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CERTIFICATE OF ANALYSIS FOR

GOLD ORE REFERENCE MATERIAL

OREAS 5Pb

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	98	96	100

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1\text{-}\alpha=0.99$, $\rho=0.95$	
		Low	High
Gold, Au (ppb)	98	96	100

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Ore Research & Exploration Pty Ltd
August, 2004

INTRODUCTION

OREAS certified reference materials (CRMs) 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 geologist they provide a means of implementing quality control in analytical data sets generated in exploration, from the grass roots level through to prospect evaluation, and in grade control at mining operations.

As a rule only source materials exhibiting a high 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 ore CRMs exhibiting homogeneity that matches or exceeds that of currently available international reference materials. In certain instances CRMs 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 CRM 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 20 to 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 gold ore standard OREAS 5Pb was prepared from gold-bearing greywacke material containing approximately 200ppb Au combined with barren siltstone material. The gold-bearing material 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-bearing standard OREAS 5Pb is given in Table 1. The constituents SiO_2 to Zr are the means of duplicate XRF analyses determined using a borate fusion method, while the remaining constituents, As to Yb, are means of twenty-five representative samples determined via INAA.

COMMINUTION AND HOMOGENISATION PROCEDURES

OREAS 5Pb was prepared from a blend of a gold-bearing greywacke sample and barren siltstone sample in the following manner:

- *a) drying each sample to constant mass at 105^0 C;*
- *b) crushing and screening each sample;*
- *c) milling gold-bearing greywacke to 100% minus 25 microns;*
- *d) milling barren siltstone to 98% minus 75 microns;*
- *e) thorough homogenisation*
- *f) bagging into 20kg sublots*
- *g) packaging into 60g lots sealed in laminated foil pouches.*

Throughout the bagging stage twenty-five 1kg test units were taken at regular intervals, sealed in laminated plastic bags and set aside for analysis.

ANALYSIS OF OREAS 5Pb

To maintain anonymity, the 17 laboratories have been randomly designated the letter codes A through Q. With the exception of Laboratory Q, each laboratory received four 110g samples with instructions to carry out one 30 to 50g fire assay determination for gold on each sample. Apart from Lab C and I (ICPOES finish) and Lab F (ICPMS finish) the remaining laboratories employed a flame AAS finish (Labs A, F, K, N, O, P using solvent extraction AAS).

For each laboratory two 110g subsamples were scoop-split from each of two separate 1kg 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 enables a comparative assessment of within- and between-unit homogeneity.

For the determination of a statistical tolerance interval, a 10g scoop split was taken from each of the twenty-five random test units and submitted to Laboratory Q for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 4g.

Individual assay results for the fire assay and INAA methods are presented in Tables 2 and 3 together with the mean, median and standard deviation (absolute and relative) for each data set. Interlaboratory agreement of the means of all but two data set is good, lying within 9.2% relative of the recommended value of 98 ppb Au. The exception to this is Labs H, O and P, which have been relegated to outlying status with a bias of +19.0%, -10.0% and -10.3% respectively.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 5Pb

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 and I to N 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)

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

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

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.

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 1. Indicative major and trace element composition of gold ore reference material OREAS 5Pb; SiO_2 to Total as weight percent; rest in ppm; SiO_2 to LOI by fusion XRF; As to Yb by INAA.

Constituent	Concentration (XRF)	Constituent	Concentration (INAA)
SiO_2	78.0	As	46
TiO_2	0.54	Ba	492
Al_2O_3	10.95	Ce	63
Fe_2O_3	4.54	Co	3
MnO	< 0.01	Cr	74
MgO	0.50	Cs	7
CaO	< 0.01	Eu	1
Na_2O	0.18	Hf	4
K_2O	2.14	La	31
P_2O_5	0.04	Rb	111
SO_3	0.04	Sb	21
LOI	2.96	Sc	11
Total	99.8	Sm	5
		Th	12
		Yb	2

Table 2. Analytical results for gold in OREAS standard 5Pb (Std.Dev. and Rel.Std.Dev. are one sigma values; Std.Dev. and Rel.Std.Dev. are one sigma values; PDM[3] - percent deviation of lab mean from corrected mean of means; outliers in bold; values in ppb).

Sample No.	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I	Lab J	Lab K
1	96	100	97	105	99	104	99	114	91	100	93
2	96	**95**	98	101	99	99	101	**122**	98	100	100
3	97	100	98	100	100	97	98	116	96	102	97
4	95	101	98	102	100	95	101	115	102	100	96
Mean	96.0	99.0	97.8	102.0	99.5	99.0	99.8	**116.8**	96.8	100.5	96.5
Median	96.0	100.0	98.0	101.5	99.5	98.2	100.0	115.5	97.0	100.0	96.5
Std.Dev.	0.8	2.7	0.5	2.2	0.6	3.8	1.5	3.6	4.6	1.0	2.9
Rel.Std.Dev.	0.85%	2.74%	0.51%	2.12%	0.58%	3.82%	1.50%	3.08%	4.73%	1.00%	2.99%
PDM[3]	-2.16%	0.90%	-0.38%	3.95%	1.41%	0.93%	1.66%	19.0%	-1.40%	2.43%	-1.65%

Table 2. continued

Sample No.	Lab L	Lab M	Lab N	Lab O	Lab P
1	91	98	101	91	87
2	91	99	101	88	89
3	91	94	102	84	86
4	**96**	95	**93**	90	90
Mean	92.3	96.5	99.3	**88.3**	**88.0**
Median	91.0	96.5	101.0	89.0	88.0
Std.Dev.	2.5	2.4	4.2	3.1	1.8
Rel.Std.Dev.	2.71%	2.47%	4.22%	3.51%	2.07%
PDM[3]	-5.98%	-1.65%	1.15%	-10.1%	-10.3%

Table 3. Analytical results for gold (ppb) in OREAS 5Pb by instrumental neutron activation analysis on 4 g analytical subsample weights (abbreviations as for Table 2).

Sample No.	Lab Q
1	102.4
2	92.9
3	91.1
4	94.1
5	98.7
6	95.4
7	99.8
8	99.7
9	95.1
10	97.9
11	96.5
12	94.4
13	97.3
14	97.1
15	98.1
16	97.8
17	93.3
18	94.9
19	97.4
20	96.0
21	98.2
22	98.7
23	94.7
24	99.3
25	97.5
Mean	96.7
Median	97.3
Std.Dev.	2.5
Rel.Std.Dev.	2.63%
PDM[3]	-1.42%

Table 4. Recommended value and 95% confidence interval

Constituent	**Recommended value**	**95% Confidence interval**	
		Low	**High**
Gold, Au (ppb)	98	96	100

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 4 g 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 ±2 ppb 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 homogeneity of the material under test. The meaning of this tolerance interval may be illustrated for gold (refer Table 5), where 99% of the time at least 95% of 50g-sized subsamples will have concentrations lying between 96 and 100 ppb. 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 5. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppb)	98	96	100

Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a routine laboratory being monitored by this standard in a QA/QC program. For many years it has been customary to employ the 95% Confidence Interval of the Recommended Value of the CRMs as the “window of acceptability” for routine analysis. This convention appears to have become established more out of pragmatism than sound statistical practice as no alternative tools were available to the geochemist. The 95% Confidence Interval is provided by producers of CRMs to give an estimate of the reliability of the Recommended Value. Its magnitude is inversely proportional to the number of participating laboratories and to interlaboratory consensus. It is based on the variance of the mean of means of all laboratory data sets after removal of outliers. Because means of data sets are employed, variability contributed by analytical precision and CRM inhomogeneity is not taken into account. The 95% Confidence Interval is not therefore an ideal tool for the monitoring of performance.

Table 6. Proposed performance gates for 5Pb

Constituent	Recommended value	Performance Gates					
		1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppb)	98	95	101	92	105	88	108

A preferred performance gate parameter is one that incorporates errors attributable to bias, precision and inhomogeneity. It is simply calculated from the standard deviation of the pooled individual analyses generated from the certification program. All individual and lab dataset (batch) outliers should be removed prior to determination of the standard deviation. These outliers can only be removed after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. Performance gates have been calculated for one, two and three standard deviations of the accepted pool of certification data and are presented in Table 6. As a guide these intervals may be regarded as informational (1σ), warning or rejection for multiple outliers (2σ), or rejection for individual outliers (3σ) in QC monitoring although their precise application should be at the discretion of the QC manager concerned.

PARTICIPATING LABORATORIES

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ALS Chemex Laboratories Pty Ltd, North Vancouver, Ontario, Canada
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Becquerel Laboratories, Lucas Heights, NSW, Australia
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PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 5Pb has been prepared and certified and is supplied by:

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It is available in unit sizes of 60g (laminated foil packets) and 500g (jars).

INTENDED USE

OREAS 5Pb 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 5Pb 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 5Pb refers to the concentration level of gold after removal of hygroscopic moisture by drying in air to constant mass at 105^0 C. 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 cooperation of all participating laboratories is gratefully acknowledged.

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