



# ROCKLABS

WORLD LEADERS IN SAMPLE PREPARATION EQUIPMENT AND REFERENCE MATERIALS FOR USE IN GOLD ASSAYING

ROCKLABS LIMITED

161 NEILSON STREET, ONEHUNGA, PO BOX 18-142, GLEN INNES, AUCKLAND, NEW ZEALAND

Tel: +64 9 634 7696

Fax: +64 9 634 6896

Email: sales@rocklabs.com

Website: www.rocklabs.com

# Certificate of Analysis

## Reference Material SP49

### Recommended Values and 95% Confidence Intervals

**Gold Concentration: 18.34 (+/- 0.12) µg/g**

**Silver Concentration: 60.2 (+/- 1.0) µg/g**

The above values apply only to product in jars or sachets which have an identification number within the following range: **190 640 – 192 121**.

**Prepared and Certified By:**

Malcolm Smith BSc, FNZIC  
Malcolm Smith Reference Materials Ltd  
40 Oakford Park Crescent, Greenhithe  
North Shore City 0632  
**NEW ZEALAND**  
Email: Malcolm@MSRML.co.nz  
Telephone: +64 9 444 3534

**Date of Certification:**

19 June 2009

**Certificate Status:**

Original

**Available Packaging:**

This reference material has been packed in wide-mouthed jars that contain 2.5 kg of product. The contents of some jars may be subsequently repacked into sealed polyethylene sachets.

**Origin of Reference Material:**

Feldspar minerals, basalt and iron pyrites with minor quantities of finely divided gold and silver-containing minerals that have been screened to ensure there is no gold nugget effect.

**Supplier of Reference Material:**

ROCKLABS Ltd  
P O Box 18 142  
Auckland 1743  
**NEW ZEALAND**  
Email: sales@rocklabs.com  
Website: www.rocklabs.com  
Telephone: +64 9 634 7696

**Description:**

The reference material is a light grey powder that has been well mixed and a homogeneity test carried out after the entire batch was packaged into wide-mouthed jars. There is no soil component. The product contains crystalline quartz and therefore dust from it should not be inhaled.

The approximate chemical composition is:  
(Uncertified Values)

	%
SiO <sub>2</sub>	54.77
Al <sub>2</sub> O <sub>3</sub>	13.98
Na <sub>2</sub> O	3.91
K <sub>2</sub> O	3.94
CaO	4.43
MgO	3.75
TiO <sub>2</sub>	0.96
MnO	0.07
P <sub>2</sub> O <sub>5</sub>	0.25
Fe <sub>2</sub> O <sub>3</sub>	4.6
Fe	3.2
S	3.5

**Intended Use:**

This reference material is designed to be included with every batch of samples analysed and the results plotted for quality monitoring purposes.

**Stability:**

The container (jar or sachet) and its contents should not be heated to temperatures higher than 50 °C. Iron pyrites are likely to oxidize in the air but tests have shown that the increase in weight of an exposed reference material of similar matrix, in the Auckland climate, is less than 0.1 % per year.

**Method of Preparation:**

Pulverized feldspar minerals, basalt rock and barren iron pyrites were blended with finely pulverized and screened, gold and silver-containing minerals. Once the powders were uniformly mixed the composite was placed into 1482 wide-mouthed jars, each bearing a unique number. 40 jars were randomly selected from the packaging run and material from these jars was used for both homogeneity assessment and assignment of gold and silver values.

**Homogeneity Assessment for Gold:**

An independent laboratory carried out all gold analyses by fire assay of 30 g portions, using a gravimetric finish. The homogeneity assessment was carried out after the material had been packaged into jars.

### **Homogeneity Assessment for Gold continued:**

The contents of five randomly selected jars were compacted by vibration (to simulate the effect of freighting) and five samples removed successively from top to bottom from each jar. In addition, five samples were removed from the last jar in the series. A sample was also removed from the top of each of the 40 jars randomly selected from the 1482 jars in the batch. The results of analysis of the 70 samples (randomly ordered and then consecutively numbered before being sent to the laboratory) produced a relative standard deviation of 2.7 %. Six of these results were anomalously low and analysis of the slags/crucibles and cupels used in their analyses showed higher than normal quantities of gold. If results from these six samples are excluded the relative standard deviation drops to 0.8 %.

### **Analytical Methodology:**

Once homogeneity had been established, two sub-samples were submitted to a number of well-recognized laboratories in order to assign gold and silver values by consensus testing. The sub-samples were drawn from the 40 randomly selected jars and each laboratory received samples from two different jars. Indicative concentration ranges were given. All laboratories used fire assay for the gold analysis, with some using an instrument finish and some a gravimetric finish. A number of different analytical procedures were used for the silver analyses.

### **Calculation of Certified Value:**

40 laboratories returned results for gold and 27 for silver. Statistical analysis to identify outliers was carried out using the principles detailed in sections 7.3.2 – 7.3.4, ISO 5725-2: 1994. Assessment of each laboratory's performance was carried out on the basis of z-scores, partly based on the concept described in ISO/IEC Guide 43-1. Details of the criteria used in these examinations are available on request. As a result of these statistical analyses, four sets of results were excluded for the purpose of assigning a gold concentration value and nil sets were excluded for silver. Recommended values were thus calculated from the average of the remaining n = 36 sets of replicate results for gold and n = 27 for silver. The 95 % confidence interval was estimated using the formula:-

$$X \pm ts/\sqrt{n}$$

(where X is the estimated average, s is the estimated standard deviation of the laboratory averages, and t is the 0.025 tail-value from Student's t-distribution with n-1 degrees of freedom). The recommended values are provided at the beginning of the certificate in µg/g (ppm) units. A summary of the results used to calculate the recommended value is listed on page 4 for silver and page 5 for gold. The names of the laboratories that submitted results are listed on page 6. The results are listed in increasing order of the individual laboratory averages. A code also indicates the method used by the laboratory. The code starts with the sample weight used (where this is known) and finishes with either the instrument used (eg AAS or ICP-OES) or gravimetric finish (indicated by "grav"). Codes for the digestions are: fa = fire assay; Hfar = hydrofluoric acid and aqua regia; ar = aqua regia; 4ac = 4 acids: hydrofluoric, perchloric, nitric, hydrochloric; 3ac = 3 acids: perchloric, aqua regia. Statistical analysis for gold was carried out separately on two groups of samples – those

analysed with an instrument finish and those completed by gravimetry. There was no significant difference in the calculated gold average for each group. This applied with outliers included as well as with outliers excluded. Therefore the assigned value was calculated from the combined sets of results. No statistical examination was carried out to determine whether there was any significant difference obtained for silver by the different methods used.

Independent statistician, Tim Ball, has carried out statistical analysis of the consensus test results.

### Summary of Results Used to Calculate Silver Value (Listed in increasing order of individual laboratory averages)

<b>Silver (ppm)</b>			
<b>Method</b>	<b>Sample 1</b>	<b>Sample 2</b>	<b>Average</b>
30gfa/grav	52	59	55.5
1gar/AAS	59	55	57.0
15gfa/grav	57.67	56.69	57.180
ar/AAS	58	57	57.5
2gHFar/AAS	57	58	57.5
0.4g4ac/ICP-AES	57	59	58.0
0.3g4ac/AAS	57	60	58.5
ar/AAS	58	59	58.5
0.2gBr2,HNO3,HCl/ICP-OES	62	56	59.0
2g4ac/AAS	59	59	59.0
1g3ac/AAS	60	58	59.0
0.5g4ac/AAS	60	59	59.5
4ac/ICP-MS	59	60	59.5
30gfa/grav	59.560	59.640	59.600
30gfa/grav	60	60	60.0
0.5g4ac/AAS	61.2	59.3	60.25
30gfa/grav	61	60	60.5
0.2g4ac/AAS	58	63	60.5
fa/grav	60.95	60.88	60.915
0.5gHF/ar/AAS	60.80	62.05	61.425
1gar/AAS	62.2	62.8	62.50
25gfa/grav	61.2	63.8	62.50
ar/AAS	63.25	62.08	62.67
ar/AAS	62.77	63.94	63.355
0.5gar/AAS	63.2	65.4	64.30
30gfa/grav	65	65	65.0
30gfa/grav	64.70	65.3	65.00
Average of 27 sets = 60.2 ppm Standard deviation of 27 sets = 2.5 ppm			
<b><i>Note: this standard deviation should not be used as a basis to set control limits when plotting results from an individual laboratory.</i></b>			
Relative standard deviation = 4.2 % 95% Confidence interval for average = 1.0 ppm			

## Summary of Results Used to Calculate Gold Value

(Listed in increasing order of individual laboratory averages)

<b>Gold (ppm)</b>			
<b>Method</b>	<b>Sample 1</b>	<b>Sample 2</b>	<b>Average</b>
30gfa/grav	17.47	17.73	17.600
30gfa/grav	17.85	17.42	17.635
30gfa/ICP-ES	17.79	17.82	17.805
30gfa/AAS	17.9	18.0	17.95
50gfa/AAS	17.90	18.05	17.975
30gfa/AAS	18.05	18.00	18.025
30gfa/AAS	17.9	18.2	18.05
15gfa/grav	17.966	18.309	18.138
30gfa/AAS	18.133	18.258	18.196
30gfa/grav	18.080	18.33	18.205
30gfa/AAS	18.25	18.20	18.225
50gfa/AAS	18.435	18.065	18.250
fa/grav	18.10	18.45	18.275
30gfa/AAS	18.38	18.21	18.295
30gfa/AAS	18.25	18.35	18.300
30gfa/grav	18.55	18.05	18.300
30gfa/grav	18.32	18.30	18.310
fa/AAS	18.330	18.320	18.325
30gfa/grav	18.33	18.34	18.335
15gfa/grav	18.21	18.48	18.345
fa/grav	18.350	18.350	18.350
30gfa/grav	18.35	18.40	18.375
50gfa/AAS	18.65	18.15	18.400
25gfa/grav	18.4	18.4	18.40
30gfa/AAS	18.45	18.50	18.475
10gfa/AAS	18.22	18.74	18.480
25gfa/ICP	18.6	18.4	18.50
fa/grav	18.462	18.571	18.517
fa/grav	18.25	18.79	18.520
30gfa/grav	18.7	18.5	18.60
40gfa/ICP-OES	18.20	19.00	18.600
30gfa/grav	18.757	18.667	18.712
fa/grav	18.747	18.867	18.807
30gfa/AAS	18.8	18.9	18.85
20gfa/AAS	18.6	19.1	18.85
50gfa/AAS	19.25	19.45	19.350
Average of 36 sets = 18.34 ppm			
Standard deviation of 36 sets = 0.34 ppm			
<b><i>Note: this standard deviation should not be used as a basis to set control limits</i></b>			
<b><i>when plotting results from an individual laboratory.</i></b>			
Relative standard deviation = 1.9 %			
95% Confidence interval for average = 0.12 ppm			

## Participating Laboratories

<b>Australia</b>	ALS Chemex, Kalgoorlie ALS Chemex, Perth Amdel Ltd, Adelaide Amdel Ltd, Kalgoorlie Genalysis Laboratory Services Pty Ltd, Perth SGS Australia Pty Ltd, Perth SGS Australia Pty Ltd, Townsville Standard and Reference Laboratories, Perth Ultra Trace Pty Ltd, Perth
<b>Burkina Faso</b>	ALS Chemex, Ouagadougou
<b>Canada</b>	Accurassay Laboratories, Thunder Bay Acme Analytical Laboratories Ltd, Vancouver ALS Chemex, Val d'Or ALS Chemex, Vancouver Assayers Canada, Vancouver Bourlamaque Assay Laboratories Ltd, Val d'Or International Plasma Labs Ltd, Richmond Loring Laboratories Ltd, Calgary SGS Mineral Services, Lakefield Techni-Lab S.G.B. Abitibi Inc, Quebec TSL Laboratories Inc, Saskatoon
<b>Chile</b>	Acme Analytical Laboratories Ltd, Santiago
<b>Kyrgyzstan</b>	Alex Stewart Assay and Environmental Laboratories Ltd, Kara-Balta
<b>Mali</b>	ALS Chemex, Bamako
<b>New Zealand</b>	Amdel NZ Ltd, Macraes, Otago Amdel Ltd, Reefton SGS Minerals, Waihi
<b>Peru</b>	ALS Chemex, Lima Minera Yanacocha SRL – Newmont, Lima Inspectorate Services Peru S.A.C., Callao
<b>Russia</b>	Irgiredmet JSC, Analytical Centre, Irkutsk
<b>South Africa</b>	AB Analytical Laboratory Services, Boksburg Anglo Research, Johannesburg Performance Laboratories, Randfontein SGS South Africa (Pty) Ltd, Barberton SGS South Africa (Pty) Ltd, Southdale
<b>United States of America</b>	ALS Chemex, Reno Barrick Goldstrike Mines Inc, Carlin Newmont Mining Corporation, Carlin Laboratory Newmont Mining Corporation, Lone Tree Laboratory

## Instructions and Recommendations for Use:

Weigh out quantity usually used for analysis and analyze by normal procedure. Do not dry before weighing.

We quote a 95% confidence interval for our estimate of the declared value. This confidence interval reflects our uncertainty in estimating the true values for the gold and silver content of the reference material. The interval is chosen such that, if the same procedure as used here to estimate the declared value were used again and again, then 95% of the trials would give intervals that contained the true value. It is a reflection of how precise the trial has been in estimating the declared value. It **does not** reflect the variability any particular laboratory will experience in its own repetitive testing.

Some users in the past have misinterpreted this confidence interval as a guide as to how different an individual test result should be from the declared value. Some mistakenly use this interval, or the standard deviation from the consensus test, to set limits for control charts on their own routine test results using the reference material. Such use inevitably leads to many apparent out-of-control points, leading to doubts about the laboratory's testing, or of the reference material itself.

A much better way of determining the laboratory performance when analysing the reference material is to accumulate a history of the test results obtained, and plot them on a control chart. The appropriate centre line and control limits for this chart should be based on the average level and variability exhibited in the laboratory's **own** data. This chart will provide a clear picture of the long-term stability or otherwise of the laboratory testing process, providing good clues as to the causes of any problems. To help our customers do this more simply for themselves, we can provide a free Excel template that will produce sensible graphs, with intelligently chosen limits, from the customer's own data.


## Legal Notice:

This certificate and the reference material described in it have been prepared with due care and attention. However ROCKLABS Ltd, Malcolm Smith Reference Materials Ltd and Tim Ball Ltd accept no liability for any decisions or actions taken following the use of the reference material.

## References:

For further information on the preparation and validation of this reference material please contact Malcolm Smith.

### Certifying Officer



M G Smith BSc, FNZIC

### Independent Statistician



Tim Ball BSc (Hons)