CERTIFICATE OF ANALYSIS FOR

Graphitic Carbon Ore

CERTIFIED REFERENCE MATERIAL

OREAS 722

Table 1. Certified Value, SD, 95% Confidence and Tolerance Limits for OREAS 722.

Constituent (wt.%)	Certified Value	1SD	95% Confidence Limits		95% Tolerance Limits	
			Low	High	Low	High
Infrared Combustion*						
TGC, Total graphitic carbon	2.03	0.093	1.98	2.08	1.98	2.08

*HCl leach of carbonates, roasting to remove organic carbon, residue by (Leco) infrared combustion furnace;

Note: intervals may appear asymmetric due to rounding.
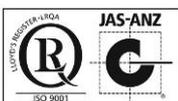
INTRODUCTION

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

Certified Reference Material OREAS 722 has been prepared from crystalline vein graphite rich ore blended with granodiorite. The vein type graphite material was sourced from the Queens Graphite Mine (QGM) in the Matale/Kurunegala Project area in central Sri Lanka. The barren I-type hornblende-bearing granodiorite was sourced from the Late Devonian Lysterfield granodiorite complex located in eastern Melbourne, Australia.

COMMINUTION AND HOMOGENISATION PROCEDURES

The material constituting OREAS 722 was prepared in the following manner:

- Drying to constant mass at 105°C;
- Milling of graphite ore to 100% minus 30 microns;
- Crushing and milling of granodiorite to 98% minus 75 microns;
- Preliminary homogenisation and check assaying of graphite ore;
- Final homogenisation by blending the source materials in specific ratios to achieve target grades;
- Packaging in 10g units in laminated foil pouches and 500g units in plastic jars.

ANALYTICAL PROGRAM

Fifteen commercial analytical laboratories participated in the program to characterise Total Graphitic Carbon (TGC) by hydrochloric acid leach (~50% HCl) to remove carbonates followed by roasting (400-500°C) to remove organic carbon, then analysis of the residue by infrared combustion furnace (Leco - CS Analyser).

For the round robin program ten 200g sample (lots) 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 10g scoop splits from each of three separate 200g lots. This format enabled nested ANOVA treatment of the results to evaluate homogeneity, i.e. to ascertain whether between-unit variance is greater than within-unit variance (see 'ANOVA Study' section below.

Table 1 presents the certified values together with their associated 1SD's, 95% confidence and tolerance limits. Table 2 provides performance gate intervals for the certified values based on their 1SD's. Tabulated laboratory results 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 (PDM[3]) are presented in the detailed certification data file for this CRM (**OREAS 722 DataPack.xlsx**).

Table 2. Indicative Values for OREAS 722.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Infrared Combustion								
C	wt.%	2.06	S	wt.%	0.055			
Coulometry								
TGC	wt.%	2.08						
Proximate Analysis								
Ash	wt.%	97.20	H_2O-	wt.%	0.190			
C-Fixed	wt.%	0.780	Volatile	wt.%	1.85			
Borate Fusion XRF								
Al_2O_3	wt.%	15.10	MgO	wt.%	2.36	SO_3	wt.%	0.160
BaO	ppm	1200	MnO	wt.%	0.070	TiO_2	wt.%	0.825
CaO	wt.%	3.73	Na_2O	wt.%	2.77	V_2O_5	ppm	215
Fe_2O_3	wt.%	5.52	P_2O_5	wt.%	0.238			
K_2O	wt.%	3.83	SiO_2	wt.%	62.64			
Laser Ablation ICP-MS								
Ag	ppm	< 0.1	Gd	ppm	5.27	Sb	ppm	0.55
As	ppm	19.5	Hf	ppm	7.77	Sc	ppm	12.5
Ba	ppm	1065	La	ppm	38.6	Sm	ppm	6.87
Bi	ppm	0.18	Mo	ppm	7.70	Th	ppm	21.2
Cd	ppm	< 0.1	Nd	ppm	31.8	Tl	ppm	0.15
Ce	ppm	75	Ni	ppm	48.0	U	ppm	5.97
Co	ppm	14.1	Pb	ppm	21.0	W	ppm	5.00
Cu	ppm	48.0	Pr	ppm	8.98	Zn	ppm	78
Eu	ppm	1.38	Re	ppm	0.013			
Thermogravimetry								
LOI^{1000}	wt.%	2.62						

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.

STATISTICAL ANALYSIS

Certified Values, Confidence Limits, Standard Deviations and Tolerance Limits (Table 1) have been determined for each analyte following removal of individual, laboratory dataset (batch) and 3SD outliers (single iteration). For individual outliers within a laboratory batch the z-score test is used in combination with a second method that determines the per cent deviation of the individual value from the batch median. Outliers in general are selected on the basis of z-scores > 2.5 and with per cent deviations (i) > 3 and (ii) more than three times the average absolute per cent deviation for the batch. In certain instances statistician's prerogative has been employed in discriminating outliers. Each laboratory data set mean is tested for outlying status based on z-score discrimination and rejected if > 2.5. After individual and laboratory data set (batch) outliers have been eliminated a non-iterative 3 standard deviation filter is applied, with those values lying

outside this window also relegated to outlying status. The Certified Values are the means of accepted laboratory means after outlier filtering.

The 95% Confidence Limits are inversely proportional to the number of participating laboratories and inter-laboratory agreement. It is a measure of the reliability of the certified value. A 95% confidence interval indicates a 95% probability that the true value of the analyte under consideration lies between the upper and lower limits. *95% Confidence Limits should not be used as control limits for laboratory performance.*

Indicative (uncertified) values (Table 2) are provided for total carbon and sulphur by Infrared combustion furnace, the major and trace elements by borate fusion with XRF (Al_2O_3 to V_2O_5), laser ablation with ICP-MS (Ag to Zn) and LOI at 1000° C by thermogravimetry. These indicative values are the means of duplicate assays from Bureau Veritas, Perth. Additional indicative values by Proximate Analysis are also provided by Mintek in Randburg for Ash, C-Fixed, H_2O and Volatiles.

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. The SD's 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 SD values thus include all sources of measurement uncertainty: between-lab variance, within-run variance (precision errors) and CRM variability. OREAS prepared reference materials have a level of homogeneity such that the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

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 any 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.

Table 3 shows **Performance Gates** 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. Pooled-Lab Performance Gates for OREAS 722.

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
Infrared Combustion*											
TGC, wt.%	2.03	0.093	1.84	2.22	1.75	2.31	4.58%	9.17%	13.75%	1.93	2.13

*HCl leach of carbonates, roasting to remove organic carbon, residue by (Leco) infrared combustion furnace;

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 total graphitic carbon (TGC), where 99% of the time ($1-\alpha=0.99$) at least 95% of subsamples ($\rho=0.95$) will have concentrations lying between 1.98 and 2.08 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). *Please note that tolerance limits pertain to the homogeneity of the CRM only and should not be used as control limits for laboratory performance.*

The homogeneity of OREAS 722 has also been evaluated in a **nested ANOVA** of the round robin program. Each of the sixteen round robin laboratories received six samples per CRM and these samples were made up of paired samples from three different, non-adjacent sampling intervals. The purpose of the ANOVA evaluation is to test that no statistically significant difference exists in the variance between-units to that of the variance within-units. This allows an assessment of homogeneity across the entire prepared batch of OREAS 722. The test was performed using the following parameters:

- 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 where 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 datasets were filtered for both individual and laboratory data set (batch) outliers prior to the calculation of the *p*-value. This process derived a *p*-value of 0.93 which is an insignificant result and the Null Hypothesis is retained.

It is important to note that ANOVA is not an absolute measure of homogeneity. Rather, it establishes whether or not the analytes are distributed in a similar manner throughout the packaging run of OREAS 722 and whether the variance between two subsamples from the same unit is statistically distinguishable to the variance from two subsamples taken from any two separate units. A reference material therefore, can possess poor absolute homogeneity yet still pass a relative homogeneity test if the within-unit heterogeneity is

large and similar across all units. Based on the statistical analysis of the results of the inter-laboratory certification program it can be concluded that OREAS 722 is fit-for-purpose as a certified reference material (see 'Intended Use' below).

PARTICIPATING LABORATORIES

1. Actlabs, Ancaster, Ontario, Canada
2. ALS, Brisbane, QLD, Australia
3. ALS, Johannesburg, South Africa
4. ALS, Loughrea, Galway, Ireland
5. ALS, Vancouver, BC, Canada
6. Bureau Veritas Commodities Canada Ltd, Vancouver, BC, Canada
7. Bureau Veritas Geoanalytical, Adelaide, SA, Australia
8. Bureau Veritas Geoanalytical, Perth, WA, Australia
9. Inspectorate (BV), Lima, Peru
10. Intertek Genalysis, Perth, WA, Australia
11. Labtium Oy, Saarenkylä, Rovaniemi, Finland
12. MINTEK Analytical Services, Randburg, South Africa
13. SGS Australia Mineral Services, Perth, WA, Australia
14. SGS Geosol Laboratorios Ltda, Vespasiano, Minas Gerais, Brazil
15. SGS Lakefield Research Ltd, Lakefield, Ontario, Canada
16. Shiva Analyticals Ltd, Bangalore North, Karnataka, India

PREPARER AND SUPPLIER

Certified reference material OREAS 722 is prepared, certified and supplied by:

ORE Research & Exploration Pty Ltd	Tel:	+613-9729 0333
37A Hosie Street	Fax:	+613-9729 8338
Bayswater North VIC 3153	Web:	www.ore.com.au
AUSTRALIA	Email:	info@ore.com.au

It is packaged in 10g units in robust single-use laminated foil pouches and 500g units in plastic jars.

INTENDED USE

OREAS 722 is a Total Graphitic Carbon (TGC) certified reference material and is intended for the following uses:

- For the monitoring of laboratory performance in the analysis of TGC in geological samples;
- For the verification of analytical methods for TGC;
- For the calibration of instruments used in the determination of the concentration of TGC.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 722 has been prepared from crystalline vein graphite rich ore blended with granodiorite. It contains negligible reactive sulphide and in its unopened state and under normal conditions of storage it has a shelf life beyond ten years. Its stability will be monitored at regular intervals and purchasers notified if any changes are observed.

INSTRUCTIONS FOR CORRECT USE

The Total Graphitic Carbon certified value for OREAS 722 is the concentration level in its packaged state. It should not be dried prior to weighing and analysis.

INFORMATION FOR QUARANTINE

OREAS 722 is biologically inactive and sterile (does not contain any organic matter or vegetation) due to the materials being sourced from depths greater than 3 metres.

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 value presented in this report is 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 9001:2015 by Lloyd's Register Quality Assurance Ltd for its quality management system including development, manufacturing, certification and supply of CRMs.

CERTIFYING OFFICER

16th October, 2017

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

REFERENCES

ISO Guide 30 (2015), Terms and definitions used in connection with reference materials.

ISO Guide 31 (2015), Reference materials – Contents of certificates and labels.

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

ISO Guide 35 (2017), Certification of reference materials - General and statistical principals.