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CERTIFICATE OF ANALYSIS FOR
GOLD ORE REFERENCE MATERIAL
OREAS 6Pb

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	1.425	1.399	1.451

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99, \rho=0.95$	
		Low	High
Gold, Au (ppm)	1.425	1.420	1.430

Prepared by:
Ore Research & Exploration Pty Ltd
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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 material used to produce gold ore standard OREAS 6Pb 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 6Pb is given in Table 1. The constituents SiO₂ 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 at Becquerel Laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing greywacke material comprising OREAS 6Pb 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 rolls crusher*
- e) *milling to 100% minus 40 micron*
- f) *homogenisation in a ribbon blender*
- g) *bagging into 20kg sublots*

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 6Pb

At the time of writing eighteen out of twenty-one laboratories had reported their results and these respondents are listed in the section headed Participating Laboratories. It should be noted that eventual inclusion of these outstanding results may lead to minor revision the recommended value.

To maintain anonymity laboratories have been randomly designated the letter codes A through R. With the exception of Laboratory R, each laboratory received four 110g samples with instructions to carry out one 30 to 50g fire assay determination for gold on each sample. One laboratory (M) employed a partial digest method (aqua regia digest / AAS) and their results were therefore not included in the determination of the recommended value. Apart from Lab B (ICPOES finish) and Lab F (gravimetric finish) the remaining laboratories employed a flame AAS finish.

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 enabled 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 R for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 0.5g.

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 one data set is good, lying within 6.5% relative of the recommended value of 1.43 ppm Au. The exception to this is Lab L which has been relegated to outlying status with a negative bias of 16.6%.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 6Pb

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 K and N to Q according to the formulae

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

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

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 ;
- \bar{x}_i is the mean for laboratory i ;
- \dot{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 $(p-1)$

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

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

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:

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

where

- T_{ij} is the test criterion for the j th result of laboratory i ;
- s_i is the standard deviation of laboratory i .

Table 1. Indicative major and trace element composition of gold ore reference material OREAS 6Pb; SiO₂ to Total as weight percent; rest in parts per million; SiO₂ to Zr by fusion XRF except C and S by Leco furnace; As to Yb by INAA.

Constituent	Concentration (XRF)	Constituent	Concentration (INAA)
SiO ₂	72.9	As	1263
TiO ₂	0.57	Ce	87
Al ₂ O ₃	13.5	Co	6
Fe ₂ O ₃	5.41	Cr	277
MnO	0.01	Cs	10
MgO	0.64	Eu	1.5
CaO	0.01	Hf	4.9
Na ₂ O	0.17	La	44
K ₂ O	3.57	Lu	0.36
P ₂ O ₅	0.80	Rb	192
LOI	2.64	Sb	122
Total	100.2	Sc	14
Ba	680	Sm	7.2
C (Leco)	400	Th	16
Ni	27	W	17
S (Leco)	400	Yb	3
V	90		
Zr	170		

Table 2. Analytical results for gold (ppm) in OREAS 6Pb by lead fire assay with flame AAS, ICPOES or gravimetric finish (STDEV - one sigma standard deviation; RSD – one sigma relative standard deviation; outliers in bold).

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	1.47	1.40	1.48	1.34	1.46	1.39	1.40	1.46	1.43	1.46	1.40
2	1.49	1.48	1.45	1.405	1.47	1.40	1.38	1.47	1.45	1.41	1.40
3	1.50	1.43	1.45	1.405	1.46	1.36	1.38	1.49	1.46	1.45	1.41
4	1.48	1.48	1.44	1.405	1.42	1.32	1.38	1.48	1.41	1.44	1.45
Mean:	1.49	1.45	1.46	1.39	1.45	1.37	1.39	1.48	1.43	1.44	1.41
Median:	1.49	1.46	1.45	1.41	1.46	1.38	1.38	1.48	1.44	1.45	1.40
STDEV:	0.01	0.04	0.02	0.03	0.02	0.04	0.01	0.01	0.02	0.02	0.03
RSD:	0.87%	2.73%	1.19%	2.34%	1.53%	2.63%	0.72%	0.88%	1.41%	1.38%	1.84%

Table 2. continued

Sample No.	Lab L	Lab M	Lab N	Lab O	Lab P	Lab Q
1	1.09	1.39	1.53	1.31	1.38	1.37
2	1.20	1.37	1.49	1.36	1.32	1.40
3	1.27	1.38	1.53	1.35	1.38	1.37
4	1.18	1.36	1.51	1.35	1.41	1.36
Mean:	1.19	1.37	1.51	1.34	1.37	1.38
Median:	1.19	1.37	1.52	1.35	1.38	1.37
STDEV:	0.07	0.01	0.02	0.02	0.04	0.02
RSD:	6.26%	1.07%	1.41%	1.65%	2.97%	1.26%

Table 3. Analytical results for gold (ppm) in OREAS 6Pb by instrumental neutron activation analysis on 0.5g analytical subsample weights (abbreviations as for Table 2).

Sample No.	Lab R
1	1.49
2	1.46
3	1.47
4	1.50
5	1.48
6	1.48
7	1.49
8	1.45
9	1.52
10	1.50
11	1.49
12	1.49
13	1.51
14	1.49
15	1.50
16	1.48
17	1.50
18	1.52
19	1.47
20	1.47
21	1.50
22	1.51
23	1.50
24	1.48
25	1.51
Mean:	1.49
Median:	1.49
STDEV:	0.02
RSD:	1.22%

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 4. Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	1.425	1.399	1.451

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 0.5 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

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

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

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

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 ±0.005 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 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 1.420 and 1.430ppm. 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 5. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	1.425	1.420	1.430

PARTICIPATING LABORATORIES

Acme Analytical Laboratories Ltd, Vancouver, Ontario, Canada
Activation Laboratories, Ancaster, Ontario, Canada
ALS Chemex Laboratories Pty Ltd, North Vancouver, Ontario, Canada
ALS Chemex Laboratories Pty Ltd, Sparks, Nevada, USA
ALS Chemex Laboratories Pty Ltd, Val d'Or, Quebec, Canada
ALS Chemex Laboratories Pty Ltd, Santiago, Chile
Amdel Laboratories Ltd, Thebarton, SA, Australia
Becquerel Laboratories, Lucas Heights, NSW, Australia
CIMM, Vitacura, Santiago, Chile
Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia
Indo Assay Laboratories, Kalimantan Timur, Indonesia
Intertek Utama Services, Jakarta Selatan, Indonesia
Lakefield-Geosol Ltd, Belo Horizonte, Brazil
OMAC Laboratories, Loughrea, Ireland
Set Point Laboratories, Wynberg, South Africa
SGS Analabs, Welshpool, WA, Australia
Standard and Reference Laboratories, Malaga, WA, Australia
Ultra Trace, Bentley, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 6Pb has been prepared and certified and is supplied by:

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It is available in unit sizes of 60g laminated foil packets or 0.5kg jars.

INTENDED USE

OREAS 6Pb 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 6Pb 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 6Pb 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 1.29% 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

ACKNOWLEDGMENTS

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REFERENCES

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