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CERTIFICATE OF ANALYSIS FOR

GOLD REFERENCE MATERIAL

OREAS 2Pa

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	854	837	871

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99$, $\rho=0.95$	
		Low	High
Gold, Au (ppb)	854	847	861

Prepared by:
Ore Research & Exploration Pty Ltd
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REPORT 2Pa

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-bearing standard OREAS 2Pa was taken from the flanks of 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 2Pa is given in Table 1. The constituents SiO_2 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-seven replicate analyses determined via INAA at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 2Pa 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 sublot 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 2Pa.

ANALYSIS OF OREAS 2Pa

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 three data sets is good, lying within 6% relative of the raw mean of means of 854 ppb Au. The exceptions to this are laboratories A, D and H which are 8.5%, 6.7% and 6.3% higher, 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 763 ± 69 ppb Au (95% confidence) are, as anticipated, on average 11% 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 2Pa

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

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

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

where

x_{ij} *is the jth result reported by laboratory i;*
p *is the number of participating laboratories;*

n_i *is the number of results reported by laboratory i;*

$\overline{x}_i$ *is the mean for laboratory i*;

$\ddot{x}$ *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)

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

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

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.

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 2Pa; SiO2 to Total as weight percent; rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO_2	73.8	As	760
TiO_2	0.54	Ba	605
Al_2O_3	12.5	Ce	72
Fe_2O_3	4.97	Co	3
MnO	<0.01	Cr	463
MgO	0.66	Cs	8
CaO	0.65	Hf	4
Na_2O	0.11	La	34
K_2O	3.20	Rb	140
P_2O_5	0.06	Sb	72
SO_3	0.03	Sc	12
H_2O^+	2.95	Sm	7
Total	99.47	Th	13
		W	8
		Yb	2
		Zn	1340

Table 2. Analytical results for gold (ppm) in OREAS 2Pa 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	0.954	0.850	0.820	0.920	0.870	0.830	0.843	0.880	0.820	0.930
2	1	0.908	0.850	0.830	0.910	0.870	0.840	0.848	0.925	0.830	0.890
3	1	0.932	0.850	0.780	0.910	0.900	0.780	0.848	0.895	0.820	0.870
1	2	0.925	0.870	0.830	0.910	0.810	0.820	0.863	0.900	0.820	0.920
2	2	0.926	0.860	0.820	0.910	0.830	0.810	0.833	0.955	0.810	0.900
3	2	0.919	0.860	0.780	0.910	0.820	0.850	0.837	0.895	0.860	0.880
Mean:		0.927	0.857	0.810	0.912	0.850	0.822	0.845	0.908	0.827	0.898
Median:		0.926	0.855	0.820	0.910	0.850	0.825	0.846	0.898	0.820	0.895
Std. Dev.:		0.015	0.008	0.024	0.004	0.035	0.025	0.011	0.027	0.018	0.023
RSD:		1.7%	1.0%	2.9%	0.4%	4.1%	3.0%	1.2%	3.0%	2.1%	2.6%

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	0.832	0.840	0.820	0.850	0.890	0.842	0.798	0.820	0.860
2	1	0.819	0.800	0.820	0.860	0.900	0.862	0.813	0.830	0.870
3	1	0.810	0.870	0.810	0.840	0.920	0.876	0.805	0.840	0.900
1	2	0.815	0.760		0.860	0.890	0.843	0.802	0.800	0.880
2	2	0.840	0.850		0.820	0.890	0.857	0.849	0.830	0.860
3	2	0.868	0.850		0.840	0.860	0.866	0.792	0.840	0.860
Mean:		0.831	0.828	0.817	0.845	0.892	0.858	0.810	0.827	0.872
Median:		0.826	0.845	0.820	0.845	0.890	0.860	0.804	0.830	0.865
Std. Dev.:		0.021	0.041	0.006	0.015	0.019	0.013	0.020	0.015	0.016
RSD:		2.6%	4.9%	0.7%	1.8%	2.2%	1.6%	2.5%	1.8%	1.8%

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

Unit No.	Lab T
1	0.830
2	0.871
3	0.833
4	0.885
5	0.843
6	0.854
7	0.894
8	0.854
9	0.848
10	0.862
11	0.865
12	0.848
13	0.876
14	0.844
15	0.833
16	0.846
17	0.864
18	0.864
19	0.887
20	0.846
21	0.858
22	0.871
23	0.863
24	0.858
25	0.878
26	0.877
27	0.874
Mean:	0.860
Median:	0.862
Std. Dev.:	0.017
RSD:	2.0%

Table 4. Analytical results for gold (ppm) in OREAS 2Pa 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	0.71	0.82	0.68	0.78	0.84	0.68
2	0.80	0.80	0.70	0.78	0.90	0.68
3	0.69	0.81	0.70	0.78	0.88	0.70
Mean:	0.733	0.810	0.693	0.780	0.873	0.687
Median:	0.710	0.810	0.700	0.780	0.880	0.680
Std. Dev.:	0.059	0.010	0.012	0.000	0.031	0.012
RSD:	8.0%	1.2%	1.7%	0.0%	3.5%	1.7%

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:

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

where

T_{ij} *is the test criterion for the jth 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 (ppb)	854	837	871

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

$$\textit{Lower limit is } \ddot{x} - k_2'(n, p, 1-\alpha)s$$
$$\textit{Upper limit is } \ddot{x} + k_2'(n, p, 1-\alpha)s$$

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

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

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 ± 7ppb 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 847 and 861ppb. 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 (IS0 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 (ppb)	854	847	861

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

Table 7. ANOVA table.

Source	Sum of squares	Degrees of freedom	Mean square
Between units	0.017776	38	0.000468 (MS1)
Within units	0.023491	54	0.000435 (MS2)

to the within-unit mean square for which:

$$Test\ statistic = MS1/MS2 = 1.075$$

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

1.71 at the 5% significance level
2.15 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 2Pa 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 2Pa has been prepared and certified and is supplied by:

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It is available in unit sizes of 60g foil packets and 1kg jars.

INTENDED USE

OREAS 2Pa 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 2Pa 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 2Pa refers to the concentration level of gold after removal of hygroscopic moisture by drying in air to constant mass at 105^0 C. In its undried state a hygroscopic moisture content of 0.42% 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

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