ORE RESEARCH & EXPLORATION

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CERTIFICATE OF ANALYSIS FOR

HEMATITE ORE
REFERENCE MATERIAL
OREAS 405

Table 1. Fusion XRF - Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 405

Constituent (wt.%)	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Fusion XRF						
Iron, Fe (wt.%)	58.02	0.297	57.89	58.16	57.86	58.18
Aluminium Oxide, Al_2O_3 (wt.%)	2.26	0.042	2.24	2.28	2.24	2.28
Calcium Oxide, CaO (wt.%)	0.196	0.006	0.194	0.199	0.192	0.201
Chromium Oxide, Cr_2O_3 (ppm)	102	13	97	107	IND	IND
Manganese Oxide, MnO (wt.%)	0.030	0.002	0.029	0.030	0.027	0.032
Phosphorus, P (wt.%)	0.111	0.002	0.110	0.112	0.109	0.113
Potassium Oxide, K_2O (wt.%)	0.020	0.001	0.019	0.020	IND	IND
Silicon Dioxide, SiO_2 (wt.%)	8.37	0.062	8.35	8.40	8.33	8.42
Sulphur, S (wt.%)	0.018	0.002	0.017	0.019	IND	IND
Titanium Oxide, TiO_2 (wt.%)	0.214	0.007	0.211	0.217	0.210	0.218
Thermogravimetry at 1000° C						
Loss On Ignition, LOI (wt.%)	5.61	0.095	5.56	5.66	5.56	5.67

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 405

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Infrared Combustion								
S	wt.%	0.030						
Fusion XRF								
As	ppm	10.1	MgO	wt.%	0.062	V	ppm	28.9
Ba	ppm	33.5	Na_2O	wt.%	0.014	Zn	ppm	17.4
Cl	ppm	49.1	Ni	ppm	39.2	Zr	ppm	67
Co	ppm	7.50	Pb	ppm	15.4			
Cu	ppm	19.9	Sr	ppm	21.5			

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 405 is one of a suite of six CRMs sourced from hematite iron ore samples from the Spinifex Ridge deposit owned by Moly Mines Limited. Areas of enriched iron occur within the banded iron formation of the Gorge Creek Group located approximately 170km east of Port Hedland in Western Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 405 was prepared in the following manner:

- drying to constant mass at 105°C;
- crushing and multi stage milling;
- homogenisation;
- packaging in 10g units into laminated foil pouches and in 1kg units into plastic jars.

ANALYTICAL PROGRAM

Seventeen commercial analytical laboratories participated in the program to characterise the elements reported in Table 1 via lithium borate fusion with x-ray fluorescence for the standard iron ore suite including Fe, P, SiO_2, Al_2O_3, CaO, MgO, MnO, S, TiO_2, K_2O, Na_2O and LOI at 1000°C via thermogravimetry. Two laboratories used infra-red combustion furnace to determine sulphur and this data was not included with the XRF data but an

indicative value for sulphur via IR combustion is presented (see Table 2). Table 2 shows indicative values for a number of elements where data was insufficient for certification (further explained in 'Statistical Analysis'). All analytes were requested to be reported on a dry basis without the addition of sodium nitrate to the flux and iron content to be determined by direct measurement XRF, not by closure to 100%, or any other assumed total.

For the round robin program ten 500g test units were taken at predetermined intervals during the bagging stage, immediately following final blending, and are considered representative of the entire batch. The six samples received by each laboratory were obtained by taking two 25g scoop splits from each of three separate 500g test units. This format enabled nested ANOVA treatment of the results to evaluate homogeneity.
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 certification data file for this CRM (**Datapack for OREAS 405.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). 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 2) 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 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. 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.

Performance Gates in Table 3 are 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 percent 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.

Table 3. Performance Gates for OREAS 405

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
Fusion XRF											
Al_2O_3, wt.%	2.26	0.042	2.18	2.34	2.13	2.39	1.86%	3.73%	5.59%	2.15	2.37
CaO, wt.%	0.196	0.006	0.184	0.209	0.177	0.216	3.24%	6.47%	9.71%	0.187	0.206
Cr_2O_3, ppm	102	13	75	129	62	142	13.16%	26.33%	39.49%	97	107
Fe, wt.%	58.02	0.297	57.43	58.62	57.13	58.91	0.51%	1.03%	1.54%	55.12	60.92
K_2O, wt.%	0.020	0.001	0.018	0.022	0.017	0.023	5.31%	10.61%	15.92%	0.019	0.021
MnO, wt.%	0.030	0.002	0.026	0.033	0.025	0.034	5.48%	10.97%	16.45%	0.028	0.031
P, wt.%	0.111	0.002	0.106	0.116	0.104	0.119	2.23%	4.45%	6.68%	0.106	0.117
S, wt.%	0.018	0.002	0.015	0.021	0.013	0.023	9.26%	18.52%	27.78%	0.017	0.019
SiO_2, wt.%	8.37	0.062	8.25	8.50	8.19	8.56	0.75%	1.49%	2.24%	7.96	8.79
TiO_2, wt.%	0.214	0.007	0.200	0.228	0.194	0.234	3.17%	6.35%	9.52%	0.203	0.225
Thermogravimetry at 1000° C											
LOI, wt.%	5.61	0.095	5.42	5.80	5.33	5.89	1.69%	3.37%	5.06%	5.33	5.89

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 iron (Fe), where 99% of the time (1-α=0.99) at least 95% of subsamples (ρ=0.95) will have concentrations lying between 57.86 and 58.18 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 (IS0 Guide 35).

ANOVA Treatment of all results was undertaken to evaluate the homogeneity of certified analytes in OREAS 405. All labs participated in the ANOVA study where each received paired samples of three different, non-adjacent, sampling units. For example, the ten samples that any one of the eight participating labs could have received is:

- Sample 1 (from sampling interval 1)
- Sample 2 (from sampling interval 4)
- Sample 3 (from sampling interval 7)
- Sample 4 (from sampling interval 1)
- Sample 5 (from sampling interval 4)
- Sample 6 (from sampling interval 7)

For the purpose of the ANOVA investigation these intervals were considered test units where the aim was to test whether between-unit variance was greater than within-unit variance. This approach permitted an assessment of homogeneity across the entire batch of OREAS 405. The test was performed using the following parameters:

- Significance Level α = 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. Each dataset was filtered for both individual and laboratory outliers prior to calculation of *p*-values. This process derived the *p*-values as shown in Table 4 and indicate no evidence that between-unit variance is greater than within-unit variance. Conclusion: do not reject H_0. Note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes that the analytes are uniformly distributed throughout OREAS 405 and that the variance between two subsamples from the same unit is identical to the variance from two subsamples taken from any two separate units.

Table 4. Results of ANOVA Treatment showing *p*-values for all Certified Values of OREAS 405

Constituent	*p*-value
Fusion XRF	
Iron, Fe (wt.%)	0.888
Aluminium Oxide, Al_2O_3 (wt.%)	0.999
Calcium Oxide, CaO (wt.%)	0.959
Chromium Oxide, Cr_2O_3 (ppm)	0.875
Manganese Oxide, MnO (wt.%)	NA
Phosphorus, P (wt.%)	0.818
Potassium Oxide, K_2O (wt.%)	NA
Silicon Dioxide, SiO_2 (wt.%)	0.998
Sulphur, S (wt.%)	0.595
Titanium Oxide, TiO_2 (wt.%)	0.734
Thermogravimetry at 1000° C	
Loss On Ignition, LOI (wt.%)	0.556

NA=Not Applicable due to results being close to LLD

Based on the statistical analysis of the results of the interlaboratory certification program it can be concluded that OREAS 405 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 405 has been prepared, certified and is supplied by:

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It is available in 10g units in single-use laminated foil pouches and in 1kg units in plastic jars.

PARTICIPATING LABORATORIES

Acme Analytical Laboratories, Vancouver, BC, Canada
Activation Laboratories, Ancaster, Ontario, Canada
ALS, Brisbane, QLD, Australia
ALS, Callao, Lima, Peru
ALS, Perth, WA, Australia
ALS, Vancouver, BC, Canada
BV Amdel, Adelaide, SA, Australia
BV Amdel, Cardiff, NSW, Australia
BV Ultra Trace, Perth, WA, Australia
Intertek Genalysis, Perth, WA, Australia
OMAC Laboratories, Loughrea, County Galway, Ireland
Rio Tinto Cape Lambert Operations, Wickham, WA, Australia
SGS, Lakefield, Ontario, Canada
SGS, Booysens, Gauteng, South Africa
SGS, Perth, WA, Australia
SGS, Vespasiano, MG, Brazil
UIS, Centurion, Gauteng, South Africa

INTENDED USE

OREAS 405 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 405 is an oxidised reference material and is stable in the laminated foil pouches. Under normal conditions of storage it has a shelf life beyond ten years.

INSTRUCTIONS FOR THE CORRECT USE OF THE REFERENCE MATERIAL

The certified values for lithium borate fusion XRF and for LOI are on a dry basis. This requires the removal of hygroscopic moisture by drying in air to constant mass at 105°C. If the reference material is not dried prior to analysis, the certified values should be corrected to the moisture-bearing basis.

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 P/L**)

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.

ISO 9516-1:2003: Iron Ores - Determination of various elements by X-ray fluorescence spectrometry - Part 1: Comprehensive procedure.