

Table A5. Fusion results for Co in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	PF*OES	NR	PF*MS	-	-	PF*OES	-	PF*OES	-
1	22	40	NR	27	NR	NR	40	NR	NR	NR
2	22	40	NR	26	NR	NR	40	NR	NR	NR
3	22	40	NR	25	NR	NR	40	NR	NR	NR
4	23	40	NR	28	NR	NR	40	NR	NR	NR
5	23	40	NR	35	NR	NR	40	NR	NR	NR
Mean	22	40		28			40			
Median	22	40		27			40			
Std.Dev.	1	0		4			0			
Rel.Std.Dev.	2.45%	0.00%		14.1%			0.00%			
PDM <sup>3</sup>	-30.5%	24.1%		-12.5%			24.1%			

Table A6. Fusion results for Cu in OREAS 110 (abbreviations as in Table A1; values in wt.%).

Replicate	Lab	Lab	Lab	Lab						
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	-	PF*OES	-						
1	0.154	0.154	0.169	0.162	0.140	0.160	0.159	NR	0.357	NR
2	0.155	0.158	0.163	0.153	0.140	0.160	0.154	NR	0.338	NR
3	0.157	0.162	0.169	0.150	0.150	0.160	0.161	NR	0.323	NR
4	0.163	0.160	0.168	0.152	0.160	0.160	0.164	NR	0.351	NR
5	0.161	0.158	0.171	0.165	0.140	0.160	0.159	NR	0.349	NR
Mean	0.158	0.158	0.168	0.156	0.146	0.160	0.159		0.343	
Median	0.157	0.158	0.169	0.153	0.140	0.160	0.159		0.349	
Std.Dev.	0.004	0.003	0.003	0.007	0.009	0.000	0.004		0.014	
Rel.Std.Dev.	2.45%	1.87%	1.68%	4.26%	6.13%	0.00%	2.29%		3.98%	
PDM <sup>3</sup>	-1.02%	-0.77%	5.07%	-2.02%	-8.54%	0.23%	-0.14%		115%	

Table A7. Fusion results for Fe in OREAS 110 (abbreviations as in Table A1; values in wt.%).

Replicate	Lab	Lab	Lab	Lab						
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	-	PF*OES	-						
1	27.3	26.8	25.8	25.8	21.5	24.0	24.4	NR	22.9	NR
2	26.8	26.4	25.5	24.5	21.8	24.0	24.4	NR	23.1	NR
3	26.3	27.0	26.0	23.9	22.0	24.4	24.5	NR	23.4	NR
4	26.4	26.5	25.4	24.5	22.3	23.8	24.6	NR	23.1	NR
5	27.3	27.1	26.2	26.9	20.9	23.7	24.5	NR	21.8	NR
Mean	26.8	26.8	25.8	25.1	21.7	24.0	24.5		22.9	
Median	26.8	26.8	25.8	24.5	21.8	24.0	24.5		23.1	
Std.Dev.	0.5	0.3	0.3	1.2	0.5	0.3	0.1		0.6	
Rel.Std.Dev.	1.78%	1.14%	1.29%	4.83%	2.52%	1.12%	0.34%		2.67%	
PDM <sup>3</sup>	8.75%	8.50%	4.52%	1.85%	-12.0%	-2.77%	-0.74%		-7.31%	

Table A8. Fusion results for Pb in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	- 1	J
	PF*OES	PF*MS	PF*MS	PF*MS	-	-	PF*OES	-	PF*OES	-
1	37.0	40.0	51.0	54.0	NR	NR	<100	NR	NR	NR
2	37.0	30.0	51.0	61.0	NR	NR	<100	NR	NR	NR
3	36.0	40.0	50.0	47.0	NR	NR	<100	NR	NR	NR
4	36.0	40.0	54.0	69.0	NR	NR	<100	NR	NR	NR
5	37.0	30.0	63.0	62.0	NR	NR	<100	NR	NR	NR
Mean	36.6	36.0	53.8	58.6						
Median	37.0	40.0	51.0	61.0						
Std.Dev.	0.5	5.5	5.4	8.4						
Rel.Std.Dev.	1.50%	15.2%	9.96%	14.3%						
PDM <sup>3</sup>	-19.9%	-21.2%	17.8%	28.3%						

Table A9. Fusion results for Sb in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	PF*MS	PF*MS	PF*MS	-	-	-	-	PF*MS	-
1	17.0	15.0	16.2	20.9	NR	NR	NR	NR	<10	NR
2	18.0	15.0	16.4	19.8	NR	NR	NR	NR	12.0	NR
3	16.0	15.0	15.5	19.4	NR	NR	NR	NR	12.0	NR
4	17.0	15.0	16.4	19.6	NR	NR	NR	NR	<10	NR
5	17.0	15.0	16.3	23.2	NR	NR	NR	NR	<10	NR
Mean	17.0	15.0	16.2	20.6					12.0	
Median	17.0	15.0	16.3	19.8					12.0	
Std.Dev.	0.7	0.0	0.4	1.6					0.0	
Rel.Std.Dev.	4.16%	0.00%	2.34%	7.66%					0.00%	
PDM <sup>3</sup>	5.9%	-6.54%	0.69%	28.2%					-25.2%	

Table A10. Fusion results for Zn in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	PF*OES	PF*OES	PF*OES	PF*OES	-	PF*OES	PF*OES	-	PF*OES	-
1	80.0	80.0	94.0	130.0	NR	<100	<100	NR	109.0	NR
2	70.0	80.0	85.0	130.0	NR	100.0	<100	NR	96.0	NR
3	70.0	80.0	78.0	140.0	NR	<100	<100	NR	100.0	NR
4	70.0	80.0	86.0	140.0	NR	<100	<100	NR	103.0	NR
5	70.0	60.0	83.0	130.0	NR	100.0	<100	NR	110.0	NR
Mean	72.0	76.0	85.2	134.0		100.0			103.6	
Median	70.0	80.0	85.0	130.0		100.0			103.0	
Std.Dev.	4.5	8.9	5.8	5.5		0.0			5.9	
Rel.Std.Dev.	6.21%	11.8%	6.81%	4.09%		0.00%			5.73%	
PDM <sup>3</sup>	-24.0%	-19.8%	-10.1%	41.4%		5.52%			9.32%	

Table A11. 4-acid results for Ag in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*MS	4A*MS	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	0.54	0.50	0.60	NR	0.50	0.50	<0.5	0.50	<1	0.90
2	1.53	0.50	0.80	NR	0.50	0.60	<0.5	0.60	<1	1.04
3	0.50	0.50	0.60	NR	0.50	0.80	<0.5	0.60	<1	0.82
4	0.68	0.50	0.60	NR	0.50	0.70	<0.5	0.60	<1	0.95
5	0.75	0.50	0.60	NR	0.50	0.60	<0.5	0.60	<1	0.93
Mean	0.80	0.50	0.64		0.50	0.64		0.58		0.93
Median	0.68	0.50	0.60		0.50	0.60		0.60		0.93
Std.Dev.	0.42	0.00	0.09		0.00	0.11		0.04		0.08
Rel.Std.Dev.	52.6%	0.00%	14.0%		0.00%	17.8%		7.71%		8.51%
PDM <sup>3</sup>	38.0%	-13.7%	10.4%		-13.7%	10.4%		0.07%		60.2%

Table A12. 4-acid results for As in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*MS	4A*MS	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	140	105	101	112	103	92	111	104	101	102
2	130	110	100	113	108	96	108	106	97	104
3	120	105	97	106	110	97	102	100	99	99
4	110	105	96	108	111	89	103	105	98	102
5	120	105	97	111	106	93	103	108	96	102
Mean	124	106	98	110	108	93	105	105	98	102
Median	120	105	97	111	108	93	103	105	98	102
Std.Dev.	11	2	2	3	3	3	4	3	2	2
Rel.Std.Dev.	9.19%	2.11%	2.21%	2.65%	2.98%	3.44%	3.71%	2.84%	1.92%	1.73%
PDM <sup>3</sup>	20.4%	2.94%	-4.63%	6.83%	4.49%	-9.30%	2.36%	1.58%	-4.96%	-1.13%

Table A13. 4-acid results for Cd in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*MS	4A*MS	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	<10	<0.5	0.10	0.30	0.30	<0.5	<0.5	<1	<2	<1
2	<10	<0.5	0.10	0.20	0.30	<0.5	<0.5	<1	<2	<1
3	<10	<0.5	0.20	0.10	0.30	<0.5	<0.5	<1	<2	<1
4	<10	<0.5	0.10	0.10	0.30	<0.5	<0.5	<1	<2	<1
5	<10	<0.5	0.20	0.40	0.30	<0.5	<0.5	<1	<2	<1
Mean			0.14	0.22	0.30					
Median			0.10	0.20	0.30					
Std.Dev.			0.05	0.13	0.00					
Rel.Std.Dev.			39.1%	59.3%	0.00%					
PDM <sup>3</sup>			-36.4%	0.00%	36.4%					

Table A14. 4-acid results for Co in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*MS	4A*OES	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	30.0	25.0	22.5	25.0	24.2	19.0	21.0	41.0	25.1	16.2
2	30.0	30.0	22.5	25.8	25.0	19.0	20.0	42.0	25.4	18.3
3	30.0	30.0	22.1	23.7	26.1	19.0	19.0	41.0	25.5	20.8
4	30.0	30.0	21.6	24.8	25.8	17.0	18.0	44.0	25.1	20.4
5	30.0	30.0	22.1	25.2	25.8	18.0	22.0	43.0	24.8	19.5
Mean	30.0	29.0	22.2	24.9	25.4	18.4	20.0	42.2	25.2	19.0
Median	30.0	30.0	22.1	25.0	25.8	19.0	20.0	42.0	25.1	19.5
Std.Dev.	0.0	2.2	0.4	0.8	0.8	0.9	1.6	1.3	0.3	1.9
Rel.Std.Dev.	0.00%	7.71%	1.68%	3.08%	3.06%	4.86%	7.91%	3.09%	1.11%	9.74%
PDM <sup>3</sup>	26.0%	21.8%	-6.95%	4.56%	6.57%	-22.7%	-16.0%	77.2%	5.67%	-20.1%

Table A15. 4-acid results for Cu in OREAS 110 (abbreviations as in Table A1; values in wt.%).

Replicate	Lab									
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*OES	4A*OES	4A*OES	4A*AAS	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	0.172	0.154	0.166	0.162	0.151	0.160	0.160	0.166	0.167	0.167
2	0.165	0.160	0.163	0.157	0.154	0.166	0.167	0.164	0.160	0.167
3	0.172	0.157	0.162	0.143	0.157	0.161	0.155	0.163	0.163	0.166
4	0.174	0.157	0.171	0.157	0.155	0.155	0.161	0.170	0.163	0.165
5	0.175	0.159	0.168	0.163	0.153	0.160	0.162	0.166	0.160	0.167
Mean	0.172	0.157	0.166	0.156	0.154	0.160	0.161	0.166	0.162	0.166
Median	0.172	0.157	0.166	0.157	0.154	0.160	0.161	0.166	0.163	0.167
Std.Dev.	0.004	0.002	0.004	0.008	0.002	0.004	0.004	0.003	0.003	0.001
Rel.Std.Dev.	2.28%	1.46%	2.35%	5.11%	1.56%	2.44%	2.57%	1.62%	1.63%	0.70%
PDM <sup>3</sup>	5.65%	-3.10%	2.18%	-3.71%	-5.33%	-1.25%	-1.00%	2.08%	-0.02%	2.44%

Table A16. 4-acid results for Fe in OREAS 110 (abbreviations as in Table A1; values in wt.%).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	I	J
	4A*MS	4A*OES	4A*OES	4A*OES	4A*MS	4A*AAS	4A*OES	4A*OES	4A*OES	4A*OES
1	26.4	26.4	27.1	27.5	23.6	21.4	22.6	27.1	25.2	24.8
2	25.8	26.7	27.2	26.6	24.0	22.4	22.8	26.5	24.1	24.9
3	26.3	26.8	26.8	25.1	24.0	21.7	21.6	26.7	24.5	24.7
4	26.5	26.7	26.4	26.8	23.5	21.0	22.3	26.8	24.3	24.6
5	26.7	26.6	27.0	27.3	23.9	21.5	22.6	27.7	24.1	24.9
Mean	26.3	26.6	26.9	26.7	23.8	21.6	22.4	27.0	24.4	24.8
Median	26.4	26.7	27.0	26.8	23.9	21.5	22.6	26.8	24.3	24.8
Std.Dev.	0.3	0.2	0.3	0.9	0.3	0.5	0.5	0.5	0.5	0.1
Rel.Std.Dev.	1.28%	0.57%	1.21%	3.54%	1.08%	2.38%	2.11%	1.73%	1.95%	0.44%
PDM <sup>3</sup>	5.08%	6.28%	7.35%	6.36%	-5.09%	-13.8%	-10.7%	7.56%	-2.60%	-1.13%

Table A17. 4-acid results for Pb in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	I	J
	4A*MS	4A*MS	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	40.0	33.0	39.0	34.0	37.4	56.0	35.0	20.0	35.9	36.3
2	40.0	33.0	36.0	34.0	37.6	45.0	31.0	17.0	34.8	36.9
3	40.0	33.0	35.0	32.0	37.0	38.0	29.0	14.0	35.3	37.7
4	40.0	34.0	36.0	33.0	38.2	40.0	35.0	20.0	33.8	38.0
5	40.0	33.0	35.0	34.0	36.4	38.0	35.0	20.0	34.9	36.7
Mean	40.0	33.2	36.2	33.4	37.3	43.4	33.0	18.2	34.9	37.1
Median	40.0	33.0	36.0	34.0	37.4	40.0	35.0	20.0	34.9	36.9
Std.Dev.	0.0	0.4	1.6	0.9	0.7	7.6	2.8	2.7	0.8	0.7
Rel.Std.Dev.	0.00%	1.35%	4.54%	2.68%	1.80%	17.5%	8.57%	14.7%	2.20%	1.93%
PDM <sup>3</sup>	10.5%	-8.30%	-0.01%	-7.75%	3.08%	19.9%	-8.85%	-49.7%	-3.52%	2.54%

Table A18. 4-acid results for Sb in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab	Lab
No.	Α	В	С	D	Е	F	G	Н	I	J
	4A*MS	4A*MS	4A*MS	4A*OES	4A*MS	4A*OES	4A*OES	4A*OES	4A*OES	4A*OES
1	30.0	15.0	15.0	16.6	15.9	22.0	16.0	21.0	13.7	15.6
2	10.0	14.2	15.2	16.0	17.0	23.0	17.0	19.0	12.8	14.3
3	10.0	14.4	15.4	14.2	16.5	24.0	9.0	21.0	13.9	15.3
4	10.0	13.6	14.9	15.8	17.3	21.0	14.0	20.0	14.4	14.7
5	10.0	14.6	15.5	16.7	15.6	25.0	16.0	17.0	13.5	15.5
Mean	14.0	14.4	15.2	15.9	16.5	23.0	14.4	19.6	13.6	15.1
Median	10.0	14.4	15.2	16.0	16.5	23.0	16.0	20.0	13.7	15.3
Std.Dev.	8.9	0.5	0.2	1.0	0.7	1.6	3.2	1.7	0.6	0.6
Rel.Std.Dev.	63.9%	3.61%	1.54%	6.33%	4.35%	6.87%	22.3%	8.54%	4.33%	3.72%
PDM <sup>3</sup>	-8.18%	-5.82%	-0.52%	4.02%	7.95%	50.8%	-5.56%	28.5%	-10.5%	-1.12%

Table A19. 4-acid results for Zn in OREAS 110 (abbreviations as in Table A1; values in ppm).

Replicate	Lab	Lab								
No.	Α	В	С	D	Е	F	G	Н	1	J
	4A*OES	MAR*OES	4A*OES							
1	80.0	72.0	72.0	75.0	75.0	74.0	72.0	64.0	71.4	62.7
2	70.0	72.0	69.0	74.0	78.0	79.0	74.0	73.0	70.4	62.6
3	70.0	76.0	70.0	67.0	75.0	74.0	69.0	69.0	71.6	63.1
4	70.0	74.0	74.0	74.0	78.0	67.0	72.0	72.0	70.1	63.0
5	70.0	72.0	73.0	77.0	79.0	67.0	73.0	71.0	68.1	63.1
Mean	72.0	73.2	71.6	73.4	77.0	72.2	72.0	69.8	70.3	62.9
Median	70.0	72.0	72.0	74.0	78.0	74.0	72.0	71.0	70.4	63.0
Std.Dev.	4.5	1.8	2.1	3.8	1.9	5.2	1.9	3.6	1.4	0.2
Rel.Std.Dev.	6.21%	2.44%	2.90%	5.15%	2.43%	7.16%	2.60%	5.11%	1.98%	0.32%
PDM <sup>3</sup>	-0.02%	1.65%	-0.58%	1.92%	6.92%	0.26%	-0.02%	-3.07%	-2.36%	-12.6%