CERTIFICATE OF ANALYSIS FOR

GOLD ORE REFERENCE MATERIAL

OREAS 17c

SUMMARY STATISTICS

Constituent	Certified Value	1SD
Gold, Au (ppm)	3.04	0.08

Prepared by:
Ore Research & Exploration Pty Ltd
November 2009

REPORT 09-801-17c

INTRODUCTION

OREAS reference materials (RM's) 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 explorationist, they provide an important control in analytical data sets related to exploration from the grass roots level through to resource definition. To the mine geologist, they provide a tool for grade control in routine mining operations. To the analyst, they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

SOURCE MATERIALS

Reference material OREAS 17c 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 material OREAS 17c is given in Table 1. The constituents SiO_2 to LOI are the means of duplicate XRF analyses determined using a borate fusion method, S and C are means of duplicate IR combustion furnace analyses, while the remaining constituents, Ag to Zr, are means of duplicate analyses determined by 4-acid digestion with ICP-MS finish.

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 certified value and confidence interval are based on a round robin program incorporating a total of 128 analyses at 19 laboratories.

COMMINUTION AND HOMOGENISATION PROCEDURES

The materials comprising OREAS 17c were prepared in the following manner:

   a)   jaw crushing to minus 3mm
   b)   drying to constant mass at 105°C
   c)   milling of the barren material to 98% minus 75 micron
   d)   milling of the gold–bearing material to 100% minus 20 microns
   e)   blending in appropriate proportions to achieve the desired grade
   f)   bagging into 25kg sublots
   g)   packaging into 60g units in laminated foil pouches and 1kg units in plastic jars

ANALYSIS OF OREAS 17c

Nineteen 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 S. Each laboratory received two scoop-split 110 gram subsamples from each of three 1kg test units taken at regular intervals during the bagging stage. They were instructed to dry the six samples at 105°C to constant mass and perform one 25-30 gram fire assay gold determination on each sample with new pots. The nested

design of the interlaboratory programme is amenable to analysis of variance (ANOVA) and enables a comparative assessment of within- and between-unit homogeneity (see 'ANOVA study' section).

Table 1. Approximate major and trace element composition of gold-bearing reference material OREAS 17c; wt.% - weight percent; ppm - parts per million.

Constituent	wt.%	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO_2	49.1	Ag	0.5	Gd	5	Sb	4.5
TiO_2	1.41	As	2055	Hf	3	Sc	20.5
Al_2O_3	12.8	Ba	398	Ho	0.82	Sm	4.83
Fe_2O_3	14.1	Be	1.0	In	0.05	Sn	1
MnO	0.33	Bi	0.1	La	18.1	Sr	333
MgO	6.63	Cd	<0.5	Li	10.3	Ta	0.85
CaO	8.40	Ce	32.7	Lu	0.26	Tb	0.76
Na_2O	2.40	Co	44	Mo	4.3	Te	<0.2
K_2O	0.71	Cs	2	Nb	13	Th	3.5
P_2O_5	0.41	Cu	130	Nd	19.7	U	1.05
LOI	2.72	Dy	5	Ni	136	W	3.8
C	0.56	Er	2.3	Pb	10.5	Y	20.5
S	1.59	Eu	1.6	Pr	4.84	Yb	1.8
		Ga	18.5	Rb	21.5	Zn	139
						Zr	123

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 'Lab S' 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 excellent with all labs but one lying within ~2.5% relative of the corrected mean of means of 3.04 ppm Au.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 17c

Certified Value and Confidence Limits
The certified value was determined from the mean of means of accepted replicate values of accepted laboratory data sets A to S 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;
>
> $\bar{x}_i$ 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 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

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

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

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. Results for gold in OREAS 17c (FA - fire assay; AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission spectrometry; GRAV - gravimetry; 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; outliers in bold and left justified; sample charge weights shown in row 3; values in ppm).

Replicate No.	Lab A FA*GRAV 30g	Lab B FA*AAS 30g	Lab C FA*AAS 30g	Lab D FA*AAS 25g	Lab E FA*AAS 30g	Lab F FA*AAS 30g	Lab G FA*OES 30g	Lab H FA*AAS 30g	Lab I FA*OES 40g
1	3.19	2.93	**2.97**	**2.99**	2.88	3.19	2.98	3.12	2.95
2	3.19	2.97	3.08	3.08	2.95	3.09	**2.88**	3.00	3.03
3	3.18	2.96	3.10	3.08	2.87	3.12	2.97	3.06	3.03
4	3.04	2.96	3.08	3.09	2.83	3.13	2.99	3.06	2.97
5	3.07	2.95	3.09	3.03	2.89	3.08	3.01	3.15	3.02
6	3.07	2.97	3.13	3.09	2.91	3.12	3.03	3.13	3.04
Mean	3.12	2.96	3.08	3.06	2.89	3.12	2.98	3.09	3.01
Median	3.13	2.96	3.09	3.08	2.89	3.12	2.99	3.09	3.03
Std.Dev.	0.07	0.01	0.05	0.04	0.04	0.04	0.05	0.05	0.04
Rel.Std.Dev.	2.25%	0.45%	1.78%	1.34%	1.39%	1.24%	1.75%	1.77%	1.24%
PDM[3]	2.76%	-2.76%	1.17%	0.68%	-4.97%	2.70%	-2.07%	1.60%	-1.08%

Table 2 continued.

Replicate No.	Lab J FA*AAS 30g	Lab K FA*AAS 30g	Lab L FA*AAS 30g	Lab M FA*AAS 30g	Lab N FA*AAS 30g	Lab O FA*AAS 30g	Lab P FA*AAS 40g	Lab Q FA*AAS 30g	Lab R FA*AAS 30g
1	3.12	3.08	3.03	3.05	2.97	2.94	3.05	2.99	2.90
2	3.03	3.08	3.05	3.09	3.05	2.79	3.04	2.97	**2.77**
3	3.06	3.12	3.13	3.16	3.04	2.98	3.06	2.94	3.04
4	3.14	3.09	3.07	3.14	3.06	2.86	3.03	2.93	3.04
5	2.98	3.09	3.04	3.10	3.01	2.92	3.09	2.93	2.86
6	3.08	3.06	3.12	3.10	3.00	2.90	2.99	2.99	3.01
Mean	3.07	3.09	3.07	3.11	3.02	2.90	3.04	2.96	2.94
Median	3.07	3.09	3.06	3.10	3.03	2.91	3.05	2.96	2.95
Std.Dev.	0.06	0.02	0.04	0.04	0.03	0.07	0.03	0.03	0.11
Rel.Std.Dev.	1.92%	0.64%	1.38%	1.25%	1.14%	2.29%	1.09%	0.97%	3.76%
PDM[3]	0.95%	1.55%	1.11%	2.21%	-0.59%	-4.64%	0.13%	-2.67%	-3.35%

The z-score test is used in combination with a second method of individual outlier detection that determines the percent deviation of the individual value from the median. Outliers in general are selected on the basis of z-scores > 2.5 and with percent deviations > 1.5%. In certain instances statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set is tested for outlying status based on z-score discrimination and rejected if $|z_i|$ > 2.5. After individual and lab data set outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status.

Table 3. Analytical results for gold in OREAS 17c by INAA (instrumental neutron activation analysis on 0.5 gram analytical subsample weights; other abbreviations as for Table 2).

Replicate No.	Lab S INAA 0.5g
1	3.22
2	3.07
3	3.26
4	3.12
5	3.14
6	3.15
7	3.21
8	3.17
9	3.29
10	3.24
11	3.29
12	3.09
13	3.04
14	3.10
15	3.15
16	3.13
17	3.19
18	3.16
19	3.16
20	3.27
Mean	3.17
Median	3.16
Std.Dev.	0.07
Rel.Std.Dev.	2.29%
PDM[3]	4.38%

Individual outliers and, more rarely, laboratory means deemed to be outlying are shown left justified and in bold in the tabulated results (Tables 2 and 3) and have been omitted in the determination of the certified value.

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 certified value, i.e. the narrower the confidence interval the greater the certainty in the certified value.

Table 4. Certified Value and 95% Confidence Interval

Constituent	Certified	95% Confidence Interval	
	Value	Low	High
Gold, Au (ppm)	3.04	3.00	3.08

Note: intervals may appear asymmetric due to rounding

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

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

where

n *is the number of results reported by laboratory Q;*

$1 - \alpha$ *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.

Table 5. Certified Value and Tolerance Interval.

Constituent	Certified Value	Tolerance Interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	3.04	3.01	3.07

Note: intervals may appear asymmetric due to rounding

From the INAA data set an estimated tolerance interval of ±0.03 ppm at an analytical subsample weight of 30 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 30g-sized subsamples will have concentrations lying between 3.01 and 3.07 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).

ANOVA Study
The sampling format for OREAS 17c was structured to enable nested ANOVA treatment of the round robin results. During the bagging stage immediately following final homogenization, samples were taken at 20 intervals representative of the entire batch of OREAS 17c. The data from eighteen labs were used in the ANOVA study (all data except the INAA data from Lab S) where each received paired samples of three different, non-adjacent, sampling units. For example, the six samples that any one of the sixteen participating labs could have received is:

- Sample 1 (from sampling interval 1)
- Sample 2 (from sampling interval 6)
- Sample 3 (from sampling interval 11)
- Sample 4 (from sampling interval 1)
- Sample 5 (from sampling interval 6)
- Sample 6 (from sampling interval 11)

The purpose of the ANOVA investigation was to compare the within-unit variance with that of the between-unit variance. This approach permitted an assessment of homogeneity across the entire batch of OREAS 17c. The test was performed using the following parameters:

- Significance Level α = P (type I error) = 0.05
- Null Hypothesis, H_0: Between-unit variance is no greater than within-unit variance (reject H_0 if p-value < 0.05)
- Alternative Hypothesis, H_1: Between-unit variance is greater than within-unit variance

P-values are a measure of probability whereby values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The dataset was filtered for both individual and laboratory outliers prior to calculation of the p-value. This derived a p-value of 0.52 and indicates no evidence that between-unit variance is greater than within-unit variance. Conclusion: do not reject H_0. Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that gold is uniformly

distributed throughout OREAS 17c and that the variance between two subsamples from the same unit is identical to the variance from two subsamples taken from any two separate units.

Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected for a particular analyte from a laboratory being monitored by this standard in a QA/QC program. They incorporate errors attributable to measurement (analytical bias and precision) and standard variability. For an effective standard 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 after removal of all individual and lab dataset (batch) outliers as well as application of a non-iterative 3 standard deviation filter. These outliers can only be removed if they can be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM. 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 (1SD), 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. It is important to note that performance gates calculated from a single submission round robin, as in the present case, do not take reproducibility errors (batch-to-batch bias) into consideration and will accordingly be more constrained than those incorporating a temporal dimension.

For the second method a simple ±5% error bar on the certified value is used as the window of acceptability (refer Table 6). 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 6. Performance Gates for OREAS 17c

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Au (ppm)	3.04	0.08	2.87	3.21	2.79	3.29	2.74%	5.49%	8.23%	2.89	3.19

Note: intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

Accurassay Laboratories, Thunder Bay, ON, Canada
Acme Analytical Laboratories, Vancouver, BC, Canada
Activation Laboratories, Ancaster, ON, Canada
Amdel Laboratories, Adelaide, SA, Australia
Alaska Assay Laboratories, Fairbanks, AK, United States of America
ALS Chemex, Perth, WA, Australia
ALS Chemex, Townsville, QLD, Australia
ALS Chemex, La Serena, Chile, South America
ALS Chemex, Sparks, Nevada, USA
ALS Chemex, Val-d'or, Quebec, Canada
ALS Chemex, Vancouver, BC, Canada
Genalysis Laboratory Services, Perth, WA, Australia

Intertek Testing Services, Jakarta, Indonesia
OMAC Laboratories, Loughrea, County Galway, Ireland
SGS Lakefield Research, Lakefield, ON, Canada
SGS, Townsville, QLD, Australia
SGS Australia, Perth, WA, Australia
Ultra Trace, Perth, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The gold ore reference material, OREAS 17c 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 plastic jars.

INTENDED USE

OREAS 17c is a reference material intended for the following:

i)   for the monitoring of laboratory performance in the analysis of gold in geological samples;
ii)   for the calibration of instruments used in the determination of the concentration of gold;
iii)   for the verification of analytical methods for gold;
iv)   for the preparation of secondary reference materials of similar composition;

STABILITY AND STORAGE INSTRUCTIONS

OREAS 17c has been prepared from gold ore diluted with barren alkali olivine basalt. The CRM is considered to have long-term stability under normal storage conditions.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The certified value for OREAS 17c refers to the concentration level of Au after removal of hygroscopic moisture by drying in air to constant mass at 105° C. If the reference material is not dried by the user prior to analysis, the certified 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

Craig Hamlyn (B.Sc. Hons.), Geology

REFERENCES

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