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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD ORE REFERENCE MATERIAL

OREAS 52Pb

SUMMARY STATISTICS

Constituent	Recommended value	95% Confidence interval		Tolerance interval 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Gold, Au (ppb)	307	299	315	301	313
Copper, Cu (ppm)	3338	3301	3375	3286	3390

Prepared by:
Ore Research & Exploration Pty Ltd
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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 50Pb 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 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) jaw crushing to minus 3mm
b) drying to constant mass at 105^{0}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 52Pb

The indicative major and trace element composition of OREAS 52Pb is given in Table 1. The constituents are the means of duplicate analyses by borate fusion XRF, four acid ICP-OES/MS, Leco and thermo-gravimetry and are uncertified values.

Table 1. Indicative major and trace element composition of reference material OREAS 52Pb; SiO_2 to S in weight percent ; rest in parts per million.

Constituent	wt.%	Constituent	ppm	Constituent	ppm	Constituent	ppm
SiO_2	68.7	Ag	1.3	Gd	5.1	Sb	1.4
TiO_2	0.26	As	2.5	Hf	2.5	Sc	7
Al_2O_3	13.6	Ba	791	Ho	0.60	Sm	6.3
Fe_2O_3	5.05	Be	2.1	In	0.36	Sn	9
MnO	0.05	Bi	1.8	La	28.8	Sr	157
MgO	0.91	Cd	<1	Li	44	Ta	0.85
CaO	1.85	Ce	59	Lu	0.21	Tb	0.75
SO3	0.89	Co	10	Mo	2	Te	<1
K_2O	4.62	Cs	9	Nb	10	Th	11.7
P_2O_5	0.11	Cu	3338	Nd	26.5	U	2.5
Na_2O	3.42	Dy	3.5	Ni	17	W	2.5
LOI	0.57	Er	1.3	Pb	28	Y	14
Total	100.0	Eu	0.85	Pr	7.3	Yb	1.3
C	0.17	Ga	19.3	Rb	173	Zn	71
S	0.33					Zr	49

Seventeen 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 17. 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 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 twenty 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 graphite furnace AAS, solvent extraction AAS, flame AAS or ICPOES finish at sixteen laboratories (Table 2), while a seventeenth determined gold in twenty samples via instrumental neutron activation analysis (INAA) using 0.5gm analytical subsample weights (Table 3). Copper was determined mostly by four acid (HF-HNO_3-$HClO_4$-HCl) digest with ICPOES or AAS finish at fifteen labs (Table 4).

Table 2. Analytical results for gold in standard OREAS 52Pb (FA*AAS - fire assay / atomic absorption spectrometry; FA*OES - fire assay / inductively coupled plasma optical emission spectrometry; SX – solvent extraction; GF – graphite furnace; 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*OES	Lab 2 FA*OES	Lab 3 FASX*AAS	Lab 4 FA*AAS	Lab 5 FAGF*AAS	Lab 6 FA*OES	Lab 7 FA*AAS	Lab 8 FA*AAS
1	**308**	300	298	320	311	297	304	313
2	315	301	300	327	305	272	308	321
3	319	311	288	322	307	306	306	315
4	320	314	300	323	301	302	305	317
5	322	313	281	319	307	321	**310**	317
6	320	308	286	322	302	317	305	311
Mean	317	308	292	322	306	303	306	316
Median	320	310	293	322	306	304	306	316
Std.Dev.	5	6	8	3	4	17	2	4
Rel.Std.Dev.	1.62%	1.96%	2.81%	0.87%	1.20%	5.77%	0.73%	1.11%
PDM[3]	3.35%	0.26%	-4.85%	4.92%	-0.50%	-1.48%	-0.23%	2.81%

Table 2. Continued.

Replicate Number	Lab 9 FASX*AAS	Lab 10 FA*OES	Lab 11 FA*OES	Lab 12 FA*AAS	Lab 13 FASX*AAS	Lab 14 FAGF*AAS	Lab 15 FAGF*AAS	Lab 16 FA*AAS
1	306	263	310	**310**	294	323	283	310
2	308	263	309	319	297	**305**	283	327
3	296	267	305	322	296	327	282	324
4	302	286	310	315	301	323	284	315
5	301	276	314	320	**315**	325	285	335
6	310	274	307	320	296	**308**	284	321
Mean	304	272	309	318	300	319	284	322
Median	304	271	310	320	297	323	284	323
Std.Dev.	5	9	3	4	8	9	1	9
Rel.Std.Dev.	1.70%	3.30%	0.99%	1.39%	2.60%	2.97%	0.37%	2.75%
PDM[3]	-1.05%	-11.6%	0.69%	3.46%	-2.35%	3.73%	-7.67%	4.87%

Table 3. Analytical results for gold in standard OREAS 52Pb 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 17 INAA
1	291
2	354
3	289
4	338
5	308
6	336
7	311
8	311
9	325
10	290
11	308
12	328
13	310
14	337
15	283
16	323
17	322
18	323
19	360
20	328
Mean	319
Median	322
Std.Dev.	21
Rel.Std.Dev.	6.59%
PDM[3]	3.77%

Table 4. Analytical results for copper in standard OREAS 52Pb (4AD*OES - four acid digest / inductively coupled plasma optical emission spectrometry; 4AD*AAS - four acid digest / atomic absorption spectrometry; AR – aqua regia digestion; other abbreviations as in Table 2; values in parts per million).

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*OES	Lab 7 4A*AAS
1	3280	3290	3292	**3370**	2790	**3363**	3374
2	3400	3270	**3171**	3310	2980	3322	3383
3	3430	3380	3256	3270	2850	3305	3370
4	3350	3330	3231	3270	3050	3326	3380
5	3490	3430	3262	3310	2900	**3274**	3385
6	3350	3280	3261	3250	2790	3309	**3408**
Mean	3383	3330	3246	3297	**2893**	3317	3383
Median	3375	3310	3259	3290	2875	3316	3381
Std.Dev.	73	64	41	43	105	29	13
Rel.Std.Dev.	2.16%	1.91%	1.27%	1.31%	3.64%	0.88%	0.39%
PDM[3]	1.37%	-0.23%	-2.76%	-1.23%	-13.3%	-0.64%	1.36%

Table 4. Continued.

Replicate Number	Lab 8 4A*OES	Lab 9 4A*OES	Lab 10 4A*OES	Lab 11 4A*AAS	Lab 12 4A*AAS	Lab 13 AR*AAS	Lab 14 AR*OES	Lab 15 4A*AAS
1	3320	3377	3350	3200	3400	3160	3549	**3300**
2	3420	3372	3310	3260	3400	3260	3557	3400
3	3380	3316	3310	3270	3400	3270	3525	3400
4	3390	3322	3340	3230	3300	**3510**	3414	3370
5	3350	3345	3380	3240	3400	3320	3352	3400
6	3410	3360	3310	3190	3400	3250	3356	**3470**
Mean	3378	3349	3333	3232	3383	3295	3459	3390
Median	3385	3353	3325	3235	3400	3265	3470	3400
Std.Dev.	38	26	29	32	41	117	96	55
Rel.Std.Dev.	1.11%	0.76%	0.86%	0.99%	1.21%	3.56%	2.78%	1.63%
PDM[3]	1.22%	0.33%	-0.13%	-3.18%	1.37%	-1.28%	3.63%	1.57%

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 52Pb

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).

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

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

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

The 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:

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

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

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 52Pb.

Constituent	Recommended value	95% Confidence interval	
		Low	High
Gold, Au (ppb)	307	299	315
Copper, Cu (ppm)	3338	3301	3375

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* exp*ected within the tolerance* lim*its*;

k'_2 *is the factor for two – sided tolerance* lim*its* (m, α *unknown*);

s''_g *is the corrected grand s* tan*dard 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 3286 and 3390 ppm 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

$1 - \left(\frac{s_i}{2s'_g} \right)$ *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:

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

where $\bar{x}'_i$ *is the transformed mean for 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 INAA data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 52Pb.

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

Constituent	Recommended value	Tolerance interval $1-\alpha=0.99$, $\rho=0.95$	
		Low	High
Gold, Au (ppb)	307	301	313
Copper, Cu (ppm)	3338	3286	3390

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 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 52Pb

Constituent	Recommended value	Performance Gates					
		1σ		2σ		3σ	
		Low	High	Low	High	Low	High
Gold, Au (ppb)	307	290	324	272	342	255	359
Copper, Cu (ppm)	3338	3261	3414	3185	3490	3109	3567

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. Data from ANSTO was not included in these calculations. 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 Analytical Laboratories, Ancaster, Ontario, Canada
ALS Chemex, Garbutt, Qld, Australia
ALS Chemex, La Serena, Chile, Sth America
ALS Chemex, Reno, Nevada, USA
ALS Chemex, Val-D'or, Quebec, Canada
ALS Chemex, Vancouver, BC, Canada
Amdel Laboratories, Thebarton, SA, Australia
Amdel Laboratories, Wangara, WA, 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, Garbutt, QLD, Australia
SGS, Welshpool, Perth, WA, Australia
Ultra Trace, Canning Vale, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The copper-gold ore reference material, OREAS 52Pb has been prepared and certified and is supplied by:

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It is available in unit sizes of 60g laminated foil packets and 1kg jars.

INTENDED USE

OREAS 52Pb 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 52Pb 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 52Pb refer 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 an indicative hygroscopic moisture content of 0.33% 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

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