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CERTIFICATE OF ANALYSIS FOR

GOLD-SILVER CERTIFIED

REFERENCE MATERIAL OREAS 61d

SUMMARY STATISTICS

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppm)	4.76	4.69	4.83	4.74	4.78
Silver, Ag (ppm)	9.27	9.00	9.54	8.91	9.63

Note: values may appear asymmetric due to rounding

Prepared by:
Ore Research & Exploration Pty Ltd
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REPORT 06-615B_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 61d 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 61d 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.

COMMINUTION AND HOMOGENISATION PROCEDURES

The Cracow and Gosowong material comprising OREAS 61d 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 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 61d

Sixteen laboratories were included in the certification of OREAS 61d 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 61d; wt.% - weight percent; ppm - parts per million.

Constituent	wt.%	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO_2	59.6	As	10	Hf	2.0	Sc	18
TiO_2	0.952	Ba	346	Ho	0.63	Sm	3.6
Al_2O_3	14.0	Be	0.75	In	0.05	Sn	1.5
Fe_2O_3	7.68	Bi	<0.1	La	8.2	Sr	284
MnO	0.131	Cd	1.5	Li	33	Ta	<0.5
MgO	3.61	Ce	32	Lu	0.22	Tb	0.51
CaO	4.83	Co	25	Mo	14	Te	4.5
K_2O	2.43	Cs	2.8	Nb	10	Th	3.3
P_2O_5	0.237	Cu	103	Nd	16	U	0.85
Na_2O	2.02	Dy	3.1	Ni	53	W	<5
LOI	2.77	Er	1.6	Pb	20	Y	17
Total	99.3	Eu	1.2	Pr	3.9	Yb	1.6
C	0.32	Ga	14	Rb	73	Zn	104
S	0.74	Gd	3.3	Sb	1.5	Zr	105

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.

Table 2. Analytical results for gold in OREAS 61d (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^3 - 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	4.90	4.61	5.09	4.73	**5.03**	4.65	**5.01**	4.71	5.25
2	4.83	**4.68**	4.93	4.96	4.96	4.64	4.55	4.79	5.26
3	4.84	4.61	5.01	4.92	4.97	4.57	4.76	4.71	5.20
4	4.82	4.63	5.08	4.67	4.93	4.62	4.69	4.71	5.14
5	4.87	**4.49**	4.90	4.76	4.95	4.70	4.75	4.77	5.08
6	4.93	4.60	4.94	4.92	4.95	4.68	4.81	4.75	4.93
Mean	4.87	4.60	4.99	4.83	4.97	4.64	4.76	4.74	**5.14**
Median	4.86	4.61	4.98	4.84	4.96	4.65	4.76	4.73	5.17
Std.Dev.	0.04	0.06	0.08	0.12	0.03	0.05	0.15	0.04	0.12
Rel.Std.Dev.	0.89%	1.36%	1.62%	2.51%	0.69%	0.99%	3.17%	0.76%	2.40%
PDM^3	2.22%	-3.28%	4.88%	1.41%	4.32%	-2.44%	0.05%	-0.48%	8.05%

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	4.84	4.77	**4.74**	**5.09**	4.90	4.71	4.92
2	4.92	4.77	4.78	4.56	4.47	4.62	4.82
3	4.66	4.75	4.78	4.67	4.59	4.67	4.89
4	4.77	4.76	4.79	4.46	4.43	4.65	4.95
5	4.86	4.74	**4.84**	4.59	4.65	4.68	4.97
6	4.90	4.75	4.80	4.68	4.71	4.61	4.90
Mean	4.83	4.76	4.79	4.68	4.63	4.66	4.91
Median	4.85	4.76	4.79	4.63	4.62	4.66	4.91
Std.Dev.	0.10	0.01	0.03	0.22	0.17	0.04	0.05
Rel.Std.Dev.	2.00%	0.25%	0.64%	4.68%	3.70%	0.81%	1.07%
PDM[3]	1.38%	-0.06%	0.60%	-1.77%	-2.83%	-2.16%	3.13%

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[3] – percent deviation of lab mean from corrected mean of means) for each data set. Interlaboratory agreement of the means of all but one data set is good, lying within 5% of the recommended value of 4.76 ppm Au. The exception to this is Lab I, which has been relegated to outlying status with a positive bias of 8.05%.

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 10% of the recommended value of 9.27 ppm Ag. The exceptions to this are Lab H (-20.8% bias) and Lab N (-11.9% bias), both of which have been relegated to outlying status.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 61d

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:

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

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

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)

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

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

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

The 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:

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

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

Table 3. Analytical results for gold (ppm) in OREAS 61d 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	4.65
2	4.63
3	4.68
4	4.66
5	**4.76**
6	4.65
7	4.64
8	4.62
9	4.63
10	4.66
11	4.71
12	4.70
13	4.68
14	4.68
15	4.65
16	4.65
17	**4.50**
18	4.70
19	4.63
20	4.67
Mean	4.66
Median	4.66
Std.Dev.	0.05
Rel.Std.Dev	1.07%
PDM[3]	-2.14%

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 61d (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	10.0	8.80	9.60	NR	9.10	9.10	9.50	7.83	8.82
2	10.0	8.50	9.60	NR	9.00	9.10	9.70	7.14	9.16
3	9.00	8.50	9.20	NR	9.10	9.30	9.50	7.70	9.01
4	9.00	8.80	**9.00**	NR	9.20	9.30	9.30	**6.90**	8.91
5	10.0	8.90	9.50	NR	9.10	9.40	9.20	7.26	8.82
6	10.0	8.90	9.50	NR	9.00	9.40	9.40	7.21	9.18
Mean	9.67	8.73	9.40		9.08	9.27	9.43	**7.34**	8.98
Median	10.00	8.80	9.50		9.10	9.30	9.45	7.24	8.96
Std.Dev.	0.52	0.19	0.24		0.08	0.14	0.18	0.35	0.16
Rel.Std.Dev.	5.34%	2.13%	2.61%		0.83%	1.47%	1.86%	4.82%	1.79%
PDM[3]	4.27%	-5.79%	1.40%		-2.02%	-0.04%	1.76%	-20.8%	-3.10%

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	9.90	8.40	9.00	9.40	**8.00**	9.20	9.40
2	9.80	8.50	8.90	9.40	8.20	9.20	9.85
3	10.30	8.20	9.10	**9.90**	8.10	9.30	9.65
4	10.2	8.20	9.10	9.60	8.30	9.30	9.65
5	10.2	8.60	9.00	9.40	8.20	9.20	9.95
6	10.2	8.20	9.00	9.50	8.20	9.20	9.75
Mean	10.10	8.35	9.02	9.53	**8.17**	9.23	9.71
Median	10.20	8.30	9.00	9.45	8.20	9.20	9.70
Std.Dev.	0.20	0.18	0.08	0.20	0.10	0.05	0.19
Rel.Std.Dev	1.98%	2.11%	0.83%	2.06%	1.26%	0.56%	1.97%
PDM[3]	8.95%	-9.93%	-2.74%	2.84%	-11.9%	-0.40%	4.72%

Table 5. Recommended values and 95% confidence intervals

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	4.76	4.69	4.83
Silver, Ag (ppm)	9.27	9.00	9.54

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 tolerance intervals 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 jth raw result reported by laboratory i*;

x'_{ij} *is the jth 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

$$\text{Lower limit is } \ddot{x} - k'_2(n, p, 1-\alpha)s''_g$$
$$\text{Upper limit is } \ddot{x} + k'_2(n, p, 1-\alpha)s''_g$$

where

n the number of results
1-α 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, α 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 8.91 and 9.63 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).

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

$$1-(\frac{s_i}{2s'_g}) \text{ is the weighting factor for laboratory } i;$$

$$s'_g \text{ is the grand standard deviation computed from the transformed (i.e. means-adjusted) results}$$

according to the formula

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

$$\text{where } \bar{x}'_i \text{ is the transformed mean for laboratory } i$$

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 $s_l / 2s_g' > 1$ (i.e. where the weighting factor $1- s_l / 2s_g' < 0$). 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)	4.76	4.74	4.78
Silver, Ag (ppm)	9.27	8.91	9.63

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 61d.

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 ±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 61d

Constituent	Recommended value	1SD	Performance gates					
			2SD Interval		3SD Interval		5% Interval	
			Low	High	Low	High	Low	High
Gold, Au (ppm)	4.76	0.14	4.47	5.04	4.33	5.19	4.52	5.00
Silver, Ag (ppm)	9.27	0.48	8.31	10.2	7.83	10.7	8.81	9.73

Note: values may appear asymmetric due to rounding

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 61d 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 61d 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 61d 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 61d refer to the concentration levels of gold and silver in packaged form. Drying in air to constant mass at 105^0C has established a hygroscopic moisture content of 1.19%. 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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