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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD STANDARD

OREAS 53P

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)	380	371	389	376	384
Copper, Cu (%)	0.413	0.404	0.422	0.406	0.420

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 53P 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 53P

The indicative major and trace element composition of OREAS 53P 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 53P; SiO_2 to Total in weight percent (Total includes traces); rest in parts per million.

Constituent	Concentration	Constituent	Concentration
SiO_2	58.03	Ba	843
TiO_2	0.51	Ce	27
Al_2O_3	16.54	Co	14
Fe_2O_3	5.45	Cr	36
MnO	0.13	Ga	20
MgO	2.59	La	17
CaO	3.50	Nb	2
Na_2O	4.72	Ni	27
K_2O	4.36	Rb	60
P_2O_5	0.26	Sc	18
SO_3	0.83	Sr	754
LOI	2.21	Th	13
Total	99.71	V	178
		Y	17
		Zn	63
		Zr	79

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 53P (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	390	430	364	370	403	345	363
2	380	380	360	370	381	340	366
3	380	370	360	370	387	338	361
4	390	360	382	370	387	355	359
5	390	400	368	380	383	343	357
6	390	390	382	380	382	357	366
Mean	387	388	369	373	387	346	362
Std. Dev.	5	25	10	5	8	8	4
Rel.Std.Dev.	1.34%	6.39%	2.79%	1.38%	2.11%	2.28%	1.02%
PDM[3]	1.76%	2.20%	-2.83%	-1.75%	1.89%	-8.86%	-4.73%

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	397	362	420	394	360	391	
2	396	367	350	396	372	394	
3	397	354	380	398	362	392	Refer to
4	392	366	410	398	368	388	Table 3
5	399	371	400	407	367	392	
6	410	359	420	392	383	394	
Mean	399	363	397	398	369	392	390
Std. Dev.	6	6	27	5	8	2	14
Rel.Std.Dev.	1.53%	1.68%	6.89%	1.31%	2.23%	0.57%	3.48%
PDM[3]	4.87%	-4.43%	4.39%	4.61%	-2.98%	3.12%	2.75%

Table 3. Analytical results for gold in standard OREAS 53P 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	390
2	386
3	373
4	411
5	406
6	401
7	381
8	401
9	376
10	401
11	401
12	411
13	407
14	380
15	391
16	363
17	402
18	378
19	391
20	376
21	377
22	403
23	382
24	376
25	399
Mean	390
Std. Dev.	14
Rel.Std.Dev.	3.48%

Table 4. Analytical results for copper in standard OREAS 53P (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	4050	3730	4280	4300	4220	4075	4150
2	4130	4110	4190	4330	4230	4125	**4370**
3	4010	3950	4200	4380	4220	4100	4130
4	4070	3710	4270	4340	4240	4100	4120
5	3870	3860	4350	4360	4150	4125	4100
6	4060	3880	4300	4390	4160	4100	4140
Mean	4032	3873	4265	4350	4203	4104	4168
Std. Dev.	88	148	61	33	38	19	100
Rel.Std.Dev.	2.19%	3.82%	1.43%	0.77%	0.91%	0.46%	2.41%
PDM[3]	-2.45%	-6.28%	3.20%	5.25%	1.71%	-0.69%	0.86%

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	4300	4200	4151	4120	4200	4020	4000
2	4300	4000	4159	4030	4150	4030	3920
3	4600	4100	4156	4200	4120	4000	3970
4	4300	4000	4252	4210	4130	4040	4030
5	4500	4100	4224	4050	4110	4050	3810
6	4500	4200	4159	4060	4170	4050	3940
Mean	4417	4100	4184	4112	4147	4032	3945
Std. Dev.	133	89	43	78	34	19	77
Rel.Std.Dev.	3.01%	2.18%	1.03%	1.91%	0.82%	0.48%	1.96%
PDM[3]	6.87%	-0.79%	1.23%	-0.51%	0.34%	-2.45%	-4.54%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 53P

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 53P.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	380	371	389
Copper, Cu (wt. %)	0.413	0.404	0.422

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 0.406% and 0.420%. 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 53P.

Table 6. Recommended values and tolerance limits for OREAS 53P.

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99$, $\rho=0.95$	
		Low	High
Gold, Au (ppb)	380	376	384
Copper, Cu (wt. %)	0.413	0.406	0.420

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 53P

Constituent	Recommended value	Performance Gates					
		1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppb)	380	361	399	341	419	322	438
Copper, Cu (ppm)	0.413	0.398	0.429	0.383	0.444	0.367	0.459

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 53P has been prepared and certified and is supplied by:

Ore Research & Exploration Pty Ltd
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It is available in unit sizes of 60g laminated foil packets.

INTENDED USE

OREAS 53P 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 53P 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 53P 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 0.83% 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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