

CERTIFICATE OF ANALYSIS FOR

GOLD ORE

CERTIFIED REFERENCE MATERIAL

OREAS 208

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 208

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits*	
			Low	High	Low	High
Fire Assay						
Gold, Au (ppm)	9.248	0.438	9.052	9.444	9.212	9.284
Aqua Regia Digestion						
Gold, Au (ppm)	8.810	0.358	8.629	8.991	8.772	8.847

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 208 was prepared from gold-bearing Magdala ore from the Stawell Gold Mine, west-central 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 208 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 208 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and milling of the ore to 100% minus 30 microns;
- packaging in 60g units under nitrogen and sealed in laminated foil pouches.

ANALYTICAL PROGRAM

Twenty commercial analytical laboratories participated in the program to characterise gold by fire assay with AAS (13 labs), ICP-OES (6 labs) or gravimetric (1 lab) 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 208. 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 208

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Pb Fire Assay								
Pd	ppb	14	Pt	ppb	5			
Thermogravimetry								
LOI	wt.%	8.46						
Borate Fusion ICP								
Al	wt.%	4.79	K	wt.%	0.494	Sr	ppm	133
Ba	ppm	796	La	ppm	21.2	Ta	ppm	0.30
Ca	wt.%	5.25	Lu	ppm	0.51	Tb	ppm	0.88
Ce	ppm	26.0	Mg	wt.%	2.26	Th	ppm	4.30
Cr	ppm	137	Mn	wt.%	0.627	Ti	wt.%	0.315
Cs	ppm	4.50	Na	wt.%	0.638	Tl	ppm	< 0.5
Dy	ppm	5.97	Nb	ppm	4.05	Tm	ppm	0.60
Er	ppm	4.00	Nd	ppm	20.5	U	ppm	2.05
Eu	ppm	1.34	P	wt.%	0.273	V	ppm	207
Fe	wt.%	14.93	Pr	ppm	4.75	W	ppm	7.00
Ga	ppm	12.8	Rb	ppm	21.8	Y	ppm	42.1
Gd	ppm	5.58	Si	wt.%	21.32	Yb	ppm	3.49
Hf	ppm	1.60	Sm	ppm	4.82	Zr	ppm	60
Ho	ppm	1.36	Sn	ppm	1.00			
4-Acid Digestion								
Ag	ppm	1.05	Cu	ppm	242	Pb	ppm	19.0
Cd	ppm	< 0.5	Mo	ppm	4.50	Sc	ppm	18.5
Co	ppm	23.0	Ni	ppm	71	Zn	ppm	124
Infrared Combustion								
C	wt.%	1.47	S	wt.%	5.16			
Aqua Regia Digestion								
As	ppm	> 250	Hg	ppb	24	Se	ppm	6.30
Bi	ppm	0.32	Sb	ppm	11.0	Te	ppm	0.41

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 208 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 208

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	9.248	0.438	8.372	10.125	7.934	10.563	4.74%	9.48%	14.21%	8.786	9.711
Aqua Regia Digestion											
Au, ppm	8.810	0.358	8.093	9.527	7.734	9.885	4.07%	8.14%	12.21%	8.369	9.250

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 0.65% (or 0.12% at a 30g charge weight) confirms the high level of gold homogeneity in OREAS 208.

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 9.212 and 9.284ppm. 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 208 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, Booyens, 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 208 has been prepared and certified by:

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It has been packaged in 60g units under nitrogen and sealed in laminated foil pouches.

INTENDED USE

OREAS 208 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 208 has been prepared from primary gold ore. 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 208 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.