

CERTIFICATE OF ANALYSIS FOR

GOLD ORE REFERENCE MATERIAL

OREAS 7Pa

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	3.00	2.94	3.06

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99, \rho=0.95$	
		Low	High
Gold, Au (ppm)	3.00	2.98	3.02

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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 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 7Pa 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 7Pa is given in Table 1. The constituents SiO₂ to Total are the means of duplicate XRF analyses determined using a borate fusion method, while the remaining constituents, As to Yb, are means of twenty-seven replicate analyses determined via INAA at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 7Pa 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 7Pa.

ANALYSIS OF OREAS 7Pa

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 3.00 ppm Au. The exceptions to this are laboratories A, B, M and P which are 6.4% higher, 6.5% lower, 7.3% lower and 6.6% 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 2.67 ± 0.19 ppm 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 7Pa

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

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 ;

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 ($p-1$):

Confidence limits

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

Constituent	Concentration	Constituent	Concentration
SiO ₂	64.8	As	2184
TiO ₂	0.83	Ba	946
Al ₂ O ₃	18.8	Ce	114
Fe ₂ O ₃	4.74	Cr	288
MnO	<0.01	Cs	14
MgO	0.89	Hf	5
CaO	0.03	La	63
Na ₂ O	<0.05	Rb	214
K ₂ O	5.23	Sb	162
P ₂ O ₅	0.10	Sc	17
SO ₃	0.04	Sm	12
H ₂ O ⁺	4.01	Th	21
Total	99.47	W	25
		Yb	4

Table 2. Analytical results for gold (ppm) in OREAS 7Pa 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	3.070	2.910	2.920	3.120	3.110	2.950	3.150	3.130	2.990	3.020
2	1	3.290	2.810	2.610	3.170	3.210	3.060	3.090	3.180	2.900	3.090
3	1	3.340	2.710	2.680	3.120	3.160	2.890	3.080	3.100	3.050	2.990
1	2	3.050	2.940	2.990	3.120	3.190	3.050	3.100	3.110	2.920	2.960
2	2	3.300	2.740	3.070	3.080	3.150	2.980	3.140	3.120	2.850	3.100
3	2	3.070	2.690	2.900	3.150	3.180	2.990	3.090	3.160	2.990	2.990
Mean:		3.187	2.800	2.862	3.127	3.167	2.987	3.108	3.133	2.950	3.025
Median:		3.180	2.775	2.910	3.120	3.170	2.985	3.095	3.125	2.955	3.005
Std. Dev.:		0.136	0.105	0.179	0.031	0.035	0.063	0.029	0.031	0.073	0.058
RSD:		4.3%	3.8%	6.3%	1.0%	1.1%	2.1%	0.9%	1.0%	2.5%	1.9%

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	2.760	2.940	2.840	3.080	3.040	2.783	2.920	2.890	3.200
2	1	2.940	3.090	2.670	3.040	3.180	2.789	2.940	2.920	2.800
3	1	2.820	3.020	2.820	3.050	3.140	2.805	2.890	2.950	3.200
1	2	2.930	3.030		3.040	3.050	2.790	2.860	3.020	3.200
2	2	2.850	2.910		3.030	3.050	2.807	2.880	2.960	3.100
3	2	3.070	3.000		3.040	3.080	2.813	2.980	2.960	3.100
Mean:		2.895	2.998	2.777	3.047	3.090	2.798	2.912	2.950	3.100
Median:		2.890	3.010	2.820	3.040	3.065	2.798	2.905	2.955	3.150
Std. Dev.:		0.109	0.065	0.093	0.018	0.057	0.012	0.044	0.044	0.155
RSD:		3.8%	2.2%	3.3%	0.6%	1.9%	0.4%	1.5%	1.5%	5.0%

Table 3. Analytical results for gold (ppm) in OREAS 7Pa by instrumental neutron activation analysis on 1g analytical subsample weights (abbreviations as for Table 2).

Unit No.	Lab T
1	2.924
2	2.965
3	2.986
4	3.053
5	2.946
6	2.979
7	2.927
8	3.128
9	2.953
10	2.923
11	3.061
12	2.982
13	2.991
14	3.054
15	2.948
16	2.980
17	2.959
18	2.958
19	2.930
20	3.012
21	3.042
22	3.023
23	3.015
24	2.947
25	2.935
26	2.936
27	3.008
Mean:	2.984
Median:	2.979
Std. Dev.:	0.051
RSD:	1.7%

Table 4. Analytical results for gold (ppm) in OREAS 7Pa 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	2.46	2.76	2.69	2.70	2.88	2.49
2	2.38	2.90	2.81	2.76	3.05	2.48
3	2.39	2.81	2.60	2.46	2.94	2.56
Mean:	2.410	2.823	2.700	2.640	2.957	2.510
Median:	2.390	2.810	2.690	2.700	2.940	2.490
Std. Dev.:	0.044	0.071	0.105	0.159	0.086	0.044
RSD:	1.8%	2.5%	3.9%	6.0%	2.9%	1.7%

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:

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)	3.00	2.94	3.06

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

Lower limit is

Upper limit is

where

and s is computed according to the formula

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 ± 0.02 ppm 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 2.98 and 3.02ppm. 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)	3.00	2.98	3.02

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 within-unit mean square for which:

$$\text{Test statistic} = MS1/MS2 = 1.227$$

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.

Table 7. ANOVA table.

Source	Sum of squares	Degrees of freedom	Mean square
Between units	0.308673	38	0.008123 (MS1)
Within units	0.357419	54	0.006619 (MS2)

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 7Pa 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 7Pa 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 7Pa 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 7Pa 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 7Pa refers to the concentration level of gold after removal of hygroscopic moisture by drying in air to constant mass at 105⁰ C. In its undried state a hygroscopic moisture content of 0.76% 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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