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**CERTIFICATE OF ANALYSIS FOR**

**COPPER ORE**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 904**

**Table 1. 4-Acid ICP-OES/MS - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 904**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Four Acid Digestion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	6.30	0.315	6.13	6.47	6.15	6.45
Antimony, Sb (ppm)	1.48	0.106	1.42	1.54	1.41	1.55
Arsenic, As (ppm)	98	4.9	96	101	95	102
Barium, Ba (ppm)	194	9.0	190	198	185	203
Beryllium, Be (ppm)	7.86	0.768	7.46	8.25	7.53	8.18
Bismuth, Bi (ppm)	4.05	0.174	3.97	4.14	3.91	4.20
Calcium, Ca (wt.%)	0.046	0.005	0.044	0.048	0.042	0.050
Cerium, Ce (ppm)	86	8.5	82	90	82	90
Cesium, Cs (ppm)	3.79	0.181	3.68	3.89	3.65	3.92
Chromium, Cr (ppm)	54	3.8	52	56	51	56
Cobalt, Co (ppm)	83	7.0	80	87	81	85
Copper, Cu (wt.%)	0.612	0.021	0.601	0.622	0.598	0.625
Gallium, Ga (ppm)	16.7	0.85	16.3	17.1	16.1	17.3
Germanium, Ge (ppm)	0.18	0.03	0.14	0.23	IND	IND
Hafnium, Hf (ppm)	5.0	0.35	4.8	5.2	4.8	5.2
Indium, In (ppm)	0.22	0.017	0.21	0.23	0.21	0.23
Iron, Fe (wt.%)	6.68	0.291	6.53	6.83	6.47	6.89
Lanthanum, La (ppm)	43.2	2.90	41.6	44.9	41.6	44.9
Lead, Pb (ppm)	10.6	1.2	9.9	11.2	10.1	11.1
Lithium, Li (ppm)	16.7	0.66	16.4	17.0	16.1	17.3
Lutetium, Lu (ppm)	0.47	0.025	0.44	0.49	0.45	0.48
Magnesium, Mg (wt.%)	0.556	0.034	0.538	0.574	0.538	0.574
Manganese, Mn (wt.%)	0.041	0.002	0.040	0.042	0.040	0.042
Molybdenum, Mo (ppm)	2.12	0.184	2.04	2.21	2.02	2.23
Nickel, Ni (ppm)	40.1	1.83	39.3	41.0	38.7	41.6
Phosphorus, P (wt.%)	0.098	0.008	0.093	0.102	0.094	0.101
Potassium, K (wt.%)	3.31	0.184	3.21	3.42	3.20	3.43
Rubidium, Rb (ppm)	130	20	118	141	124	135
Scandium, Sc (ppm)	11.2	0.81	10.8	11.6	10.7	11.6
Selenium, Se (ppm)	3.30	0.60	2.94	3.66	IND	IND
Silver, Ag (ppm)	0.551	0.053	0.529	0.573	0.526	0.575
Sodium, Na (wt.%)	0.034	0.004	0.032	0.036	0.032	0.035
Strontium, Sr (ppm)	27.2	1.88	26.2	28.1	26.4	28.0
Sulphur, S (wt.%)	0.063	0.005	0.060	0.065	0.060	0.065
Tantalum, Ta (ppm)	0.54	0.09	0.48	0.61	0.50	0.59
Terbium, Tb (ppm)	1.00	0.11	0.91	1.09	0.96	1.05
Thallium, Tl (ppm)	0.52	0.026	0.50	0.53	0.50	0.53
Thorium, Th (ppm)	14.3	1.5	13.5	15.1	13.7	14.9
Tin, Sn (ppm)	2.83	0.174	2.73	2.93	2.66	3.01
Tungsten, W (ppm)	2.12	0.36	1.92	2.32	1.92	2.32
Uranium, U (ppm)	8.43	0.721	8.03	8.82	8.12	8.74
Vanadium, V (ppm)	76	3.0	74	77	73	78
Ytterbium, Yb (ppm)	3.14	0.229	2.98	3.30	2.98	3.30
Yttrium, Y (ppm)	31.5	3.4	29.8	33.2	30.2	32.8
Zinc, Zn (ppm)	26.3	3.5	24.5	28.1	24.8	27.8
Zirconium, Zr (ppm)	171	9.0	166	176	166	177

Note: intervals may appear asymmetric due to rounding.

**Table 2. Aqua Regia ICP-OES/MS - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 904**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Aqua Regia Digestion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	1.25	0.105	1.19	1.31	1.20	1.29
Antimony, Sb (ppm)	0.78	0.11	0.72	0.84	0.75	0.80
Arsenic, As (ppm)	91	5.3	89	94	89	94
Barium, Ba (ppm)	68	5.5	65	71	64	72
Beryllium, Be (ppm)	6.54	0.362	6.34	6.73	6.22	6.85
Bismuth, Bi (ppm)	3.74	0.370	3.56	3.92	3.62	3.86
Cadmium, Cd (ppm)	0.058	0.008	0.053	0.063	IND	IND
Calcium, Ca (wt.%)	0.0404	0.0016	0.0396	0.0412	0.0377	0.0431
Cerium, Ce (ppm)	70	4.9	67	73	68	71
Cesium, Cs (ppm)	0.62	0.061	0.58	0.66	0.59	0.64
Chromium, Cr (ppm)	17.5	2.5	16.1	18.9	16.1	18.9
Cobalt, Co (ppm)	82	4.8	80	85	80	84
Copper, Cu (wt.%)	0.630	0.018	0.622	0.639	0.622	0.638
Gallium, Ga (ppm)	3.40	0.47	3.12	3.69	3.27	3.54
Indium, In (ppm)	0.17	0.016	0.16	0.18	0.17	0.18
Iron, Fe (wt.%)	6.40	0.418	6.18	6.62	6.24	6.56
Lanthanum, La (ppm)	33.9	2.64	32.5	35.3	32.9	34.9
Lead, Pb (ppm)	8.49	0.94	7.98	9.00	8.12	8.85
Lutetium, Lu (ppm)	0.21	0.02	0.18	0.23	0.19	0.23
Magnesium, Mg (wt.%)	0.143	0.019	0.132	0.153	0.129	0.156
Manganese, Mn (wt.%)	0.041	0.003	0.040	0.042	0.040	0.042
Molybdenum, Mo (ppm)	2.02	0.23	1.88	2.15	1.97	2.06
Nickel, Ni (ppm)	36.6	1.57	35.8	37.4	35.4	37.8
Phosphorus, P (wt.%)	0.095	0.004	0.093	0.097	0.092	0.098
Potassium, K (wt.%)	0.603	0.054	0.571	0.635	0.577	0.629
Rubidium, Rb (ppm)	22.4	1.44	21.5	23.4	21.5	23.4
Scandium, Sc (ppm)	3.83	0.52	3.53	4.13	3.67	3.98
Selenium, Se (ppm)	2.81	0.29	2.73	2.90	2.67	2.96
Silver, Ag (ppm)	0.366	0.035	0.350	0.382	0.350	0.382
Strontium, Sr (ppm)	16.5	0.92	16.0	17.0	15.7	17.3
Sulphur, S (wt.%)	0.034	0.007	0.030	0.037	0.031	0.037
Terbium, Tb (ppm)	0.70	0.027	0.67	0.73	0.68	0.73
Thallium, Tl (ppm)	0.15	0.02	0.14	0.16	IND	IND
Thorium, Th (ppm)	7.56	0.696	7.16	7.96	7.31	7.81
Tin, Sn (ppm)	0.58	0.11	0.51	0.66	IND	IND
Uranium, U (ppm)	5.20	0.54	4.89	5.50	5.00	5.39
Vanadium, V (ppm)	21.7	0.89	21.3	22.2	20.5	22.9
Ytterbium, Yb (ppm)	1.41	0.084	1.33	1.50	IND	IND
Yttrium, Y (ppm)	17.2	1.24	16.4	17.9	16.7	17.7
Zinc, Zn (ppm)	22.4	2.05	21.4	23.3	21.1	23.6

Note: intervals may appear asymmetric due to rounding.

**Table 3. Copper Soluble - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 904**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Acid Leach 5% H<sub>2</sub>SO<sub>4</sub></b>						
Copper Soluble, Cu-Sol (wt.%)	0.519	0.028	0.503	0.534	0.510	0.527

Note: intervals may appear asymmetric due to rounding.

**Table 4. Fire Assay - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 904**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits*	
			Low	High	Low	High
<b>Fire Assay</b>						
Gold, Au (ppb)	45	4.3	43	47	44	47

\*Tolerance Limits calculated from 20 x INAA analyses on 1.2g subsamples; Note: intervals may appear asymmetric due to rounding.

**Table 5. Indicative Values for OREAS 904**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Four Acid Digestion ICP-OES/MS</b>								
Cd	ppm	0.07	Ho	ppm	1.1	Sm	ppm	7.1
Dy	ppm	5.6	Nb	ppm	6.3	Te	ppm	0.04
Er	ppm	3.4	Nd	ppm	36	Ti	wt.%	0.19
Eu	ppm	1.4	Pr	ppm	9.7	Tm	ppm	0.5
Gd	ppm	6.6	Re	ppb	< 2			
<b>Aqua Regia Digestion ICP-OES/MS</b>								
Au	ppm	0.02	Hg	ppm	0.04	Sm	ppm	5.4
B	ppm	12	Ho	ppm	0.6	Ta	ppm	< 0.01
Dy	ppm	3.0	Li	ppm	3.5	Te	ppm	0.03
Er	ppm	1.6	Na	wt.%	0.01	Ti	wt.%	0.007
Eu	ppm	1.0	Nb	ppm	0.09	Tm	ppm	0.2
Gd	ppm	4.9	Nd	ppm	32	W	ppm	0.6
Ge	ppm	0.1	Pr	ppm	7.7	Zr	ppm	17
Hf	ppm	0.6	Re	ppb	< 1			
<b>Fire Assay</b>								
Pd	ppb	< 2	Pt	ppb	< 10			

## 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 904 is a medium grade oxide copper ore certified reference material. It is one of a suite of four transitional to oxide copper CRMs prepared from CST's Lady Annie Mine, located 120 kms northwest of Mount Isa, Queensland, Australia. Mineralisation at Lady Annie is hosted in dolomitic, carbonaceous and argillaceous sandstones and siltstones. The oxide deposits consist primarily of near surface malachite mineralisation with minor cuprite, chrysocolla and chalcocite extending from surface to a depth of 60 to 100 m. The oxide copper deposit is underlain by deeper transition and sulphide mineralisation. The primary copper sulphide mineralisation at depth is dominated by chalcocite and chalcopyrite and appears to be structurally controlled, being commonly associated with fault-related silicification.

## COMMUNITION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 904 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing;
- milling to 100% minus 30 microns;
- homogenisation;
- packaging into 10g and 60g units in laminated foil pouches and into 1kg jars.

## ANALYTICAL PROGRAM

Nineteen commercial analytical laboratories participated in the program to characterise the elements reported in Tables 1 to 5. The following methods were employed:

- Four acid digestion with ICP-OES and ICP-MS finish (18 laboratories);
- Aqua regia digestion with ICP-OES and ICP-MS finish (19 laboratories);
- 5% H<sub>2</sub>SO<sub>4</sub> acid leach with AAS or ICP-OES finish (14 laboratories);
- Fire assay for gold with ICP-OES, ICP-MS, AAS or solvent extraction AAS finish (17 laboratories);
- Instrumental neutron activation analysis for Au on 20 x 1g subsamples to confirm homogeneity (1 lab).

For the round robin program twenty 1kg test units were taken at predetermined intervals during the bagging stage after final blending 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.

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 the detailed certification data for this CRM (**Datapack for OREAS 904.xlsx**).

## 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 Tables 1-4). 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 5) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) interlaboratory consensus is poor; or iii) a significant proportion of results are outlying or reported as less than detection limits.

**Standard Deviation** values (1SDs) are reported in Tables 1-4 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.

As a guide two or more analytical results lying outside the 2SD window may be regarded as warning or rejection, and rejection for single results lying outside the 3SD window in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

**Tolerance Limits** (ISO Guide 3207) for analytes other than gold 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 1.2 grams was employed and confirms the high level of gold homogeneity in OREAS 904.

The meaning of tolerance limits may be illustrated for gold by INAA, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $p=0.95$ ) will have concentrations lying between 44 and 47 ppb. 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 904 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 904 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## **PARTICIPATING LABORATORIES**

Acme Analytical Laboratories, Vancouver, BC, Canada  
Activation Laboratories, Ancaster, Ontario, Canada  
Activation Laboratories, Thunder Bay, Ontario, Canada  
ALS, Brisbane, QLD, Australia  
ALS, Callao, Lima, Peru  
ALS, Johannesburg, Gauteng, South Africa  
ALS, La Serena, Coquimbo, Chile  
ALS, Perth, WA, Australia  
ALS, Vancouver, BC, Canada  
BV Amdel, Adelaide, SA, Australia  
BV Ultra Trace, Perth, WA, Australia  
Intertek Genalysis, Perth, WA, Australia  
McPhar Geoservices (Phil) Inc., Manila, Philippines  
SGS Mineral Services, Lakefield, Ontario, Canada  
SGS Mineral Services, Perth, WA, Australia  
SGS Mineral Services, Toronto, Ontario, Canada  
SGS Mineral Services, Townsville, QLD, Australia  
SGS Mineral Services, Vancouver, BC, Canada  
Zarazma Mineral Studies, Tehran, Iran

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

Medium grade oxide copper ore reference material OREAS 904 has been prepared, certified and is supplied by:

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OREAS 904 has been packaged in single-use laminated foil pouches and is available in unit sizes of 10g and 60g. 1kg units in plastic jars are also available upon request.

## **INTENDED USE**

OREAS 904 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of analytes reported in Tables 1-4 in geological samples
- for the verification of analytical methods for analytes reported in Tables 1-4
- for the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1-4

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 904 has been sourced from medium grade oxide copper ore. 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.