

**CERTIFICATE OF ANALYSIS FOR**

**NICKEL SULPHIDE ORE REFERENCE MATERIAL**

**OREAS 74b**

**Table 1. Fusion XRF - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 74b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion XRF</b>						
Aluminium, Al (wt.%)	3.55	0.048	3.52	3.59	3.52	3.59
Calcium, Ca (wt.%)	3.09	0.015	3.08	3.11	3.07	3.12
Chromium, Cr (ppm)	981	56.8	943	1018	961	1000
Cobalt, Co (ppm)	502	12.5	498	505	491	512
Copper, Cu (ppm)	947	91.2	848	1046	907	987
Iron, Fe (wt.%)	12.40	0.180	12.27	12.53	12.27	12.53
Magnesium, Mg (wt.%)	9.23	0.135	9.12	9.34	9.17	9.30
Nickel, Ni (wt.%)	3.39	0.093	3.31	3.47	3.36	3.43
Phosphorus, P (wt.%)	0.0195	0.0015	0.0189	0.0201	0.0195	0.0196
Potassium, K (wt.%)	0.691	0.014	0.682	0.700	0.679	0.704
Silicon, Si (wt.%)	19.36	0.208	19.22	19.51	19.26	19.47
Sodium, Na (wt.%)	0.692	0.017	0.676	0.708	0.670	0.714
Titanium, Ti (wt.%)	0.1555	0.0030	0.1549	0.1560	0.1512	0.1597

Note: intervals may appear asymmetric due to rounding.

**Table 2. Fusion ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 74b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	3.54	0.132	3.46	3.61	3.48	3.59
Arsenic, As (ppm)	614	35.3	596	633	587	642
Barium, Ba (ppm)	210	6.9	205	216	205	216
Calcium, Ca (wt.%)	3.09	0.108	3.04	3.15	3.03	3.15
Cerium, Ce (ppm)	30.9	1.04	30.0	31.7	28.6	33.1
Cesium, Cs (ppm)	2.73	0.239	2.51	2.94	2.58	2.88
Chromium, Cr (ppm)	978	67.4	935	1021	943	1012
Cobalt, Co (ppm)	499	23.9	483	515	486	512
Copper, Cu (ppm)	1021	94.7	962	1081	991	1052
Dysprosium, Dy (ppm)	1.96	0.146	1.77	2.15	1.80	2.12
Erbium, Er (ppm)	1.18	0.088	1.09	1.27	IND	IND
Europium, Eu (ppm)	0.53	0.038	0.49	0.56	IND	IND
Gadolinium, Gd (ppm)	1.90	0.149	1.75	2.06	1.64	2.16
Gallium, Ga (ppm)	8.52	0.331	8.22	8.82	8.01	9.03
Holmium, Ho (ppm)	0.40	0.04	0.36	0.45	IND	IND
Iron, Fe (wt.%)	12.60	0.467	12.33	12.88	12.39	12.81
Lanthanum, La (ppm)	17.4	0.75	16.6	18.3	16.4	18.4
Lead, Pb (ppm)	24.1	2.5	22.2	26.0	22.3	25.9
Lithium, Li (ppm)	29.3	1.71	27.5	31.1	27.3	31.2
Magnesium, Mg (wt.%)	9.38	0.263	9.23	9.53	9.25	9.51
Manganese, Mn (wt.%)	0.093	0.003	0.092	0.094	0.090	0.096
Neodymium, Nd (ppm)	11.9	0.69	11.1	12.6	11.2	12.5
Nickel, Ni (wt.%)	3.43	0.119	3.36	3.49	3.33	3.53
Niobium, Nb (ppm)	3.56	0.266	3.31	3.82	3.27	3.86
Potassium, K (wt.%)	0.718	0.051	0.683	0.753	0.676	0.759
Praseodymium, Pr (ppm)	3.39	0.260	3.07	3.71	3.10	3.68
Rubidium, Rb (ppm)	31.3	2.10	29.4	33.2	29.9	32.6
Samarium, Sm (ppm)	2.23	0.186	2.11	2.34	1.89	2.56
Silicon, Si (wt.%)	19.68	0.373	19.46	19.89	19.40	19.96
Strontium, Sr (ppm)	54.2	4.45	50.6	57.8	50.3	58.0
Sulphur, S (wt.%)	6.61	0.203	6.46	6.77	6.48	6.74
Terbium, Tb (ppm)	0.31	0.015	0.29	0.32	IND	IND
Thallium, Tl (ppm)	0.60	0.07	0.52	0.68	IND	IND
Thorium, Th (ppm)	7.60	0.493	7.20	8.00	7.12	8.09
Thulium, Tm (ppm)	0.19	0.03	0.16	0.22	IND	IND
Titanium, Ti (wt.%)	0.152	0.007	0.147	0.156	0.147	0.156
Uranium, U (ppm)	2.40	0.219	2.21	2.60	2.22	2.58
Vanadium, V (ppm)	62.1	10.8	52.7	71.5	55.8	68.4
Ytterbium, Yb (ppm)	1.23	0.17	1.05	1.40	IND	IND
Yttrium, Y (ppm)	11.3	0.71	10.8	11.8	10.7	11.9
Zinc, Zn (ppm)	133	11.1	125	141	120	146
Zirconium, Zr (ppm)	63.0	5.74	57.0	69.1	52.7	73.3

Note: intervals may appear asymmetric due to rounding.

**Table 3. 4-Acid ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 74b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Four Acid ICP-OES/MS</b>						
Aluminium, Al (wt.%)	3.58	0.140	3.51	3.65	3.49	3.67
Antimony, Sb (ppm)	2.69	0.182	2.56	2.81	2.55	2.82
Arsenic, As (ppm)	603	55.6	573	633	586	620
Barium, Ba (ppm)	213	9.6	208	219	203	224
Beryllium, Be (ppm)	0.81	0.12	0.71	0.90	0.70	0.91
Bismuth, Bi (ppm)	1.35	0.085	1.32	1.39	1.25	1.46
Cadmium, Cd (ppm)	0.60	0.10	0.56	0.64	0.56	0.65
Calcium, Ca (wt.%)	3.07	0.118	3.00	3.14	3.01	3.14
Cerium, Ce (ppm)	33.5	1.75	32.4	34.7	32.2	34.9
Cesium, Cs (ppm)	2.97	0.152	2.85	3.08	2.78	3.15
Cobalt, Co (ppm)	490	23.7	477	502	478	501
Copper, Cu (ppm)	998	38.4	978	1018	978	1019
Gallium, Ga (ppm)	9.0	0.66	8.5	9.5	8.7	9.4
Hafnium, Hf (ppm)	1.87	0.109	1.80	1.93	IND	IND
Indium, In (ppm)	0.067	0.004	0.063	0.070	0.000	0.000
Iron, Fe (wt.%)	12.32	0.679	11.96	12.67	12.06	12.57
Lanthanum, La (ppm)	18.8	1.47	17.9	19.7	18.1	19.5
Lead, Pb (ppm)	24.9	2.24	23.6	26.3	23.6	26.3
Lithium, Li (ppm)	27.7	2.15	26.4	29.0	26.7	28.7
Magnesium, Mg (wt.%)	9.31	0.441	9.07	9.55	9.10	9.52
Manganese, Mn (wt.%)	0.091	0.004	0.088	0.093	0.089	0.093
Nickel, Ni (wt.%)	3.38	0.093	3.33	3.43	3.30	3.45
Niobium, Nb (ppm)	4.20	0.383	3.95	4.45	3.95	4.45
Phosphorus, P (wt.%)	0.0191	0.0015	0.0181	0.0200	0.0182	0.0199
Potassium, K (wt.%)	0.729	0.034	0.710	0.748	0.709	0.748
Rhenium, Re (ppm)	0.0072	0.0014	0.0060	0.0084	0.0000	0.0000
Rubidium, Rb (ppm)	33.2	2.82	31.3	35.0	32.1	34.3
Scandium, Sc (ppm)	10.2	0.82	9.7	10.7	9.9	10.5
Selenium, Se (ppm)	4.64	0.56	4.23	5.05	3.59	5.69
Silver, Ag (ppm)	0.568	0.044	0.543	0.593	0.515	0.621
Sodium, Na (wt.%)	0.719	0.033	0.699	0.739	0.699	0.739
Strontium, Sr (ppm)	56.1	2.82	54.4	57.7	54.1	58.1
Tantalum, Ta (ppm)	0.33	0.031	0.32	0.35	0.30	0.37
Tellurium, Te (ppm)	0.38	0.04	0.35	0.42	0.34	0.43
Thallium, Tl (ppm)	0.59	0.033	0.57	0.61	0.55	0.63
Thorium, Th (ppm)	8.28	0.426	8.02	8.55	7.98	8.59
Tin, Sn (ppm)	1.44	0.136	1.37	1.50	IND	IND
Titanium, Ti (wt.%)	0.156	0.007	0.152	0.161	0.152	0.161
Tungsten, W (ppm)	4.75	0.61	4.43	5.08	4.04	5.47
Uranium, U (ppm)	2.45	0.161	2.35	2.54	2.27	2.62
Vanadium, V (ppm)	60.7	2.45	59.3	62.1	58.8	62.6
Yttrium, Y (ppm)	9.93	0.580	9.56	10.30	9.62	10.23
Zinc, Zn (ppm)	136	9.8	132	141	130	143
Zirconium, Zr (ppm)	65	4.5	63	68	63	68

Note: intervals may appear asymmetric due to rounding.

**Table 4. IR Furnace - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 74b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>IR Combustion Furnace</b>						
Sulphur, S (wt.%)	6.57	0.161	6.48	6.65	6.45	6.68

Note: intervals may appear asymmetric due to rounding.

**Table 5. Thermograv - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 74b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Thermogravimetry</b>						
Loss On Ignition, LOI (wt.%)	6.63	0.338	6.36	6.89	6.54	6.71

Note: intervals may appear asymmetric due to rounding.

**Table 6. Indicative Values for OREAS 74b**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Fusion XRF</b>								
As	ppm	644	Mn	wt.%	0.09	Sr	ppm	86
Ba	ppm	221	Rb	ppm	41	V	ppm	67
Cl	ppm	314	S	wt.%	6.2	Zn	ppm	156
<b>Fusion ICP-OES/MS</b>								
Ag	ppm	< 1	Hf	ppm	1.8	Sb	ppm	2.6
B	ppm	< 30	In	ppm	< 0.1	Sc	ppm	9.9
Be	ppm	1.2	Lu	ppm	0.2	Se	ppm	4.9
Bi	ppm	1.5	Mo	ppm	2.6	Sn	ppm	< 2
Cd	ppm	0.8	Na	wt.%	0.71	Ta	ppm	0.3
Ge	ppm	2.8	P	wt.%	0.02	W	ppm	4.5
<b>Four Acid ICP-OES/MS</b>								
Dy	ppm	1.9	Hg	ppm	< 2	Pr	ppm	3.5
Er	ppm	1.1	Ho	ppm	0.4	Sm	ppm	2.2
Eu	ppm	0.5	Lu	ppm	0.2	Tb	ppm	0.3
Gd	ppm	2.1	Mo	ppm	3.6	Tm	ppm	0.2
Ge	ppm	1.3	Nd	ppm	12	Yb	ppm	1.0
<b>Infra-red combustion furnace</b>								
C	wt.%	0.05						

## 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.

## SOURCE MATERIALS

Reference material OREAS 74b is one of a suite of seven nickel sulphide CRMs prepared from high grade massive nickel sulphide ore and barren ultramafic material sourced from Xstrata Nickel's Prospero and Tapinos Nickel mines, located in the Kathleen Valley area approximately 30km north of Leinster in Western Australia within the Agnew-Wiluna portion of the Norseman-Wiluna greenstone belt. It is a typical komatiite-associated, massive sulphide deposit representing an in-situ accumulation of massive and semi-massive primary magmatic Ni-Fe sulphides with minor by-products including Cu, Co and platinum group elements (PGE's).

## COMMUNITION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 74b was prepared in the following manner:

- drying to constant mass at 75°C (Ni ore) and 105°C (barren ultramafic);
- crushing;
- milling of the nickel ore to 100% minus 30 microns;
- milling of the barren ultramafic to 98% minus 75 microns;
- combining in appropriate proportions to achieve the desired grade;
- homogenisation;
- packaging into 10g units in laminated foil pouches.

## ANALYTICAL PROGRAM

Nineteen commercial analytical laboratories participated in the program to characterise the elements reported in Tables 1 to 6. The following methods were employed:

- Lithium borate fusion with X-ray fluorescence (8 laboratories)
- Sodium peroxide fusion or lithium borate fusion with ICP-OES and ICP-MS (14 laboratories)
- Four acid digestion with ICP-OES and ICP-MS (16 laboratories)
- Infra-red combustion furnace for sulphur (14 laboratories)
- Thermogravimetry for Loss On Ignition (9 laboratories)

For the round robin program twenty 800g test units were taken at predetermined intervals during the bagging stage and are considered representative of the entire batch. The six samples received by each laboratory were obtained by taking two 20g scoop splits from each of three separate 800g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity. All test portions distributed to the laboratories were nitrogen flushed and vacuum sealed to prevent oxidation.

Results, together with uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM<sup>3</sup>) are presented in the detailed certification report for this CRM (Hamlyn, 2011).

## STATISTICAL ANALYSIS

**Certified Values, Standard Deviations, Confidence and Tolerance Limits** have been determined for each analytical method following removal of individual and laboratory outliers (see Tables 1-5). 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.

Indicative values (Table 6) are provided where i) the number of laboratories reporting a particular analyte is insufficient (< 5) to support certification; ii) interlaboratory consensus is poor; or iii) a significant proportion of results are outlying or reported as less than detection limits.

**Standard Deviation** values (1SDs) are reported in Tables 1-5 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.

As a guide two or more analytical results lying outside the 2SD window may be regarded as warning or rejection, and rejection for single results lying outside the 3SD window in QC monitoring, although their precise application should be at the discretion of the QC manager concerned.

**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 nickel by lithium borate fusion XRF, where 99% of the time ( $1-\alpha=0.99$ ) at least 95% of subsamples ( $\rho=0.95$ ) will have concentrations lying between 3.36 and 3.43 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).

The homogeneity of OREAS 74b has also been evaluated in an ANOVA study for all certified analytes. This study indicates no evidence that between-unit variance is greater than within-unit variance.

Based on the statistical analysis of the results of the interlaboratory certification program it can be concluded that OREAS 74b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

A detailed report covering statistical treatment and tabulation of the analytical results is available on request as a separate pdf document (Certification Report for OREAS 74b).

## **PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL**

Nickel sulphide ore reference material OREAS 74b has been prepared, certified and is supplied by:

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Due to the presence of reactive sulphides OREAS 74b has been packaged under nitrogen and is available in unit sizes of 10g (single-use laminated foil pouches).

## **INTENDED USE**

OREAS 74b is intended for the following uses:

- for the monitoring of laboratory performance in the analysis of analytes reported in Tables 1-5 in geological samples
- for the verification of analytical methods for analytes reported in Tables 1-5
- for the calibration of instruments used in the determination of the concentration of analytes reported in Tables 1-5

## **STABILITY AND STORAGE INSTRUCTIONS**

OREAS 74b has been sourced from samples of high grade nickel ore and waste rock. It has been packaged under nitrogen in robust laminated foil sachets to prevent oxidation of the sulphides. In its unopened state and under normal conditions of storage it has a shelf life beyond five years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed. After sampling the open sachets should be re-sealed and stored in a nitrogen-purged desiccator.

## **INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL**

The certified values for XRF and for LOI are on a dry basis whilst all other certified values are reported on an "as received" basis. This obviates the need for drying at elevated temperatures, as this can result in oxidation of the sulphide minerals. A moisture content of ~0.3 wt.% has been determined for OREAS 74b in its packaged state.



## 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

Craig Hamlyn (B.Sc. Hons - Geology), Technical Manager - **ORE**

## PARTICIPATING LABORATORIES

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Activation Laboratories, Thunder Bay, Ontario, Canada  
ALS, Brisbane, QLD, Australia  
ALS, Callao, Lima, Peru  
ALS, Perth, WA, Australia  
ALS, Vancouver, BC, Canada  
BV Amdel, Adelaide, SA, Australia  
BV Kalassay, Perth, WA, Australia  
BV Ultra Trace, Perth, WA, Australia  
Intertek Genalysis, Perth, WA, Australia  
Intertek Testing Services, Beijing, China  
Intertek Testing Services, Jakarta, Indonesia  
OMAC Laboratories, Loughrea, County Galway, Ireland  
SGS Mineral Services, Lakefield, Ontario, Canada  
SGS Mineral Services, Perth, WA, Australia  
SGS Mineral Services, Toronto, Ontario, Canada  
Shiva Analyticals, Bangalore North, Karnataka, India  
Zarazma Mineral Studies, Tehran, Iran

## 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.  
Hamlyn, C. L. (2011), Certification Report for OREAS 74b.