OREAS
Certified Reference Materials

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CERTIFICATE OF ANALYSIS FOR

NICKEL LATERITE ORE CERTIFIED REFERENCE MATERIAL

OREAS 195

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 195.

Constituent	Certified Value	Within-Lab SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
Al_2O_3, Aluminium(III) oxide (wt.%)	3.13	0.031	3.11	3.15	3.10	3.15
CaO, Calcium oxide (wt.%)	0.390	0.005	0.387	0.393	0.388	0.392
Co, Cobalt (ppm)	477	10	469	486	469	486
Cr_2O_3, Chromium(III) oxide (ppm)	9585	113	9504	9666	9474	9695
Fe_2O_3, Iron(III) oxide (wt.%)	18.29	0.086	18.19	18.40	18.23	18.36
MgO, Magnesium oxide (wt.%)	19.01	0.114	18.90	19.12	18.93	19.09
MnO, Manganese oxide (wt.%)	0.288	0.004	0.284	0.291	0.286	0.290
Ni, Nickel (wt.%)	2.94	0.029	2.92	2.97	2.93	2.96
SiO_2, Silicon dioxide (wt.%)	44.00	0.190	43.81	44.18	43.86	44.14
TiO_2, Titanium dioxide (wt.%)	0.037	0.005	0.035	0.039	IND	IND
Zn, Zinc (ppm)	300	7	291	308	297	302
Borate / Peroxide Fusion ICP						
Al_2O_3, Aluminium(III) oxide (wt.%)	3.07	0.063	3.02	3.13	3.03	3.12
CaO, Calcium oxide (wt.%)	0.397	0.017	0.380	0.415	0.385	0.409
Co, Cobalt (ppm)	465	19	459	471	456	475
Cr_2O_3, Chromium(III) oxide (ppm)	9383	279	9296	9469	9221	9544
Fe_2O_3, Iron(III) oxide (wt.%)	18.16	0.414	17.81	18.51	17.95	18.37
MgO, Magnesium oxide (wt.%)	18.88	0.481	18.55	19.22	18.69	19.08
MnO, Manganese oxide (wt.%)	0.285	0.007	0.281	0.289	0.284	0.286
Na_2O, Sodium oxide (wt.%)	0.031	0.002	0.029	0.032	IND	IND
Ni, Nickel (wt.%)	2.89	0.048	2.86	2.93	2.86	2.92
SiO_2, Silicon dioxide (wt.%)	43.30	0.840	42.64	43.96	42.75	43.85
TiO_2, Titanium dioxide (wt.%)	0.037	0.003	0.034	0.040	0.035	0.038
Zn, Zinc (ppm)	293	18	270	316	283	303
Thermogravimetry						
LOI^{1000}, Loss On Ignition @1000°C (wt.%)	9.71	0.123	9.52	9.90	9.66	9.76
Infrared Combustion						
C, Carbon (wt.%)	0.076	0.013	0.064	0.088	IND	IND

Note: Intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 195.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF								
As	ppm	*200*	K_2O	wt.%	*0.009*	SO_3	wt.%	*0.003*
BaO	ppm	*93*	Na_2O	wt.%	*0.034*	Sr	ppm	*< 20*
Cl	ppm	*< 50*	P_2O_5	wt.%	*0.005*	V_2O_5	ppm	*140*
Cu	ppm	*40.8*	Pb	ppm	*< 10*	Zr	ppm	*< 20*
Borate / Peroxide Fusion ICP								
As	ppm	*< 100*	P_2O_5	wt.%	*< 0.01*	V_2O_5	ppm	*126*
BaO	ppm	*81*	Pb	ppm	*48.0*	Y	ppm	*34.5*
Cu	ppm	*47.6*	Sc	ppm	*17.3*	Zr	ppm	*< 5*
K_2O	wt.%	*0.012*	SO_3	wt.%	*0.031*			
Nb	ppm	*< 5*	Sr	ppm	*3.00*			
Thermogravimetry								
H_2O-	wt.%	*5.79*						
Infrared Combustion								
S	wt.%	*< 0.01*						

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.

SOURCE MATERIALS

OREAS 195 has been prepared from saprolitic ore material sourced from Anglo American Brazil Limitada's Codemin Nickel Mine located in the state of Goiás, ~300 kms from the port of Santos in Brazil. It is one of a suite of thirteen nickel laterite CRMs (OREAS 182 to OREAS 195) sourced from two nickel laterite mines in Brazil.

Table 1 above contains 25 certified values, including values by fusion XRF, fusion ICP, LOI at 1000°C and C by infrared combustion furnace. The analytical data for these analytes have been processed by robust statistical procedures to determine certified values, 95% confidence intervals and tolerance limits and the pooled repeatability standard deviation. Non-certified 'Indicative Values' for 27 additional elements are provided in Table 2 above. Performance gates (based on the pooled repeatability SD) are also provided as a guide to QC monitoring in Table 3.

Tabulated round robin laboratory results of all elements together with analytical method codes, uncorrected means, medians, standard deviations, relative standard deviations and per cent deviation of laboratory means from the corrected mean of means (PDM[3]) are presented in the detailed certification data (**OREAS 195 DataPack.xlsx**). The parameter PDM[3] is a measure of laboratory accuracy while the relative standard deviation is an effective measure of analytical precision where homogeneity of the test material has been confirmed.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 195 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing to achieve a particle size of 99.8% minus 75 microns;
- homogenisation;
- packaging into 10g units in laminated foil pouches and 1kg units in plastic jars.

ANALYTICAL PROGRAM

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

- Ni, Co, Al_2O_3, CaO, Cl, Cu, Cr_2O_3, Fe_2O_3, K_2O, MgO, MnO, Na_2O, P_2O_5, SiO_2, SO_3, TiO_2 and Zn by lithium borate fusion with X-ray fluorescence (17 laboratories);
- Ni, Co, Al_2O_3, CaO, Cu, Cr_2O_3, Fe_2O_3, K_2O, MgO, MnO, Na_2O, P_2O_5, SiO_2, SO_3, TiO_2 and Zn by lithium borate or sodium peroxide fusion with ICP-OES (12 laboratories)*;
- carbon and sulphur by infrared combustion furnace (11 laboratories);
- loss on ignition (LOI) at 1000°C (17 laboratories).

Due to the hygroscopic nature of nickel laterites, the laboratories were instructed to dry all samples thoroughly at 105°C prior to analysis and place in a desiccator with fresh desiccant. The samples were then to be cooled to room temperature before weighing for analysis. Alternatively, samples could be corrected to dry basis by allowing the samples to equilibrate to lab atmosphere before weighing for analysis and correction for moisture by determination at 105°C on a separate portion.

For the round robin certification program a total of twenty 300g test units were taken at predetermined intervals following homogenisation and are considered representative of the entire prepared batch. To evaluate and compensate for the effects of batch-to-batch variation at individual laboratories, samples were submitted to the laboratories in three batches of four 20g sample pulps at weekly intervals. The four samples received by each laboratory were obtained by taking two 20g scoop splits from each of two separate 300g test units. This enabled a nested ANOVA to be undertaken (see ‘ANOVA Test’ section below) to compare within and between unit variance in addition to characterisation of the CRM.

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*Departures from a fusion ICP method were Lab 05, whom used a modified aqua regia digestion with ICP to determine Ni, Co, Cu, SO_3 and Zn, and Lab 08, whom used 4-acid digestion with ICP to determine Co and Cu. Given both methods are total for these analytes and no systematic bias is present, the author believes it acceptable to combine this data with the fusion ICP data on this occasion.

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. There are four sources of measurement error:

- within-laboratory within-batch variance or analytical precision (repeatability)
- within-laboratory between-batch variance (reproducibility)
- between-laboratory variance
- CRM variability

Performance gates (Table 3) have been calculated from the same filtered data set used to determine the certified value. 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.

For routine submissions (assessing the data quality of a sample batch at one laboratory) the Within-Lab SD can be used as a guide to QC monitoring. Within-Lab SD's include precision errors and batch-to-batch variance but exclude between-laboratory variance. It is calculated from the square root of the average variance for *p* laboratories and is known as the pooled repeatability standard deviation (NIST/SEMATECH e-Handbook of Statistical Methods, 2012).

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.

In QC monitoring performance gates are generally constructed for two and three standard deviations either side of the certified value. As a guide these intervals may be regarded as warning for an individual 2SD outlier, or rejection for multiple 2SD outliers or an individual 3SD outlier. Their precise application however, should always be at the discretion of the QC manager concerned. A second method utilises a ±5% error bar on the certified value as the window of acceptability.

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. Both methods should be used with caution 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.

Table 3. Within-Lab Performance Gates for OREAS 195

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
Borate Fusion XRF											
Al_2O_3, wt.%	3.13	0.031	3.07	3.19	3.04	3.22	1.00%	2.00%	3.00%	2.97	3.29
CaO, wt.%	0.390	0.005	0.379	0.401	0.374	0.406	1.40%	2.79%	4.19%	0.370	0.409
Co, ppm	477	10	458	496	449	506	1.99%	3.98%	5.98%	453	501
Cr_2O_3, ppm	9585	113	9359	9811	9246	9924	1.18%	2.36%	3.54%	9106	10064
Fe_2O_3, wt.%	18.29	0.086	18.12	18.47	18.04	18.55	0.47%	0.94%	1.41%	17.38	19.21
MgO, wt.%	19.01	0.114	18.78	19.24	18.67	19.35	0.60%	1.20%	1.80%	18.06	19.96
MnO, wt.%	0.288	0.004	0.280	0.295	0.277	0.299	1.27%	2.54%	3.80%	0.273	0.302
Ni, wt.%	2.94	0.029	2.89	3.00	2.86	3.03	0.99%	1.97%	2.96%	2.80	3.09
SiO_2, wt.%	44.00	0.190	43.62	44.38	43.43	44.57	0.43%	0.86%	1.30%	41.80	46.20
TiO_2, wt.%	0.037	0.005	0.027	0.047	0.022	0.052	13.30%	26.61%	39.91%	0.035	0.039
Zn, ppm	300	7	286	314	279	321	2.34%	4.69%	7.03%	285	315
Borate / Peroxide Fusion ICP											
Al_2O_3, wt.%	3.07	0.063	2.95	3.20	2.89	3.26	2.04%	4.09%	6.13%	2.92	3.23
CaO, wt.%	0.397	0.017	0.363	0.431	0.346	0.448	4.31%	8.62%	12.92%	0.377	0.417
Co, ppm	465	19	427	504	408	523	4.12%	8.24%	12.36%	442	489
Cr_2O_3, ppm	9383	279	8824	9941	8545	10220	2.97%	5.95%	8.92%	8913	9852
Fe_2O_3, wt.%	18.16	0.414	17.33	18.99	16.92	19.40	2.28%	4.56%	6.84%	17.25	19.07
MgO, wt.%	18.88	0.481	17.92	19.85	17.44	20.33	2.55%	5.10%	7.65%	17.94	19.83
MnO, wt.%	0.285	0.007	0.270	0.300	0.263	0.307	2.60%	5.19%	7.79%	0.271	0.299
Na_2O, wt.%	0.031	0.002	0.026	0.035	0.024	0.037	6.85%	13.71%	20.56%	0.029	0.032
Ni, wt.%	2.89	0.048	2.80	2.99	2.75	3.03	1.65%	3.29%	4.94%	2.75	3.04
SiO_2, wt.%	43.30	0.840	41.62	44.98	40.78	45.82	1.94%	3.88%	5.82%	41.14	45.47
TiO_2, wt.%	0.037	0.003	0.030	0.043	0.027	0.046	8.73%	17.46%	26.19%	0.035	0.039
Zn, ppm	293	18	258	328	240	346	5.98%	11.96%	17.94%	278	308
Thermogravimetry											
LOI^{1000}, wt.%	9.71	0.123	9.47	9.96	9.34	10.08	1.27%	2.54%	3.80%	9.23	10.20
Infrared Combustion											
C, wt.%	0.076	0.013	0.050	0.102	0.037	0.115	17.28%	34.56%	51.84%	0.072	0.080

Note: Intervals may appear asymmetric due to rounding.

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 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 2.929 and 2.959 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).

It should be noted that estimates of tolerance by this method are considered conservative as a significant proportion of the observed variance, even in those laboratories exhibiting the best analytical precision, can presumably be attributed to measurement error. Despite the limitations of this method, the tolerance intervals presented in Table 1 are considered to confirm a high level of homogeneity for this CRM.

ANOVA Test

All laboratories and all 3 rounds of sample submission were included in the ANOVA study for nickel, cobalt, iron oxide and magnesium oxide. The sampling format for OREAS 195 was structured to enable nested ANOVA treatment of the round robin results. During the bagging stage, immediately following homogenization, twenty 300g samples were taken at regular intervals representative of the entire batch of OREAS 195. For each round of sample submissions, each laboratory received paired samples from two different, non-adjacent 300g samples. For example, the samples that any one of the seventeen (XRF) laboratories could have received are:

Round 1 (week 1)	Round 2 (week 2)	Round 3 (week 3)
Sample 1: Unit 1	Sample 1: Unit 10	Sample 1: Unit 6
Sample 2: Unit 11	Sample 2: Unit 20	Sample 2: Unit 16
Sample 3: Unit 1	Sample 3: Unit 10	Sample 3: Unit 6
Sample 4: Unit 11	Sample 4: Unit 20	Sample 4: Unit 16

The purpose of the ANOVA investigation was to compare the within-unit variance with that of the between-unit variance. This approach permitted an assessment of homogeneity across the entire batch of OREAS 195. The test was performed using the following parameters:

- Significance Level $\alpha = P$ (type I error) = 0.05
- Null Hypothesis, H_0: Between-unit variance is no greater than within-unit variance (reject H_0 if p-value < 0.05)
- Alternative Hypothesis, H_1: Between-unit variance is greater than within-unit variance

P-values are a measure of probability whereby values less than 0.05 indicate a greater than 95% probability that the observed differences in within-unit and between-unit variances are real. The dataset was filtered for both individual and batch (lab round) outliers prior to the calculation of the p-value. This process derived p-values of 1.00 for nickel, 0.559 for cobalt, 1.00 for iron oxide and 1.00 for magnesium oxide and indicates no evidence that between-unit variance is greater than within-unit variance. Conclusion: do not reject H_o.

Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that the metals are distributed in a similar manner throughout OREAS 195 and that the variance between two subsamples from the same unit is statistically indistinguishable to the variance from two subsamples taken from any two separate units.

PARTICIPATING LABORATORIES

1. Acme (BV), Vancouver, BC, Canada
2. Actlabs, Ancaster, Ontario, Canada
3. ALS, Brisbane, QLD, Australia
4. ALS, Lima, Peru

5. ALS, Perth, WA, Australia
6. ALS, Vancouver, BC, Canada
7. Amdel (BV), Adelaide, SA, Australia
8. Amdel (BV), Cardiff, NSW, Australia
9. Inspectorate (BV), Kendari, Sulawesi , Indonesia
10. Intertek Genalysis, Perth, WA, Australia
11. Intertek Testing Services, Jakarta, Indonesia
12. Ni Lab, Pouembout, New Caledonia
13. SGS Australia Mineral Services, Perth (Newburn), WA, Australia
14. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
15. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
16. SGS Mineral Services, Toronto, Ontario, Canada
17. Société le Nickel (SLN), Noumea, New Caledonia
18. UIS Analytical Services, Centurion , South Africa
19. Ultra Trace Pty Ltd (BV), Perth, WA, Australia

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 195 has been prepared, certified and is supplied by:

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It is available in unit sizes of 10g (single-use laminated foil pouches) and 1kg (plastic jars).

INTENDED USE

OREAS 195 is intended for the following uses:

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

STABILITY AND STORAGE INSTRUCTIONS

OREAS 195 has been sourced from a sample of saprolitic nickel ore. It has been packaged in robust laminated foil pouches and plastic jars. In its unopened state and under normal conditions of storage it has a shelf life beyond ten years. Once opened the jars should be re-sealed after sampling and the contents consumed within two years.

INSTRUCTIONS FOR CORRECT USE

All certified values are reported on a dry basis after removal of hygroscopic moisture by drying in air at 105°C to constant mass. Users departing from these conventions should correct for moisture content.

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.

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) for a particular analytical method, analyte or analyte suite, and sample matrix. Most of these laboratories have and maintain ISO 17025 accreditation. The certified and non-certified (indicative) values presented in this report are calculated from the means of accepted data following robust statistical treatment as detailed in this report.

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.

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.

CERTIFYING OFFICER

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

REFERENCES

ISO Guide 30 (1992), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2000), Reference materials – Contents of certificates and labels.

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