

**CERTIFICATE OF ANALYSIS FOR**

**NICKEL SULPHIDE ORE REFERENCE MATERIAL**

**OREAS 77b**

**Table 1. Fusion XRF - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 77b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion XRF</b>						
Aluminium, Al (wt.%)	1.86	0.024	1.84	1.88	1.84	1.89
Calcium, Ca (wt.%)	3.11	0.016	3.10	3.13	3.08	3.15
Chromium, Cr (ppm)	341	36	311	371	333	349
Cobalt, Co (ppm)	1604	36.7	1581	1627	1575	1632
Copper, Cu (ppm)	3163	216.8	2875	3452	3095	3232
Iron, Fe (wt.%)	29.21	0.506	28.79	29.63	28.97	29.44
Magnesium, Mg (wt.%)	2.67	0.045	2.62	2.71	2.64	2.69
Manganese, Mn (wt.%)	0.068	0.002	0.066	0.070	0.067	0.069
Nickel, Ni (wt.%)	11.24	0.187	11.06	11.43	11.13	11.36
Potassium, K (wt.%)	0.344	0.014	0.335	0.353	0.333	0.354
Silicon, Si (wt.%)	9.24	0.095	9.17	9.32	9.18	9.30
Sodium, Na (wt.%)	0.406	0.040	0.363	0.450	0.389	0.423
Titanium, Ti (wt.%)	0.065	0.002	0.064	0.067	IND	IND

Note: intervals may appear asymmetric due to rounding.

**Table 2. Fusion ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 77b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Fusion ICP-OES/MS</b>						
Aluminium, Al (wt.%)	1.84	0.090	1.79	1.90	1.80	1.89
Antimony, Sb (ppm)	8.2	1.1	7.1	9.4	IND	IND
Arsenic, As (ppm)	2083	115.8	2014	2152	2024	2141
Barium, Ba (ppm)	124	10.2	116	132	117	130
Bismuth, Bi (ppm)	3.4	0.6	2.8	4.0	IND	IND
Calcium, Ca (wt.%)	3.09	0.130	3.02	3.15	2.99	3.18
Cerium, Ce (ppm)	25.7	1.18	25.0	26.5	23.9	27.6
Cesium, Cs (ppm)	2.18	0.207	1.99	2.37	2.08	2.28
Chromium, Cr (ppm)	336	33.4	320	351	327	344
Cobalt, Co (ppm)	1608	61.4	1570	1647	1568	1649
Copper, Cu (ppm)	3302	176.3	3191	3412	3218	3386
Dysprosium, Dy (ppm)	1.33	0.115	1.20	1.46	IND	IND
Erbium, Er (ppm)	0.81	0.10	0.73	0.90	IND	IND
Europium, Eu (ppm)	0.38	0.06	0.34	0.43	IND	IND
Gadolinium, Gd (ppm)	1.51	0.23	1.28	1.75	IND	IND
Gallium, Ga (ppm)	4.5	0.9	3.7	5.3	4.2	4.8
Holmium, Ho (ppm)	0.27	0.03	0.25	0.30	IND	IND
Iron, Fe (wt.%)	29.81	0.622	29.42	30.21	29.39	30.24
Lanthanum, La (ppm)	15.4	1.48	13.8	17.0	14.7	16.2
Lead, Pb (ppm)	58	5.2	53	63	55	61
Lithium, Li (ppm)	20.4	1.34	19.4	21.4	IND	IND
Magnesium, Mg (wt.%)	2.65	0.106	2.59	2.71	2.62	2.68
Manganese, Mn (wt.%)	0.067	0.004	0.065	0.070	0.065	0.069
Neodymium, Nd (ppm)	9.4	0.65	8.7	10.1	8.7	10.1
Nickel, Ni (wt.%)	11.33	0.413	11.05	11.60	11.12	11.54
Potassium, K (wt.%)	0.369	0.051	0.332	0.405	0.340	0.397
Praseodymium, Pr (ppm)	2.89	0.31	2.57	3.22	2.59	3.19
Rubidium, Rb (ppm)	18.3	0.98	17.4	19.2	18.0	18.6
Samarium, Sm (ppm)	1.66	0.18	1.51	1.82	IND	IND
Silicon, Si (wt.%)	9.49	0.188	9.38	9.59	9.29	9.68
Strontium, Sr (ppm)	33.7	4.2	30.1	37.2	31.1	36.3
Sulphur, S (wt.%)	22.22	0.720	21.62	22.81	21.79	22.64
Terbium, Tb (ppm)	0.24	0.04	0.21	0.26	IND	IND
Thorium, Th (ppm)	6.14	0.356	5.88	6.41	5.81	6.48
Titanium, Ti (wt.%)	0.062	0.003	0.060	0.064	IND	IND
Tungsten, W (ppm)	2.67	0.48	2.26	3.08	IND	IND
Uranium, U (ppm)	1.72	0.30	1.47	1.97	1.53	1.91
Ytterbium, Yb (ppm)	0.80	0.13	0.66	0.94	IND	IND
Yttrium, Y (ppm)	7.2	0.66	6.7	7.6	6.6	7.7
Zinc, Zn (ppm)	202	19.9	187	217	185	219
Zirconium, Zr (ppm)	33	3.9	28	37	28	37

Note: intervals may appear asymmetric due to rounding.

**Table 3. 4-Acid ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 77b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Four Acid ICP-OES/MS</b>						
Aluminium, Al (wt.%)	1.94	0.106	1.88	2.00	1.89	1.99
Antimony, Sb (ppm)	9.10	0.95	8.48	9.72	8.69	9.51
Arsenic, As (ppm)	2054	124.3	1980	2127	2004	2103
Barium, Ba (ppm)	118	8.2	112	123	112	124
Beryllium, Be (ppm)	0.47	0.07	0.43	0.51	0.40	0.55
Bismuth, Bi (ppm)	3.44	0.146	3.36	3.51	3.28	3.60
Cadmium, Cd (ppm)	1.20	0.14	1.10	1.29	1.07	1.32
Calcium, Ca (wt.%)	3.06	0.094	3.00	3.12	3.00	3.12
Cerium, Ce (ppm)	27.7	2.9	25.7	29.7	26.3	29.1
Cesium, Cs (ppm)	2.32	0.083	2.27	2.37	2.21	2.43
Chromium, Cr (ppm)	280	34	261	299	269	291
Cobalt, Co (ppm)	1551	56.2	1523	1580	1512	1591
Copper, Cu (ppm)	3426	120.2	3367	3484	3346	3505
Gallium, Ga (ppm)	4.61	0.294	4.40	4.82	4.39	4.83
Hafnium, Hf (ppm)	1.15	0.073	1.11	1.19	IND	IND
Indium, In (ppm)	0.112	0.015	0.100	0.124	0.105	0.120
Iron, Fe (wt.%)	29.85	0.966	29.38	30.32	29.07	30.63
Lanthanum, La (ppm)	15.8	2.0	14.6	17.1	15.2	16.5
Lead, Pb (ppm)	61	5.2	58	64	59	64
Lithium, Li (ppm)	18.8	1.71	17.7	19.9	17.8	19.7
Magnesium, Mg (wt.%)	2.59	0.099	2.54	2.65	2.54	2.65
Manganese, Mn (wt.%)	0.064	0.002	0.063	0.066	0.062	0.066
Nickel, Ni (wt.%)	11.30	0.301	11.15	11.46	11.11	11.50
Niobium, Nb (ppm)	3.26	0.40	2.98	3.54	3.13	3.39
Potassium, K (wt.%)	0.361	0.012	0.355	0.367	0.352	0.370
Rhenium, Re (ppm)	0.022	0.002	0.020	0.023	0.019	0.024
Rubidium, Rb (ppm)	19.1	1.41	18.2	20.0	18.4	19.9
Scandium, Sc (ppm)	3.51	0.42	3.25	3.78	3.33	3.69
Silver, Ag (ppm)	1.62	0.16	1.50	1.73	1.51	1.72
Sodium, Na (wt.%)	0.434	0.019	0.423	0.445	0.421	0.447
Strontium, Sr (ppm)	34.4	1.57	33.3	35.4	32.9	35.9
Tantalum, Ta (ppm)	0.28	0.04	0.26	0.30	0.26	0.30
Tellurium, Te (ppm)	1.35	0.27	1.15	1.55	1.28	1.42
Thallium, Tl (ppm)	1.37	0.074	1.33	1.42	1.32	1.42
Thorium, Th (ppm)	6.61	0.311	6.40	6.82	6.36	6.86
Tin, Sn (ppm)	1.59	0.147	1.50	1.69	1.40	1.78
Titanium, Ti (wt.%)	0.064	0.002	0.062	0.065	0.062	0.066
Tungsten, W (ppm)	3.07	0.194	2.99	3.16	2.86	3.29
Uranium, U (ppm)	1.71	0.087	1.66	1.77	1.62	1.81
Vanadium, V (ppm)	33.6	2.92	32.4	34.8	31.8	35.4
Yttrium, Y (ppm)	6.55	0.390	6.28	6.83	6.28	6.83
Zinc, Zn (ppm)	205	17.9	194	216	198	212
Zirconium, Zr (ppm)	37.9	2.43	36.4	39.5	36.3	39.6

Note: intervals may appear asymmetric due to rounding.

**Table 4. IR Furnace - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 77b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>IR Combustion Furnace</b>						
Sulphur, S (wt.%)	22.29	0.850	21.76	22.82	21.92	22.66

Note: intervals may appear asymmetric due to rounding.

**Table 5. Thermograv - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 77b**

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
<b>Thermogravimetry</b>						
Loss On Ignition, LOI (wt.%)	8.87	0.154	8.73	9.01	8.75	9.00

Note: intervals may appear asymmetric due to rounding.

**Table 6. Indicative Values for OREAS 77b**

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
<b>Fusion XRF</b>								
As	ppm	2068	Mo	ppm	< 10	V	ppm	36
Ba	ppm	99	P	wt.%	0.009	Zn	ppm	271
Cl	ppm	241	S	wt.%	21.8			
<b>Fusion ICP-OES/MS</b>								
Ag	ppm	1.5	Lu	ppm	0.09	Sn	ppm	1.1
B	ppm	< 10	Mo	ppm	3.8	Ta	ppm	0.3
Be	ppm	0.8	Na	wt.%	0.44	Te	ppm	2.0
Cd	ppm	1.2	Nb	ppm	2.4	Tl	ppm	1.5
Ge	ppm	5.6	P	wt.%	0.02	Tm	ppm	0.1
Hf	ppm	1.0	Sc	ppm	3.5	V	ppm	36
In	ppm	0.08	Se	ppm	16			
<b>Four Acid ICP-OES/MS</b>								
Dy	ppm	1.3	Ho	ppm	0.3	Se	ppm	13
Er	ppm	0.8	Lu	ppm	0.1	Sm	ppm	1.7
Eu	ppm	0.3	Mo	ppm	6.7	Tb	ppm	0.2
Gd	ppm	1.6	Nd	ppm	9.4	Tm	ppm	0.1
Ge	ppm	3.8	P	wt.%	0.01	Yb	ppm	0.7
Hg	ppm	< 10	Pr	ppm	3.0			
<b>Infra-red combustion furnace</b>								
C	wt.%	< 0.02						

## 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 77b 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).

## COMMINATION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 77b 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 in 10g units sealed under nitrogen, 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.

Tabulated results of all elements 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 (PDM3) are presented in the detailed certification data for this CRM (**OREAS 77b Datapack.xlsx**).

## 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 ( $p=0.95$ ) will have concentrations lying between 11.13 and 11.36 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 77b 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 77b is fit-for-purpose as a certified reference material (see 'Intended Use' below).

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

Nickel sulphide ore reference material OREAS 77b has been prepared, certified and is supplied by:

ORE Research & Exploration Pty Ltd  
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AUSTRALIA

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Due to the presence of reactive sulphides OREAS 77b has been packaged under nitrogen and is available in unit sizes of 10g (single-use laminated foil pouches).

## **INTENDED USE**

OREAS 77b 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 77b 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 ~0.2 wt.% has been determined for OREAS 77b 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**

Acme Analytical Laboratories, Vancouver, BC, Canada  
Activation Laboratories, Ancaster, Ontario, Canada  
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.