ORE RESEARCH & EXPLORATION PTY LTD
Unit 23, 993 North Road, Murrumbeena, Vic 3163 AUSTRALIA
Telephone: 61-03-9570 5300 Facsimile: 61-03-9570 5221

CERTIFICATE OF ANALYSIS FOR

GOLD REFERENCE MATERIAL

OREAS 2Ca

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	599	580	618

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppb)	599	542	656

Prepared by:
Ore Research & Exploration Pty Ltd
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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 transparent 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 2Ca 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 2Ca is given in Table 1. The constituents SiO_2 to Total are the means of duplicate XRF analyses determined by the borate fusion method, while the remaining constituents, As to Zn, are means of twenty-five replicate analyses determined via INAA at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 2Ca 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) *screening to minus 3mm*
f) *milling of approximately one third in a gamma mill*
g) *homogenisation in a ribbon blender*
h) *bagging into 20kg sublots*

Throughout the bagging stage twenty-five 1kg samples were taken at random intervals, sealed in laminated plastic bags and set aside for analysis. These random sampling intervals were determined using tables of random numbers.

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 3mm reference material OREAS 2Ca.

For homogeneity testing a 250g scoop split was taken from each of the twenty-five 1kg chip samples and pulverised separately for 3 minutes in a vibratory ring mill. One 30g scoop split was then taken from each of the 250g pulps for gold assay via instrumental neutron activation analysis on 1g subsamples. The remaining 750g portions of the twenty-five chip samples were each pulverised separately for 3 minutes in a vibratory ring mill and split into 200g subsamples for distribution to the laboratories participating in the round robin.

ANALYSIS OF OREAS 2Ca

Seventeen laboratories participated in the analytical program and are listed in the section headed Participating Laboratories.

To ensure anonymity these laboratories have been randomly designated the letter codes A through Q. With the exception of Laboratory Q, each laboratory received three 200g subsamples with instructions to carry out duplicate fire assays for gold (using 50g charges) on each subsample. Laboratories D, E, F, G, N & L 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 selected from different 750g pulps produced from the 1kg random samples taken during the bagging stage. The results therefore provide an assessment of both the within- and between-unit homogeneity and are amenable to analysis of variance treatment. Laboratory Q received twenty-five 50g subsamples split from the twenty-five 250g pulps produced from the 1kg random samples described above. The instructions were to complete one assay per subsample via instrumental neutron activation analysis on an analytical subsample weight of 30g.

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

Individual assay results for the fire assay/AAS and INAA methods are presented in Tables 2 and 3 together with the mean, median and standard deviations (absolute and relative) given for each data set. Interlaboratory agreement of the means of all but three data sets is good, lying within 5% relative of the raw mean of means of 598 ppb Au. The exceptions to this are laboratories E, N and O which are 12.6% higher, 11.5% lower and 14.2% 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 563 ± 47 ppb Au (95% confidence) are, as anticipated, on average 6% 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 2Ca

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

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

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

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;*

$\bar{x}_i$ *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)

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

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

Constituent	Concentration	Constituent	Concentration
SiO2	69.9	As	580
TiO2	0.71	Ba	744
Al2O3	15.2	Ce	100
Fe2O3	5.29	Co	5
MnO	0.01	Cr	844
MgO	0.82	Cs	10
CaO	0.04	Hf	6
Na2O	<0.05	La	48
K2O	4.11	Rb	179
P2O5	0.10	Sb	76
SO3	<0.01	Sc	16
H2O+	3.44	Sm	9
Total	99.62	Th	17
		U	5
		W	11
		Yb	3
		Zn	293

Table 2. Analytical results for gold (ppm) in OREAS 2Ca 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
1	1	0.531	0.58	0.58	0.60	0.65	0.613	0.594	0.55
2	1	0.634	0.59	0.58	0.59	0.68	0.604	0.638	0.56
3	1	0.593	0.59	0.60	0.62	0.68	0.620	0.628	0.57
1	2	0.529	0.60	0.57	0.59	0.65	**0.522**	0.588	0.59
2	2	0.607	0.59	0.58	0.60	0.72	0.606	0.579	0.57
3	2	0.595	0.59	0.59	0.60	0.66	0.636	0.622	0.57
Mean:		0.582	0.590	0.583	0.600	0.673	0.600	0.608	0.568
Median:		0.594	0.590	0.580	0.600	0.670	0.610	0.608	0.570
Std. Dev.:		0.042	0.006	0.010	0.011	0.027	0.040	0.024	0.013
RSD:		7.3	1.1	1.8	1.8	3.9	6.7	4.0	2.3

Table 2. Continued.

Unit	Replicate	Lab I	Lab J	Lab K	Lab L	Lab M	Lab N	Lab O	Lab P
1	1	0.63	0.608	0.59	0.57	0.580	0.506	0.68	0.60
2	1	0.61	0.588	0.58	0.60	0.590	0.570	0.70	0.61
3	1	0.62	0.612	0.59	0.56	0.560	0.541	0.68	0.59
1	2	0.62	0.576	0.61	0.60	0.590	0.504	0.68	0.61
2	2	0.61	0.607	0.58	0.60	0.600	0.563	0.68	0.59
3	2	0.61	0.613	0.60	0.51	0.570	0.492	0.68	0.60
Mean:		0.617	0.601	0.592	0.573	0.582	0.529	0.683	0.600
Median:		0.615	0.608	0.590	0.585	0.585	0.524	0.680	0.600
Std. Dev.:		0.008	0.015	0.012	0.036	0.015	0.033	0.008	0.009
RSD:		1.3	2.5	2.0	6.2	2.5	6.3	1.2	1.5

Table 3. Analytical results for gold (ppm) in OREAS 2Ca by instrumental neutron activation analysis on 30g analytical subsample weights (abbreviations as for Table 2).

Unit No.	Lab Q
1	0.575
2	0.653
3	0.554
4	0.565
5	0.561
6	0.574
7	0.600
8	0.565
9	0.563
10	0.556
11	0.586
12	0.570
13	0.562
14	0.564
15	0.581
16	0.636
17	0.580
18	0.566
19	0.569
20	0.603
21	0.607
22	0.572
23	0.596
24	0.579
25	0.580
Mean:	0.581
Median:	0.574
Std. Dev.:	0.024
RSD:	4.1

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

Unit	Lab D	Lab E	Lab F	Lab G	Lab L	Lab N
1	0.50	0.47	0.53	0.61	0.64	0.52
2	0.51	0.70	0.56	0.61	0.62	0.52
3	0.50	0.46	0.61	0.63	0.61	0.53
Mean:	0.503	0.543	0.567	0.617	0.623	0.523
Median:	0.500	0.470	0.560	0.610	0.620	0.520
Std. Dev.:	0.006	0.136	0.040	0.012	0.015	0.006
RSD:	1.1	25.0	7.1	1.9	2.5	1.1

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

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.

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 3 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)	599	580	618

Statement of Homogeneity

The variability of replicate assays from each laboratory is a result of both measurement and subsampling errors. In the determination of tolerance limits it is desirable 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 sampling error and measurement error becomes negligible. This approach has been adopted for pulp reference materials where no additional processing is involved prior to taking the analytical subsample. In the case of chip RM's, however, this method is inappropriate as two stages of subsampling and an intervening pulverisation stage are involved. Instead homogeneity has been determined by taking a 30g subsample from each of the twenty-five 250g pulps prepared from the 1kg random chip samples described earlier and assaying via INAA. From these results (Table 3) an estimated tolerance interval of ± 57ppb, calculated for an analytical subsample weight of 50g (from the sampling constant relationship of Ingamells and Switzer, 1973), was obtained using tables of factors for two-sided tolerance limits for normal distributions (ISO Guide 3207) in which

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

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 outliers were removed from the results prior to the calculation of tolerance intervals.

The meaning of these tolerance limits may be illustrated for gold (refer Table 6), where 99% of the time at least 95% of 250g-sized subsamples will have concentrations lying between 542 and 656ppb. 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). Obviously, if OREAS 2Ca is subsampled in weights less than or greater than 250g, the anticipated tolerance interval will be greater than or less than, respectively, that of ± 57ppb.

Table 6. Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppb)	599	542	656

It should be noted that these tolerance intervals are based on the assumption that all of the observed variablilty is attributable to sampling error. In reality, the contribution of measurement error may be significant and therefore, the estimate of homogeneity should be regarded as conservative.

PARTICIPATING LABORATORIES

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 Assay Laboratories Pty Ltd, Balcatta, WA, Australia
Australian Laboratory Services Pty Ltd, Bendigo, VIC, 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
Western Mining Corporation Ltd, Kalgoorlie, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 2Ca has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd
Unit 23, 993 North Road
Murrumbeena VIC 3163
AUSTRALIA

Telephone (03) 9570 5300 International 61-3-9570 5300
Facsimile (03) 9570 5221 International 61-3-9570 5221

It is available in unit sizes of 1kg.

INTENDED USE

OREAS 2Ca is a reference material intended for the verification of sample preparation and analytical methods for gold.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 2Ca 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 2Ca 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 0.54% 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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