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**CERTIFICATE OF ANALYSIS FOR  
COPPER-GOLD ORE REFERENCE MATERIAL  
OREAS 53Pb**

**SUMMARY STATISTICS**

<b>Constituent</b>	<b>Certified value</b>	<b>95% Confidence interval</b>		<b>Tolerance interval 1-<math>\alpha</math>=0.99, <math>\rho</math>=0.95</b>	
		<b>Low</b>	<b>High</b>	<b>Low</b>	<b>High</b>
Gold, Au (ppm)	0.623	0.612	0.634	0.611	0.635
Copper, Cu (wt.%)	0.546	0.540	0.553	0.540	0.552

*Prepared by:  
Ore Research & Exploration Pty Ltd  
October 2006*

## INTRODUCTION

OREAS certified reference materials (CRMs) 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 53Pb is one of four new porphyry copper-gold standards prepared from ore and waste rock samples from a porphyry copper-gold deposit, 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 porphyry 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) *jaw crushing to minus 3mm*
- b) *drying to constant mass at 105°C*
- c) *milling of the waste rock material to 98% minus 75 micron*
- d) *milling of the ore to 100% minus 20 micron*
- e) *blending in appropriate proportions to achieve the desired grade*
- f) *bagging into 25kg sublots*

## ANALYSIS OF OREAS 53Pb

The indicative major and trace element composition of OREAS 53Pb is given in Table 1. The constituents are the means of duplicate analyses by borate fusion XRF and 4 acid ICPOES/MS and are uncertified values.

Table 1. Indicative major and trace element composition of reference material OREAS 53Pb (SiO<sub>2</sub> to P<sub>2</sub>O<sub>5</sub> by borate fusion XRF; C & S by Leco; Co, Cu, Ni, Sc, Zn, Na by 4 acid ICP-OES; rest by 4 acid ICP-MS.

Constituent	wt. %	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO <sub>2</sub>	59.5	Ag	1.8	Hf	2.3	Sc	12
TiO <sub>2</sub>	0.47	As	7	Ho	0.58	Sm	3.3
Al <sub>2</sub> O <sub>3</sub>	15.3	Ba	714	In	0.38	Sn	7
Fe <sub>2</sub> O <sub>3</sub>	7.57	Be	1.8	La	16.9	Sr	433
MnO	0.06	Bi	2.3	Li	17.5	Ta	0.35
MgO	2.37	Cd	<1	Lu	0.28	Tb	0.46
CaO	2.52	Ce	31.0	Mo	3.8	Te	<1
SO <sub>3</sub>	1.5	Co	20	Nb	6.3	Th	5.3
K <sub>2</sub> O	4.12	Cs	1.6	Nd	14.1	U	2.0
P <sub>2</sub> O <sub>5</sub>	0.23	Dy	2.7	Ni	19.5	W	3
Na <sub>2</sub> O	4.26	Er	1.6	Pb	15	Y	14
LOI	2.31	Eu	0.88	Pr	3.6	Yb	1.7
Total	100.1	Ga	14.9	Rb	76	Zn	76
C	0.46	Gd	3.1	Sb	1.7	Zr	63
S	0.60						

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 the INAA lab, 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 nineteen 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 (20-50g charge with new pots) with flame AAS or ICPOES finish at fourteen laboratories (Table 2), while one laboratory determined gold in nineteen samples via instrumental neutron activation analysis (INAA) using 0.5gm analytical subsample weights (Table 3). For copper laboratories were requested to use a four acid (HF-HNO<sub>3</sub>-HClO<sub>4</sub>-HCl) digestion with ICPOES or AAS finish. Three laboratories departed from this scheme, one employing 3 acid and two an aqua regia digestion (Table 4). As there is no indication of the presence of acid insoluble copper all results were used.

Table 2. Analytical results for gold in standard OREAS 53Pb (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; PDM<sup>3</sup> is percent deviation of lab mean from corrected mean of means; outliers in bold; values in parts per million.

Replicate Number	Lab 1 FA*OES	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	0.611	0.661	0.650	0.638	0.610	0.598	0.610
2	0.603	0.670	0.640	0.636	0.610	0.594	0.591
3	0.613	0.645	0.640	0.640	0.610	0.598	0.614
4	0.614	0.653	<b>0.670</b>	0.640	0.610	0.594	0.614
5	0.607	0.657	0.650	0.634	0.610	0.595	0.597
6	0.598	0.666	0.640	0.640	0.620	0.592	0.590
Mean	0.608	0.659	0.648	0.638	0.612	0.595	0.603
Median	0.609	0.659	0.645	0.639	0.610	0.595	0.604
Std.Dev.	0.006	0.009	0.012	0.003	0.004	0.002	0.011
Rel.Std.Dev.	1.03%	1.37%	1.80%	0.40%	0.67%	0.40%	1.88%
PDM <sup>3</sup>	-2.52%	5.67%	4.01%	2.35%	-1.87%	-4.52%	-3.32%

Table 2. Continued.

Replicate Number	Lab 8 FA*OES	Lab 9 FA*AAS	Lab 10 FA*AAS	Lab 11 FA*AAS	Lab 12 FA*AAS	Lab 13 FA*AAS	Lab 14 FA*AAS
1	0.630	<b>0.623</b>	<b>0.635</b>	0.600	0.610	0.633	0.590
2	0.623	0.657	0.621	0.600	0.600	0.634	0.610
3	0.611	0.660	0.624	0.600	0.620	<b>0.611</b>	0.600
4	0.613	0.658	0.620	0.600	<b>0.580</b>	0.633	0.600
5	0.618	0.665	0.621	0.600	0.620	0.634	0.620
6	0.619	0.643	0.620	0.640	0.620	0.644	0.610
Mean	0.619	0.651	0.624	0.607	0.608	0.632	0.605
Median	0.619	0.658	0.621	0.600	0.615	0.634	0.605
Std.Dev.	0.007	0.016	0.006	0.016	0.016	0.011	0.010
Rel.Std.Dev.	1.11%	2.39%	0.93%	2.69%	2.63%	1.73%	1.73%
PDM <sup>3</sup>	-0.70%	4.44%	0.02%	-2.68%	-2.41%	1.31%	-2.94%

Table 3. Analytical results for gold in standard OREAS 53Pb via instrumental neutron activation analysis using a 0.5g analytical subsample weight (abbreviations as in Table 2; values in parts per million).

Replicate Number	Lab 15 INAA
1	0.651
2	<b>0.746</b>
3	0.702
4	0.645
5	0.682
6	0.665
7	0.634
8	0.647
9	0.595
10	0.651
11	0.615
12	0.612
13	0.589
14	0.649
15	0.633
16	0.637
17	0.625
18	0.572
19	0.612
20	0.638
Mean	0.640
Median	0.637
Std.Dev.	0.039
Rel.Std.Dev.	6.17%
PDM <sup>3</sup>	2.65%

Table 4. Analytical results for copper in standard OREAS 53Pb (4AD - four acid digestion; 3A – three acid digestion; AR – aqua regia digestion; OES - inductively coupled plasma optical emission spectrometry; AAS - atomic absorption spectrometry; other abbreviations as in Table 2; values in weight percent).

Replicate Number	Lab 1 4A*OES	Lab 2 4A*AAS	Lab 3 4A*OES	Lab 4 4A*AAS	Lab 5 4A*OES	Lab 6 4A*AAS	Lab 7 4A*OES
1	<b>5580</b>	5340	5219	5570	5346	5390	5470
2	5390	5330	5220	5560	5265	5430	5660
3	5460	<b>5400</b>	5176	5580	5312	5400	5580
4	5440	<b>5240</b>	5324	5570	5314	5450	5470
5	5410	5340	5336	5570	5357	5410	5610
6	5360	5330	5354	5580	5354	5420	5620
Mean	5440	5330	5272	5572	5325	5417	5568
Median	5425	5335	5272	5570	5330	5415	5595
Std.Dev.	77	51	75	8	35	22	80
Rel.Std.Dev.	1.42%	0.96%	1.43%	0.14%	0.66%	0.40%	1.44%
PDM <sup>3</sup>	-0.40%	-2.42%	-3.49%	2.01%	-2.52%	-0.83%	1.95%

Table 4. Continued.

Replicate Number	Lab 8 4A*OES	Lab 9 4A*AAS	Lab 10 4A*AAS	Lab 11 3A*AAS	Lab 12 AR*AAS	Lab 13 MAR*OES	Lab 14 4A*AAS
1	5410	5800	5400	5400	5600	5420	<b>5420</b>
2	5410	5800	5500	5500	5600	5430	5590
3	5430	5400	<b>5700</b>	5500	5700	5410	5570
4	5410	5300	5500	5600	5500	5390	5660
5	5430	5800	5400	5500	5500	5380	5600
6	5420	5800	5400	5500	5400	5350	5650
Mean	5418	5650	5483	5500	5550	5397	5582
Median	5415	5800	5450	5500	5550	5400	5595
Std.Dev.	10	235	117	63	105	29	87
Rel.Std.Dev.	0.18%	4.15%	2.13%	1.15%	1.89%	0.55%	1.55%
PDM <sup>3</sup>	-0.80%	3.44%	0.39%	0.69%	1.61%	-1.20%	2.19%

## STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 53Pb

### Certified 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 ( $p-1$ ).

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

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

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

The distributions 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 certified values.

Table 5. Certified values and 95% confidence intervals for OREAS 53Pb

Constituent	Certified value	95% Confidence interval	
		Low	High
Gold, Au (ppm)	0.623	0.612	0.634
Copper, Cu (wt.%)	0.546	0.540	0.553

### 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 0.540 and 0.552 wt.%. 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 20-50gm 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 INAA data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 53Pb.

Table 6. Certified values and tolerance limits for OREAS 53Pb.

Constituent	Certified value	Tolerance interval $1-\alpha=0.99, \rho=0.95$	
		Low	High
Gold, Au (ppm)	0.623	0.611	0.635
Copper, Cu (wt.%)	0.546	0.540	0.552

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  $1 - s_j / 2s_g' < 0$ ).

### Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this standard in a QA/QC program. They take into account 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.

Table 7. Performance gates for OREAS 53Pb

Constituent	Certified value	Performance Gates					
		1 $\sigma$		2 $\sigma$		3 $\sigma$	
		Low	High	Low	High	Low	High
Gold, Au (ppm)	0.623	0.602	0.644	0.581	0.666	0.559	0.687
Copper, Cu (wt.%)	0.546	0.533	0.560	0.519	0.573	0.506	0.587

The performance gates are calculated from the standard deviation of the pooled individual analyses generated from the certification program. All individual and lab dataset (batch) outliers are removed prior to determination of the standard deviation. These outliers can only be removed if they can be confidently deemed to be analytical rather than arising from inhomogeneity of the standard.

Performance gates have been calculated for one, two and three standard deviations of the accepted pool of certification data and are presented in Table 7. The gold INAA data was not included in these calculations. As a guide these intervals may be regarded as informational (1 $\sigma$ ), warning or rejection for multiple outliers (2 $\sigma$ ), or rejection for individual outliers (3 $\sigma$ ) in QC monitoring although their precise application should be at the discretion of the QC manager concerned.

## PARTICIPATING LABORATORIES

Acme Analytical Laboratories, Vancouver, BC, Canada  
 Actlabs Analytical Laboratories, Ancaster, Ontario, Canada  
 ALS Chemex, Garbutt, Qld, Australia  
 ALS Chemex, La Serena, Chile, Sth America  
 ALS Chemex, Val-D'or, Quebec, Canada  
 ALS Chemex, Vancouver, BC, Canada  
 Amdel Laboratories, Thebarton, SA, Australia  
 ANSTO Laboratories, Lucas Heights, NSW, Australia  
 Genalysis Laboratory Services, Maddington, WA, Australia  
 Intertek Testing Services, Jakarta, Indonesia  
 McPhar Geoservices (Phil.) Inc., Makati, Philippines  
 OMAC Laboratories Ltd, Loughrea, Co Galway, Ireland  
 SGS, Kalimantan Timur, Indonesia  
 SGS, Garbutt, QLD, Australia  
 Ultra Trace, Canning Vale, WA, Australia

## PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The copper-gold ore reference material, OREAS 53Pb has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd  
6 – 8 Gatwick Road  
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It is available in unit sizes of 60g laminated foil packets and 1kg jars.

## INTENDED USE

OREAS 53Pb 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 53Pb 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 certified values for OREAS 53Pb refer 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 an indicative hygroscopic moisture content of 0.61% has been established. If the reference material is not dried by the user prior to analysis, the moisture content should be verified and the certified values 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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