

CERTIFICATE OF ANALYSIS FOR  
**GOLD REFERENCE MATERIAL**  
**OREAS 10P**

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	6.81	6.70	6.92

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99, \rho=0.95$	
		Low	High
Gold, Au (ppm)	6.81	6.72	6.90

*Prepared by:*  
*Ore Research & Exploration Pty Ltd*  
*May, 1996*

## 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 prospect evaluation.

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 10P was obtained from the Wonga mine in Stawell, west-central Victoria. It consists of a quartz-sericite-carbonate schist assemblage containing approximately 2% sulphides, mainly arsenopyrite and pyrite. Some of the gold is refractory in occurrence, being hosted by arsenopyrite.

The approximate major and trace element composition of this reference material is given in Table 1. The constituents SiO<sub>2</sub> to Total are the means of duplicate XRF analyses determined using a borate fusion method, while the remaining constituents, As to Zn, are means of twenty-three replicate analyses determined via INAA at Becquerel Laboratories.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 10P was prepared in the following manner:

- a) *primary crushing in a large (36 x 51cm) jaw crusher*
- b) *drying in a gas-fired rotary drier*
- c) *secondary crushing in a small (10 x 20cm) jaw crusher*
- d) *tertiary crushing in a roller crusher*

- e) *milling in a gamma mill*
- f) *screening to minus 75 micron in an air classifier*
- g) *homogenisation in a ribbon blender*
- h) *bagging into 20kg sublots*

The oversize fraction from the screening stage was re-milled and screened until a negligible amount remained. Throughout the bagging stage twenty-seven 1kg test units were taken at random intervals (determined using tables of random numbers), sealed in laminated plastic bags and set aside for analysis.

Prior to bottling in 1kg units each 20kg subplot was further homogenised in a tumble blender to counter the possibility of unmixing during handling. The resultant material constitutes the minus 75 micron reference material OREAS 10P.

## **ANALYSIS OF OREAS 10P**

Nineteen 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 Z. With the exception of Laboratory T, each laboratory received three 200g 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. Six laboratories (letter codes U to Z) were also instructed to conduct single gold determinations on each subsample using an aqua regia digest. In all instances a flame AAS finish was employed.

For each laboratory the three 200g 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 30g scoop split was taken from each of the twenty-seven random test units and submitted to Laboratory T for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 1g.

In all instances laboratories were requested to ensure rigorous analytical procedures were adhered to.

Individual assay results for the fire assay and INAA methods are presented in Tables 2 and 3 together with the mean, median and standard deviations (absolute and relative) for each data set. Interlaboratory agreement of the means of all but four data sets is good, lying within 6% relative of the raw mean of means of 6.71 ppm Au. The exceptions to this are laboratories D and G, which are both 6.0% higher, and N and P, which are 9.2% and 14.8% lower, respectively, than the raw mean of means.

Supplementary aqua regia/flame AAS data from six laboratories are reported in Table 4 and with a mean of means of  $6.52 \pm 0.30$  ppm Au (95% confidence) are, as anticipated, on average 4.3% lower than for fire assay determinations. The results obtained by the aqua regia digest method were not included in the determination of the recommended value.

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 10P

### 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 S according to the formulae

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

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

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  $i$ ;

$\bar{x}$  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 ( $p-1$ )

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

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

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 was based on the test criterion,  $T$ , and reference to tables of critical values of  $T$  at the 1% level of significance (ASTM E 178-94) as follows:

$$T_{ij} = \left| (x_{ij} - \bar{x}_i) \right| / s_i$$

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 10P; SiO<sub>2</sub> to Total as weight percent; rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO <sub>2</sub>	67.5	As	5011
TiO <sub>2</sub>	0.67	Ba	434
Al <sub>2</sub> O <sub>3</sub>	13.1	Ce	76
Fe <sub>2</sub> O <sub>3</sub>	6.64	Co	24
MnO	0.21	Cr	1824
MgO	2.38	Cs	6
CaO	1.45	Hf	3
Na <sub>2</sub> O	0.62	La	39
K <sub>2</sub> O	4.11	Rb	146
P <sub>2</sub> O <sub>5</sub>	0.07	Sb	6
SO <sub>3</sub>	1.07	Sc	12
H <sub>2</sub> O <sup>+</sup>	1.44	Sm	7
Total	99.26	Th	17
		Yb	2
		Zn	110

Table 2. Analytical results for gold (ppm) in OREAS 10P by 50g fire assay/ flame AAS (Std. Dev. - one sigma standard deviation; RSD – one sigma relative standard deviation; outliers in bold).

Unit	Replicate	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J
1	1	6.280	<b>6.140</b>	6.400	7.140	6.950	6.760	7.060	7.100	6.570	6.820
2	1	6.620	6.680	6.430	7.080	6.870	6.760	7.060	6.950	6.510	6.890
3	1	6.630	6.620	6.680	7.080	6.910	6.750	7.020	7.200	6.720	6.810
1	2	6.280	6.560	6.740	7.200	6.990	6.960	7.060	7.050	6.720	6.830
2	2	6.400	6.720	6.740	7.200	6.740	6.870	7.220	7.100	6.630	6.850
3	2	6.590	6.600	6.440	6.960	6.720	6.680	7.220	7.100	6.790	6.790
Mean:		6.467	6.553	6.572	7.110	6.863	6.797	7.107	7.083	6.657	6.832
Median:		6.495	6.610	6.560	7.110	6.890	6.760	7.060	7.100	6.675	6.825
Std. Dev.:		0.167	0.210	0.164	0.091	0.111	0.101	0.089	0.082	0.105	0.035
RSD:		2.6%	3.2%	2.5%	1.3%	1.6%	1.5%	1.3%	1.2%	1.6%	0.5%

Table 2. Continued.

Unit	Replicate	Lab K	Lab L	Lab M	Lab N	Lab O	Lab P	Lab Q	Lab R	Lab S
1	1	6.550	7.040	6.500	6.440	6.960	5.734	6.890	6.590	7.200
2	1	6.510	7.000	6.800	6.030	6.990	5.568	6.860	6.640	7.000
3	1	6.640	6.340	6.800	6.050	7.000	5.692	6.830	6.670	7.000
1	2	6.600	6.680		6.480	7.000	5.786	6.910	6.650	7.000
2	2	6.450	6.940		5.860	6.990	5.722	6.790	6.520	7.200
3	2	6.300	6.760		5.670	7.000	5.778	6.890	6.640	7.200
Mean:		6.508	6.793	6.700	<b>6.088</b>	6.990	<b>5.713</b>	6.862	6.618	7.100
Median:		6.530	6.850	6.800	6.040	6.995	5.728	6.875	6.640	7.100
Std. Dev.:		0.122	0.263	0.173	0.319	0.015	0.079	0.045	0.055	0.110
RSD:		1.9%	3.9%	2.6%	5.2%	0.2%	1.4%	0.7%	0.8%	1.5%

Table 3. Analytical results for gold (ppm) in OREAS 10P by instrumental neutron activation analysis on 1g analytical subsample weights (abbreviations as for Table 2).

Unit No.	Lab T
1	6.431
2	6.489
3	6.534
4	6.217
5	6.492
6	6.692
7	6.645
8	6.559
9	6.698
10	6.166
11	6.454
12	6.617
13	6.397
14	6.688
15	6.894
16	6.811
17	6.189
18	6.301
19	6.773
20	6.591
21	6.143
22	6.389
23	6.500
Mean:	6.507
Median:	6.500
Std. Dev.:	0.210
RSD:	3.2%

Table 4. Analytical results for gold (ppm) in OREAS 10P by aqua regia digest / flame AAS on 25-30g analytical subsample weights (abbreviations as for Table 2).

Unit	Lab U	Lab V	Lab W	Lab X	Lab Y	Lab Z
1	6.510	6.220	6.820	6.180	6.500	6.570
2	6.450	6.100	7.140	6.660	<b>5.300</b>	6.950
3	6.290	5.960	7.100	6.240	6.800	6.160
Mean:	6.417	6.093	7.020	6.360	6.200	6.560
Median:	6.450	6.100	7.100	6.240	6.500	6.570
Std. Dev.:	0.114	0.130	0.174	0.262	0.794	0.395
RSD:	1.8%	2.1%	2.5%	4.1%	12.8%	6.0%

where

$T_{ij}$  is the test criterion for the  $j$ th result of laboratory  $i$ ;  
 $s_i$  is the standard deviation of laboratory  $i$ .

The same principles were applied in testing for outlying laboratory means. Individual and mean outliers are shown in bold type in Table 2 and have been omitted in the determination of recommended values.

Table 5. Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	6.81	6.70	6.92

### 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 1g 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'<sub>2</sub>* 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.**

From the INAA data set an estimated tolerance interval of ±0.09ppm at an analytical subsample weight of 50g was obtained (using the sampling constant relationship of Ingamells and Switzer, 1973) and is considered to reflect the actual inhomogeneity of the material under test. The meaning of this tolerance interval may be illustrated for gold (refer Table 6), where 99% of the time at least 95% of 50g-sized subsamples will have

concentrations lying between 6.72 and 6.90ppm. 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).

Table 6. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Gold, Au (ppm)	6.81	6.72	6.90

The two-stage nested design adopted for the interlaboratory programme entailed each laboratory completing two replicate fire assay determinations on each of the three units received. This enabled the homogeneity to be also evaluated using an Analysis of Variance (ANOVA) approach. The results of this treatment, modified for unbalanced data, are summarised in Table 7. The between-unit mean square is of similar magnitude to the

Table 7. ANOVA table.

Source	Sum of squares	Degrees of freedom	Mean square
Between units	0.740667	36	0.020574 (MS1)
Within units	0.700358	51	0.013733 (MS2)

within-unit mean square for which:

$$\text{Test statistic} = MS1/MS2 = 1.498$$

(Note: the results of Laboratory N have been excluded from this analysis as they are considered to exhibit poor analytical precision and produce an erroneous bias. If included, a test statistic of 2.097 is obtained.)

and the critical values for the F-test (one-sided) are:

1.75 at the 5% significance level  
2.22 at the 1% significance level.

We may conclude, therefore, that there is no evidence to indicate that the between-units variance is greater than that within units and that the homogeneity of the entire batch of OREAS 10P is of an acceptable level.

## **PARTICIPATING LABORATORIES**

Amdel Laboratories Ltd, Darwin, NT, Australia  
Amdel Laboratories Ltd, Thebarton, SA, Australia  
Amdel Laboratories Ltd, Wangara, WA, Australia  
Ammtec Limited, Balcatta, WA, Australia  
Analabs Pty Ltd, Cooee, TAS, Australia  
Analabs Pty Ltd, East Brisbane, QLD, Australia  
Analabs Pty Ltd, Townsville, QLD, Australia  
Analabs Pty Ltd, Welshpool, WA, Australia  
Anglo American Research Laboratories Pty Ltd, Johannesburg, South Africa  
Assaycorp Pty Ltd, Pine Creek, NT, Australia  
Australian Laboratory Services Pty Ltd, Bendigo, VIC, Australia  
Australian Laboratory Services Pty Ltd, Stafford, QLD, Australia  
Australian Laboratory Services Pty Ltd, Malaga, WA, Australia  
Becquerel Laboratories, Lucas Heights, NSW, Australia  
Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia  
Minlab, Malaga, WA, Australia  
SGS Australia Pty Ltd, Queens Park, WA, Australia  
Ultra Trace, Bentley, WA, Australia  
Western Mining Corporation Ltd, Kalgoorlie, WA, Australia

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

The gold ore reference material, OREAS 10P has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd  
3 London Drive  
Bayswater VIC 3153  
AUSTRALIA

Telephone	(03) 9762 1808	International	+613-9762 1808
Facsimile	(03) 9762 3808	International	+613-9762 3808
Email	info@ore.com.au		
Web	www.ore.com.au		

It is available in unit sizes of 60 grams and 1 kg.

## **INTENDED USE**

OREAS 10P 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 10P has been prepared from a gold-bearing quartz-sericite-carbonate schist containing approximately 2% sulphides (mainly arsenopyrite and pyrite). Although its sulphide content is low, care should be exercised in minimising exposure to air to reduce the possibility of oxidation.

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

The recommended value for OREAS 10P refers to the concentration level of gold after removal of hygroscopic moisture by drying in air to constant mass at 105<sup>0</sup> C. In its undried state a hygroscopic moisture content of 0.40% has been established. 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

## **ACKNOWLEDGMENTS**

The costs of development of this reference material were sponsored in part by the Australian Minerals Industry Research Association Limited (AMIRA Project 388) through funds contributed by Acacia Resources Limited, BHP Minerals, MIM Exploration Pty Ltd and Western Mining Corporation Limited. The gold ore sample from which OREAS 10P was prepared was provided by Stawell Gold Mines. The assistance of these organisations and the cooperation of all participating laboratories is warmly acknowledged.

## **REFERENCES**

ASTM E178-94 (1994), Standard practice for dealing with outlying observations.

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.