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CERTIFICATE OF ANALYSIS FOR

COPPER-GOLD-SILVER CONCENTRATE

CERTIFIED REFERENCE MATERIAL

OREAS 991

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 991

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Classical Wet Chemistry						
Cu, Copper (wt.%)	20.66	0.053	20.59	20.73	20.61	20.71
Reduced Charge Fire Assay with Gravimetry						
Au, Gold (ppm)	47.04	0.22	46.70	47.37	46.87	47.21
Lab's Preferred Method						
Ag, Silver (ppm)	48.14	0.90	47.23	49.05	47.16	49.12

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 991

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
4-Acid Digestion								
As	ppm	*170*	Co	ppm	*122*	Ni	ppm	*32.0*
Bi	ppm	*< 50*	Fe	wt.%	*26.92*	Pb	ppm	*123*
Ca	wt.%	*1.08*	Mg	wt.%	*0.495*	S	wt.%	*30.77*
Cd	ppm	*< 10*	Mo	ppm	*490*	Sb	ppm	*80*
Thermogravimetry								
H_2O-	wt.%	*0.528*						

SOURCE MATERIALS

OREAS 991 is a matrix-matched certified reference material (MMCRM) prepared from copper-gold-silver concentrate samples supplied by Newcrest Mining Limited's Cadia Valley Operations near Orange, New South Wales, Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 991 was prepared in the following manner:

- drying under a nitrogen environment to constant mass at 105° C;
- multi stage milling to 100% <30 microns;
- homogenisation;
- packaging in 50g units sealed under nitrogen into labelled laminated foil pouches.

ANALYTICAL PROGRAM

Six umpire laboratories participated in the program to characterise copper, gold and silver (see Table 1). The following methods were employed:

- Copper via short iodide titration (4 labs) or electro-gravimetry (2 labs);
- Gold via reduced charge (10-15g) fire assay with gravimetric finish and full corrections for slag, cupel and silver losses (6 labs);
- Gold by reduced subsample INAA for homogeneity confirmation and evaluation (1 lab);
- Silver via the laboratory's preferred method. This included 4-acid digestion with AAS (3 labs) or ICP-OES finish (1 lab), 3-acid digestion with ICP-OES finish (1 lab) and fire assay with gravimetric finish (1 lab).

For the round robin program samples were taken at 10 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire batch of OREAS 991. To evaluate batch to batch variation at individual laboratories, samples were submitted in four batches at weekly intervals. Each batch consisted of 4 x 50g samples selected in a manner to maximise representation of the 10 sampling intervals.

Laboratories were given strict pre-assay sample instructions relating to moisture correction. These instructions included:

- Equilibration of sample material to lab atmosphere for a minimum of 2 hours;
- Hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Au, Cu and Ag as per ISO 9599;

The laboratories were also requested to report metal concentrations on both a dry and moisture-bearing basis and include all results for moisture determinations. All certified values provided in this certificate are on dry basis. Table 1 presents these certified values together with their associated 1SD's, 95% confidence and tolerance limits. Table 2 shows indicative values for additional analytes and Table 3 provides performance gate intervals for the certified values based on their 1SD's. Gold homogeneity has been evaluated and confirmed by INAA on twenty ~1.0 gram sample portions. Tabulated results of all elements (including Au INAA analyses) together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of lab means from the corrected mean of means (PDM^3) are presented in an Excel-compatible file for this CRM (**OREAS 991 Datapack.xlsx**).

STATISTICAL ANALYSIS

Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying outside this window also relegated to outlying status. For Tolerance Limits only individual outliers have been removed.

Certified Values are the means of accepted laboratory means after outlier filtering. The INAA data is omitted from determination of the certified value for Au and is used solely for the calculation of Tolerance Limits and homogeneity evaluation of OREAS 991. Indicative (uncertified) values (Table 2) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) inter-laboratory consensus is poor; or iii) a significant proportion of results are outlying or reported as less than detection limit.

95% Confidence Limits are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. *95% Confidence Limits should not be used as control limits for laboratory performance.*

Standard Deviation values (1SDs) are reported in Table 1 and provide an indication of a level of performance that might reasonably be expected from a laboratory being monitored by this CRM in a QA/QC program. They take into account errors attributable to measurement uncertainty and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. There are four sources of measurement error:

- within-laboratory within-batch variance or analytical precision (repeatability);
- within-laboratory between-batch variance (reproducibility);
- between-laboratory variance and
- CRM variability.

Performance gates (Table 3) have been calculated from the same filtered data set used to determine the certified value. 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.

For routine submissions (assessing the data quality of a sample batch at one laboratory) the Within-Lab SD can be used as a guide to QC monitoring. Within-Lab SD's include precision errors and batch-to-batch variance but exclude between-laboratory variance. It is calculated from the square root of the average variance for *p* laboratories and is known as the pooled repeatability standard deviation (NIST/SEMATECH e-Handbook of Statistical Methods, 2012).

In QC monitoring performance gates are generally constructed for two and three standard deviations either side of the certified value. As a guide these intervals may be regarded as warning for an individual 2SD outlier, or rejection for multiple 2SD outliers or an individual 3SD outlier. Their precise application however, should always be at the discretion of the QC manager concerned. A second method utilises a ±5% error bar on the certified value as the window of acceptability.

Standard deviation is also shown in relative per cent for one, two and three relative standard deviations (1RSD, 2RSD and 3RSD) to facilitate an appreciation of the magnitude of these numbers and a comparison with the 5% window. 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 991

Constituent	Certified Value	Absolute Standard Deviations					Relative Standard Deviations			5% window	
		1SD	2SD Low	2SD High	3SD Low	3SD High	1RSD	2RSD	3RSD	Low	High
Classical Wet Chemistry											
Cu, wt.%	20.66	0.053	20.56	20.77	20.50	20.82	0.26%	0.51%	0.77%	19.63	21.69
Reduced Charge Fire Assay with Gravimetry											
Au, ppm	47.04	0.22	46.60	47.47	46.38	47.69	0.46%	0.93%	1.39%	44.68	49.39
Lab's Preferred Method											
Ag, ppm	48.14	0.90	46.35	49.94	45.45	50.84	1.86%	3.73%	5.59%	45.74	50.55

Note: intervals may appear asymmetric due to rounding

Tolerance Limits (ISO Guide 3207) were determined using an analysis of precision errors method and are considered a conservative estimate of true homogeneity. The meaning of tolerance limits may be illustrated for copper where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 20.61 and 20.71 wt.%. 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 (ISO Guide 35).

For gold the tolerance has been determined by INAA using the reduced analytical subsample method which utilises the known relationship between standard deviation and analytical subsample weight (Ingamells and Switzer, 1973). In this approach the latter parameter is substantially reduced to a point where most of the variability in replicate assays is due to inhomogeneity of the reference material and measurement error becomes negligible. In this instance a subsample weight of 1 gram was employed and the 1RSD of 0.592% (or 0.114% at a 15g charge weight) confirms the high level of gold homogeneity in OREAS 991.

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 991 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

*Activation Laboratories, Ancaster, ON, Canada
AH Knight, Merseyside, United Kingdom
Independent Assays Laboratory, Perth, WA, Australia
Inspectorate Int. Ltd., Witham, ESS, United Kingdom
**Intertek Genalysis, Perth, WA, Australia
Ledoux & Company, Teaneck, NJ, United States of America
LSI, Rotterdam, Netherlands
Newcrest Services Laboratory, Orange, NSW, Australia

*used only for Au homogeneity evaluation (see Tolerance Limits); **used only for indicative values (see Table 2).

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 991 has been prepared and certified by:

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It has been packaged in 50g units sealed under nitrogen into laminated foil pouches.

INTENDED USE

OREAS 991 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of Cu, Au and Ag in metallurgical plant concentrate samples;
- for the verification of analytical methods for Cu, Au and Ag;
- for the calibration of instruments used in the determination of the concentration of Cu, Au and Ag;
- for the preparation of internal reference materials of similar composition for Cu, Au and Ag.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 991 was prepared from concentrate samples supplied by Newcrest Mining Limited's Cadia Valley Operations near Orange, New South Wales. To ensure a long shelf life it has been sealed under nitrogen in robust laminated foil pouches. In its unopened state under normal conditions of storage it has a shelf life beyond five years.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The certified values for OREAS 991 refer to the concentration levels of Cu, Au and Ag on a dry basis. All analyses were performed on the samples after equilibration with the laboratory atmosphere for a minimum of 2 hours and hygroscopic moisture analysis at 105°C determined on a separate subsample and weighed for analysis at the same time as the sample aliquots for Cu, Au and Ag as per ISO 9599. The data was then corrected to dry basis based on the moisture value. Moisture content varied amongst the labs from 0.36 – 0.63% with an average of 0.53% (excluding one lab which reported a mean of 0.85%).

HANDLING INSTRUCTIONS

Fine powders pose a risk to eyes and lungs and therefore standard precautions such as the use of safety glasses and dust masks are advised.

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

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REFERENCES

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