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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD STANDARD

OREAS 52P

SUMMARY STATISTICS

Recommended Values, 95% Confidence and Tolerance Intervals

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppb)	183	176	190	179	187
Copper, Cu (%)	0.387	0.382	0.392	0.377	0.397

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 52P 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 Synclinorial 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 52P

The indicative major and trace element composition of OREAS 52P 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 52P; SiO_2 to Total in weight percent (Total includes traces); rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO_2	54.44	Ba	762
TiO_2	0.61	Co	16
Al_2O_3	19.34	Cr	26
Fe_2O_3	5.33	Ga	22
MnO	0.09	Nb	3
MgO	2.43	Nd	17
CaO	3.43	Ni	32
Na_2O	5.20	Rb	82
K_2O	4.50	Sc	19
P_2O_5	0.45	Sr	837
SO_3	0.45	Th	17
LOI	2.84	V	166
Total	99.68	Y	20
		Zn	92
		Zr	85

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^3) are presented in Tables 2 to 4. The parameter PDM^3 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-five samples via instrumental neutron activation analysis (INAA) using 0.5gm analytical subsample weights (Table 3). Copper was determined via four acid (HF-HNO_3-$HClO_4$-HCl) digest with ICPOES or AAS finish (Table 4).

Table 2. Analytical results for gold in standard OREAS 52P (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^3 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	180	185	170	170	180	157	173
2	190	180	165	170	181	160	177
3	190	200	171	170	177	165	174
4	180	210	166	170	189	168	176
5	190	190	168	170	190	165	176
6	200	180	171	170	183	170	174
Mean	188	191	169	170	183	164	175
Std. Dev.	8	12	3	0	5	5	2
Rel.Std.Dev.	4.00%	6.29%	1.54%	0.00%	2.82%	2.97%	0.89%
PDM^3	2.75%	4.12%	-8.07%	-7.25%	0.02%	-10.4%	-4.52%

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	202	162	210	187	187	184	
2	199	166	200	196	181	182	
3	194	175	200	199	172	188	Refer to
4	199	176	200	195	176	192	table 3
5	199	175	190	200	188	189	
6	196	170	210	195	170	188	
Mean	198	171	202	195	179	187	194
Std. Dev.	3	6	8	5	8	4	13
Rel.Std.Dev.	1.41%	3.35%	3.73%	2.35%	4.24%	1.92%	6.67%
PDM^3	8.12%	-6.89%	10.03%	6.57%	-2.34%	2.11%	5.79%

Table 3. Analytical results for gold in standard OREAS 52P 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	212
2	194
3	175
4	192
5	191
6	198
7	166
8	191
9	204
10	193
11	195
12	205
13	189
14	205
15	196
16	189
17	200
18	199
19	210
20	188
21	177
22	162
23	207
24	207
25	203
Mean	194
Std. Dev.	13
Rel.Std.Dev.	6.67%

Table 4. Analytical results for copper in standard OREAS 52P (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	3690	3650	3990	3920	3870	3800	3870
2	3780	3500	4160	3960	3860	3825	3870
3	3740	3270	4050	4000	3870	3850	3810
4	3780	3330	3790	3960	3940	3900	3910
5	3800	3530	3980	4070	3800	3850	3820
6	3840	3490	3940	3970	3910	3800	3920
Mean	3772	**3462**	3985	3980	3875	3838	3867
Std. Dev.	52	139	122	51	48	38	45
Rel.Std.Dev.	1.37%	4.01%	3.07%	1.28%	1.23%	0.99%	1.16%
PDM[3]	-2.61%	-10.61%	2.90%	2.77%	0.06%	-0.91%	-0.16%

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	4000	3700	3918	3960	3780	3730	3840
2	4200	3900	3913	3880	3800	3670	3850
3	4100	3900	3889	3920	3830	3740	3910
4	4000	3800	3827	3900	3800	3770	3850
5	4100	3700	3831	3890	3780	3780	3900
6	3800	3700	3847	3900	3790	3780	3990
Mean	4033	3783	3871	3908	3797	3745	3890
Std. Dev.	137	98	41	29	19	42	57
Rel.Std.Dev.	3.39%	2.60%	1.06%	0.73%	0.49%	1.13%	1.46%
PDM[3]	4.15%	-2.31%	-0.05%	0.92%	-1.96%	-3.30%	0.45%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 52P

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

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

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

where

x_{ij} *is the jth 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;*

$\ddot{x}$ *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).

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

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

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

$$S = 1.483 \underset{j=1.....n}{\text{median}} / x_j - \underset{i=1.....n}{\text{median}} (x_i) /$$

$$z_i = \frac{x_i - T}{S}$$

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 52P.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	183	176	190
Copper, Cu (wt. %)	0.387	0.382	0.392

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 jth raw result reported by laboratory i*;

x'_{ij} *is the jth 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

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

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 0.377% and 0.397%. 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 (IS0 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

$$s''_g = \frac{\sum_{i=1}^{p}\left(s_i\left(1 - \frac{s_i}{s'_g}\right)\right)}{\sum_{i=1}^{p}\left(1 - \frac{s_i}{s'_g}\right)}$$

where

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

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

according to the formula

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

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

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 52P.

Table 6. Recommended values and tolerance limits for OREAS 52P.

Constituent	Recommended value	Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High
Gold, Au (ppb)	183	179	187
Copper, Cu (wt. %)	0.387	0.377	0.397

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 $s_l / 2s_g' > 1$ (i.e. where the weighting factor $1 - s_l / 2s_g' < 0$).

Performance Gates

Performance gates provide an indication of a level of performance that might reasonably be expected from a routine laboratory being monitored by this standard in a QA/QC program. They incorporate errors attributable to bias, precision and inhomogeneity and are simply calculated from the standard deviation of the pooled individual analyses (fire assay data only) 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 after the absolute homogeneity of the CRM has been independently established, i.e. the outliers must be confidently deemed to be analytical rather than arising from inhomogeneity of the CRM.

Table 7. Proposed performance gates for OREAS 52P

Constituent	Recommended value	Performance Gates					
		1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppb)	183	170	196	157	209	144	222
Copper, Cu (wt. %)	0.387	0.377	0.398	0.366	0.409	0.355	0.419

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. As a guide these intervals may be regarded as informational (1σ), warning or rejection for multiple outliers (2σ), or rejection for individual outliers (3σ) 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 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 52P has been prepared and certified and

is supplied by:

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It is available in unit sizes of 60g laminated foil packets.

INTENDED USE

OREAS 52P 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 52P 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 52P refers to the concentration levels of gold and copper after removal of hygroscopic moisture by drying in air to constant mass at 105^0 C. In its packaged state a hygroscopic moisture content of 1.10% 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

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