

CERTIFICATE OF ANALYSIS FOR

MANGANESE ORE

CERTIFIED REFERENCE MATERIAL

OREAS 171

Table 1. Certified Values, SD's, 95% Confidence and Tolerance Limits for OREAS 171.

Constituent	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Borate Fusion XRF						
Al ₂ O ₃ , Aluminium oxide (wt.%)	7.32	0.123	7.25	7.39	7.23	7.40
BaO, Barium oxide (wt.%)	0.247	0.011	0.241	0.253	0.239	0.254
CaO, Calcium oxide (wt.%)	0.081	0.007	0.078	0.084	IND	IND
Fe, Iron (wt.%)	3.66	0.034	3.64	3.68	3.63	3.69
K ₂ O, Potassium oxide (wt.%)	2.05	0.030	2.04	2.07	2.04	2.07
MgO, Magnesium oxide (wt.%)	0.217	0.016	0.208	0.226	0.204	0.231
Mn, Manganese (wt.%)	35.10	0.167	35.02	35.18	34.84	35.36
Na ₂ O, Sodium oxide (wt.%)	0.236	0.015	0.227	0.244	0.226	0.245
P, Phosphorus (ppm)	663	24	648	677	647	678
SiO ₂ , Silicon dioxide (wt.%)	24.82	0.170	24.74	24.90	24.59	25.05
TiO ₂ , Titanium dioxide (wt.%)	0.336	0.009	0.331	0.341	0.326	0.345
V ₂ O ₅ , Vanadium oxide (ppm)	418	45	386	450	402	433
Thermogravimetry						
Loss On Ignition 1000°C (wt.%)	10.54	0.148	10.45	10.63	10.48	10.59

Note: intervals may appear asymmetric due to rounding.

Table 2. Indicative Values for OREAS 171.

Constituent	Unit	Value	Constituent	Unit	Value
Borate Fusion XRF					
Cr ₂ O ₃	ppm	133	Pb	ppm	< 50
Cu	ppm	42.5	S	ppm	49.5

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

OREAS 171 is a supergene manganese ore matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration Pty Ltd. The material was sourced from the Groote Eylandt Mining Company Pty Ltd (GEMCO) Mn deposit which is hosted within a shallow dipping Lower Cretaceous sedimentary rock unit. Groote Eylandt is in the Gulf of Carpentaria off the coast of Australia's Northern Territory. A portion (22%) of barren siltstone sourced from a quarry north of Melbourne, Australia was added to lower the grade. OREAS 171 is one of a suite of four Mn ore MMCRMs certified by borate fusion XRF covering a Mn grade range of 35-46%.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 171 was prepared in the following manner:

- drying to constant mass at 105°C (Mn ore and barren siltstone);
- crushing and milling to 98% minus 75 microns (Mn ore and barren siltstone);
- sieving at 150 microns (Mn ore and barren siltstone);
- blending the ore component with barren siltstone in appropriate proportions to achieve the desired grades;
- packaging in 10g units sealed in laminated foil pouches and 1kg units in plastic jars.

Fourteen 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 manganese ore XRF suite including Al₂O₃, BaO, CaO, Fe, K₂O, MgO, Mn, Na₂O, P, SiO₂, TiO₂ and V₂O₅. LOI was determined at 1000°C via thermogravimetry. Table 2 shows indicative values for a number of additional elements where data was provided but insufficient for certification (further explained in the 'Statistical Analysis' section). All analytes were requested to be reported on a dry basis and manganese content to be determined by direct measurement XRF, not by closure to 100%, or any other assumed total.

For the round robin program eight 200g test units were taken at predetermined intervals during the bagging stage, immediately following final blending, and are considered

representative of the entire 242kg batch. To maximise representation, the six samples received by each laboratory were obtained by taking one 10g subsample from each of six of the eight available test units. Results, together with uncorrected means, medians, standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM³) are presented in the certification data file for this CRM (**Datapack for OREAS 171.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) inter-laboratory 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.

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. To produce more generally achievable SD's the 'pooled' SD's provided in this report include inter-lab bias. This 'one size fits all' approach may require revision at the discretion of the QC manager concerned following careful scrutiny of QC control charts.

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 171

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 ₂ O ₃ , wt.%	7.32	0.123	7.07	7.57	6.95	7.69	1.68%	3.37%	5.05%	6.95	7.69
BaO, wt.%	0.247	0.011	0.224	0.269	0.213	0.280	4.52%	9.04%	13.57%	0.234	0.259
CaO, wt.%	0.081	0.007	0.067	0.095	0.060	0.102	8.50%	17.00%	25.49%	0.077	0.085
Fe, wt.%	3.66	0.034	3.59	3.73	3.56	3.76	0.94%	1.87%	2.81%	3.48	3.84
K ₂ O, wt.%	2.05	0.030	1.99	2.11	1.97	2.14	1.44%	2.88%	4.32%	1.95	2.16
MgO, wt.%	0.217	0.016	0.186	0.249	0.170	0.265	7.31%	14.62%	21.93%	0.206	0.228
Mn, wt.%	35.10	0.167	34.77	35.44	34.60	35.60	0.48%	0.95%	1.43%	33.35	36.86
Na ₂ O, wt.%	0.236	0.015	0.207	0.265	0.192	0.279	6.16%	12.33%	18.49%	0.224	0.247
P, ppm	663	24	614	711	589	736	3.68%	7.37%	11.05%	629	696
SiO ₂ , wt.%	24.82	0.170	24.48	25.16	24.31	25.33	0.68%	1.37%	2.05%	23.58	26.06
TiO ₂ , wt.%	0.336	0.009	0.317	0.354	0.308	0.364	2.77%	5.55%	8.32%	0.319	0.353
V ₂ O ₅ , ppm	418	45	327	509	282	554	10.85%	21.69%	32.54%	397	439
Thermogravimetry at 1000° C											
LOI, wt.%	10.54	0.148	10.24	10.84	10.09	10.98	1.41%	2.82%	4.23%	10.01	11.07

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 manganese (Mn), where 99% of the time (1- α =0.99) at least 95% of subsamples (ρ =0.95) will have concentrations lying between 34.84 and 35.36 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).

Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 171 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 171 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

ALS, Brisbane, QLD, Australia
ALS, Johannesburg, South Africa
ALS, Vancouver, BC, Canada
Bureau Veritas Geoanalytical, Adelaide, SA, Australia
Bureau Veritas Geoanalytical, Cardiff, NSW, Australia
Bureau Veritas Geoanalytical, Perth, WA, Australia
Bureau Veritas Mineral Processing, Darwin, WA, Australia
Intertek Genalysis, Perth, WA, Australia
Intertek Minerals (IMI), Jakarta, Indonesia
Intertek Testing Services, Darwin, NT, Australia
LCT, Sao Paulo, Sao Paulo, Brazil
SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
SGS South Africa Pty Ltd, Booyens, Gauteng, South Africa

INTENDED USE

OREAS 171 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 171 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 CORRECT USE

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.

REFERENCES

ISO Guide 3207 (1975), Statistical interpretation of data - Determination of a statistical tolerance interval.

ISO Guide 35 (2006), Certification of reference materials - General and statistical principals.

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

A handwritten signature in blue ink, appearing to read 'Craig Hamlyn'.

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