

CERTIFICATE OF ANALYSIS FOR
GOLD ORE REFERENCE MATERIAL
OREAS 7Pb

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	2.77	2.75	2.79

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99, \rho=0.95$	
		Low	High
Gold, Au (ppm)	2.77	2.76	2.78

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INTRODUCTION

OREAS reference materials (RM) are intended to provide a low cost method of evaluating and improving the quality of precious and base metal analysis of geological samples. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. To the explorationist they provide an important control in analytical data sets related to exploration from the grass roots level through to resource definition.

As a rule only source materials exhibiting an exceptional level of homogeneity of the element(s) of interest are used in the preparation of these materials. This has enabled Ore Research & Exploration to produce a range of gold RM exhibiting homogeneity that matches or exceeds that of currently available international reference materials. In many instances RM produced from a single source are sufficiently homogeneous to produce a relatively coarse-grained form designed to simulate drill chip samples. These have a grain size of minus 3mm and are designated with a "C" suffix to the RM identification number. These standards are packaged in 1kg units following homogenisation and are intended for submission to analytical laboratories in subsample sizes of as little as 250g. They offer the added advantages of providing a check on both sample preparation and analytical procedures while acting as a blind standard to the assay laboratory. The more conventional pulped standards have a grain size of minus 75 microns and a higher degree of homogeneity. These standards are distinguished by a "P" suffix to the standard identification number. In line with ISO recommendations successive batch numbers are now designated by the lower case suffixes "a", "b", "c", "d", etc.

SOURCE MATERIALS

The material used to produce gold ore standard OREAS 7Pb was taken from a mineralised shear zone within Ordovician flysch sediments in the Blackwood area of central Victoria. The sedimentary succession hosting the shear zone consists predominantly of medium-grained greywackes together with subordinate interbedded siltstone and slate. Hydrothermal alteration in the vicinity of the mineralisation is indicated by the development of phyllite. The shear zone, in which gold grades attain a maximum, is manifested by foliated sericitic and chloritic fault gouge and goethitic quartz veins.

Although no ore mineragraphy or scanning electron microscopy has been undertaken to determine the nature of occurrence of the gold, the very homogeneous distribution on a mesoscopic scale and uniform concentration gradient away from the ore zone suggests the gold is extremely fine-grained and evenly disseminated. Limited percussion drilling indicates that sulphides are rare to absent in the shear zone.

The approximate major and trace element composition of this oxidised, quartz-veined metagreywacke comprising gold ore standard OREAS 7Pb is given in Table 1. The constituents SiO₂ to Total are the means of triplicate XRF analyses determined using a borate fusion method, while the remaining constituents, As to Yb, are means of twenty replicate analyses determined via INAA at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 7Pb was prepared in the following manner:

- a) *primary crushing to minus 10mm*
- b) *drying at 105°C to constant mass*
- c) *secondary crushing to minus 2mm*
- d) *milling to 100% minus 30 micron*
- e) *final homogenisation*
- f) *bagging into 20kg sublots*
- g) *packaging into 60g units using laminated foil pouches*

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 7Pb; SiO₂ to Total as weight percent; rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO ₂	70.5	As	2106
TiO ₂	0.66	Ba	720
Al ₂ O ₃	14.9	Ce	104
Fe ₂ O ₃	5.16	Cr	200
MnO	0.009	Cs	12
MgO	0.76	Hf	5
CaO	0.02	La	56
Na ₂ O	0.12	Rb	220
K ₂ O	4.22	Sb	163
P ₂ O ₅	0.081	Sc	16
SO ₃	0.07	Sm	8
H ₂ O ⁺	2.95	Th	18
Total	99.45	W	25
		Yb	3

ANALYSIS OF OREAS 7Pb

Seventeen laboratories participated in the analytical program and are listed in the section headed Participating Laboratories. To maintain anonymity laboratories have been randomly designated the letter codes A through Q. With the exception of Laboratory Q, each laboratory received two 120g subsamples with instructions to carry out duplicate fire assays for gold on each subsample. The duplicate assays were to be performed on separate test portions using 50g charges. For each laboratory the two 120 gram subsamples were scoop-split from separate test units taken during the bagging stage. This two-stage nested design for the interlaboratory programme was amenable to analysis of variance (ANOVA) treatment and enabled a comparative assessment of within- and between-unit homogeneity.

For the determination of a statistical tolerance interval, a 10 gram scoop split was taken from each of the twenty test units and submitted to Laboratory Q for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 0.5 gram.

Individual assay results for the fire assay and INAA methods are presented in Tables 2 and 3 together with the mean, median, standard deviations (absolute and relative) and percent deviation of the lab mean from the corrected mean of means for each data set (PDM³). Interlaboratory agreement of the means of all but two data sets is good, lying within 3.0% relative of the mean of means of 2.77 ppm Au. The exceptions to this are laboratories E and M, which are 11.1% and 9.58% lower, respectively, than the corrected mean of means.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 7Pb

Recommended Value and Confidence Limits

The recommended value was determined from the mean of means of accepted replicate values of accepted laboratory data sets A to Q according to the formulae

$$\bar{x}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} x_{ij}$$

$$\dot{x} = \frac{1}{p} \sum_{i=1}^p \bar{x}_i$$

where

x_{ij} is the j th result reported by laboratory i ;

p is the number of participating laboratories;

n_i is the number of results reported by laboratory i ;

\bar{x}_i is the mean for laboratory i ;

\dot{x} is the mean of means.

The confidence limits were obtained by calculation of the variance of the consensus value (mean of means) and reference to Student's- t distribution with degrees of freedom ($p-1$):

$$\hat{V}(\ddot{x}) = \frac{1}{p(p-1)} \sum_{i=1}^p (\bar{x}_i - \dot{x})^2$$

$$\text{Confidence limits} = \dot{x} \pm t_{1-x/2}(p-1) \left(\hat{V}(\ddot{x}) \right)^{1/2}$$

where $t_{1-x/2}(p-1)$ is the $1-x/2$ fractile of the t -distribution with $(p-1)$ degrees of freedom.

The distribution of the values are assumed to be symmetrical about the mean in the calculation of the confidence limits.

The test for rejection of individual outliers from each laboratory data set was based on z scores (rejected if $|z_i| > 2.5$) computed from the robust estimators of location and scale, T and S , respectively, according to the formulae

$$S = 1.483 \frac{\text{median } |x_j - \text{median}(x_i)|}{\sqrt{j=1, \dots, n \quad i=1, \dots, n}}$$

$$z_i = \frac{x_i - T}{S}$$

where

T is the median value in a data set;

S is the median of all absolute deviations from the sample median multiplied by 1.483, a correction factor to make the estimator consistent with the usual parameter of a normal distribution.

Table 2. Analytical results for gold (ppm) in OREAS 7Pb by 50g fire assay/flare AAS (Std. Dev. - one sigma standard deviation; RSD – one sigma relative standard deviation; PDM³ – percent deviation of lab mean from corrected mean of means; outliers in bold).

Replicate	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	2.87	2.77	2.80	2.79	2.32	2.77	2.63	2.72	2.81	2.82
2	2.82	2.73	2.79	2.78	2.50	2.73	2.78	2.69	2.83	2.80
3	2.80	2.80	2.80	2.78	2.55	2.72	2.71	2.68	2.78	2.78
4	2.79	2.76	2.73	2.79	2.47	2.80	2.80	2.67	2.79	2.79
Mean	2.82	2.77	2.78	2.78	2.46	2.75	2.73	2.69	2.80	2.80
Median	2.81	2.77	2.80	2.78	2.49	2.75	2.75	2.69	2.80	2.80
Std.Dev.	0.04	0.03	0.03	0.01	0.10	0.04	0.08	0.02	0.02	0.02
Rel.Std.Dev.	1.26%	1.04%	1.21%	0.25%	4.02%	1.44%	2.82%	0.80%	0.79%	0.61%
PDM ³	1.86%	-0.13%	0.41%	0.52%	-11.1%	-0.58%	-1.39%	-2.84%	1.23%	1.04%

Table 2. Continued.

Replicate	Lab K	Lab L	Lab M	Lab N	Lab O	Lab P
1	2.93	2.83	2.49	2.75	2.75	2.77
2	2.81	2.79	2.51	2.74	2.7	2.74
3	2.79	2.81	2.51	2.71	2.68	2.74
4	2.87	2.87	2.51	2.91	2.72	2.78
Mean	2.85	2.83	2.50	2.78	2.71	2.76
Median	2.84	2.82	2.51	2.75	2.71	2.76
Std.Dev.	0.06	0.03	0.01	0.09	0.03	0.02
Rel.Std.Dev.	2.22%	1.21%	0.46%	3.24%	1.10%	0.65%
PDM ³	2.94%	2.04%	-9.58%	0.32%	-2.03%	-0.38%

The same principles were applied in testing for outlying laboratory means. In certain instances statistician's prerogative has been employed in discriminating outliers. Individual and mean outliers are shown in bold type in Tables 2 and 3, and have been omitted in the determination of recommended values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It is a measure of the reliability of the recommended value, i.e. the narrower the confidence interval the greater the certainty in the recommended value.

Table 3. Analytical results for gold (ppm) in OREAS 7Pb by instrumental neutron activation analysis on 0.5 gram analytical subsample weights (abbreviations as for Table 2).

Unit No.	Lab Q
1	2.73
2	2.71
3	2.73
4	2.75
5	2.73
6	2.74
7	2.73
8	2.71
9	2.77
10	2.70
11	2.75
12	2.71
13	2.72
14	2.73
15	2.76
16	2.71
17	2.69
18	2.71
19	2.76
20	2.76
Mean	2.73
Median	2.73
Std.Dev.	0.02
Rel.Std.Dev.	0.84%
PDM ³	-1.42%

Table 4. Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	2.77	2.75	2.79

Statement of Homogeneity

The variability of replicate assays from each laboratory is a result of both measurement and subsampling errors. In the determination of a statistical tolerance interval it is therefore necessary to eliminate, or at least substantially minimise, those errors attributable to measurement. One way of achieving this is by substantially reducing the analytical subsample weight to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible. This approach was adopted in the INAA data set (Table 3) where a 0.5 gram subsample weight was employed. The homogeneity was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO Guide 3207) in which

$$\text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha)s$$

$$\text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha)s$$

where

n is the number of results reported by laboratory *Q*;

1 - α is the confidence level;

p is the proportion of results expected within the tolerance limits;

k'₂ is the factor for two-sided tolerance limits (*m*, *σ* unknown);

and *s* is computed according to the formula

$$s = \left[\frac{\sum_{j=1}^n (x_j - \bar{x})^2}{n - 1} \right]^{1/2}$$

No individual outliers were removed from the results prior to the calculation of tolerance intervals.

Table 5. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	2.77	2.76	2.78

From the INAA data set an estimated tolerance interval of ±0.01 ppm at an analytical subsample weight of 50 gram was obtained (using the sampling constant relationship of Ingamells and Switzer, 1973) and is considered to reflect the actual homogeneity of the material under test. The meaning of this tolerance interval may be illustrated for gold (refer Table 5), where 99% of the time at least 95% of 50g-sized subsamples will have concentrations lying between 2.76 and 2.78 ppm. 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).

Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a routine laboratory being monitored by this standard in a QA/QC program. They incorporate errors attributable to bias, precision and inhomogeneity and are simply calculated from the standard deviation of the pooled individual analyses (fire assay data only) generated from the certification program. All individual and lab dataset (batch) outliers are removed prior to determination of the standard deviation. 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.

Table 6. Proposed performance gates for 7Pb

Constituent	Recommended value	Performance Gates					
		1 σ		2 σ		3 σ	
		Low	High	Low	High	Low	High
Gold, Au (ppm)	2.77	2.71	2.82	2.66	2.88	2.61	2.93

Performance gates have been calculated for one, two and three standard deviations of the accepted pool of certification data and are presented in Table 6. As a guide these intervals may be regarded as informational (1 σ), warning or rejection for multiple outliers (2 σ), or rejection for individual outliers (3 σ) in QC monitoring although their precise application should be at the discretion of the QC manager concerned.

PARTICIPATING LABORATORIES

Acme Analytical Laboratories Ltd, Vancouver, BC, Canada
 Activation Laboratories, Ancaster, Ontario, Canada
 Amdel Laboratories Ltd, Thebarton, SA, Australia
 ALS Chemex, Garbutt, QLD, Australia
 ALS Chemex, Reno, Nevada, USA
 ALS Chemex, Val-d'or, Quebec, Canada
 ALS Chemex, Vancouver, BC, Canada
 Becquerel Laboratories, Lucas Heights, NSW, Australia
 Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia
 Intertek Testing Services, Jakarta, Indonesia
 SGS Lakefield Geosol, Belo Horizonte MG, Brazil
 McPhar Laboratories, Legaspi Village, Makati City, Philippines
 OMAC Laboratories Ltd, Loughrea, County Galway, Ireland
 PT Indo Assay Laboratories, Balikpapan, Kalimantan Timur, Indonesia
 SGS Analabs, Welshpool, WA, Australia
 Standard & Reference Laboratories, Malaga, WA, Australia
 Ultra Trace, Canning Vale, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 7Pb has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd
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It is available in unit sizes of 60g foil packets and 1kg jars.

INTENDED USE

OREAS 7Pb is a reference material intended for the following:

- i) for the calibration of instruments used in the determination of the concentration of gold;
- ii) for the verification of analytical methods for gold;
- iii) for the preparation of secondary reference materials of similar composition;
- iv) as an arbitration sample for commercial transactions.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 7Pb has been prepared from gold-bearing metasediments within the oxidised zone of a mineralised shear zone. It is therefore considered to have long-term stability under normal storage conditions.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The recommended value for OREAS 7Pb refers to the concentration level of gold after removal of hygroscopic moisture by drying in air to constant mass at 105⁰ C. If the reference material is not dried by the user prior to analysis, the recommended value should be corrected to the moisture-bearing basis.

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: Dr Paul Hamlyn

REFERENCES

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

ISO Guide 35 (1985), *Certification of reference materials - General and statistical principals.*

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