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CERTIFICATE OF ANALYSIS FOR

POLYMETALLIC SULPHIDE ORE
REFERENCE MATERIAL OREAS 33

Summary Statistics

Constituent	Recommended	95% Confidence Interval		No. of
	value	Low	High	Labs
Arsenic, As (ppm)	1528	1478	1578	18
Cadmium, Cd (ppm)	94	91	97	20
Copper, Cu (ppm)	3464	3397	3531	22
Gold (fire assay), Au (ppm)	0.521	0.510	0.531	27
Iron, Fe (%)	16.54	16.13	16.95	22
Lead, Pb (%)	7.15	7.06	7.24	18
Manganese, Mn (ppm)	348	326	370	19
Molybdenum, Mo (ppm)	~3	~2	~4	11
Silver, Ag (ppm)	73.5	72.0	75.0	17
Zinc, Zn (%)	4.06	3.98	4.14	24

Prepared by:
Ore Research & Exploration Pty Ltd
July 2003

SOURCE MATERIAL

Base metal sulphide reference material OREAS 33 was prepared from a blend of Pb-Zn-Ag sulphide ore from Pasminco Limited's Elura Mine, central New South Wales, Australia and copper and gold-bearing material from Mt Lyell, Queenstown, Tasmania, Australia and Blackwood, central Victoria, Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

a) drying for 24 hours at 65^0 C;
b) crushing;
c) milling to minus 40 microns;
d) homogenisation;
e) packaging into 50g lots sealed under N_2 in laminated foil pouches.

ANALYSIS OF OREAS 33

Thirty commercial laboratories participated in the analytical program. 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 a separate Excel spreadsheet. 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 indicated in the table captions or at the head of each laboratory data set.

Each laboratory received a set of five 110g samples chosen at spaced intervals from a pool of 50 samples taken at regular intervals during packaging of the standard. Each set of samples is therefore considered reasonably representative of the entire batch.

Ag, As, Cd, Cu, Fe, Pb, Mn, Mo and Zn were determined mainly by total acid digest or, where this method was unavailable, aqua regia digest with ICPOES or AAS as the reading method. The aqua regia results have been treated separately from the total digest results throughout. Gold was determined in replicate assays using a fire assay technique (40-50g charge with new pots) together with flame furnace AAS or ICPOES finish at 27 laboratories and by aqua regia digest with similar finish at 17 laboratories. The homogeneity of gold, which may be considered a yardstick for the overall standard homogeneity, was determined by instrumental neutron activation analysis (INAA) on a reduced analytical subsample weight of 0.5g. The twenty INAA subsamples were taken at regular intervals during packaging and are considered representative of the entire batch.

STATISTICAL EVALUATION OF ANALYTICAL DATA FOR OREAS 33

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 and have been omitted in the determination of recommended values.

Table 1. Recommended values and 95% confidence intervals for standard OREAS 33.

Constituent	Recommended value	95% Confidence Interval		No. of Labs
		Low	High	
Arsenic, As (ppm)	1528	1478	1578	18
Cadmium, Cd (ppm)	94	91	97	20
Copper, Cu (ppm)	3464	3397	3531	22
Gold, Au (ppm)	0.521	0.510	0.531	27
Iron, Fe (%)	16.54	16.13	16.95	22
Lead, Pb (%)	7.15	7.06	7.24	18
Manganese, Mn (ppm)	348	326	370	19
Molybdenum, Mo (ppm)	~3	~2	~4	11
Silver, Ag (ppm)	73.5	72.0	75.0	17
Zinc, Zn (%)	4.06	3.98	4.14	24

Table 2. Indicative aqua regia digest values and 95% confidence intervals for standard OREAS 33.

Constituent	Recommended value	95% Confidence Interval		No. of Labs
		Low	High	
Arsenic, As (ppm)	1432	1167	1697	3
Cadmium, Cd (ppm)	95	89	101	5
Copper, Cu (ppm)	3332	3124	3540	6
Gold (aqua regia), Au (ppm)	0.473	0.449	0.497	17
Iron, Fe (%)	~16.63	*IND	*IND	2
Lead, Pb (%)	6.89	6.41	7.37	3
Manganese, Mn (ppm)	354	270	438	5
Molybdenum, Mo (ppm)	~2	*IND	*IND	4
Silver, Ag (ppm)	73.8	71.4	76.2	9
Zinc, Zn (%)	3.94	3.56	4.32	4

*IND - indeterminate

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* 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.

Table 3. Recommended values and tolerance limits for standard OREAS 33.

Constituent	Recommended value	Tolerance limits 1-α = 0.99, ρ = 0.95		No. of Labs
		Low	High	
Arsenic, As (ppm)	1528	1505	1551	23
Cadmium, Cd (ppm)	94	93	95	25
Copper, Cu (ppm)	3464	3428	3500	29
Gold, Au (ppm)	0.521	0.516	0.525	1
Iron, Fe (%)	16.54	16.31	16.76	26
Lead, Pb (%)	7.15	7.07	7.23	25
Manganese, Mn (ppm)	348	344	352	26
Molybdenum, Mo (ppm)	~3	*IND	*IND	-
Silver, Ag (ppm)	73.5	72.5	74.5	27
Zinc, Zn (%)	4.06	4.02	4.10	28

*IND – indeterminate

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, 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 3428 and 3500ppm. 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

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

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. A weighting factor of zero was applied to those data sets where $s_I / 2s_g' > 1$ (i.e. where the weighting factor $1- s_I / 2s_g' < 0$). 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 0.5g (compared to 40-50g 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 50g subsample weight can then be determined from the observed standard deviation of the 0.5g 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 for the gold data sets (particularly the 0.5 g INAA data) is consistent with the very narrow calculated tolerance interval and is confirmation of the excellent homogeneity of gold in OREAS 33.
For elements other than gold, outliers were removed prior to the calculation of tolerance intervals and a weighting factor of zero was applied to those data sets where $s_l / 2s_g' > 1$ (i.e. where the weighting factor $1- s_l / 2s_g' < 0$).

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PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

The base metal sulphide ore reference material, OREAS 33 has been prepared and certified and is supplied by:

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It is available in unit sizes of 50g foil packets sealed under nitrogen.

INTENDED USE

OREAS 33 is a reference material intended for the following:

i) for the monitoring and evaluation of laboratory performance;
ii) for the calibration of instruments used in the determination of the concentration of gold;
iii) for the verification of analytical methods for gold;
iv) for the preparation of secondary reference materials of similar composition;
v) as an arbitration sample for commercial transactions.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 33 is a sulphide-bearing reference material and is unstable under prolonged exposure to oxygen and moisture. To enhance its shelf life it has been sealed under a nitrogen atmosphere. Once opened any unused portion should be stored in a desiccator.

INSTRUCTIONS FOR CORRECT USE

The recommended values for OREAS 33 refer to the concentration levels of the certified elements at hygroscopic equilibrium. The material has been packaged in this state.

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

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.
Kleeman, A. W. (1967), *J. Geol. Soc. Australia*, **14**, 43.

Appendix A

Analytical data for OREAS 33

(Refer to relevant Excel spreadsheet)