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CERTIFICATE OF ANALYSIS FOR

NICKEL SULPHIDE ORE REFERENCE MATERIAL

OREAS 70b

Table 1. Fusion XRF - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 70b

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fusion XRF						
Aluminium, Al (wt.%)	3.81	0.039	3.78	3.84	3.78	3.84
Calcium, Ca (wt.%)	3.07	0.022	3.05	3.09	3.05	3.09
Chromium, Cr (ppm)	1243	43.8	1216	1271	1234	1252
Iron, Fe (wt.%)	5.51	0.086	5.44	5.58	5.46	5.56
Magnesium, Mg (wt.%)	13.54	0.086	13.48	13.61	13.45	13.63
Manganese, Mn (wt.%)	0.115	0.003	0.112	0.117	0.114	0.116
Nickel, Ni (wt.%)	0.223	0.006	0.218	0.229	0.219	0.227
Phosphorus, P (wt.%)	0.024	0.002	0.023	0.025	0.024	0.024
Potassium, K (wt.%)	0.585	0.010	0.577	0.593	0.578	0.592
Silicon, Si (wt.%)	22.42	0.171	22.28	22.56	22.33	22.51
Sodium, Na (wt.%)	0.742	0.016	0.726	0.757	0.725	0.759
Titanium, Ti (wt.%)	0.178	0.004	0.175	0.180	0.175	0.180

Note: intervals may appear asymmetric due to rounding.

Table 2. Fusion ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 70b

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fusion ICP-OES/MS						
Aluminium, Al (wt.%)	3.80	0.162	3.71	3.88	3.72	3.87
Arsenic, As (ppm)	143	20	135	151	133	153
Barium, Ba (ppm)	202	11.1	194	210	194	210
Calcium, Ca (wt.%)	3.08	0.101	3.03	3.13	3.01	3.14
Cerium, Ce (ppm)	27.5	2.62	25.5	29.5	24.6	30.3
Cesium, Cs (ppm)	3.37	0.293	3.10	3.65	3.22	3.53
Chromium, Cr (ppm)	1252	119.9	1180	1323	1214	1289
Cobalt, Co (ppm)	83	6.7	79	88	80	86
Copper, Cu (ppm)	52	6	48	56	48	55
Dysprosium, Dy (ppm)	1.92	0.149	1.79	2.05	1.79	2.05
Erbium, Er (ppm)	1.18	0.107	1.08	1.29	IND	IND
Europium, Eu (ppm)	0.54	0.037	0.51	0.56	IND	IND
Gadolinium, Gd (ppm)	1.93	0.23	1.70	2.16	1.65	2.21
Gallium, Ga (ppm)	9.71	0.412	9.39	10.03	9.12	10.29
Holmium, Ho (ppm)	0.41	0.05	0.36	0.46	IND	IND
Iron, Fe (wt.%)	5.66	0.234	5.53	5.80	5.58	5.75
Lanthanum, La (ppm)	15.5	1.7	13.8	17.2	14.7	16.3
Lead, Pb (ppm)	13.2	1.8	11.8	14.6	IND	IND
Lithium, Li (ppm)	35.3	5.2	28.5	42.1	32.5	38.2
Magnesium, Mg (wt.%)	13.61	0.434	13.36	13.86	13.39	13.83
Manganese, Mn (wt.%)	0.116	0.004	0.114	0.118	0.112	0.120
Neodymium, Nd (ppm)	11.3	1.4	9.9	12.8	10.6	12.1
Nickel, Ni (wt.%)	0.222	0.008	0.217	0.227	0.215	0.229
Niobium, Nb (ppm)	3.27	0.307	2.89	3.65	3.06	3.47
Phosphorus, P (wt.%)	0.025	0.003	0.022	0.028	IND	IND
Potassium, K (wt.%)	0.617	0.056	0.584	0.650	0.578	0.657
Praseodymium, Pr (ppm)	3.13	0.35	2.75	3.52	2.98	3.29
Rubidium, Rb (ppm)	34	2.9	31	36	32	35
Samarium, Sm (ppm)	2.00	0.155	1.85	2.15	1.57	2.42
Silicon, Si (wt.%)	22.78	0.458	22.51	23.04	22.38	23.18
Strontium, Sr (ppm)	72	5.7	67	77	70	75
Sulphur, S (wt.%)	0.308	0.026	0.291	0.324	0.285	0.330
Terbium, Tb (ppm)	0.32	0.03	0.29	0.34	IND	IND
Thorium, Th (ppm)	6.45	0.578	6.01	6.88	5.97	6.92
Thulium, Tm (ppm)	0.18	0.02	0.16	0.21	IND	IND
Titanium, Ti (wt.%)	0.176	0.006	0.171	0.181	0.166	0.186
Uranium, U (ppm)	1.87	0.32	1.62	2.12	1.64	2.10
Vanadium, V (ppm)	69	6.8	64	74	62	76
Ytterbium, Yb (ppm)	1.16	0.105	1.07	1.25	IND	IND
Yttrium, Y (ppm)	10.7	0.64	10.2	11.2	10.0	11.3
Zinc, Zn (ppm)	107	9.5	101	114	100	114
Zirconium, Zr (ppm)	67	9	61	74	59	76

Note: intervals may appear asymmetric due to rounding.

Table 3. 4-Acid ICP - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 70b

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Four Acid ICP-OES/MS						
Aluminium, Al (wt.%)	3.87	0.182	3.78	3.96	3.79	3.95
Antimony, Sb (ppm)	0.56	0.06	0.53	0.59	0.50	0.62
Arsenic, As (ppm)	148	8.4	145	150	136	159
Barium, Ba (ppm)	202	9.3	197	207	196	208
Beryllium, Be (ppm)	1.04	0.19	0.92	1.15	0.94	1.14
Bismuth, Bi (ppm)	0.84	0.15	0.78	0.90	0.71	0.97
Cadmium, Cd (ppm)	0.36	0.04	0.34	0.38	0.33	0.39
Calcium, Ca (wt.%)	3.05	0.149	2.97	3.14	2.99	3.12
Cerium, Ce (ppm)	28.2	1.97	26.9	29.5	26.5	29.9
Cesium, Cs (ppm)	3.44	0.271	3.25	3.63	3.30	3.59
Cobalt, Co (ppm)	78	5.0	76	81	76	81
Copper, Cu (ppm)	52	4.3	50	55	50	55
Gallium, Ga (ppm)	10.1	0.89	9.5	10.6	9.7	10.4
Hafnium, Hf (ppm)	1.86	0.20	1.73	1.98	IND	IND
Indium, In (ppm)	0.047	0.003	0.044	0.049	0.041	0.052
Iron, Fe (wt.%)	5.52	0.273	5.38	5.66	5.41	5.64
Lanthanum, La (ppm)	15.3	1.41	14.5	16.2	14.6	16.1
Lead, Pb (ppm)	13.7	1.6	12.8	14.6	12.8	14.6
Lithium, Li (ppm)	34.4	2.61	32.8	35.9	33.3	35.5
Magnesium, Mg (wt.%)	13.40	0.783	12.98	13.82	13.15	13.65
Manganese, Mn (wt.%)	0.115	0.004	0.113	0.117	0.112	0.117
Molybdenum, Mo (ppm)	3.30	0.53	3.07	3.53	2.45	4.15
Nickel, Ni (wt.%)	0.218	0.010	0.212	0.223	0.213	0.223
Niobium, Nb (ppm)	3.68	0.335	3.47	3.88	3.44	3.91
Phosphorus, P (wt.%)	0.0224	0.0015	0.0216	0.0233	0.0213	0.0236
Potassium, K (wt.%)	0.620	0.029	0.604	0.637	0.602	0.639
Scandium, Sc (ppm)	12.4	1.08	11.7	13.1	11.9	12.8
Silver, Ag (ppm)	0.175	0.035	0.152	0.198	IND	IND
Sodium, Na (wt.%)	0.769	0.033	0.750	0.788	0.747	0.791
Strontium, Sr (ppm)	74	6.1	70	78	72	77
Sulphur, S (wt.%)	0.309	0.020	0.299	0.320	0.298	0.320
Tantalum, Ta (ppm)	0.30	0.04	0.28	0.31	0.25	0.34
Thallium, Tl (ppm)	0.33	0.04	0.31	0.35	0.31	0.35
Thorium, Th (ppm)	6.91	0.278	6.77	7.04	6.52	7.29
Tin, Sn (ppm)	1.21	0.18	1.10	1.32	IND	IND
Titanium, Ti (wt.%)	0.181	0.006	0.177	0.185	0.176	0.186
Tungsten, W (ppm)	4.92	0.72	4.72	5.13	3.86	5.99
Uranium, U (ppm)	1.72	0.108	1.64	1.79	1.56	1.87
Vanadium, V (ppm)	67	2.6	66	68	65	69
Yttrium, Y (ppm)	9.85	0.520	9.51	10.18	9.41	10.29
Zinc, Zn (ppm)	112	9.0	107	117	108	116
Zirconium, Zr (ppm)	66	5.1	63	69	63	69

Note: intervals may appear asymmetric due to rounding.

Table 4. IR Furnace - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 70b

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
IR Combustion Furnace						
Sulphur, S (wt.%)	0.286	0.020	0.273	0.298	0.273	0.298

Note: intervals may appear asymmetric due to rounding.

Table 5. Thermograv - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 70b

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Thermogravimetry						
Loss On Ignition, LOI (wt.%)	6.69	0.330	6.43	6.95	6.63	6.75

Note: intervals may appear asymmetric due to rounding.

Table 6. Indicative Values for OREAS 70b

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Fusion XRF								
As	ppm	127	Nb	ppm	< 10	Th	ppm	20
Ba	ppm	196	Pb	ppm	< 50	U	ppm	< 10
Bi	ppm	< 80	Rb	ppm	< 50	W	ppm	< 10
Cl	ppm	386	S	wt.%	0.3	Zn	ppm	103
Co	ppm	84	Sb	ppm	< 50	Zr	ppm	< 100
Cu	ppm	< 60	Sn	ppm	< 50			
Mo	ppm	< 10	Sr	ppm	62			
Fusion ICP-OES/MS								
Ag	ppm	< 1	Hg	ppm	< 20	Se	ppm	1.5
B	ppm	28	In	ppm	< 0.2	Sn	ppm	1.0
Be	ppm	1.5	Lu	ppm	0.20	Ta	ppm	0.36
Bi	ppm	< 2	Mo	ppm	3.0	Te	ppm	< 6
Cd	ppm	< 2	Na	wt.%	0.76	Tl	ppm	0.35
Ge	ppm	2.0	Sb	ppm	< 2	W	ppm	4.7
Hf	ppm	1.9	Sc	ppm	11			
Four Acid ICP-OES/MS								
Dy	ppm	1.8	Ho	ppm	0.38	Se	ppm	1.0
Er	ppm	1.1	Lu	ppm	0.17	Sm	ppm	2.0
Eu	ppm	0.5	Nd	ppm	10	Tb	ppm	0.33
Gd	ppm	2.0	Pr	ppm	3.1	Te	ppm	0.07
Ge	ppm	0.2	Rb	ppm	36	Tm	ppm	0.18
Hg	ppm	< 1	Re	ppm	0.001	Yb	ppm	1.0
Infra-red combustion furnace								
C	wt.%	0.07						

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 70b 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).

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 70b 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^3) 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 2189 and 2274 ppm. 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 (IS0 Guide 35).
The homogeneity of OREAS 70b 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 70b 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 70b).

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Nickel sulphide ore reference material OREAS 70b has been prepared, certified and is supplied by:

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Due to the presence of reactive sulphides OREAS 70b has been packaged under nitrogen and is available in unit sizes of 10g (single-use laminated foil pouches).

INTENDED USE

OREAS 70b 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 70b 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 ~0.4 wt.% has been determined for OREAS 72b 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.

Hamlyn, C. L. (2011), Certification Report for OREAS 70b.