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**CERTIFICATE OF ANALYSIS FOR**

**GOLD ORE REFERENCE MATERIAL**

**OREAS 15d**

**SUMMARY STATISTICS**

Constituent	Recommended Value	1SD
Gold, Au (ppm)	1.559	0.042

*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 explorationist, they provide an important control in analytical data sets related to exploration from the grass roots level through to resource definition. 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 15d 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 15d is given in Table 1. The constituents SiO<sub>2</sub> to Total 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 recommended value and confidence interval are based on a round robin program incorporating a total of 116 analyses at 17 laboratories.

## COMMINUTION AND HOMOGENISATION PROCEDURES

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

- a) jaw crushing to minus 3mm
- b) drying to constant mass at 105<sup>o</sup>C
- c) milling of the barren material to 98% minus 75 micron
- d) milling of the gold-bearing material to 100% minus 20 micron
- 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 15d

Eighteen 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. With the exception of Laboratory S, each laboratory received two scoop-split 120 gram subsamples from each of three 1kg test units taken at regular intervals during the bagging stage. They were instructed to carry out one 30-50 gram

fire assay gold determination with new pots on each subsample. 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 15d; wt.% - weight percent; ppm - parts per million.

Constituent	wt. %	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO <sub>2</sub>	52.86	Ag	0.5	Gd	4.9	Sb	2.3
TiO <sub>2</sub>	1.61	As	2445	Hf	3.2	Sc	21
Al <sub>2</sub> O <sub>3</sub>	13.25	Ba	252	Ho	0.81	Sm	4.7
Fe <sub>2</sub> O <sub>3</sub>	12.17	Be	0.9	In	0.06	Sn	1
MnO	0.188	Bi	0.4	La	16.9	Sr	357
MgO	6.47	Cd	<0.5	Li	8.3	Ta	1
CaO	7.95	Ce	34.1	Lu	0.23	Tb	0.75
Na <sub>2</sub> O	2.74	Co	43	Mo	2	Te	0.3
K <sub>2</sub> O	0.743	Cs	0.8	Nb	17.8	Th	2.9
P <sub>2</sub> O <sub>5</sub>	0.326	Cu	68	Nd	18.2	U	0.8
LOI	1.28	Dy	4.3	Ni	147	W	1.5
Total	100.4	Er	2.1	Pb	12	Y	20.2
C	0.20	Eu	1.6	Pr	4.29	Yb	1.73
S	0.62	Ga	17.9	Rb	21.1	Zn	107
						Zr	120

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<sup>3</sup>). Interlaboratory agreement of the means is good with all labs lying within 4% relative of the corrected mean of means of 1.56 ppm Au.

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 15d

### 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

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

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

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 ( $p-1$ ):

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

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

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

$$S = 1.483 \frac{\text{median } |x_j - \text{median}(x_i)|}{j=1, \dots, n \quad i=1, \dots, n}$$

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

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.

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 entire 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.

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 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 2. Analytical results for gold in OREAS 15d (FA - fire assay; AAS - flame atomic absorption spectrometry; OES - inductively coupled plasma optical emission 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; outliers in bold and left justified; sample charge weights shown in row 3; values in ppm).

Replicate No.	Lab A FA*AAS 50g	Lab B FA*OES 30g	Lab C FA*AAS 25g	Lab D FA*AAS 30g	Lab E FA*AAS 40g	Lab F FA*OES 30g	Lab G FA*AAS 50g	Lab H FA*OES 40g	Lab I FA*AAS 50g
1	1.450	1.585	<b>1.530</b>	1.620	1.590	1.510	1.500	1.550	1.640
2	1.470	<b>1.605</b>	1.560	1.600	1.560	1.560	1.490	1.555	1.600
3	1.400	1.575	1.570	1.610	1.580	1.530	1.490	1.550	1.570
4	1.450	<b>1.550</b>	1.580	1.600	1.540	1.550	1.510	1.550	1.570
5	1.380	1.580	1.570	1.620	1.510	1.540	1.460	<b>1.570</b>	1.590
6	1.410	1.575	1.580	1.630	<b>1.430</b>	1.560	1.510	1.540	1.600
Mean	<b>1.427</b>	1.578	1.565	1.613	1.535	1.542	1.493	1.553	1.595
Median	1.430	1.578	1.570	1.615	1.550	1.545	1.495	1.550	1.595
Std.Dev.	0.035	0.018	0.019	0.012	0.059	0.019	0.019	0.010	0.026
Rel.Std.Dev.	2.45%	1.13%	1.20%	0.75%	3.84%	1.26%	1.25%	0.64%	1.62%
PDM <sup>3</sup>	-8.50%	1.23%	0.37%	3.47%	-1.55%	-1.12%	-4.22%	-0.43%	2.30%

Table 2. Continued

Replicate No.	Lab J FA*AAS 30g	Lab K FA*AAS 30g	Lab L FA*AAS 50g	Lab M FA*AAS 30g	Lab N FA*AAS 50g	Lab O FA*AAS 50g	Lab P FA*OES 40g	Lab Q FA*AAS 30g	Lab R FA*AAS 30g
1	1.523	1.575	1.470	1.560	1.520	1.560	1.580	1.530	1.540
2	1.525	1.615	1.510	1.600	1.520	1.630	1.592	1.560	<b>1.470</b>
3	1.517	1.630	1.495	1.620	1.500	1.570	1.571	1.510	1.510
4	1.528	1.595	1.500	1.595	1.520	1.600	1.550	1.530	1.520
5	1.522	1.615	1.505	1.580	1.510	1.570	1.612	1.550	1.530
6	1.528	1.600	1.490	1.570	1.510	1.520	1.642	1.520	1.520
Mean	1.524	1.605	1.495	1.588	1.513	1.575	1.591	1.533	1.515
Median	1.524	1.608	1.498	1.588	1.515	1.570	1.586	1.530	1.520
Std.Dev.	0.004	0.019	0.014	0.022	0.008	0.037	0.032	0.019	0.024
Rel.Std.Dev.	0.27%	1.20%	0.95%	1.38%	0.54%	2.37%	2.04%	1.21%	1.60%
PDM <sup>3</sup>	-2.27%	2.94%	-4.12%	1.82%	-2.94%	1.01%	2.05%	-1.66%	-2.83%

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

Replicate No.	Lab S INAA
1	1.600
2	1.600
3	1.640
4	1.640
5	1.600
6	1.630
7	1.590
8	1.600
9	1.630
10	1.620
11	1.620
12	1.630
13	1.590
14	<b>1.700</b>
15	1.620
16	1.610
17	1.620
18	1.640
19	1.640
20	1.620
Mean	1.622
Median	1.620
Std.Dev.	0.025
Rel.Std.Dev.	1.53%
PDM <sup>3</sup>	4.03%

Table 4. Recommended Value and 95% Confidence Interval

Constituent	Recommended Value	95% Confidence Interval	
		Low	High
Gold, Au (ppm)	1.559	1.540	1.579

### 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

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

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

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'<sub>2</sub>* 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. Recommended Value and Tolerance Interval.

Constituent	Recommended Value	Tolerance Interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppm)	1.559	1.551	1.567

From the INAA data set an estimated tolerance interval of  $\pm 0.01$  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 1.551 and 1.567 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).

### **ANOVA Study**

The sampling format for OREAS 15d 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 15d. Eight labs were chosen for the ANOVA study (Labs A to H) where each received paired samples of three different, non-adjacent, sampling units. For example, the six samples that any one of the eight participating labs could have received is:

- Sample 1 (from sampling interval 1)
- Sample 2 (from sampling interval 1)
- Sample 3 (from sampling interval 6)
- Sample 4 (from sampling interval 6)
- Sample 5 (from sampling interval 11)
- 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 15d. The test was performed using the following parameters:

- Significance Level  $\alpha = P$  (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 0.64 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 15d 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  $\pm 5\%$  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 15d

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)	1.559	0.042	1.475	1.643	1.433	1.685	2.69%	5.38%	8.07%	1.481	1.637

Note: intervals may appear asymmetric due to rounding

## PARTICIPATING LABORATORIES

Acme Analytical Laboratories Ltd, Vancouver, BC, Canada  
 Activation Laboratories, Ancaster, Ontario, Canada  
 Amdel Laboratories, Perth, WA, Australia  
 Amdel Laboratories Ltd, Thebarton, SA, Australia  
 ALS Chemex, Brisbane, 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 Pty Ltd, Perth, WA, Australia  
 Intertek Testing Services, Jakarta, Indonesia  
 McPhar Laboratories, Legaspi Village, Makati City, Philippines  
 OMAC Laboratories Ltd, Loughrea, County Galway, Ireland  
 PT Indo Assay Laboratories, Balikpapan, Kalimantan Timur, Indonesia  
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 Ultra Trace Pty Ltd, Perth, WA, Australia



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

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

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

## **INTENDED USE**

OREAS 15d 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 15d 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 recommended value for OREAS 15d refers to the concentration level of gold in its packaged state. Therefore it should not be dried prior to weighing and analysis.

## **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 35 (2006), *Rference materials - General and statistical principals for certification.*

ISO Guide 3207 (1975), *Statistical interpretation of data - Determination of a statistical tolerance interval.*