



97 Broadway NEDLANDS 6009  
Western Australia  
PO Box 3258  
Broadway Nedlands WA 6009  
Phone: (08) 9386 3304  
Facsimile: (08) 9386 3202  
Internet: <http://www.daa.com.au>  
ABN 68 009 304 956

## Certificate of Analysis

Reference Material: AUOP-5

Recommended Value	95% Confidence Interval		Inter-Laboratory Standard Deviation	Inter-Unit Standard Deviation
	Lower Limit	Upper Limit		
5.3738	5.2720	5.4756	0.1838	0.0885

Table 1 Recommended Values and 95% Confidence Interval of Gold Concentration (in ppm, or  $\mu\text{g/g}$ ). Based on the results of 15 laboratories.

### 1. Date of Certification

July 2012

### 2. Certificate Status

Original

### 3. Supplier of Reference Material

Western Refractories Pty Ltd  
12 Reggio Road  
Kewdale 6105 Western Australia

### 4. Prepared and Certified by

Dr John Henstridge BSc, PhD, CStat, Astat  
Principal Consultant Statistician  
Data Analysis Australia  
97 Broadway  
Nedlands, WA, 6009

Dr John Henstridge

## 5. Available Packaging

This reference material is packed in 2kg PE screw cap jars, labelled with the identification code, the expected value and standard deviation of the gold concentration, health warning and the manufacturer's contact details. They are also available in cartons of six jars, and each carton will include this certificate of analysis.

## 6. Origin of Reference Material

This reference material is based on a feldspar from the Pippingarra region, near Port Hedland, Western Australia.

The reference material has been well mixed. The entire batch was tested to ensure that the gold was evenly distributed throughout the material.

## 7. Intended Use

Gold Assay standards provided with associated geochemistry data.

## 8. Instructions for Use

Weigh out the usual quantity used in the analysis for total gold by the normal procedure. Homogeneity testing has shown that consistent results can be obtained for gold when using samples of 30 to 40 grams.

## 9. Method of Test Material Preparation

Gold (99.99% fine gold at <10µm particle size) was added to the matrix material to give the required grade. Several dilution stages, mixing at each stage, were used in conjunction with splitting to give one tonne of final material.

Following mixing, the reference material was discharged from a homogenous mixer through a vibrasonic sieve into large bags. A two-kilogram sample was taken after every 100 kilograms of the material was discharged. These two-kilogram samples were numbered in order of being discharged, then divided into subsamples of approximately 250 grams.

These subsamples were placed into zip lock bags, and then labelled with a random identifier. To ensure laboratory confidentiality, the labelling was done by staff from Data Analysis Australia.

The laboratories participating in this certification process were sent either three or four of the 250 gram subsamples. The laboratories were also sent a number of 250 gram subsamples from three other reference materials that were being analysed for certification. An indication of the expected gold concentration was given in broad terms, with a range of four parts per million.

## 10. Homogeneity Tests

The homogeneity test was incorporated into the methodology by ensuring that each laboratory received at least three subsamples of the reference material, taken from

samples spaced out across the ordering (see Analytical Methodology). There was no evidence that this reference material was non-homogenous across the two-kilogram samples.

## 11. Analytical Methodology

This reference material was analysed for certification along with three other reference materials, which allows the use of more powerful statistical techniques in the certification. Performing the certification in this manner also provides the following advantages:

- “Blinding” of the laboratories, in that they did not know which reference material each sample corresponded to, so the laboratory did not have prior knowledge as to what result they should obtain. The inclusion of the expected range of the gold concentration was to assist the laboratories in their analysis, and did not provide enough information for the laboratories to know which reference material was in each sample.
- Improved ability to detect bias between laboratories, such as if one laboratory gives results that are consistently higher/lower than other laboratories for all standards; and
- Ability to detect bias in the results between laboratories for each standard separately.

Eighteen laboratories were randomly allocated an identifying letter from A to R. The set of subsamples was designed in such a way that each laboratory received at least three subsamples of each reference material. These subsamples were taken from different ordered samples in such a way to eliminate the confounding of laboratory against the order of the sample. Table 2 shows the set of subsamples received by each laboratory, where AUOP-5 has been labelled as reference material D.

The subsamples were labelled with random identifiers so the laboratories were unaware of the reference material and ordered two-kilogram sample that the subsample came from. This provided a means of determining whether a laboratory had an overall bias or a problem of precision.

Lab	Ordered One Kilogram Sample									
	1	2	3	4	5	6	7	8	9	10
A	A	AB	BD		C	D	B	CD	AC	AD
B	D	A	AC	D	B	CD	A	BC	B	C
C	C	D	B	C	AD	BCD		AB	A	B
D	B	C	A	BD	CD	ABC	D	A		AD
E	A	B	D	ACD	BC	AB	C		D	C
F	D	A	C	BC	AB	A	BD	D	C	B
G	CD		BD	AB	AD		AC	C	B	AD
H	BC	D	AC	A	CD		B	BD	A	C
I	AB	C	B	D	BC	D	A	ACD		B
J	A	B	A	C	AB	CD	D	BC	D	AD
K		A	D	B	A	BC	CD	AB	CD	C
L	D	D	C	A	D	AB	BC	A	BC	B
M	C	CD	B	D	C	A	AB	D	AB	AD
N	B	BC	AD	C	B	D	AD	C	A	C
O	AD	AB	C	BD	A	C	C	B	D	B
P	CD	AD	B	AC		B	B	A	CD	AD
Q	BC	CD	AD	B		A	AD		BC	C
R	AB	BC	C	A	D		C	D	ABD	B

Table 2 Set of subsamples by laboratory. The reference materials being tested have been labelled A through to D, with AUOP-5 labelled D.

## 12. Calculation of Certified Values

Results for Gold via Fire Assay were returned by 18 laboratories. These results were used to calculate the recommended values and 95% confidence intervals for gold concentrations in each of the materials.

The Analysis of Variance (ANOVA) technique was used to assess the homogeneity of the reference materials across the one-kilogram samples and also to identify outliers in the gold concentration. Outliers were detected and removed by comparing individual results, sample results and overall laboratory results. Once the outliers were removed and the homogeneity of the reference material was confirmed, the remaining results were used to calculate the certified values.

The results from Laboratories A, D and N showed a consistent downward bias across all reference materials. All results from these laboratories were therefore excluded from the analysis. For this reference material, the remaining fifteen laboratories returned a total of 123 results across 52 subsamples three results were removed as outliers. No further results were removed as outliers. A summary of the results from the laboratories is given in Table 3, showing average values with and without outliers. A laboratory result marked with an asterisk signifies that some of that laboratory's results were identified as outliers and removed prior to the calculation of the recommended value, confidence limits and standard deviation.

The recommended value was calculated as an average of the laboratory average gold concentrations, and the confidence interval was created using the average and standard deviation of the laboratory averages. The confidence interval is calculated using the following formula:

$$X \pm (t * s / \sqrt{n})$$

where  $X$  is the average and  $s$  is the standard deviation of the laboratory averages,  $n$  is the number of laboratory averages used and  $t$  is the 0.025 critical value of the Student's  $t$ -distribution. The recommended values and confidence limits are given in Table 1 on the first page of this certificate.

Laboratory	Average Gold Concentration ( $\mu\text{g/g}$ )	
	Fire Assay (Outliers Removed)	Fire Assay
B	4.8800	4.8800
C	5.3867	5.3867
E	5.6083	5.6083
F	5.3733	5.3733
G*	5.4400	5.2650
H	5.4867	5.4867
I	5.2700	5.2700
J	5.1988	5.1988
K*	5.4018	5.4800
L	5.5983	5.5983
M*	5.4957	5.4163
O	5.1967	5.1967
P	5.3413	5.3413
Q	5.4794	5.4794
R	5.4500	5.4500

**Table 3 Average gold concentrations ( $\mu\text{g/g}$ ) by laboratory and method. A \* indicates that outliers were removed.**

The scale of inhomogeneity was determined through the Analysis of Variance and is presented in Table 1 as the Inter-Unit Standard Deviation. In determining the likely precision of a single assay using this standard, it is necessary to combine this with the measurement error that may depend upon laboratory practice and should be determined for each laboratory through the analysis of duplicates. The combining of standard deviations should be done through the summing of the corresponding variances. If the inter-unit standard deviation is not significantly different from zero this indicates that the reference material is homogenous.

Note that when making comparisons between laboratories, between batches or between duplicates the appropriate consideration of errors should be used. When in doubt appropriate statistical guidance should be obtained.

## 13. Notice

The reference material AUOP-5 and this certificate have been prepared with the appropriate care and attention. This certificate conforms to the requirements outlined in the ISO Guide 35, Reference materials – General and statistical principles for certification, 2006 edition.

The Purchaser by receipt hereof releases and indemnifies Data Analysis Australia and Western Refractories from and against all liability and costs arising from the use of this material.

## 14. Participating Laboratories

The laboratories that agreed to participate are shown in Table 4.

Laboratory	State/Province	Country
Acme Analytical Laboratories Ltd	British Columbia	Canada
Activation Laboratories	Ontario	Canada
ALS Minerals Nevada	Nevada	United States of America
ALS Minerals Orange	New South Wales	Australia
ALS Minerals Perth	Western Australia	Australia
ALS Minerals Townsville	South Australia	Australia
ALS Minerals Vancouver	British Columbia	Canada
Amdel Ltd, Adelaide	South Australia	Australia
Ammtec	Western Australia	Australia
Genalysis (Adelaide)	South Australia	Australia
Genalysis (Perth)	Western Australia	Australia
ITS Jakarta	Jakarta	Indonesia
SGS Kalgoorlie	Western Australia	Australia
SGS Perth	Western Australia	Australia
SGS Townsville	Queensland	Australia
SGS West Wyalong	New South Wales	Australia
Stewart Assay And Environmental Laboratories Lic	Chuy	Kyrgystan
Ultra Trace	Western Australia	Australia

**Table 4 Participating laboratories. (Not related to the order of laboratories in other tables.)**

To obtain additional copies of this certificate, please contact Western Refractories or visit their website <http://www.assaysupplies.com.au>.