

CERTIFICATE OF ANALYSIS FOR

GOLD-SILVER ORE

CERTIFIED REFERENCE MATERIAL

OREAS 62e

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 62e

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fire Assay						
Au, Gold (ppm)	9.13	0.41	8.97	9.30	*9.09	*9.18
4-Acid Digestion						
Ag, Silver (ppm)	9.86	0.34	9.73	9.98	9.58	10.13
S, Sulphur (wt.%)	0.429	0.014	0.412	0.446	0.408	0.451
Aqua Regia Digestion						
Ag, Silver (ppm)	9.86	0.37	9.71	10.01	9.60	10.12
Au, Gold (ppm)	9.37	0.62	9.04	9.69	*9.32	*9.42

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 62e 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 62e 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 62e 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 62e 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 (20 labs), ICP-OES (5 labs) or gravimetric (1 lab) 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 (7 labs), AAS finish (4 labs), graphite furnace AAS (2 labs), solvent extraction AAS (1 lab) or ICP-OES (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 62e. 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 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 (PDM³) are presented in the detailed certification data for this CRM (**OREAS 62e 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.

Table 2. Indicative Values for OREAS 62e

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Pd	ppm	< 0.005	Pt	ppm	< 0.005			
4-Acid Digestion								
Al	wt.%	5.61	Ga	ppm	10.0	Sb	ppm	< 5
As	ppm	11.3	K	wt.%	1.78	Sc	ppm	12.5
Ba	ppm	343	La	ppm	10.0	Sr	ppm	346
Be	ppm	0.94	Li	ppm	50	Th	ppm	< 20
Bi	ppm	< 2	Mg	wt.%	1.17	Ti	wt.%	0.281
Ca	wt.%	4.45	Mn	wt.%	0.085	Tl	ppm	< 10
Cd	ppm	< 0.5	Mo	ppm	5.25	U	ppm	< 10
Co	ppm	11.5	Na	wt.%	1.38	V	ppm	104
Cr	ppm	18.9	Ni	ppm	9.83	W	ppm	< 10
Cu	ppm	68	P	wt.%	0.066	Zn	ppm	71
Fe	wt.%	3.31	Pb	ppm	16.7			
Aqua Regia Digestion								
Al	wt.%	1.75	Ga	ppm	9.44	Sb	ppm	< 2
As	ppm	11.5	Hg	ppm	0.60	Sc	ppm	8.00
B	ppm	< 10	K	wt.%	0.149	Se	ppm	0.50
Ba	ppm	30.0	La	ppm	13.3	Sr	ppm	112
Be	ppm	0.51	Mg	wt.%	1.07	Te	ppm	3.37
Bi	ppm	< 2	Mn	wt.%	0.082	Th	ppm	< 20
Ca	wt.%	3.86	Mo	ppm	5.56	Ti	wt.%	0.140
Cd	ppm	1.17	Na	wt.%	0.091	Tl	ppm	< 10
Co	ppm	11.1	Ni	ppm	9.61	U	ppm	< 10
Cr	ppm	17.4	P	wt.%	0.062	V	ppm	77
Cu	ppm	65	Pb	ppm	15.2	W	ppm	< 10
Fe	wt.%	2.95	S	wt.%	0.448	Zn	ppm	65
Thermogravimetry								
LOI	wt.%	6.10						
Borate Fusion								
Al	wt.%	5.53	K	wt.%	1.75	Sr	ppm	349
Ba	ppm	355	La	ppm	12.1	Ta	ppm	0.10
Ca	wt.%	4.38	Lu	ppm	0.17	Tb	ppm	0.35
Ce	ppm	24.8	Mg	wt.%	1.17	Th	ppm	3.09
Cr	ppm	30.0	Mn	wt.%	0.093	Ti	wt.%	0.294
Cs	ppm	4.55	Na	wt.%	1.49	Tl	ppm	0.60
Dy	ppm	2.02	Nb	ppm	2.35	Tm	ppm	0.17
Er	ppm	1.18	Nd	ppm	12.5	U	ppm	0.79
Eu	ppm	0.74	P	wt.%	0.065	V	ppm	130
Fe	wt.%	3.24	Pr	ppm	3.09	W	ppm	2.00
Ga	ppm	12.5	Rb	ppm	73	Y	ppm	11.2
Gd	ppm	2.30	Si	wt.%	30.71	Yb	ppm	1.12
Hf	ppm	2.10	Sm	ppm	2.59	Zr	ppm	73
Ho	ppm	0.43	Sn	ppm	< 1			
Infrared Combustion								
C	wt.%	1.15	S	wt.%	0.415			

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 62e

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
Fire Assay											
Au, ppm	9.13	0.41	8.31	9.95	7.90	10.36	4.49%	8.97%	13.5%	8.68	9.59
4-Acid Digestion											
Ag, ppm	9.86	0.34	9.17	10.54	8.83	10.88	3.47%	6.95%	10.4%	9.36	10.35
S, wt.%	0.429	0.014	0.401	0.458	0.387	0.472	3.32%	6.64%	9.96%	0.408	0.451
Aqua Regia Digestion											
Ag, ppm	9.86	0.37	9.13	10.59	8.77	10.96	3.70%	7.40%	11.1%	9.37	10.36
Au, ppm	9.37	0.62	8.13	10.61	7.51	11.23	6.62%	13.2%	19.9%	8.90	9.84

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 0.787% (or 0.147% at a 30g charge weight) confirms the high level of gold homogeneity in OREAS 62e.

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 9.09 and 9.18 ppm (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 62e 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 62e 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 62e 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 62e 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 62e 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.*