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

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

Table 2. Indicative Values for OREAS 78.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Oxidising Fusion XRF								
Al_2O_3	wt.%	0.142	Fe_2O_3	wt.%	0.295	SnO_2	ppm	< 13
As	ppm	30.0	K_2O	wt.%	< 0.001	SO_3	wt.%	72.12
BaO	ppm	< 11	MgO	wt.%	< 0.001	SrO	ppm	< 12
CaO	wt.%	0.036	MnO	wt.%	< 0.001	TiO_2	wt.%	0.010
Cl	ppm	< 10	NiO	wt.%	32.97	V_2O_5	ppm	17.9
CoO	wt.%	29.97	P_2O_5	wt.%	0.016	ZnO	ppm	448
Cr_2O_3	ppm	< 15	PbO	ppm	70	ZrO_2	ppm	< 14
CuO	wt.%	2.66	SiO_2	wt.%	0.525			
Thermogravimetry								
LOI^{1000}	wt.%	33.22	H2O-	wt.%	2.36			
Laser Ablation ICP-MS								
Ag	ppm	2.25	Ge	ppm	1.33	Sm	ppm	0.59
As	ppm	27.4	Hf	ppb	60.0	Sn	ppm	1.10
Ba	ppm	< 0.5	Ho	ppb	< 10	Sr	ppm	1.60
Be	ppm	< 0.2	In	ppm	< 0.05	Ta	ppb	< 10
Bi	ppm	0.57	La	ppm	3.75	Tb	ppb	20.0
Cd	ppm	10.5	Lu	ppb	< 10	Te	ppb	< 200
Ce	ppm	5.66	Mo	ppm	2.20	Th	ppm	0.14
Co	wt.%	23.90	Nb	ppm	0.75	Tl	ppm	0.40
Cr	ppm	2.00	Nd	ppm	2.87	Tm	ppb	< 10
Cs	ppm	0.20	Pb	wt.%	0.007	U	ppm	0.15
Cu	wt.%	2.05	Pr	ppm	0.76	V	ppm	0.80
Dy	ppm	0.095	Rb	ppm	0.55	W	ppm	14.7
Er	ppm	0.030	Re	ppb	< 10	Y	ppm	0.41
Eu	ppb	95.0	Sb	ppm	0.20	Yb	ppb	20.0
Ga	ppm	0.50	Sc	ppm	< 0.1	Zn	ppm	295
Gd	ppm	0.21	Se	ppm	5.00	Zr	ppm	1.25

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ μg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.

Note: the number of significant figures reported is not a reflection of the level of certainty of stated values. They are instead an artefact of ORE's in-house CRM-specific LIMS.

INTRODUCTION

OREAS reference materials are intended to provide a low cost method of evaluating and improving the quality of analysis of geological samples. To the geologist they provide a means of implementing quality control in analytical data sets generated in exploration from the grass roots level through to prospect evaluation, and in grade control at mining operations. To the analyst they provide an effective means of calibrating analytical equipment, assessing new techniques and routinely monitoring in-house procedures.

OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

SOURCE MATERIALS

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.

COMMINUTION AND HOMOGENISATION 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

Fourteen analytical laboratories participated in the program and were chosen for their wet-chemistry classical method capability of base metal sulphide analysis. Samples for the round robin 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.

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-) are 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.

Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits. The approximate major and trace element composition is provided in Table 2. The non-certified values contained in this table are the means of duplicate assays from one laboratory.

Table 3 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-2.0.181213_170100.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 laboratory 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.

Certified Values are the means of accepted laboratory means after outlier filtering.

Indicative (uncertified) values (Table 2) are provided for the major and trace elements determined by borate fusion XRF (Al_2O_3 to TiO_2), laser ablation with ICP-MS (Ag to Zr) and LOI at 1000°C and are the means of duplicate assays from Bureau Veritas, Perth.

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. The SD values include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. OREAS reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

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 any 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.**

In the application of SD's in monitoring performance it is important to note that not all laboratories function at the same level of proficiency and that different methods in use at a particular laboratory have differing levels of precision. Each laboratory has its own inherent SD (for a specific concentration level and analyte-method pair) based on the analytical process and this SD is not directly related to the round robin program.

The majority of data generated in the round robin program was produced by a selection of world class laboratories. The SD's thus generated are more constrained than those that would be produced across a randomly selected group of laboratories. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-lab bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

Table 3 shows **Performance Gates** 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. 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 Ni, where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 25.69 and 25.88 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).

Table 3. 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											
S, wt.%	28.61	0.440	27.74	29.49	27.30	29.93	1.54%	3.07%	4.61%	27.18	30.04

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.
Note 1: intervals may appear asymmetric due to rounding.
Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

PARTICIPATING LABORATORIES

1. AH Knight International, Merseyside, UK
2. AH Knight North America, Spartanburg, SC, USA
3. ALS Inspection UK, Merseyside, UK
4. ALS, North Vancouver, BC, Canada
5. ALS, Johannesburg, South Africa
6. Bachelet, Angleur, Belgium
7. Bureau Veritas (Ultra Trace) Geoanalytical, Perth, WA, Australia
8. Independent Assay Laboratory, Perth, WA, Australia
9. Inspectorate International, Witham, Essex, UK
10. Ledoux & Company, Teaneck, NJ, USA
11. LSI, Rotterdam, Netherlands
12. SGS, Lakefield, ON, Canada
13. SGS, Spijkenisse, Netherlands
14. SRL (BV), Perth, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 78 has been prepared, certified and supplied by:

ORE Research & Exploration Pty Ltd	Tel:	+613-9729 0333
37A Hosie Street	Fax:	+613-9729 8338
Bayswater North VIC 3153	Web:	www.ore.com.au
AUSTRALIA	Email:	info@ore.com.au

It is available in 10g units sealed under nitrogen in 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%.

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.

METROLOGICAL TRACEABILITY

The analytical samples were selected in a manner to represent the entire batch of prepared CRM. This 'representivity' was maintained in each submitted laboratory sample batch and ensures the user that the data is traceable from sample selection through to the

analytical results that underlie the consensus values. Each analytical data set has been validated by its assayer through the inclusion of internal reference materials and QC checks during analysis.

The laboratories were chosen on the basis of their competence (from past performance in inter-laboratory programs undertaken by ORE Pty Ltd) for a particular analytical method, analyte or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *"Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, **only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results** (see ISO Guide 35:2006, Clause 10)."*

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine 'field' samples in the relevant measurement process. Care should be taken to ensure 'matrix matching' as close as practically achievable. The matrix and mineralisation style of the CRM is described in the 'Source Material' section and users should select appropriate CRMs matching these attributes to their field samples.

QMS ACCREDITED

ORE Pty Ltd is accredited to ISO 9001:2008 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.

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

DOCUMENT HISTORY

Revision No	Date	Changes applied
1	3rd Sep, 2018	The Standard Deviations (SD's) were revised to bring them into line with the method used for all other OREAS CRMs (pooled SD method). The original certification used a different method (involving standardising the laboratory means) that generated SD's that were overly constrained for practical use. Indicative values have been added (see Table 2). Added major and trace element characterisation.
0	21st Jan, 2014	First publication.

REFERENCES

ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2015), Reference materials – Contents of certificates and labels.

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

ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.