O R E   R E S E A R C H   &   E X P L O R A T I O N   P T Y   L T D

6-8 Gatwick Drive, Bayswater North, Vic 3153 AUSTRALIA
Telephone: 61-3-9729 0333 Facsimile: 61-3-9729 4777

CERTIFICATE OF ANALYSIS FOR

COPPER CONCENTRATE CRM,
OREAS 99

Summary Statistics

Constituent	Recommended Value	95% Confidence Interval		Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High	Low	High
Copper, Cu (wt %)	25.89	25.82	25.96	25.85	25.93
Silver, Ag, (ppm)	67.3	66.1	68.5	65.3	69.3

Prepared by:
Ore Research & Exploration Pty Ltd
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SOURCE MATERIAL

Reference material OREAS 99 is a copper concentrate obtained from the CSA mine in Cobar, New South Wales. This reference material has been characterised for Cu and Ag.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

a) *drying to constant mass at 65^0 C;*
b) *screening;*
c) *preliminary homogenisation;*
d) *milling to 100% minus 40 microns;*
e) *final homogenisation;*
f) *packaging into 10g lots sealed under nitrogen in laminated foil pouches.*

ANALYSIS OF OREAS 99

Nine umpire laboratories participated in the analytical program to characterise Cu and Ag. 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 the Appendix, Tables A2 and A3. 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 explained, together with other abbreviations used, in Table A1 (Appendix).

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 99

Recommended Value and Confidence Limits

The recommended 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;*
>
> $\overline{x}_i$ *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).

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

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

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.*

In certain instances statistician's prerogative has been employed in discriminating outliers. Individual outliers and, more rarely, laboratory means deemed to be outlying are shown in bold in the tables and have been omitted in the determination of recommended values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It is a measure of the reliability of the recommended value, i.e. the narrower the confidence interval the greater the certainty in the recommended value.

Table 1. Recommended values and 95% confidence intervals for OREAS 99

Constituent	Recommended value	95% Confidence interval	
		Low	High
Copper, Cu (wt %)	25.89	25.82	25.96
Silver, Ag, (ppm)	67.3	66.1	68.5

Intervals may appear asymmetric due to rounding

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 elements other than gold 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

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

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, α 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 25.85 and 25.93 %. 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

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

$$s_g' \text{ is the grand standard deviation computed from the transformed (i.e. means -}$$

$$\text{adjusted) results}$$

according to the formula:

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

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

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.

Table 2. Recommended values and tolerance limits for OREAS 99

Constituent	Recommended value	Tolerance limits 1-α=0.99, ρ=0.95	
		Low	High
Copper, Cu (wt %)	25.89	25.85	25.93
Silver, Ag, (ppm)	67.3	65.3	69.3

Intervals may appear asymmetric due to rounding

Any outliers were removed prior to the calculation of s_g' and a weighting factor of zero was applied to those data sets where $s_i/2s_g' > 1$ (i.e. where the weighting factor 1- $s_i/2s_g' < 0$).

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. Two methods have been employed to calculate performance gates.

The first method uses 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 CRM. Performance gates have been calculated for one, two and three standard deviations of the accepted pool of certification data and are presented in Table 3. As a guide these intervals may be regarded as informational (1SD), warning or rejection for multiple outliers (2SD), or rejection for individual outliers (3SD) in QC monitoring although their precise application should be at the discretion of the QC manager concerned.

For the second method a ±5% error bar on the recommended value is used as the window of acceptability (refer Table 3). Both methods should be used with caution when concentration levels approach lower limits of detection of the analytical methods employed, as performance gates calculated from standard deviations tend to be excessively wide whereas those determined by the 5% method are too narrow.

Table 3. Performance gates for OREAS 99

Constituent	Recommended Value	1 SD	2 SD		3 SD	
			Low	High	Low	High
Copper, Cu (wt %)	25.89	± 0.10	25.68	26.10	25.58	26.20
Silver, Ag (ppm)	67.3	± 1.7	63.9	70.7	62.2	72.4

Note - intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

AH Knight, St Helens, Merseyside, United Kingdom

Alex Stewart Assayers Ltd, Knowsley, Merseyside, United Kingdom

ALS Chemex, Vancouver, British Columbia, Canada

Bachelet Laboratories, Angleur, Belgium

BSI Inspectorate Ltd, Witham, Essex, United Kingdom

Laboratory Services International, BV, Rotterdam, Belgium

SGS Analabs, Townsville, Queensland, Australia

Standard & Reference Laboratories, Malaga, Western Australia, Australia

Walker & Whyte, Long Island City, New York, USA

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The reference material OREAS 99 has been prepared and certified by:

Ore Research & Exploration Pty Ltd
6-8 Gatwick Road
Bayswater North VIC 3153
AUSTRALIA

Telephone	*(03) 9729 0333*	*International*	*+613-9729 0333*
Facsimile	*(03) 9729 4777*	*International*	*+613-97617878*
Email	*info@ore.com.au*	*Web*	*www.ore.com.au*

OREAS 99 has been packaged under nitrogen in laminated foil pouches in 10g units.

INTENDED USE

OREAS 99 is a reference material intended for the following:

i) for the monitoring of laboratory performance in the analysis of copper and silver in concentrates by prescribed methods;
ii) for the calibration of instruments used in the determination of the concentration of copper and silver;
iii) for the verification of analytical methods for copper and silver.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 99 has been prepared from a copper concentrate of copper sulphide ore. To prolong its shelf life it has been packaged under nitrogen in robust foil laminate pouches. In its unopened state it is considered to have long-term stability under normal storage conditions. Once opened it should be stored in a nitrogen flushed desiccator to inhibit oxidation.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The recommended values for OREAS 99 refer to the concentration levels of copper and silver after removal of hygroscopic moisture by drying in air to constant mass at the reduced temperature of 65^0 C. If the reference material is not dried prior to analysis, the recommended value 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:

Craig Hamlyn (B.Sc. Hons - Geology)

REFERENCES

ISO Guide 35 (1985), Certification of reference materials - General and statistical principals.

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

APPENDIX

Analytical Results for OREAS 99

Table A1. Key to abbreviations used in Tables A2 andA3.

Abbreviation	Explanation
Std.Dev.	one sigma standard deviation
Rel.Std.Dev.	one sigma relative standard deviation
PDM[3]	percent deviation of lab mean from corrected mean of means
GRAV	gravimetric determination
4A	four acid (HF-HNO_3-$HClO_4$-HCl) digestion
3A	three acid (HNO_3-$HClO_4$-HCl) digestion
AD	acid digest (unspecified)
SIT	short iodide titration
EG	electrogravimetry
AAS	atomic absorption spectrometry
OES	inductively coupled plasma optical emission spectrometry

Table A2. Analytical results for Copper in OREAS 99 (abbreviations as in Table A1; values in wt %)

Replicate No.	Lab A SIT	Lab B EG	Lab C SIT	Lab D SIT	Lab E EG	Lab F SIT	Lab G SIT	Lab H SIT	Lab I SIT
1	25.84	25.85	25.82	25.92	26.03	26.00	25.86	25.94	25.75
2	25.86	25.80			25.99	26.03		25.93	25.76
3	25.87	25.83			26.08	26.00			25.76
4					26.03	25.98			
5						26.07			
6						26.06			
Mean	25.86	25.83	25.82	25.92	26.03	26.02	25.86	25.94	25.76
Median	25.86	25.83	25.82	25.92	26.03	26.02	25.86	25.94	25.76
Std.Dev.	0.02	0.03			0.04	0.04		0.01	0.01
Rel.Std.Dev.	0.06%	0.10%			0.14%	0.14%		0.03%	0.02%
PDM[3]	-0.14%	-0.25%	-0.28%	0.11%	0.54%	0.51%	-0.12%	0.16%	-0.52%

Table A3. Analytical results for Silver in OREAS 99 (abbreviations as in Table A1; values in ppm)

Replicate No.	Lab A 3A*AAS	Lab B AD*OES/AAS	Lab C 4A*OES/AAS	Lab D AD*AAS	Lab E 4A*OES	Lab F 4A*AAS	Lab G 3A*AAS	Lab H FA*GRAV	Lab I 4D*AAS
1	68.2	66.0	66.0	66.40	70.0	69.0	67.0	67.0	86.0
2	68.3	67.0	66.0		70.0	72.0		66.0	86.0
3	68.1	66.0	66.0		68.0	70.0			79.0
4		66.0			66.0	70.0			
5		65.0			69.0				
6		65.0			69.0				
Mean	68.2	65.8	66.0	66.40	68.7	70.3	67.0	66.5	83.7
Median	68.2	66.0	66.0	66.40	69.0	70.0	67.0	66.5	86.0
Std.Dev.	0.100	0.753	0.000		1.51	1.26		0.71	4.04
Rel.Std.Dev.	0.15%	1.14%	0.00%		2.19%	1.79%		1.06%	4.83%
PDM[3]	1.94%	-1.59%	-1.35%	-1.34%	2.64%	5.01%	0.15%	-0.60%	25.1%