

CERTIFICATE OF ANALYSIS FOR

GOLD ORE CERTIFIED REFERENCE MATERIAL OREAS 203

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 203

Constituent	Certified	1SD	95% Confid	dence Limits	95% Tolerance Limits*		
Constituent	Value		Low	Low High		High	
Fire Assay							
Gold, Au (ppm)	0.871	0.030	0.859	0.884	0.861	0.881	
Aqua Regia Digestion							
Gold, Au (ppm)	0.825	0.062	0.793	0.857	0.815	0.835	

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

Certified Reference Material (CRM) OREAS 203 was prepared from a blend of gold-bearing Magdala ore from the Stawell Gold Mine, west-central Victoria, Australia and barren tholeiitic basalt from Epping, Victoria, Australia. The Magdala lode is intimately associated with an intensely deformed package of volcanogenic sedimentary rocks. The ore samples were taken from basalt contact lodes and are strongly chlorite-altered (+/-silica, stilpnomelane) carbonaceous mudstones located directly on the western margin of the Magdala basalt dome. Mineralisation in the ore consists of a quartz-sericite-carbonate schist assemblage containing the sulphides arsenopyrite, pyrrhotite and pyrite. OREAS 203 is one of a suite of eight CRMs ranging in gold content from 0.514 to 9.24ppm.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 203 was prepared in the following manner:

- drying to constant mass at 105 °C;
- crushing and milling of the barren material to 95% minus 75 microns;
- crushing and milling of the ore material to 100% minus 30 microns;
- blending in appropriate proportions to achieve the desired grade;
- packaging in 60g units sealed in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

Twenty commercial analytical laboratories participated in the program to characterise gold by fire assay with AAS (14 labs) or ICP-OES (6 labs) finish. Fifteen of these laboratories also determined gold via aqua regia digestion with ICP-MS (7 labs), AAS (5 labs), graphite furnace AAS (2 labs) or solvent extraction AAS (1 lab) finish. Gold has been certified separately for the fire assay and aqua regia digestion methods.

For the round robin program the samples were taken at 20 predetermined sampling intervals during packaging and are considered representative of the entire batch of OREAS 203. 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 203

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Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value			
Pb Fire Assay											
Pd	ppb	< 5	Pt	ppb	< 5						
Thermogravimetry											
LOI	wt.%	2.25									
Borate Fusion ICP											
Al	wt.%	7.16	K	wt.%	1.23	Sr	ppm	338			
Ва	ppm	<i>537</i>	La	ppm	29.6	Ta	ppm	1.35			
Ca	wt.%	4.27	Lu	ppm	0.38	Tb	ppm	0.88			
Ce	ppm	55	Mg	wt.%	3.14	Th	ppm	7.49			
Cr	ppm	230	Mn	wt.%	0.217	Ti	wt.%	0.854			
Cs	ppm	4.83	Na	wt.%	1.65	TI	ppm	< 0.5			
Dy	ppm	5.32	Nb	ppm	19.1	Tm	ppm	0.44			
Er	ppm	3.01	Nd	ppm	27.8	U	ppm	1.75			
Eu	ppm	1.74	Р	wt.%	0.159	V	ppm	166			
Fe	wt.%	8.31	Pr	ppm	6.84	W	ppm	2.00			
Ga	ppm	19.5	Rb	ppm	68	Υ	ppm	31.5			
Gd	ppm	5.86	Si	wt.%	26.18	Yb	ppm	2.62			
Hf	ppm	4.20	Sm	ppm	6.11	Zr	ppm	170			
Но	ppm	1.10	Sn	ppm	2.00						
4-Acid Digestion											
Ag	ppm	< 0.5	Cu	ppm	74	Pb	ppm	12.5			
Cd	ppm	< 0.5	Мо	ppm	1.50	Sc	ppm	15.5			
Co	ppm	31.5	Ni	ppm	110	Zn	ppm	121			
Infrared Combustion											
С	wt.%	0.237	S	wt.%	0.721						
Aqua Regia Digestion											
As	ppm	> 250	Hg	ppb	4	Se	ppm	1.45			
Bi	ppm	0.26	Sb	ppm	0.65	Te	ppm	0.040			

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 203 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 203

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	0.871	0.030	0.811	0.931	0.781	0.961	3.43%	6.87%	10.30%	0.828	0.915
Aqua Regia Digestion											
Au, ppm	0.825	0.062	0.700	0.949	0.638	1.012	7.55%	15.10%	22.65%	0.784	0.866

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 1.92% (or 0.36% at a 30g charge weight) confirms the high level of gold homogeneity in OREAS 203.

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 (p=0.95) will have concentrations lying between 0.861 and 0.881ppm. 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 203 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

ALS, Brisbane, QLD, Australia

ALS, La Serena, Chile

ALS, Lima, Peru

ALS, Perth, WA, Australia

ALS, Vancouver, BC, Canada

Acme, Santiago, Chile

Acme, Vancouver, BC, Canada

Actlabs, Ancaster, Ontario, Canada

Actlabs, Thunder Bay, Ontario, Canada

Amdel (BV), Adelaide, SA, Australia

Intertek Genalysis, Perth, WA, Australia

Intertek Testing Services, Beijing, China

Intertek Testing Services, Jakarta, Indonesia

Intertek Testing Services, Muntinlupa, Philippines

SGS, Booysens, Gauteng, South Africa

SGS, Perth, WA, Australia

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 203 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 203 is intended for the following uses:

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

STABILITY AND STORAGE INSTRUCTIONS

OREAS 203 has been prepared from gold ore diluted with barren tholeiitic basalt. 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 203 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.

