

**CERTIFICATE OF ANALYSIS FOR**

**Au-Cu-Mo-Ag-S ORE REFERENCE MATERIAL**

**OREAS 504**

**Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 504**

Method / Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fire Assay</b>						
Gold, Au (ppm)	1.48	0.04	1.46	1.50	1.47*	1.48*
<b>4-Acid Digestion</b>						
Copper, Cu (wt.%)	1.137	0.032	1.123	1.151	1.115	1.159
Molybdenum, Mo (ppm)	643	37.1	625	661	629	657
Silver, Ag (ppm)	3.13	0.21	3.01	3.24	2.99	3.26
Sulphur, S (wt.%)	1.37	0.048	1.35	1.40	1.34	1.41
<b>Aqua Regia Digestion</b>						
Copper, Cu (wt.%)	1.123	0.019	1.117	1.130	1.102	1.144
Gold, Au (ppm)	1.47	0.07	1.42	1.51	1.41	1.52
Molybdenum, Mo (ppm)	624	36.1	607	641	613	635
Sulphur, S (wt.%)	1.37	0.090	1.32	1.41	1.34	1.39

\*Tolerance Limits calculated from 20 x INAA analyses on 0.5g subsamples; Note: intervals may appear asymmetric due to rounding.

**Table 2. Indicative values for major and trace elements in OREAS 504**

Method / Constituent	Unit	Value	Method / Constituent	Unit	Value	Method / Constituent	Unit	Value
<b>Borate Fusion XRF</b>								
Al <sub>2</sub> O <sub>3</sub>	wt.%	13.2	MgO	wt.%	3.2	SiO <sub>2</sub>	wt.%	55.0
CaO	wt.%	4.2	MnO	wt.%	0.08	TiO <sub>2</sub>	wt.%	0.58
Fe <sub>2</sub> O <sub>3</sub>	wt.%	11.6	Na <sub>2</sub> O	wt.%	2.9			
K <sub>2</sub> O	wt.%	3.5	P <sub>2</sub> O <sub>5</sub>	wt.%	0.24			
LOI	wt.%	3.6	S	wt.%	1.4			
<b>Four Acid Digestion ICP-OES/MS</b>								
Al	wt.%	8.2	Ho	ppm	0.6	Sm	ppm	2.9
As	ppm	6.5	In	ppm	0.7	Sn	ppm	11
Ba	ppm	604	K	wt.%	3.1	Sr	ppm	509
Be	ppm	1.1	La	ppm	13	Ta	ppm	0.4
Bi	ppm	5.0	Li	ppm	23	Tb	ppm	0.5
Ca	wt.%	3.2	Lu	ppm	0.2	Te	ppm	0.4
Cd	ppm	1.0	Mg	wt.%	2.2	Th	ppm	4.1
Ce	ppm	25	Mn	wt.%	0.07	Ti	wt.%	0.36
Co	ppm	22	Nb	ppm	6.8	Tl	ppm	0.3
Cr	ppm	60	Nd	ppm	13	Tm	ppm	0.2
Cs	ppm	2.3	Ni	ppm	36	U	ppm	1.2
Dy	ppm	2.8	P	wt.%	0.10	V	ppm	186
Er	ppm	1.6	Pb	ppm	21	W	ppm	2.8
Eu	ppm	0.8	Pr	ppm	3.1	Y	ppm	15
Fe	wt.%	8.6	Rb	ppm	69	Yb	ppm	1.6
Ga	ppm	19	Re	ppm	< 0.1	Zn	ppm	113
Gd	ppm	3.4	Sb	ppm	1.1	Zr	ppm	55
Hf	ppm	1.6	Sc	ppm	18			
Hg	ppm	< 0.1	Se	ppm	9.8			
<b>Aqua Regia Digestion ICP-OES/MS</b>								
Ag	ppm	2.7	Hg	ppm	<0.1	Se	ppm	11
Al	wt.%	1.7	K	wt.%	0.25	Sn	ppm	9.1
As	ppm	5.5	La	ppm	9.0	Sr	ppm	91
Ba	ppm	65	Li	ppm	20	Ta	ppm	< 0.4
Be	ppm	< 1	Mg	wt.%	1.5	Te	ppm	0.3
Bi	ppm	4.8	Mn	wt.%	0.04	Th	ppm	3.2
Ca	wt.%	1.9	Na	wt.%	0.11	Ti	wt.%	0.15
Cd	ppm	0.4	Nb	ppm	0.5	U	ppm	0.8
Ce	ppm	18	Ni	ppm	35	V	ppm	130
Co	ppm	18	P	wt.%	0.09	W	ppm	1.0
Cr	ppm	48	Pb	ppm	18	Y	ppm	8.8
Fe	wt.%	7.3	Rb	ppm	16	Zn	ppm	93
Ga	ppm	8.0	Sb	ppm	0.5	Zr	ppm	11
Hf	ppm	0.6	Sc	ppm	7.9			
<b>Infra-red combustion furnace</b>								
C	wt.%	0.47	S	wt.%	1.3			
<b>Fire Assay</b>								
Pd	ppb	14	Pt	ppb	7			

## 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.

## SOURCE MATERIALS

OREAS 504 is a porphyry Au-Cu-Mo-Ag-S CRM prepared from Au-Cu ore and waste rock samples from a porphyry copper-gold deposit located in central western New South Wales, Australia with the addition of a minor quantity of Mo concentrate.

Mineralisation in the region is hosted by a sequence of late Ordovician to Early Silurian volcanics, intrusives and sediments that occur within the Bogan Gate Synclinal Zone of the Lachlan Fold Belt. The western portion of this zone is dominated by volcanics and host to the Late Ordovician Goonumbla porphyry copper-gold deposits. These volcanics are interpreted to have erupted from shallow water to partly emergent volcanic centres and show a broad range in composition from shoshonite through to latite to trachyte. Coeval sub-volcanic quartz monzonite porphyries (and attendant mineralisation) have intruded the volcanics. They are generally small, sub-vertical, pipe-like intrusives. Typically the mineralised porphyries contain plagioclase and quartz phenocrysts in a matrix of fine-grained potassium feldspar and quartz with minor biotite and hornblende.

Copper-gold mineralisation occurs as stockwork quartz veins and disseminations associated with potassic alteration. This alteration is intimately associated spatially and temporally with the small finger-like quartz monzonite porphyries that intrude the Goonumbla Volcanics. Sulphides are zoned laterally from the centres of mineralisation. The central portions are bornite-rich with minor chalcopyrite, zoning outward through equal concentrations of bornite and chalcopyrite, to an outermost chalcopyrite-rich zone. Pyrite increases outward at the expense of bornite.

The approximate major and trace element composition of OREAS 504 is given in Table 2. These constituents are the means of data from one or two laboratories.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 504 was prepared in the following manner:

- jaw crushing to minus 3mm;
- drying to constant mass at 105°C;
- multi-stage milling of ore and waste components to 100% minus 30 microns;
- combining in appropriate proportions to achieve target grades;
- homogenisation and bagging into 25kg sublots;
- packaging into 60g units in laminated foil pouches and 1kg units in plastic jars

## ANALYTICAL PROGRAM

Twenty commercial analytical laboratories participated in the program to characterise the elements reported in Table 1. The following methods were employed:

- Fire assay with ICP-OES, AAS or solvent extraction AAS finish (19 laboratories)
- Four acid digestion with ICP-OES, ICP-MS or AAS finish (19 labs; 10 for Ag)
- Aqua regia digestion with ICP-OES, ICP-MS or AAS finish (19 laboratories)

For the round robin program twenty 1kg test units were taken at predetermined intervals during the bagging stage and are considered representative of the entire batch. The six samples received by each laboratory were obtained by taking two 110g scoop splits from each of three separate 1kg test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity.

Results, together with uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification report for this CRM (Hamlyn, 2011).

## STATISTICAL ANALYSIS

**Certified Values, Standard Deviations, Confidence and Tolerance Limits** have been determined for each analytical method following removal of individual and laboratory outliers (see Table 1). Certified Values are the mean of means after outlier filtering. The 95% Confidence Limit 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. It should not be used as a control limit for laboratory performance. Indicative values (Table 2) are the means of data from one or two laboratories.

**Standard Deviation** values (1SDs) are reported in Table 1 and 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.

**Performance Gates** (Table 3) are 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 504**

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>Fire Assay</b>											
Au, ppm	1.48	0.04	1.39	1.56	1.35	1.61	2.93%	5.87%	8.80%	1.40	1.55
<b>Four Acid Digestion</b>											
Cu, wt.%	1.14	0.032	1.07	1.20	1.04	1.23	2.82%	5.63%	8.45%	1.08	1.19
Mo, ppm	643	37.1	569	717	532	754	5.77%	11.53%	17.30%	611	675
Ag, ppm	3.13	0.21	2.70	3.55	2.49	3.76	6.80%	13.60%	20.40%	2.97	3.28
S, wt.%	1.37	0.048	1.28	1.47	1.23	1.52	3.50%	7.01%	10.51%	1.31	1.44
<b>Aqua Regia Digestion</b>											
Cu, wt.%	1.12	0.019	1.09	1.16	1.07	1.18	1.69%	3.39%	5.08%	1.07	1.18
Au, ppm	1.47	0.07	1.33	1.60	1.27	1.67	4.52%	9.04%	13.55%	1.39	1.54
Mo, ppm	624	36.1	552	696	516	732	5.78%	11.56%	17.34%	593	655
S, wt.%	1.37	0.090	1.18	1.55	1.09	1.64	6.62%	13.25%	19.87%	1.30	1.43

Note: intervals may appear asymmetric due to rounding

**Tolerance Limits** (ISO Guide 3207) for copper, molybdenum, silver and sulphur were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. For gold the tolerance 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 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. In this instance a subsample weight of 0.5 grams was employed and the 1RSD of 0.777% (or 0.319% at a 30g charge weight) confirms the high level of gold homogeneity in OREAS 504.

The meaning of tolerance limits may be illustrated for copper by four acid digest, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 1.115 and 1.159 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 homogeneity of OREAS 504 has also been evaluated in an ANOVA study for all certified analytes. This study indicates no evidence that between-unit variance is greater than within-unit variance. Based on the statistical analysis of the results of the interlaboratory certification program it can be concluded that OREAS 504 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

Tabulated results of all elements (including Au INAA analyses) together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification data for this CRM (**OREAS 504 Datapack.xlsx**).

## **PARTICIPATING LABORATORIES**

Acme Analytical Laboratories SA, Santiago, Chile  
Acme Analytical Laboratories, Vancouver, BC, Canada  
Accurassay, Thunder Bay, Ontario, Canada  
Activation Laboratories, Ancaster, Ontario, Canada  
Alaska Assay Laboratories, Fairbanks, Alaska  
ALS, Brisbane, QLD, Australia  
ALS, La Serena, Coquimbo, Chile  
ALS, Perth, WA, Australia  
ALS, Val-d'Or, Quebec, Canada  
ALS, Vancouver, BC, Canada  
BV Amdel, Adelaide, SA, Australia  
BV Ultra Trace, Perth, WA, Australia  
Intertek Genalysis, Perth, WA, Australia  
Intertek Testing Services, Jakarta, Indonesia  
McPhar Geoservices (Phil) Inc., Manila, Philippines  
OMAC Laboratories, Loughrea, County Galway, Ireland  
SGS Mineral Services, Lakefield, Ontario, Canada  
SGS Mineral Services, Perth, WA, Australia  
SGS Mineral Services, Santiago, Chile  
SGS Mineral Services, Toronto, Ontario, Canada

## **PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL**

Porphyry Au-Cu-Mo-Ag-S reference material OREAS 504 has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd  
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OREAS 504 has been packaged in unit sizes of 60g (single-use laminated foil pouches) and is available in 1kg units in plastic jars upon request.

## **INTENDED USE**

OREAS 504 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of gold, copper, molybdenum, silver and sulphur in geological samples
- for the verification of analytical methods for gold, copper, molybdenum, silver and sulphur
- for the calibration of instruments used in the determination of the concentration of gold, copper, molybdenum, silver and sulphur

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 504 has been sourced from samples of Au-Cu ore and waste rock samples from a porphyry copper-gold deposit. In its unopened state and under normal conditions of storage it has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

## **INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL**

The certified values refer to the concentration level of analytes in their packaged state. The CRM should therefore 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.

## **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.

## **CERTIFYING OFFICER**

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

## **REFERENCES**

Ingamells, C. O. and Switzer, P. (1973), *Talanta* 20, 547-568.  
ISO Guide 35 (2006), *Certification of reference materials - General and statistical principals*.  
ISO Guide 3207 (1975), *Statistical interpretation of data - Determination of a statistical tolerance interval*.