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CERTIFICATE OF ANALYSIS FOR

GOLD ORE REFERENCE MATERIAL

OREAS 18Pa

Summary Statistics for OREAS 18Pa Gold Ore Standard (basalt matrix)

Constituent	Recommended Value	95% Confidence Interval		Tolerance limits $1-\alpha=0.99$, $\rho=0.95$	
		Low	High	Low	High
Gold, Au (ppm)	3.36	3.31	3.41	3.34	3.38

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

Reference material OREAS 18Pa 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 18Pa is given in Table 1. The constituents SiO_2 to Total and Ba, Ni, V and Zr are the means of duplicate XRF analyses determined using a borate fusion method, C and S are the means 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-four 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 76 analyses at 17 laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The gold-bearing basaltic material comprising OREAS 18Pa 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

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

Table 1. Indicative major and trace element composition of gold ore reference material OREAS 18Pa; SiO_2 to Total as weight percent; rest in parts per million; SiO_2 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	54.2	As	5235
TiO_2	1.37	Ce	38
Al_2O_3	11.8	Co	41
Fe_2O_3	13.1	Cr	216
MnO	0.23	Cs	<2
MgO	5.83	Eu	1.3
CaO	6.95	Hf	<31
Na_2O	2.34	La	16.7
K_2O	0.76	Lu	~0.2
P_2O_5	0.34	Rb	<40
LOI	2.06	Sb	4
Total	98.97	Sc	17
Ba	269	Sm	4.2
C (Leco)	0.31	Th	<4
Ni	159	W	<4
S (Leco)	1.73	Yb	1.5
V	140		
Zr	104		

ANALYSIS OF OREAS 18Pa

At the time of writing seventeen laboratories had reported their results and these respondents are listed in the section headed Participating Laboratories.

To maintain anonymity laboratories have been randomly designated the letter codes A through Q. Labs A to J received four 110g samples while Labs K to P received two 110g samples with instructions to Lab A to carry out one 30g INAA determination and Labs B to J to complete one 40-50g fire assay determination for gold on each sample. Apart from Labs A and Q (INAA), Lab C (ICPOES finish) and Lab G (gravimetric finish) most laboratories employed a flame AAS finish.

For laboratories A to J 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-four random test units and submitted to Laboratory Q 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 is good for all but one data set, Lab A, where bias is unacceptably high (+13.5%).

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 18Pa

Recommended Value and Confidence Limits

The recommended value was determined from the mean of means of accepted replicate values of accepted laboratory data sets B to Q according to the formulae

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

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

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)

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

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

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.

Table 2. Analytical results for gold in OREAS 18Pa (INAA - instrumental neutron activation analysis; FA*AAS - fire assay / atomic absorption spectrometry; FA*ICP - fire assay/inductively coupled plasma optical emission spectrometry; FA*GRAV - fire assay/gravimetric; STDEV and RSD are one sigma values; PDM^3 - percent deviation of lab mean from corrected mean of means; outliers in bold; values in ppm).

Sample No.	Lab A INAA (30g)	Lab B FA*AAS (50g)	Lab C FA*ICP (40g)	Lab D FA*AAS (50g)	Lab E FA*AAS (50g)	Lab F FA*AAS (50g)	Lab G FA*GRAV (50g)	Lab H FA*AAS (50g)	Lab I FA*AAS (50g)	Lab J FA*AAS (50g)	Lab K FA*AAS (50g)
1	3.84	3.43	3.46	3.38	3.27	3.43	3.46	3.29	3.36	3.56	3.64
2	3.84	3.39	3.41	3.35	3.27	3.32	3.49	3.41	3.46	3.58	3.37
3	3.87	3.34	3.34	3.34	3.49	3.39	3.49	3.34	3.25	3.53	
4	**3.72**	3.35	3.39	3.40	3.45	3.29	3.48	3.38	3.40	3.47	
Mean:	**3.82**	3.38	3.40	3.37	3.37	3.36	3.48	3.36	3.37	3.54	3.50
Median:	3.84	3.37	3.40	3.37	3.36	3.36	3.49	3.36	3.38	3.55	3.50
STDEV:	0.07	0.04	0.05	0.03	0.12	0.06	0.01	0.05	0.09	0.05	0.19
RSD:	1.74%	1.22%	1.46%	0.82%	3.42%	1.91%	0.42%	1.55%	2.63%	1.36%	5.47%

Sample No.	Lab L FA*AAS (50g)	Lab M FA*AAS (50g)	Lab N FA*AAS (50g)	Lab O FA*AAS (50g)	Lab P FA*AAS (30g)
1	3.45	3.21	3.21	3.27	3.23
2	3.45	3.22	3.43	3.36	3.17
Mean:	3.45	3.22	3.32	3.32	3.20
Median:	3.45	3.22	3.32	3.32	3.20
STDEV:	0.00	0.01	0.16	0.06	0.04
RSD:	0.00%	0.22%	4.73%	1.26%	0.00%

Table 3. Analytical results for gold (ppm) in OREAS 18Pa by instrumental neutron activation analysis on 0.5g analytical subsample weights (abbreviations as for Table 2).

Sample No.	Lab Q
1	3.22
2	3.22
3	3.19
4	3.22
5	3.17
6	3.21
7	3.21
8	3.16
9	3.26
10	3.12
11	3.22
12	3.19
13	3.18
14	3.12
15	3.26
16	3.17
17	3.18
18	3.20
19	3.27
20	3.25
21	3.10
22	3.13
23	3.11
24	3.13
Mean:	3.19
Median:	3.19
STDEV:	0.05
RSD:	1.56%

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 Tables 2 and 3 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)	3.36	3.31	3.41

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

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

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 ±0.02 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 3.34 and 3.38 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).

Table 5. Recommended value and tolerance interval.

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99$, $\rho=0.95$	
		Low	High
Gold, Au (ppm)	3.36	3.34	3.38

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 OREAS 18Pa

Constituent	Recommended value	1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppm)	3.36	3.26	3.46	3.16	3.57	3.06	3.67

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, Ontario, Canada
ALS Chemex Laboratories Pty Ltd, North Vancouver, Ontario, Canada
ALS Chemex Laboratories Pty Ltd , Garbutt, Queensland, Australia
ALS Chemex Laboratories Pty Ltd, Sparks, Nevada, USA
Amdel Laboratories, Wangara, Western Australia, Australia
Amdel Laboratories, Thebarton, South Australia, Australia
SGS Analabs, Welshpool, Western Australia, Australia
Becquerel Laboratories, Lucas Heights, New South Wales, Australia
Becquerel Laboratories, Mississauga, Ontario, Canada
Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia
Indo Assay Laboratories, Kalimantan Timur, Indonesia
Intertek Utama Services, Jakarta Selatan, Indonesia
SGS Lakefield Research, Lakefield, Ontario, Canada
Lakefield Geosol Ltda, Belo Horizonte - MG, Brazil
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OMAC Laboratories, Loughrea, County Galway, Ireland
Standard & Reference Laboratories, Malaga, Western Australia, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 18Pa has been prepared and certified and is supplied by:

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It is available in unit sizes of 60g in laminated foil packets or in 1 or 2.5kg jars.

INTENDED USE

OREAS 18Pa 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 18Pa 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 18Pa refers to the concentration level of gold in its packaged form at hygroscopic equilibrium. Drying in air to constant mass at 105^0C has established a hygroscopic moisture content of 1.25%. If the reference material is dried by the user prior to analysis, the recommended value should be corrected to the moisture-free 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.

REFERENCES

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