CERTIFICATE OF ANALYSIS FOR

NICKEL-COBALT SULPHIDE
CERTIFIED REFERENCE MATERIAL

OREAS 78

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 78

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Classical Wet Chemistry						
Co, Cobalt (wt.%)	23.74	0.338	23.51	23.97	23.62	23.86
Ni, Nickel (wt.%)	25.79	0.265	25.62	25.96	25.69	25.88
Infrared Combustion / Acid Digestion						
S, Sulphur (wt.%)	28.61	0.440	28.33	28.90	28.53	28.70
Thermogravimetry						
H_2O-, Moisture @105°C (wt.%)	2.36*	0.58*	2.01*	2.71*	2.30*	2.42*

*Indicative only; Note: intervals may appear asymmetric due to rounding.

SOURCE MATERIAL

OREAS 78 is a nickel-cobalt sulphide certified reference material (CRM) prepared from a nickel matte by-product during the refining process at a nickel refinery. It has been prepared, packaged and certified by Ore Research & Exploration P/L.

PREPARATION PROCEDURES

The materials comprising OREAS 78 were prepared in the following manner:

- drying to constant mass at 75°C;
- multi stage milling to 100% <35 microns;
- packaging in 10g units sealed under nitrogen in labelled laminated foil pouches.

ANALYTICAL PROGRAM

Thirteen analytical laboratories participated in the program and were chosen for their wet-chem classical method capability of base metal sulphide analysis. Cobalt was characterised by specialty acid digestions (mostly EDTA) with titration finishes (11 labs). Nickel was predominantly characterised by DMG precipitation with gravimetric finish (9 labs) and two laboratories used acid digestions with gravimetry and one laboratory used electrogravimetry. Sulphur has been characterised by infrared combustion furnace (7 labs) or an acid digestion with gravimetry (5 labs). Moisture content has been analysed by 13 laboratories via thermogravimetry at 105°C. The values and statistics provided for moisture (H_2O-) should be used as indicative only as moisture content is influenced by particle size distribution, the hygroscopy of the mineral constituents of the CRM and ambient temperature and humidity.

For the round robin program the samples were taken at 10 predetermined sampling intervals immediately following homogenisation and are considered representative of the entire batch of OREAS 78. One 50g sample was submitted to each laboratory for analysis. Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits. Table 2 provides performance gate intervals for the certified values based on their 1SD's. Tabulated laboratory results 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 the detailed certification data for this CRM (**OREAS 78 Datapack.xlsx**).

STATISTICAL ANALYSIS

Certified Values, Standard Deviations, Confidence and Tolerance Limits have been determined for each analyte following removal of individual and laboratory outliers (Table 1). Certified Values are the mean of means after outlier filtering. The 95% Confidence Limit is a measure of the reliability of the certified value, i.e. the narrower the Confidence Interval the greater the certainty in the Certified Value. It should not be used as a control limit 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. The Standard Deviation values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. The SD for each analyte's certified value is calculated from the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers (single iteration). 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. The standard deviation is then calculated for each analyte from the pooled accepted analyses generated from the certification program.

Performance Gates (Table 2) are calculated for two and three standard deviations. As a guide these intervals may be regarded as warning or rejection for multiple 2SD outliers, or rejection for individual 3SD outliers in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

Table 2. Performance Gates for OREAS 78

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											
Co, wt.%	23.74	0.338	23.06	24.42	22.73	24.75	1.42%	2.84%	4.27%	22.55	24.93
Ni, wt.%	25.79	0.265	25.26	26.32	24.99	26.58	1.03%	2.05%	3.08%	24.50	27.08
Infrared Combustion / Acid Digestion											
S, wt.%	28.61	0.440	27.74	29.49	27.30	29.93	1.54%	3.07%	4.61%	27.18	30.04
Thermogravimetry											
H_2O-, wt.%	2.36*	0.58*	1.21*	3.51*	0.63*	4.09*	24.37%*	48.74%*	73.11%*	2.24*	2.48*

*Indicative only; Note: intervals may appear asymmetric due to rounding.

A second method utilises a 5% window calculated directly from the certified value. 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. Caution should be exercised 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.

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 Co, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 23.62 and 23.86 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).

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 78 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

AH Knight International, Merseyside, UK
AH Knight North America, Spartanburg, SC, USA
ALS Inspection UK, Merseyside, UK
ALS, North Vancouver, BC, Canada
ALS, Johannesburg, South Africa
Bachelet, Angleur, Belgium
Independent Assay Laboratory, Perth, WA, Australia
Inspectorate International, Witham, Essex, UK
Ledoux & Company, Teaneck, NJ, USA
LSI, Rotterdam, Netherlands
SGS, Lakefield, ON, Canada
SGS, Spijkenisse, Netherlands
SRL (BV), Perth, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 78 has been prepared and certified by:

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37A Hosie Street   Fax: +613-9729 8338
Bayswater North VIC 3153   Web: www.ore.com.au
AUSTRALIA   Email: info@ore.com.au

It has been packaged in 10g units sealed under nitrogen in labelled laminated foil pouches.

INTENDED USE

OREAS 78 is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of Co, Ni and S in geological samples;
- for the verification of analytical methods for Co, Ni and S;
- for the calibration of instruments used in the determination of the concentration of Co, Ni and S.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 78 was prepared from a mixture of nickel and cobalt sulphide matte. It contains 28.6% sulphur and is reactive under normal atmospheric conditions. To inhibit oxidation and prolong its 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 CORRECT USE

The certified values for OREAS 78 refer to the concentration levels of Co, Ni and S on a dry sample basis. All analyses were performed on the samples as received (without drying) and moisture content at 105°C was determined on separate subsamples. The data was then corrected to dry basis based on the moisture value. Moisture content varied amongst the labs from 1.23 to 3.24% with an average of 2.36%. The values and statistics provided for moisture (H_2O-) should be used as indicative only as moisture content is influenced by particle size distribution, the hygroscopy of the mineral constituents of the CRM and ambient temperature and humidity.

HANDLING INSTRUCTIONS

Materials containing 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

Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager – (ORE P/L)

REFERENCES

ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.

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