ORE RESEARCH & EXPLORATION PTY LTD
6-8 Gatwick Road, Bayswater North, Vic 3153 AUSTRALIA
Telephone: +613-9729 0333 Facsimile: +613-9729 4777

CERTIFICATE OF ANALYSIS FOR

GOLD ORE REFERENCE MATERIAL

OREAS 15Pb

SUMMARY STATISTICS

Constituent	Recommended Value	95% Confidence Interval		Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppm)	1.06	1.04	1.07	1.04	1.07

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 resource definition.

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

Reference material OREAS 15Pb was prepared from a blend of barren alkali olivine basalt from Epping, Victoria, Australia and gold-bearing Magdala ore from the Stawell Gold Mine, west-central Victoria, Australia. The Magdala lode is intimately associated with an intensely deformed package of volcanogenic sedimentary rocks. Mineralisation in the ore consists of a quartz-sericite-carbonate schist assemblage containing the sulphides pyrite and arsenopyrite. The major constituents of the alkali olivine basalt are feldspar, augite, olivine and titanomagnetite.

The approximate major and trace element composition of this sulphide-bearing gold ore standard OREAS 15Pb is given in Table 1. The constituents SiO_2 to Total are the means of duplicate XRF analyses determined using a borate fusion method, S is the mean of duplicate Leco analyses, while the remaining constituents, As to Yb, are means of twenty replicate analyses determined via INAA at Becquerel Laboratories.

Gold homogeneity has been evaluated and confirmed by INAA on twenty 0.5 gram sample portions and by a nested ANOVA program using conventional fire assay. The tolerance interval is determined from the INAA data while the recommended value and confidence interval are based on a round robin program incorporating a total of 84 analyses at 17 laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing basaltic material comprising OREAS 15Pb was prepared in the following manner:

a) *jaw crushing to minus 7mm*
b) *drying to constant mass at 105^{0}C*
c) *milling of the barren basalt to 98% minus 75 micron*
d) *milling of the gold ore to 100% minus 20 micron*
e) *blending in appropriate proportions to achieve the desired grade*
f) *bagging into 25kg sublots*

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 15Pb; Wt. % - weight percent, ppm - parts per million

Constituent	Wt. %	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO_2	52.5	Ag	<1	Gd	5.3	Sb	1.7
TiO_2	1.68	As	1640	Hf	3.5	Sc	20
Al_2O_3	13.6	Ba	268	Ho	0.77	Sm	4.7
Fe_2O_3	12.0	Be	1.1	In	0.06	Sn	2
MnO	0.18	Bi	0.1	La	17	Sr	344
MgO	6.75	Cd	<1	Li	6.8	Ta	0.4
CaO	8.12	Ce	35	Lu	0.23	Tb	0.7
Na_2O	2.84	Co	42	Mo	2	Te	0.2
K_2O	0.74	Cs	1.2	Nb	9.7	Th	2.8
P_2O_5	0.33	Cu	66	Nd	20	U	0.8
Total	99.4	Dy	4.1	Ni	157	W	0.8
C	0.14	Er	2.0	Pb	8	Y	19
S	0.51	Eu	1.5	Pr	4.5	Yb	1.6
		Ga	19	Rb	19	Zn	130
						Zr	149

ANALYSIS OF OREAS 15Pb

Seventeen 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 Q. With the exception of Laboratory Q, each laboratory received two scoop-split 120 gram subsamples from each of two 1kg test units taken at regular intervals during the bagging stage. They were instructed to carry out one 40-50 gram fire assay gold determination on each subsample. 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 10 gram scoop split was taken from each of the twenty test units and submitted to Laboratory Q for gold assay via instrumental neutron activation analysis on a reduced analytical subsample weight of 0.5 gram.

Individual assay results for the fire assay and INAA methods are presented in Tables 2 and 3 together with the mean, median, standard deviations (absolute and relative) and percent deviation of the lab mean from the corrected mean of means for each data set (PDM^3). Interlaboratory agreement of the means is good with all labs lying within 5.6% relative of the corrected mean of means of 1.06 ppm Au.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 15Pb

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

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

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

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;*

$\overline{x}_i$ *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):

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

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

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 is assumed to be symmetrical about the mean in the calculation of the confidence limits.

The test for rejection of individual outliers from each laboratory data set 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

$$S = 1.483 \underset{j=1.....n}{\text{median}} / x_j - \underset{i=1.....n}{\text{median}} (x_i) /$$

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

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.

Table 2. Analytical results for gold (ppm) in OREAS 15Pb by 50g fire assay/flame AAS/OES/ES (Std. Dev. - one sigma standard deviation; RSD – one sigma relative standard deviation; PDM[3] – percent deviation of lab mean from corrected mean of means; outliers in bold).

Replicate	Lab A FA*AAS	Lab B ICP*OES	Lab C FA*AAS	Lab D FA*AAS	Lab E FA*AAS	Lab F FA*AAS	Lab G FA*AAS	Lab H ICP*ES	Lab I FA*AAS	Lab J FA*AAS
1	1.07	1.02	**1.13**	1.05	1.06	1.08	1.06	1.11	1.05	1.07
2	1.09	1.05	1.18	1.05	1.06	1.07	1.07	1.10	**1.01**	1.07
3	1.07	1.06	1.20	1.03	1.05	1.08	1.05	**1.15**	1.05	1.08
4	1.07	1.00	1.18	**1.09**	**1.01**	1.09	1.10	1.10	1.04	1.09
Mean	1.08	1.03	**1.17**	1.06	1.05	1.08	1.07	1.12	1.03	1.07
Median	1.07	1.04	1.18	1.05	1.06	1.08	1.07	1.11	1.04	1.07
Std.Dev.	0.01	0.03	0.03	0.03	0.02	0.01	0.02	0.02	0.02	0.01
Rel.Std.Dev.	0.93%	2.67%	2.55%	2.39%	2.28%	0.57%	2.02%	2.13%	1.60%	0.77%
PDM[3]	1.8%	-2.21%	11.05%	-0.08%	-1.02%	2.24%	1.34%	5.60%	-2.09%	1.77%

Table 2. Continued.

Replicate	Lab K FA*AAS	Lab L FA*AAS	Lab M FA*AAS	Lab N FA*GRAV	Lab O FA*AAS	Lab P FA*AAS
1	0.98	1.04	1.03	1.04	1.08	1.05
2	1.05	1.03	1.00	**1.06**	1.11	1.06
3	1.10	1.07	1.01	1.03	1	1.07
4	1.06	1.05	1.02	1.04	1.06	1.09
Mean	1.05	1.05	1.02	1.04	1.06	1.07
Median	1.06	1.04	1.02	1.04	1.07	1.07
Std.Dev.	0.05	0.02	0.01	0.01	0.05	0.02
Rel.Std.Dev.	4.77%	1.87%	1.27%	1.26%	4.37%	1.42%
PDM[3]	-0.79%	-1.02%	-3.87%	-1.38%	0.63%	0.9%

The same principles were applied in testing for outlying laboratory means. In certain instances statistician's prerogative has been employed in discriminating outliers. Individual and mean outliers are shown in bold type in Tables 2 and 3, and have been omitted in the determination of recommended values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It is a measure of the reliability of the recommended value, i.e. the narrower the confidence interval the greater the certainty in the recommended value.

Table 3. Analytical results for gold (ppm) in OREAS 15Pb by instrumental neutron activation analysis on 0.5 gram analytical subsample weights (abbreviations as for Table 2).

Unit No.	Lab Q INAA
1	1.19
2	1.13
3	1.21
4	1.29
5	1.26
6	1.25
7	1.28
8	1.27
9	1.15
10	1.25
11	1.29
12	1.26
13	1.21
14	1.23
15	1.30
16	1.26
17	1.36
18	1.19
19	1.17
20	1.19
Mean	**1.24**
Median	1.25
Std.Dev.	0.06
Rel.Std.Dev.	4.59%
PDM[3]	17.2%

Table 4. Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	1.06	1.04	1.07

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 gram 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.

Table 5. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	1.06	1.04	1.07

From the INAA data set an estimated tolerance interval of ±0.02 ppm at an analytical subsample weight of 50 gram 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 1.04 and 1.07 ppm. 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).

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. They incorporate errors attributable to bias, precision and inhomogeneity and are simply calculated from the standard deviation of the pooled individual analyses (fire assay data only) generated from the certification program. All individual and lab dataset (batch) outliers are 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.

Table 6. Proposed performance gates for 15Pb

Constituent	Recommended value	Performance Gates					
		1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppm)	1.06	1.03	1.09	1.00	1.12	0.97	1.14

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

Acme Analytical Laboratories Ltd, Vancouver, BC, Canada
Activation Laboratories, Ancaster, Ontario, Canada
Amdel Laboratories Ltd, Thebarton, SA, Australia
ALS Chemex, Garbutt, QLD, Australia
ALS Chemex, La Serena, Chile, South America
ALS Chemex, Reno, Nevada, USA
ALS Chemex, Val-d'or, Quebec, Canada
ALS Chemex, Vancouver, BC, Canada
Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia
Intertek Testing Services, Jakarta, Indonesia
Lakefield Geosol Ltda, Brazil, South America
McPhar Laboratories, Legaspi Village, Makati City, Philippines
OMAC Laboratories Ltd, Loughrea, County Galway, Ireland
PT Indo Assay Laboratories, Balikpapan, Kalimantan Timur, Indonesia
SGS Analabs, Welshpool, WA, Australia
Standard & Reference Laboratories, Malaga, WA, Australia
Ultra Trace, Canning Vale, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 15Pb has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd
6-8 Gatwick Road
Bayswater North, VIC 3153
AUSTRALIA

Telephone (03) 9729 0333 International +613-9729 0333
Facsimile (03) 9729 4777 International +613-9729 4777

It is available in unit sizes of 60g foil packets and 1kg jars.

INTENDED USE

OREAS 15Pb 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 15Pb has been prepared from a blend of gold-ore and barren basalt. Being characterised by a low sulphide content the sealed CRM is considered to have long-term stability under normal storage conditions.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The recommended value for OREAS 15Pb 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

REFERENCES

Ingamells, C. O. and Switzer, P. (1973), Talanta 20, 547-568.

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.