

**CERTIFICATE OF ANALYSIS FOR**

**GOLD-SILVER ORE**

**CERTIFIED REFERENCE MATERIAL**

**OREAS 60c**

**Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 60c**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fire Assay</b>						
Au, Gold (ppm)	2.47	0.08	2.44	2.50	*2.46	*2.48
<b>4-Acid Digestion</b>						
Ag, Silver (ppm)	4.87	0.22	4.78	4.96	4.68	5.06
S, Sulphur (wt.%)	0.860	0.031	0.821	0.898	0.835	0.885
<b>Aqua Regia Digestion</b>						
Ag, Silver (ppm)	4.81	0.30	4.68	4.93	4.66	4.95
Au, Gold (ppm)	2.45	0.11	2.40	2.51	*2.44	*2.47

Note: intervals may appear asymmetric due to rounding; \*determined from RSD of INAA data for 30g and 25g analytical subsample weights for fire assay and aqua regia digestion, respectively.

## 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 60c was prepared from coarse reject splits of gold-silver ore samples from the Cracow mine located 500km northwest of Brisbane in Queensland, Australia. Cracow is a low sulphidation epithermal deposit hosted by meta-andesitic volcanics. High grade gold mineralisation occurs within fissure quartz veins and is associated with zones of silicification, present as quartz lode breccia and as quartz vein breccia.

The indicative major and trace element composition of OREAS 60c is given in Table 2. These constituents are the means of duplicate analyses by borate fusion with ICP-OES and ICP-MS, four acid digestion with ICP-OES, Leco, thermo-gravimetry and volatiles by aqua regia digestion ICP-MS and are uncertified values. OREAS 60c is one of a suite of three CRMs ranging in gold content from 2.47 to 9.13ppm.

## COMMUNITION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 60c was prepared in the following manner:

- drying to constant mass at 105 °C;
- crushing and multi stage milling to 100% passing 35 microns;
- homogenisation;
- packaging in 60g units sealed in laminated foil pouches and 1kg units in plastic jars.

## ANALYTICAL PROGRAM

Twenty-six commercial analytical laboratories participated in the program to characterise Au and Ag. The following methods were employed:

- Au by 26 labs via fire assay with AAS (21 labs) or ICP-OES (5 labs) finish;
- Instrumental neutron activation analysis for Au on 20 x 1g subsamples to confirm homogeneity (1 lab);
- Ag via four acid digestion with ICP-OES (17 labs), AAS (4 labs) or ICP-MS (3 labs) finish except for 2 labs that used a 3-acid digestion;
- S was also determined via four acid digestion with ICP-OES (5 labs);
- Ag by 24 labs via aqua regia digestion with ICP-OES (18 labs), ICP-MS (4 labs) or AAS (2 labs) finish;
- Au by 15 labs via aqua regia digestion with ICP-MS (8 labs), AAS finish (4 labs), graphite furnace AAS (2 labs) or solvent extraction AAS (1 lab) finish.

For the round robin program samples were taken at 20 predetermined sampling intervals during packaging and are considered representative of the entire batch of OREAS 60c. Six 110g samples were submitted to each laboratory for analysis. Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits and Table 2 shows indicative values for major and trace element composition. The constituents within Table 2 are the means of duplicate analyses determined via the methods indicated in the table headings.

**Table 2. Indicative Values for OREAS 60c**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Pb Fire Assay</b>								
Pd	ppm	< 0.005	Pt	ppm	0.004			
<b>4-Acid Digestion</b>								
Al	wt.%	5.45	Ga	ppm	10.0	Sb	ppm	< 5
As	ppm	20.4	K	wt.%	2.31	Sc	ppm	12.6
Ba	ppm	342	La	ppm	10.0	Sr	ppm	282
Be	ppm	0.92	Li	ppm	45.0	Th	ppm	< 20
Bi	ppm	< 2	Mg	wt.%	0.957	Ti	wt.%	0.274
Ca	wt.%	4.00	Mn	wt.%	0.072	Tl	ppm	< 10
Cd	ppm	0.39	Mo	ppm	6.70	U	ppm	< 10
Co	ppm	10.8	Na	wt.%	0.892	V	ppm	104
Cr	ppm	24.3	Ni	ppm	9.71	W	ppm	< 10
Cu	ppm	73	P	wt.%	0.065	Zn	ppm	90
Fe	wt.%	3.24	Pb	ppm	18.7			
<b>Aqua Regia Digestion</b>								
Al	wt.%	1.71	Ga	ppm	10.0	Sb	ppm	< 2
As	ppm	18.9	Hg	ppm	0.65	Sc	ppm	8.00
B	ppm	10.0	K	wt.%	0.271	Se	ppm	0.75
Ba	ppm	40.0	La	ppm	13.3	Sr	ppm	122
Be	ppm	0.60	Mg	wt.%	0.873	Te	ppm	2.11
Bi	ppm	< 2	Mn	wt.%	0.070	Th	ppm	< 20
Ca	wt.%	3.73	Mo	ppm	6.78	Ti	wt.%	0.063
Cd	ppm	1.33	Na	wt.%	0.089	Tl	ppm	< 10
Co	ppm	10.9	Ni	ppm	10.6	U	ppm	< 10
Cr	ppm	24.0	P	wt.%	0.062	V	ppm	69
Cu	ppm	73	Pb	ppm	18.7	W	ppm	< 10
Fe	wt.%	2.97	S	wt.%	0.908	Zn	ppm	85
<b>Thermogravimetry</b>								
LOI	wt.%	7.38						
<b>Borate Fusion</b>								
Al	wt.%	5.45	K	wt.%	2.33	Sr	ppm	284
Ba	ppm	355	La	ppm	12.0	Ta	ppm	0.10
Ca	wt.%	3.92	Lu	ppm	0.17	Tb	ppm	0.33
Ce	ppm	24.8	Mg	wt.%	0.962	Th	ppm	3.04
Cr	ppm	< 70	Mn	wt.%	0.077	Ti	wt.%	0.288
Cs	ppm	6.36	Na	wt.%	0.972	Tl	ppm	0.95
Dy	ppm	1.93	Nb	ppm	2.35	Tm	ppm	0.17
Er	ppm	1.13	Nd	ppm	12.8	U	ppm	0.79
Eu	ppm	0.73	P	wt.%	0.063	V	ppm	120
Fe	wt.%	3.13	Pr	ppm	3.06	W	ppm	3.00
Ga	ppm	12.0	Rb	ppm	103	Y	ppm	11.1
Gd	ppm	2.25	Si	wt.%	30.92	Yb	ppm	1.07
Hf	ppm	2.00	Sm	ppm	2.67	Zr	ppm	70
Ho	ppm	0.43	Sn	ppm	< 1			
<b>Infrared Combustion</b>								
C	wt.%	1.27	S	wt.%	0.830			

Table 3 provides performance gate intervals for the certified values based on their associated standard deviations. Gold homogeneity has been evaluated and confirmed by INAA on twenty ~1.0 gram sample portions and by a nested ANOVA program for both fire assay and aqua regia digestion. 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 (**OREAS 60c Datapack.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 (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 provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) inter-laboratory 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 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 per cent 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 60c**

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	2.47	0.08	2.30	2.63	2.22	2.71	3.32%	6.63%	9.95%	2.34	2.59
<b>4-Acid Digestion</b>											
Ag, ppm	4.87	0.22	4.42	5.31	4.20	5.54	4.58%	9.16%	13.74%	4.62	5.11
S, wt. %	0.860	0.031	0.798	0.921	0.767	0.952	3.59%	7.18%	10.77%	0.817	0.903
<b>Aqua Regia Digestion</b>											
Ag, ppm	4.81	0.30	4.21	5.40	3.92	5.70	6.17%	12.33%	18.50%	4.57	5.05
Au, ppm	2.45	0.11	2.23	2.68	2.11	2.79	4.63%	9.25%	13.88%	2.33	2.58

Note: intervals may appear asymmetric due to rounding

**Tolerance Limits** (ISO Guide 3207) for Au were determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the latter parameter is substantially reduced to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible. In this instance a subsample weight of 1.0 gram was employed and the 1RSD of 0.837% (or 0.157% at a 30g charge weight) confirms the high level of gold homogeneity in OREAS 60c.

The meaning of tolerance limits may be illustrated for gold fire assay (at a conventional 30g charge weight) where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 2.46 and 2.48ppm (see Table 1). 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).

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 60c is fit-for-purpose as a certified reference material (see 'Intended Use' below).

## PARTICIPATING LABORATORIES

Accurassay, Thunder Bay, ON, Canada  
 Acme, Santiago, Chile  
 Acme, Vancouver, BC, Canada  
 Actlabs, Ancaster, Ontario, Canada  
 Actlabs, Guadalupe, Zacatecas, Mexico  
 Actlabs, Thunder Bay, Ontario, Canada  
 ALS, Johannesburg, South Africa  
 ALS, La Serena, Chile  
 ALS, Lima, Peru

ALS, Perth, WA, Australia  
ALS, Vancouver, BC, Canada  
Amdel (BV), Adelaide, SA, Australia  
Intertek Genalysis, Perth, WA, Australia  
Intertek Genalysis, Boksburg, Gauteng, South Africa  
Intertek Minerals Indonesia, Jakarta, Indonesia  
Intertek Testing Services, Beijing, China  
Intertek Testing Services, Muntinlupa, Philippines  
Newmont Metallurgical Services, Engelwood, CO, USA  
OMAC (ALS), Loughrea, County Galway, Ireland  
SGS, Booyens, Gauteng, South Africa  
SGS, Durango, Durango, Mexico  
SGS, Lakefield, Ontario, Canada  
SGS, Townsville, QLD, Australia  
SGS, Vespasiano, MG, Brazil  
Shiva Analyticals, Bangalore North, Karnataka, India  
Ultra Trace (BV), Perth, WA, Australia

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

Reference material OREAS 60c has been prepared and certified by:

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It has been packaged in 60g units into laminated foil pouches and 1kg units in plastic jars.

## **INTENDED USE**

OREAS 60c is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of gold by fire assay and gold and silver by aqua regia digestion methods in geological samples;
- for the verification of analytical methods for gold by fire assay and gold and silver by aqua regia digestion methods;
- for the calibration of instruments used in the determination of the concentration of gold by fire assay and gold and silver by aqua regia digestion methods.

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 60c has been prepared from gold-silver ore samples from the Cracow mine. In its unopened state under normal conditions of storage it has a shelf life beyond ten years.

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

The certified values for OREAS 60c refer to the concentration level in its packaged state. It 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.

### **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 P/L

### **REFERENCES**

Ingamells, C. O. and Switzer, P. (1973), *Talanta* 20, 547-568.

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

ISO Guide 35 (2006), *Certification of reference materials - General and statistical principals.*