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**CERTIFICATE OF ANALYSIS FOR**

**COPPER-GOLD STANDARD**

**OREAS 51P**

**SUMMARY STATISTICS**

Recommended Values, 95% Confidence and Tolerance Intervals

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High	Low	High
Gold, Au (ppb)	430	417	443	426	434
Copper, Cu (%)	0.728	0.716	0.740	0.717	0.739

*Prepared by:*  
*Ore Research & Exploration Pty Ltd*  
*April 2004*

## INTRODUCTION

OREAS reference materials (RMs) 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 analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures. To the explorationist they provide an important control in analytical data sets related to exploration from the grass roots level through to prospect evaluation. To the mine geologist they provide a valuable tool in grade control and QA/QC management programs. Following the implementation of new processing technology Ore Research & Exploration now produces gold RMs exhibiting a level of homogeneity previously unattainable. In certain instances RMs produced from a single source are sufficiently homogeneous to produce a relatively coarse-grained form designed to simulate drill chip samples. These have a grain size of minus 3mm and are designated with a "C" suffix to the RM identification number. These standards are packaged in 0.5-1kg units following homogenisation and are intended for submission to analytical laboratories in subsample sizes of as little as 250g. They offer the added advantages of providing a check on both sample preparation and analytical procedures while acting as a blind standard to the assay laboratory. The more conventional pulped standards have a grain size of minus 20 to minus 75 microns and a higher degree of homogeneity. These standards are distinguished by a "P" suffix to the standard identification number. In line with ISO recommendations successive batch numbers are now designated by the lower case suffixes "a", "b", "c", "d", etc.

## SOURCE MATERIALS

Reference material OREAS 51P is one of four porphyry copper-gold standards prepared from ore samples from the Northparkes Mine, central western New South Wales, Australia.

Mineralisation in the region is hosted by a sequence of late Ordovician to Early Silurian volcanics, intrusives and sediments that occur within the Bogan Gate Synclinal Zone of the Lachlan Fold Belt. The western portion of this zone is dominated by volcanics and host to the Goonumbla porphyry copper-gold deposits. The Late Ordovician Goonumbla Volcanics host the Northparkes deposits and are interpreted to have erupted from shallow water to partly emergent volcanic centres. They exhibit a broad range in composition from shoshonite through to latite to trachyte.

Coeval sub-volcanic quartz monzonite porphyries (and attendant mineralisation) have intruded the volcanics. They are generally small, sub-vertical, pipe-like intrusives. Typically the mineralised porphyries contain plagioclase and quartz phenocrysts in a matrix of fine-grained potassium feldspar and quartz with minor biotite and hornblende.

Copper-gold mineralisation occurs as stockwork quartz veins and disseminations associated with potassic alteration. This alteration is intimately associated spatially and temporally with the small finger-like quartz monzonite porphyries that intrude the Goonumbla Volcanics. Sulphides are zoned laterally from the centres of mineralisation. The central portions are bornite-rich with minor chalcopyrite, zoning outward through equal concentrations of bornite and chalcopyrite, to an outermost chalcopyrite-rich zone. Pyrite increases outward at the expense of bornite.

## COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- a) *drying;*
- b) *crushing and screening;*
- c) *preliminary homogenisation;*
- d) *milling to minus 20 microns;*
- e) *final homogenisation;*
- f) *bagging into 20kg sublots.*

## ANALYSIS OF OREAS 51P

The indicative major and trace element composition of OREAS 51P is given in Table 1. The constituents are the means of duplicate XRF analyses determined using a borate fusion method at the University of Melbourne, Victoria, Australia, and are uncertified values.

Table 1. Indicative major and trace element composition of reference material OREAS 51P; SiO<sub>2</sub> to Total in weight percent (Total includes traces); rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO <sub>2</sub>	55.94	Ba	1061
TiO <sub>2</sub>	0.59	Co	10
Al <sub>2</sub> O <sub>3</sub>	18.69	Cr	24
Fe <sub>2</sub> O <sub>3</sub>	4.96	Ga	22
MnO	0.07	Nb	3
MgO	2.09	Nd	21
CaO	2.26	Ni	30
Na <sub>2</sub> O	5.52	Rb	79
K <sub>2</sub> O	4.68	Sc	20
P <sub>2</sub> O <sub>5</sub>	0.44	Sr	642
SO <sub>3</sub>	0.56	Th	17
LOI	3.09	V	170
Total	99.76	Y	17
		Zn	67
		Zr	80

Fifteen commercial laboratories participated in the certification program for gold and copper and are listed in the section headed Participating Laboratories. To maintain anonymity laboratories have been randomly assigned a number code 1 through 15. Their results together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in Tables 2 to 4. The parameter PDM<sup>3</sup> 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 Becquerel, six 110g samples were submitted to each laboratory for analysis. These samples were duplicate scoop splits from three separate 1kg test units taken during the bagging stage. This two-stage nested design for the interlaboratory program was amenable to analysis of variance (ANOVA) treatment and enabled a comparative assessment of within- and between-unit homogeneity. The twenty-six INAA samples, on which much of the homogeneity evaluation is based, were also taken at regular intervals throughout the bagging stage and are considered representative of the entire batch.

Gold was determined in six replicate assays using a fire assay technique (40-50g charge with new pots) with flame AAS or ICPOES finish at thirteen laboratories (Table 2), while Becquerel determined gold in twenty-six samples via instrumental neutron activation analysis (INAA) using 0.5gm analytical subsample weights (Table 3). Copper was determined via four acid (HF-HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) digest with ICPOES or AAS finish (Table 4).

Table 2. Analytical results for gold in standard OREAS 51P (FA\*AAS - fire assay / atomic absorption spectrometry; FA\*OES - fire assay / inductively coupled plasma optical emission spectrometry; INAA - instrumental neutron activation analysis; Std.Dev. and Rel.Std.Dev. are one sigma values; PDM<sup>3</sup> is percent deviation of lab mean from corrected mean of means; outliers in bold; values in parts per billion.

Replicate Number	Lab 1 FA*AAS	Lab 2 FA*AAS	Lab 3 FA*AAS	Lab 4 FA*AAS	Lab 5 FA*OES	Lab 6 FA*AAS	Lab 7 FA*AAS
1	430	430	412	420	451	390	<b>426</b>
2	440	440	413	400	457	395	398
3	450	440	402	400	451	390	403
4	430	420	397	400	457	405	402
5	440	430	405	420	445	395	399
6	450	470	410	430	438	400	405
Mean	440	438	407	412	450	396	406
Std. Dev.	9	17	6	13	7	6	10
Rel.Std.Dev.	2.03%	3.93%	1.55%	3.23%	1.63%	1.48%	2.56%
PDM <sup>3</sup>	2.41%	2.02%	-5.39%	-4.18%	4.70%	-7.87%	-5.62%

Table 2. Continued.

Replicate Number	Lab 8 FA*AAS	Lab 9 FA*AAS	Lab 10 FA*AAS	Lab 11 FA*AAS	Lab 12 FA*AAS	Lab 13 FA*OES	Lab 14 INAA
1	450	408	470	455	402	440	Refer to Table 3
2	470	418	470	433	404	441	
3	463	417	450	438	404	451	
4	461	414	460	452	411	432	
5	432	398	450	449	411	449	
6	447	414	450	443	399	450	
Mean	454	412	458	445	405	444	458
Std. Dev.	14	7	10	9	5	7	16
Rel.Std.Dev.	3.01%	1.82%	2.15%	1.91%	1.20%	1.68%	3.51%
PDM <sup>3</sup>	5.63%	-4.22%	6.68%	3.58%	-5.70%	3.30%	6.68%

Table 3. Analytical results for gold in standard OREAS 51P via instrumental neutron activation analysis using a 0.5g analytical subsample weight (abbreviations as in Table 2; values in parts per billion).

Replicate Number	Lab 14 INAA
1	482
2	450
3	455
4	450
5	472
6	442
7	449
8	453
9	473
10	445
11	467
12	454
13	471
14	462
15	455
16	431
17	447
18	472
19	456
20	497
21	475
22	475
23	441
24	463
25	454
26	427
Mean	458
Std. Dev.	16
Rel.Std.Dev.	3.51%

Table 4. Analytical results for copper in standard OREAS 51P (4AD\*OES - four acid digest / inductively coupled plasma optical emission spectrometry; 4AD\*AAS - four acid digest / atomic absorption spectrometry, other abbreviations as in Table 2; values in parts per million).

Replicate Number	Lab 1 4AD*OES	Lab 2 4AD*OES	Lab 3 4AD*OES	Lab 4 4AD*AAS	Lab 5 4AD*OES	Lab 6 4AD*OES	Lab 7 4AD*OES
1	6970	5860	7460	7330	7340	7150	7380
2	7130	6480	7240	7380	7210	7300	7360
3	6970	6280	7670	7380	7300	7200	7270
4	6960	6130	7510	7140	7300	7350	7390
5	7150	6140	7590	7230	7270	7250	7180
6	6960	6320	7250	7070	7300	7250	7240
Mean	7023	<b>6202</b>	7453	7255	7287	7250	7303
Std. Dev.	91	211	176	130	44	71	86
Rel.Std.Dev.	1.29%	3.41%	2.37%	1.80%	0.60%	0.98%	1.18%
PDM <sup>3</sup>	-3.53%	-14.8%	2.37%	-0.35%	0.08%	-0.42%	0.31%

Table 4. Continued.

Replicate Number	Lab 8 4AD*AAS	Lab 9 4AD*AAS	Lab 10 4AD*OES	Lab 11 4AD*AAS	Lab 12 4AD*AAS	Lab 13 4AD*OES	Lab 15 4AD*OES
1	7800	7300	7268	7140	7020	7130	7560
2	7900	7300	7235	7170	7060	7100	7330
3	7600	7500	7318	7130	7000	7100	7720
4	7600	7300	7322	7020	7030	7140	7510
5	7800	7100	7354	7070	7030	7160	7300
6	7800	7200	7342	7120	7040	7140	7390
Mean	7750	7283	7307	7108	7030	7128	7468
Std. Dev.	122	133	46	54	20	24	159
Rel.Std.Dev.	1.58%	1.82%	0.63%	0.76%	0.28%	0.34%	2.13%
PDM <sup>3</sup>	6.45%	0.04%	0.36%	-2.36%	-3.44%	-2.09%	2.58%

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 51P

### 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

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

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

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 ( $p-1$ ).

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

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

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 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.

Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in bold italics and have been omitted in the determination of recommended values.

Table 5. Recommended values and 95% confidence intervals for OREAS 51P.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	430	417	443
Copper, Cu (wt. %)	0.728	0.716	0.740

### 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. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material analysed and, in particular, to deficiencies in accuracy of each analytical method. In determining tolerance intervals for copper that component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \bar{x}_i + \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^p n_i}$$

where

- $x_{ij}$  is the  $j$ th raw result reported by laboratory  $i$ ;
- $x'_{ij}$  is the  $j$ th transformed result reported by laboratory  $i$ ;
- $n_i$  is the number of results reported by laboratory  $i$ ;
- $p$  is the number of participating laboratories;
- $\bar{x}_i$  is the raw mean for laboratory  $i$ .

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$\begin{aligned} \text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha) s_g'' \\ \text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha) s_g'' \end{aligned}$$

where

- $n$  is the number of results;
- $1 - \alpha$  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 ( $m, \alpha$  unknown);
- $s_g''$  is the corrected grand standard deviation.

The meaning of these tolerance limits may be illustrated for copper, where 99% of the time at least 95% of subsamples will have concentrations lying between 0.717% and 0.739%. 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 corrected grand standard deviation,  $s_g''$ , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula

$$s_g'' = \frac{\sum_{i=1}^p (s_i (1 - \frac{s_i}{s_g'}))}{\sum_{i=1}^p (1 - \frac{s_i}{s_g'})}$$

where



$1 - \left( \frac{S_i}{2s'_g} \right)$  is the weighting factor for laboratory  $i$ ;

$s'_g$  is the grand standard deviation computed from the transformed (i.e. means-adjusted) results

according to the formula

$$s'_g = \left[ \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} (x'_{ij} - \bar{x}'_i)^2}{\sum_{i=1}^p n_i - 1} \right]^{1/2}$$

where  $\bar{x}'_i$  is the transformed mean for laboratory  $i$

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error.

For gold a more simplified procedure was used in the determination of homogeneity. This entailed using the high precision INAA data alone, obtained on an analytical subsample weight of 0.5gm (compared to 40-50gm 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 subsampling error. The corresponding standard deviation at a 50gm subsample weight can then be determined from the observed standard deviation of the 0.5gm data using the known relationship between the two parameters (Kleeman, 1967). The homogeneity of gold was then determined from tables of factors for two-sided tolerance limits for normal distributions. The high level of repeatability indicated by the low coefficients of variation in Table 2 and the 0.5gm Becquerel data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 51P.

Table 6. Recommended values and tolerance limits for OREAS 51P.

Constituent	Recommended value	Tolerance interval 1- $\alpha$ =0.99, $\rho$ =0.95	
		Low	High
Gold, Au (ppb)	430	426	434
Copper, Cu (wt. %)	0.728	0.717	0.739

No outliers were removed from the INAA results prior to the calculation of tolerance intervals for gold, although for copper, outliers were removed prior to the calculation of  $s'_g$

and a weighting factor of zero was applied to those data sets where  $s_j / 2s_g' > 1$  (i.e. where the weighting factor  $1 - s_j / 2s_g' < 0$ ).

## **PARTICIPATING LABORATORIES**

Acme Analytical Laboratories, Vancouver, BC, Canada  
Actlabs Pacific Pty Ltd, Redcliffe, WA, Australia  
ALS Chemex, Santiago, Chile  
ALS Chemex, Sparks, Nevada, USA  
Amdel Laboratories, Thebarton, SA, Australia  
Amdel Laboratories, Wangara, WA, Australia  
Becquerel Laboratories Inc, Lucas Heights, NSW, Australia  
Cantech Laboratories Inc, Calgary, Canada  
Cone Geochemical, Lakewood, Colorado, USA  
Genalysis Laboratory Services, Maddington, WA, Australia  
McPhar Geoservices (Phil.) Inc., Makati, Philippines  
SGS, Welshpool, WA, Australia  
SGS, Garbutt, QLD, Australia  
Ultra Trace, Canning Vale, WA, Australia  
Intertek Testing Services, Jakarta, Indonesia

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

The copper-gold ore reference material, OREAS 51P has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd  
6 – 8 Gatwick Road  
Bayswater North VIC 3153  
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It is available in unit sizes of 60g laminated foil packets.

## **INTENDED USE**

OREAS 51P is a reference material intended for the following:

- i) for the calibration of instruments used in the determination of the concentration of gold and copper;
- ii) for the verification of analytical methods for gold and copper;
- iii) for the preparation of secondary reference materials of similar composition;
- iv) as an arbitration sample for commercial transactions.

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 51P has been prepared from a sulphide-poor mineralised quartz monzonite porphyry sample. The robust foil laminate film used to package it is an effective barrier to oxygen and moisture and the sealed CRM is considered to have long-term stability under normal storage conditions.

## **INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL**

The recommended values for OREAS 51P refers to the concentration levels of gold and copper after removal of hygroscopic moisture by drying in air to constant mass at 105<sup>0</sup> C. In its packaged state a hygroscopic moisture content of 1.05% has been established. If the reference material is not dried by the user prior to analysis, the recommended values 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:** Dr Paul Hamlyn

## **REFERENCES**

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