

CERTIFICATE OF ANALYSIS FOR

GOLD REFERENCE MATERIAL

OREAS 15Pz

SUMMARY STATISTICS

Recommended value and 95% confidence interval

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm) – Fire Assay	1.27	1.25	1.29
Gold, Au (ppm) – Aqua Regia	1.21	1.16	1.26

Recommended value and tolerance interval

Constituent	Recommended value	Tolerance interval 1- α =0.99, ρ =0.95	
		Low	High
Gold, Au (ppm) – 25g Fire Assay	1.27	1.26	1.28
Gold, Au (ppm) – 50g Fire Assay	1.27	1.26	1.28

Prepared by:
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SOURCE MATERIAL

Reference material OREAS 15Pz was prepared from a blend of barren alkali olivine basalt and gold-bearing Magdala ore from the Stawell Gold Mine, west-central Victoria, Australia. Mineralisation in the ore consists of a quartz-sericite-carbonate schist assemblage containing the sulphides pyrite and arsenopyrite. Some of the gold is refractory in occurrence, being hosted by arsenopyrite.

COMMUNITION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- a) *drying;*
- b) *crushing and screening;*
- c) *preliminary homogenisation;*
- d) *check assaying*
- e) *milling of gold ore to minus 20 microns;*
- f) *homogenisation;*
- g) *check assaying;*
- h) *dilution with minus 75 micron barren basalt to achieve desired grade;*
- i) *re-homogenisation;*
- j) *packaging into 60g units sealed in laminated foil pouches.*

ANALYSIS OF OREAS 15Pz

Ten Australian commercial laboratories participated in the analytical program. The results for gold via fire assay and aqua regia digest together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM³) are presented in Tables 1 and 2. The parameter PDM³ is a measure of laboratory accuracy while the relative standard deviation is an effective measure of analytical precision where homogeneity of the test material has been confirmed. The analytical methods employed by each laboratory are given in the table captions. With the exception of the INAA work, five 110g samples were submitted to each laboratory for analysis.

Fire assay gold was determined in five replicate assays using a fire assay technique (40-50g charge with new pots) together with AAS (eight laboratories) or ICPOES (one laboratory) finish. One laboratory determined gold in twenty replicates via instrumental neutron activation analysis (INAA) using 0.5g analytical subsample weights. Gold was also determined by aqua regia following roasting (20-30g analytical subsample weight) and with AAS (six laboratories) or ICPMS (one laboratory) finish. Each set of subsamples submitted to each laboratory was taken at regular intervals during packaging of the standard in order to maximise their representation. The twenty INAA subsamples, on which much of the homogeneity evaluation is based, were also taken

at regular intervals during packaging and are considered representative of the entire batch.

Table 1. Analytical results for fire assay gold in OREAS 15Pz (INAA - instrumental neutron activation analysis; FA*AAS - fire assay / atomic absorption spectrometry; FA*OES - fire assay/inductively coupled plasma optical emission spectrometry; Std.Dev. and Rel.Std.Dev. are one sigma values; PDM³ - percent deviation of lab mean from corrected mean of means; outliers in bold; values in ppm).

Replicate No.	Lab A INAA (0.5g)	Lab B FA*AAS (50g)	Lab C FA*OES (40g)	Lab D FA*AAS (50g)	Lab E FA*AAS (50g)	Lab F FA*AAS (50g)	Lab G FA*AAS (50g)	Lab H FA*AAS (50g)	Lab I FA*AAS (50g)	Lab J FA*AAS (50g)
1	1.255	1.25	1.32	1.299	1.32	1.26	1.23	0.20*	1.29	1.26
2	1.225	1.33	1.33	1.284	1.33	1.26	1.24	1.22	1.29	1.24
3	1.218	1.30	1.29	1.292	1.30	1.24	1.25	1.17	1.32	1.27
4	1.241	1.30	1.28	1.284	1.34	1.21	1.26	1.20	1.28	1.23
5	1.283	1.28	1.30	1.292	1.33	1.23	1.25	1.25	1.27	1.26
6	1.288									
7	1.251									
8	1.292									
9	1.293									
10	1.273									
11	1.299									
12	1.263									
13	1.242									
14	1.225									
15	1.271									
16	1.278									
17	1.257									
18	1.248									
19	1.240									
20	1.256									
Mean	1.260	1.292	1.304	1.290	1.324	1.240	1.246	1.210	1.290	1.252
Median	1.257	1.300	1.300	1.292	1.330	1.240	1.250	1.210	1.290	1.260
Std.Dev.	0.024	0.029	0.021	0.006	0.015	0.021	0.011	0.034	0.019	0.016
Rel.Std.Dev.	1.92%	2.28%	1.59%	0.49%	1.15%	1.71%	0.92%	2.78%	1.45%	1.31%
PDM ³	-0.86%	1.67%	2.61%	1.53%	4.19%	-2.42%	-1.95%	-4.79%	1.51%	-1.48%

*Value excluded from stats

Table 2. Analytical results for pre-roast aqua regia gold in OREAS 15Pz (AR*AAS - aqua regia / atomic absorption spectrometry; AR*MS - fire assay/inductively coupled plasma mass spectrometry; Std.Dev. and Rel.STD.Dev. are one sigma values; abbreviations as in Table 1; values in ppm).

Replicate No.	Lab B AR*AAS	Lab C AR*MS	Lab D AR*AAS	Lab F AR*AAS	Lab G AR*AAS	Lab I AR*AAS	Lab J AR*AAS
1	1.08	1.22	1.257	1.18	1.25	1.22	1.29
2	1.09	1.17	1.269	1.22	1.26	1.25	1.24
3	1.12	1.21	1.232	1.18	1.29	1.25	1.27
4	1.11	1.20	1.211	1.33	1.25	1.31	1.26
5	1.15	1.13	1.171	1.07	1.30	1.22	1.23
Mean	1.11	1.19	1.23	1.20	1.27	1.25	1.26
Median	1.11	1.20	1.23	1.18	1.26	1.25	1.26
Std.Dev.	0.03	0.04	0.04	0.09	0.02	0.04	0.02
Rel.Std.Dev.	2.47%	3.07%	3.17%	7.81%	1.85%	2.94%	1.90%
PDM ³	-8.54%	-2.28%	1.18%	-1.45%	4.65%	3.00%	3.66%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 15Pz

Recommended Value and Confidence Limits

The certified value is the mean of means of accepted replicate values of accepted participating laboratories computed according to the formulae

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

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

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 ;

$\bar{\bar{x}}$ 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 $(p-1)$.

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

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

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

The distribution 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 and of outlying laboratory means was based primarily 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.

In certain instances “statistician’s prerogative” has been employed to discriminate 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 3. Recommended values and 95% confidence intervals for OREAS 15Pz.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppm) – Fire Assay	1.27	1.25	1.29
Gold, Au (ppm) – Aqua Regia	1.21	1.16	1.26

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. To eliminate, or at least substantially reduce, the effect of analytical error in the determination of homogeneity a procedure incorporating reduced analytical subsample weights was used. This entailed using the high precision INAA data alone, obtained on subsample weights of 0.5g (compared to 40-50g 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 sampling error. The corresponding standard deviation at a 25 or 50g subsample weight can then be determined from the observed standard deviation of the 0.5g data using the known relationship between the two parameters (Kleeman, 1967).

No outliers were removed from the results prior to the calculation of tolerance intervals. The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

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

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

where

n is the number of results;

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, α unknown);

s is the standard deviation.

The meaning of these tolerance limits may be illustrated for gold, where 99% of the time at least 95% of subsamples will have concentrations lying between 1.26 and 1.28 g/t. 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 high level of repeatability indicated by the low coefficients of variation in Table 1 (particularly the 0.5 g INAAI data) is consistent with the narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 15Pz.

Table 4. Recommended values and tolerance limits for OREAS 15Pz.

Constituent	Recommended value	Tolerance limits 1- α =0.99, ρ =0.95	
		Low	High
Gold, Au (ppm) at 25g	1.27	1.26	1.28
Gold, Au (ppm) at 50g	1.27	1.26	1.28

PARTICIPATING LABORATORIES

ALS Chemex, Garbutt, QLD, Australia
 ALS Chemex, Malaga, WA, Australia
 Amdel Laboratories, Thebarton, SA, Australia
 Amdel Laboratories, Wangara, WA, Australia
 Aminya Laboratories, Ballarat, VIC, Australia
 Analabs, Welshpool, WA, Australia
 Becquerel Laboratories, Lucas Heights, NSW, Australia
 Genalysis Laboratory Services, Maddington, WA, Australia
 Standard and Reference Laboratories, Malaga, WA, Australia
 Ultra Trace, Canning Vale, WA, Australia

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