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**CERTIFICATE OF ANALYSIS FOR**  
**GOLD-SILVER CERTIFIED**  
**REFERENCE MATERIAL OREAS 60b**

**SUMMARY STATISTICS**

<b>Constituent</b>	<b>Recommended value</b>	<b>95% Confidence interval</b>		<b>Tolerance interval 1-<math>\alpha</math>=0.99, <math>\rho</math>=0.95</b>	
		<b>Low</b>	<b>High</b>	<b>Low</b>	<b>High</b>
Gold, Au (ppm)	2.57	2.52	2.61	2.55	2.58
Silver, Ag (ppm)	4.96	4.78	5.13	4.75	5.16

Note: values may appear asymmetric due to rounding

*Prepared by:*  
*Ore Research & Exploration Pty Ltd*  
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REPORT 06-615A\_RMh

## 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.

## SOURCE MATERIALS

Reference material OREAS 60b was prepared from a blend of barren meta-andesite from Cracow, Queensland Australia and gold-bearing meta-andesite from the Gosowong gold mine in Maluku, Indonesia. Both Cracow and Gosowong are epithermal deposits hosted by andesitic volcanics. The barren Cracow material is unmineralised altered andesite of the Camboon volcanics with low sulphidation epithermal quartz veining (epithermal quartz, carbonate, adularia and minor sulphides). The Gosowong ore consists of andesitic volcanic-hosted low sulphidation epithermal quartz veining containing an assemblage of epithermal quartz, carbonate, adularia, kaolin after adularia, chlorite and minor sulphides.

The indicative major and trace element composition of gold ore standard OREAS 60b is given in Table 1. These constituents are the means of duplicate analyses by borate fusion XRF, four acid ICP-OES/MS, Leco and thermo-gravimetry and are uncertified values.

## COMMUNITION AND HOMOGENISATION PROCEDURES

The Cracow and Gosowong material comprising OREAS 60b was prepared in the following manner:

- a) *jaw crushing to minus 7mm*
- b) *drying to constant mass at 105<sup>o</sup>C*
- c) *milling of the barren Cracow material to 98% minus 75 micron*
- d) *milling of the Gosowong 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 1kg test units were taken at regular intervals, sealed in laminated plastic bags and set aside for this analytical program.

## ANALYSIS OF OREAS 60b

Sixteen laboratories were included in the certification of OREAS 60b and are listed in the section headed Participating Laboratories. To maintain anonymity they have been randomly designated the letter codes A through P (Tables 2 – 4). With the exception of Laboratory Q, each received six 100g samples with instructions to carry out one 20 to 50g fire assay determination for gold and one aqua regia digest determination for silver using their preferred finish. Apart from Labs A and P (ICPOES) and Lab K (gravimetric), a flame AAS finish was employed for gold. Silver was determined by an aqua regia digestion with an AAS, ICPOES or ICPMS finish.

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 60b; wt.% - weight percent; ppm - parts per million.

Constituent	wt.%	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO <sub>2</sub>	58.8	As	12	Hf	4.0	Sc	17
TiO <sub>2</sub>	0.682	Ba	552	Ho	0.63	Sm	4.1
Al <sub>2</sub> O <sub>3</sub>	15.5	Be	0.95	In	0.04	Sn	1.5
Fe <sub>2</sub> O <sub>3</sub>	6.14	Bi	<0.1	La	11.1	Sr	381
MnO	0.139	Cd	1	Li	32	Ta	<0.5
MgO	2.40	Ce	42	Lu	0.26	Tb	0.53
CaO	4.28	Co	15	Mo	9.8	Te	2.7
K <sub>2</sub> O	2.86	Cs	4	Nb	<5	Th	5.6
P <sub>2</sub> O <sub>5</sub>	0.25	Cu	114	Nd	19.8	U	1.4
Na <sub>2</sub> O	2.24	Dy	3.1	Ni	45	W	<5
LOI	4.46	Er	1.7	Pb	20	Y	18
Total	99.2	Eu	1.2	Pr	5.0	Yb	1.8
C	0.67	Ga	15.4	Rb	79	Zn	100
S	0.79	Gd	3.4	Sb	<1	Zr	135

For each laboratory two 100g subsamples were scoop-split from each of three 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 for gold, a 10g scoop split was taken from each of the twenty test units and submitted to Lab Q for determination via instrumental neutron activation analysis on a reduced analytical subsample weight of 0.5 gram.

Individual gold results for the fire assay and INAA methods are presented in Tables 2 and 3 together with the mean, median, standard deviation (absolute and relative) and bias (PDM<sup>3</sup> – percent deviation of lab mean from corrected mean of means) for each data set. Interlaboratory agreement of the means of all but two data sets is good, lying within 5% of the recommended value of 2.57 ppm Au. The exception to this is Lab I which has a positive bias of 6.88% and Lab O, which has a negative bias of 5.59%.

Individual silver results together with summary statistics for each data set are presented in Table 4. Interlaboratory agreement of the means of all but two data sets is good, lying within 11% of the recommended value of 4.96 ppm Ag.

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 60b

### Recommended Value and Confidence Limits

Each batch of results was treated as a separate data set in testing for outliers and in determining the consensus mean. The recommended value was determined from the mean of means of accepted replicate values of accepted laboratory data sets A to P according to the formulae:

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

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

Table 2. Analytical results for gold in OREAS 60b (FA\*AAS – fire assay / atomic absorption spectrometry; GRAV – gravimetric finish; OES – inductively coupled plasma optical emission spectrometry; MS - inductively coupled plasma mass spectrometry; Std.Dev. – one sigma standard deviation; Rel.Std.Dev. – one sigma relative standard deviation; PDM<sup>3</sup> – percent deviation of lab mean from corrected mean of means; values in parts per million).

Replicate No.	Lab A FA*OES (30g)	Lab B FA*AAS (30g)	Lab C FA*AAS (50g)	Lab D FA*AAS (50g)	Lab E FA*AAS (50g)	Lab F FA*AAS (50g)	Lab G FA*AAS (50g)	Lab H FA*AAS (20g)	Lab I FA*AAS (40g)
1	2.59	2.48	2.56	<b>1.90</b>	2.66	2.44	2.80	2.58	2.82
2	2.62	2.52	2.57	2.57	2.68	2.44	2.70	2.66	2.82
3	2.59	2.48	2.69	2.55	2.67	2.43	2.50	2.64	2.68
4	<b>2.76</b>	2.45	2.67	2.58	2.66	2.47	2.77	2.62	2.73
5	2.52	2.47	2.63	2.56	2.67	2.48	2.74	2.62	2.73
6	2.58	2.51	2.67	2.50	2.69	2.39	2.52	2.54	2.67
Mean	2.61	2.49	2.63	2.44	2.67	2.44	2.67	2.61	2.74
Median	2.59	2.48	2.65	2.56	2.67	2.44	2.72	2.62	2.73
Std.Dev.	0.08	0.03	0.06	0.27	0.01	0.03	0.13	0.04	0.06
Rel.Std.Dev.	3.08%	1.04%	2.10%	11.0%	0.44%	1.31%	4.85%	1.63%	2.32%
PDM <sup>3</sup>	1.75%	-3.12%	2.59%	-4.78%	4.15%	-4.81%	4.15%	1.68%	6.88%

Table 2. continued

Replicate No.	Lab J FA*AAS (50g)	Lab K FA*GRAV (50g)	Lab L FA*AAS (30g)	Lab M FA*AAS (50g)	Lab N FA*AAS (50g)	Lab O FA*AAS (50g)	Lab P FA*OES (40g)
1	2.49	2.58	2.59	2.32	2.48	2.42	2.64
2	2.54	2.58	2.58	2.40	2.56	2.40	2.68
3	2.48	2.56	2.59	2.37	2.51	2.40	2.71
4	2.48	2.54	2.60	2.56	2.75	2.42	2.67
5	<b>2.70</b>	2.50	2.58	2.53	2.56	2.40	2.60
6	2.60	2.54	<b>2.54</b>	2.55	2.62	<b>2.49</b>	2.67
Mean	2.55	2.55	2.58	2.46	2.58	2.42	2.66
Median	2.52	2.55	2.58	2.47	2.56	2.41	2.67
Std.Dev.	0.09	0.03	0.02	0.10	0.10	0.03	0.04
Rel.Std.Dev	3.44%	1.19%	0.83%	4.24%	3.73%	1.44%	1.41%
PDM <sup>3</sup>	-0.65%	-0.59%	0.52%	-4.29%	0.58%	-5.59%	3.73%

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  $i$ ;

$\bar{x}$  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)

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

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

where  $t_{1-x/2}(p-1)$  is the 1-x/2 fractile of the *t*-distribution with (*p*-1) degrees of freedom.

The distributions 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 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:

$$S = 1.483 \text{ median } / x_j - \text{median } (x_i) /$$

$j=1, \dots, n$                        $i=1, \dots, n$

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

Table 3. Analytical results for gold (ppm) in OREAS 60b by instrumental neutron activation analysis on 0.5 gram analytical subsample weights (abbreviations as in Table 2).

Replicate No.	Lab Q INAA (0.5g)
1	2.45
2	2.47
3	2.48
4	2.52
5	<b>2.32</b>
6	2.41
7	2.40
8	2.46
9	2.43
10	2.47
11	2.55
12	2.49
13	2.50
14	2.49
15	2.47
16	2.49
17	2.38
18	2.45
19	2.41
20	2.51
Mean	2.46
Median	2.47
Std.Dev.	0.05
Rel.Std.Dev	2.18%
PDM <sup>3</sup>	-4.20%

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.*

In certain instances statistician's prerogative has been employed in discriminating outliers. Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in bold and have been omitted in the determination of recommended values.

Table 4. Analytical results for silver in OREAS 60b (AR\*AAS - aqua regia digest / atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; MS - inductively coupled plasma mass spectrometry; NR - not reported; other abbreviations as in Table 2; values in parts per million).

Replicate No.	Lab A AR*OES	Lab B AR*OES	Lab C AR*AAS	Lab D -	Lab E AR*AAS	Lab F AR*AAS	Lab G AR*OES	Lab H AR*MS	Lab I AR*MS
1	6.00	4.50	4.90	NR	4.90	5.10	5.30	3.89	5.11
2	6.00	4.60	5.30	NR	5.00	5.30	5.00	3.74	5.25
3	6.00	4.60	5.00	NR	4.90	5.30	5.00	3.91	5.02
4	6.00	4.60	5.40	NR	5.00	5.30	5.10	3.97	5.18
5	5.00	4.90	5.00	NR	4.90	4.90	5.20	4.12	4.90
6	6.00	4.60	5.20	NR	5.00	4.90	5.30	4.32	4.76
Mean	<b>5.83</b>	4.63	5.13		4.95	5.13	5.15	<b>3.99</b>	5.04
Median	6.00	4.60	5.10		4.95	5.20	5.15	3.94	5.07
Std.Dev.	0.41	0.14	0.20		0.05	0.20	0.14	0.20	0.18
Rel.Std.Dev.	7.00%	2.95%	3.83%		1.11%	3.83%	2.68%	5.07%	3.63%
PDM <sup>3</sup>	17.7%	-6.50%	3.59%		-0.11%	3.59%	3.93%	-19.5%	1.64%

Table 4. continued

Replicate No.	Lab J AR*AAS	Lab K AR*OES	Lab L AR*AAS	Lab M AR*AAS	Lab N AR*AAS	Lab O 4A*MS	Lab P AR*MS
1	5.10	4.40	4.80	5.40	4.40	4.90	5.10
2	5.20	<b>4.70</b>	4.80	5.30	4.40	4.80	5.05
3	5.00	4.40	5.00	5.30	4.50	5.10	5.15
4	5.30	4.50	4.90	5.20	4.50	5.20	5.20
5	5.10	4.50	4.90	5.40	4.30	4.70	5.50
6	5.50	4.40	4.80	5.20	4.40	4.90	5.35
Mean	5.20	4.48	4.87	5.30	4.42	4.93	5.23
Median	5.15	4.45	4.85	5.30	4.40	4.90	5.18
Std.Dev.	0.18	0.12	0.08	0.09	0.08	0.19	0.17
Rel.Std.Dev	3.44%	2.61%	1.68%	1.69%	1.70%	3.77%	3.25%
PDM <sup>3</sup>	4.94%	-9.52%	-1.79%	6.96%	-10.9%	-0.44%	5.44%

Table 5. Recommended values and 95% confidence intervals

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	2.57	2.52	2.61
Silver, Ag (ppm)	4.96	4.78	5.13

Note: values may appear asymmetric due to rounding

### Statement of Homogeneity

The standard deviation of each laboratory data set includes error due to both 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 the tolerance interval for silver that 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 of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

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

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

where

- $n$  the number of results
- $1 - \alpha$  is the confidence level;
- $p$  is the proportion of results expected within tolerance limits;
- $k'_2$  is the factor for two-sided tolerance limits ( $m, \alpha$  unknown);
- $s_g''$  is the corrected grand standard deviation.

The meaning of these tolerance limits may be illustrated for silver, where 99% of the time at least 95% of subsamples will have concentrations lying between 4.75 and 5.16 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 (ISO Guide 35).

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

$1 - (\frac{s_i}{2s_g'})$  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

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

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. Outliers (shown in bold in Table 4) were removed prior to the calculation of tolerance intervals and a weighting factor of zero was applied to those data sets where  $s_i / 2s_g' > 1$  (i.e. where the weighting factor  $1 - s_i / 2s_g' < 0$ ). It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error.

Table 6. Recommended values and tolerance intervals

Constituent	Recommended value	Tolerance interval 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Gold, Au (ppm)	2.57	2.55	2.58
Silver, Ag (ppm)	4.96	4.75	5.16

Note: values may appear asymmetric due to rounding

For gold a more simplified procedure was used in the determination of homogeneity. This entailed using the high precision INAA data alone, obtained on an analytical subsample weight of 0.5 gram (compared to 20-50 gram for the fire assay method). By employing a sufficiently reduced subsample weight in a series of determinations by the same method, analytical error becomes negligible in comparison to subsampling error. The corresponding



standard deviation at a 50 gram subsample weight can then be determined from the observed standard deviation of the 0.5 gram data using the known relationship between the two parameters (Ingamells and Switzer, 1973). The homogeneity of gold was then determined from tables of factors for two-sided tolerance limits for normal distributions. The high level of repeatability indicated by the low coefficients of variation in Tables 2 and 3 (particularly the 0.5 gram INAA data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 60b.

### Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement (analytical bias and precision) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. Two methods have been employed to calculate performance gates.

The first method uses the standard deviation of the pooled individual analyses generated from the certification program. All individual and lab dataset (batch) outliers are removed prior to determination of the standard deviation. The outliers can only be removed if they can be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. Standard deviations and performance gates at 2SD and 3SD have been calculated from the accepted pool of certification data and are presented in Table 7.

As a guide these intervals may be regarded as warning or rejection for multiple outliers (2SD), or rejection for individual outliers (3SD) in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

For the second method a  $\pm 5\%$  error bar on the recommended value is used as the window of acceptability (refer Table 7).

Both methods should be used with caution when concentration levels approach lower limits of detection of the analytical methods employed, as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow.

Table 7. Performance gates for OREAS 60b

Constituent	Recommended value	1SD	Performance gates					
			2SD Interval		3SD Interval		5% Interval	
			Low	High	Low	High	Low	High
Gold, Au (ppm)	2.57	0.11	2.35	2.78	2.25	2.89	2.44	2.69
Silver, Ag (ppm)	4.96	0.31	4.34	5.57	4.03	5.88	4.71	5.20

## PARTICIPATING LABORATORIES

Acme Analytical Laboratories, Vancouver, BC, Canada  
 Activation Laboratories, Ancaster, Ontario, Canada  
 ALS Chemex Laboratories, Garbutt, Qld, Australia  
 ALS Chemex Laboratories, La Serena, Chile  
 ALS Chemex Laboratories, Val d'Or, Quebec, Canada  
 ALS Chemex Laboratories, North Vancouver, BC, Canada  
 ALS Chemex Laboratories, Sparks, Nevada, USA  
 Amdel Laboratories Ltd, Thebarton, SA, Australia  
 Amdel Laboratories Ltd, Perth, WA, Australia

Genalysis Laboratory Services Pty Ltd, Maddington, WA, Australia  
Intertek Testing Services, Jakarta, Indonesia  
McPhar Geoservices (Phil.) Inc., Makati, Philippines  
OMAC Laboratories, Loughrea. Co. Galway, Ireland  
SGS Geochemical Services, Perth, WA, Australia  
SGS Geochemical Services, Townsville, Qld, Australia  
Ultra Trace, Canning Vale, WA, Australia

## **PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL**

The gold ore reference material, OREAS 60b has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd  
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It is available in unit sizes of 60g laminated foil packets.

## **INTENDED USE**

OREAS 60b is a reference material intended for the following:

- i) for the calibration of instruments used in the determination of the concentration of gold and silver;
- ii) for the verification of analytical methods for gold and silver;
- iii) for the preparation of secondary reference materials of similar composition;
- iv) as an arbitration sample for commercial transactions.

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 60b has been prepared from sulphide-poor epithermal Au-Ag ore. The robust foil laminate film used to package it is an effective barrier to oxygen and moisture and 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 values for OREAS 60b refer to the concentration levels of gold and silver in packaged form. Drying in air to constant mass at 105<sup>0</sup>C has established a hygroscopic moisture content of 1.36%. If the reference material is dried by the user prior to analysis, the recommended value stated herein 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**

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