
ORE RESEARCH & EXPLORATION PTY LTD

Unit 23, 993 North Road, Murrumbena, Vic 3163 AUSTRALIA

Telephone: 61-3-9570 5300 Facsimile: 61-3-9570 5221

CERTIFICATE OF ANALYSIS FOR
GOLD REFERENCE MATERIAL
OREAS 2C BATCH 2D3

Recommended value and 95% confidence limits

Constituent	Recommended value	95% Confidence limits	
		Low	High
Gold, Au (ppb)	371	354	388

Recommended value and tolerance limits

Constituent	Recommended value	Tolerance limits $1-\alpha=0.99, \rho=0.95$	
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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.

SOURCE MATERIALS

The material used to produce gold-bearing standard OREAS 2C Batch 2D3 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 major and trace element composition of this oxidised, quartz-veined metagreywacke comprising gold ore standard OREAS 2C Batch 2D3 is given in Table 1. The constituents SiO₂ to Zr are the means of duplicate XRF analyses determined at the University of Melbourne using a low dilution borate fusion method, while the remaining constituents, As to Zn, are means of at least ten replicate analyses determined via INAA at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

Approximately 300kg of the gold-bearing greywacke was subjected to the following procedure:

- 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 pan mill*
- e) screening to minus 3mm*

Following crushing, the material was split into ten equal sublots which were homogenised separately in a tumble blender for two hours each. A tenth split from each sublot was then combined and blended for a further two hours, two 250g subsamples set aside for assay and the remainder collected in a 40 litre container. This process was repeated nine times until all the material had been recombined, homogenised, subsampled and collected in separate 40 litre containers. The resultant material constitutes the minus 3mm

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reference material OREAS 2C Batch 2D3. Each of the 250g subsamples were pulverised for 5 minutes in a vibratory ring mill and split into 100g lots prior to submission for assay.

ANALYSIS OF OREAS 2C BATCH 2D3

Seven laboratories participated in the analytical program and are listed in Appendix I.

To ensure anonymity these laboratories have been randomly designated the letter codes A through G (note: laboratory A analysed gold by two different methods and is designated as A1 and A2 in Table 2). Material was submitted to each laboratory prepackaged in 100g subsamples. Laboratories A to F were instructed to carry out four replicate assays for gold on 50g charges. Laboratories A1, B, C and D used a fire assay/carbon rod AAS procedure, A1 employed fire assay/ICP-MS, E fire assay/solvent extraction/flame AAS, and F fire assay/flame AAS. Laboratory G was instructed to complete 10 replicate assays on 30g subsamples for gold, arsenic, antimony and 26 additional elements using instrumental neutron activation analysis.

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

The results are presented in Table 2 with the analytical subsample weight and method indicated at the head of each column and the mean, median and two sigma standard deviations (absolute and relative) given for each laboratory data set. Interlaboratory agreement of the means of each data set is generally good with the exception of E which is 15.3% lower than the uncorrected global mean of 360ppb Au.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 2C BATCH 2D3

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 G (note: to eliminate over-representation on

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 2C Batch 2D3; SiO₂ to Total as weight percent; rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO ₂	64.43	As	162
TiO ₂	0.7	Ba	1270
Al ₂ O ₃	20.36	Ce	114
Fe ₂ O ₃	3.28	Co	29
MnO	0.01	Cr	116
MgO	1.27	Cs	16
CaO	0.04	Hf	4
Na ₂ O	0.23	La	54
K ₂ O	5.58	Rb	255
P ₂ O ₅	0.06	Sb	83
H ₂ O+	3.78	Sc	18
Total	99.74	Sm	10
Nb	15	Th	18
Pb	35	Yb	3
S	65	Zn	72
Sr	117		
V	137		
Y	41		
Zr	130		

Table 2. Analytical results for gold in OREAS 2C Batch 2D3 (FA*MS - fire assay/inductively coupled plasma - mass spectrometry; FA*CR - fire assay/carbon rod atomic absorption spectrometry; FA*AAS - fire assay/atomic absorption spectrometry; FA*EX - fire assay/solvent extraction/atomic absorption spectrometry; INAA - instrumental neutron activation analysis; sample weight in parentheses; Std.Dev.(x2) - two sigma standard deviation; RSD(x2) - two sigma relative standard deviation; outliers in bold).

Replicate No.	Lab A1 FA*CR (50g)	Lab A2 FA*MS (50g)	Lab B FA*CR (50g)	Lab C FA*CR (50g)	Lab D FA*CR (50g)	Lab E FA*EX (50g)	Lab F FA*AAS (50g)	Lab G INAA (30g)
1	350	354	400	400	364	300	365	369
2	350	344	400	400	378	320	348	378
3	350	337	390	365	380	300	352	374
4	350	337	390	335	372	300	378	392
5							378	366
6								381
7								377
8								385
9								364
10								360
mean	350.0	343.0	395.0	375.0	373.5	305.0	364.2	374.6
median	350.0	340.5	395.0	382.5	375.0	300.0	365.0	375.5
Std. Dev.(x2)	0.0	16.1	11.5	62.7	14.4	20.0	28.2	20.0
RSD(x2)	0.0	4.7	2.9	16.7	3.8	6.6	7.7	5.3

the part of laboratory A a weighting factor of 0.5 was applied to the means of A1 and A2) according to the formulae

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

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

where

- x_j* 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*;
- $\bar{\bar{x}}$ 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)

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

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

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 assay outliers and of outlying laboratory means 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

$$S = 1.483 \operatorname{median}_{j=1, \dots, n} |x_j - \operatorname{median}_{i=1, \dots, n}(x_i)|$$

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

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.

Individual assay and mean outliers are shown in bold type in Table 2 and have been omitted in the determination of recommended values.

Table 3. Recommended values and 95% confidence limits

Constituent	Recommended value	95% Confidence limits	
		Low	High
Gold, Au (ppb)	371	354	388

Statement of Homogeneity

The standard deviation of each laboratory data set includes error due both to the imprecision of the analytical method employed and to possible inhomogeneity of the material analysed. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material analysed and, in particular, to deficiencies in accuracy of each analytical method. In determining tolerance intervals the component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \bar{x}_i + \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^p n_i}$$

where

- x_{ij} is the j th raw result reported by laboratory i ;
- x'_{ij} is the j th transformed result reported by laboratory i ;
- n_i is the number of results reported by laboratory i ;
- p is the number of participating laboratories;
- \bar{x}_i is the raw mean for laboratory i .

The homogeneity was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$\begin{aligned} \text{Lower limit is } \bar{x} - k'_2(n,p,1-\alpha)s_g'' \\ \text{Upper limit is } \bar{x} + k'_2(n,p,1-\alpha)s_g'' \end{aligned}$$

where

- n is the number of results;
- $1-\alpha$ 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);
- s_g'' is the corrected grand standard deviation.

The corrected grand standard deviation, S_g'' , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula

$$s_g'' = \frac{\sum_{i=1}^p (s_i(1 - \frac{s_i}{s_g}))}{\sum_{i=1}^p (1 - \frac{s_i}{s_g})}$$

where

- $1 - (\frac{s_i}{s_g})$ is the weighting factor for laboratory i ;
- s_g' is the grand standard deviation computed from the transformed (i.e. means-adjusted) results

according to the formula

$$s_g' = \left[\frac{\sum_{i=1}^p \sum_{j=i}^p n_i (x_{ij}' - \bar{x}_i')^2}{\sum_{i=1}^p n_i - 1} \right]^{1/2}$$

where \bar{x}_i' is the transformed mean for laboratory i

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set.

No individual outliers were removed from the results prior to the calculation of tolerance intervals, however, a weighting factor of zero was applied to laboratory data sets where $s_i/4s_g' > 1$ (i.e. where the weighting factor $1 - s_i/4s_g' < 0$).

The meaning of these tolerance limits may be illustrated for gold (refer Table 4), where 99% of the time at least 95% of 250g-sized subsamples will have concentrations lying between 352 and 390ppm. 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). Obviously, if OREAS 2C Batch 2D3 is subsampled in weights less than or greater than 250g, the anticipated tolerance interval will be greater than or less than, respectively, that of ± 19 ppb.

Table 4. Recommended values and tolerance limits (subsample size of 250g).

Constituent	Recommended value	Tolerance limits 1- α =0.99, ρ =0.95	
		Low	High
Gold, Au (ppb)	371	352	390

REFERENCES

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.

APPENDIX I

List of Participating Laboratories:-

Analabs, Welshpool, WA, Australia

Australian Assay Laboratories, Balcatta, WA, Australia

Australian Laboratory Services, Stafford, QLD, Australia

Becquerel Laboratories, Lucas Heights, NSW, Australia

Classic Laboratories, Thebarton, SA, Australia

Genalysis Laboratory Services, Maddington, WA, Australia

SGS Australia, Queens Park, WA, Australia