

CERTIFICATE OF ANALYSIS FOR

HIGH GRADE MANGANESE ORE

CERTIFIED REFERENCE MATERIAL

OREAS 170a

Table 1. Certified Values, SDs, 95% Confidence and Tolerance Limits for OREAS 170a.

Constituent	Certified Value	95% Confidence Limits		95% Tolerance Limits	
		Low	High	Low	High
Borate Fusion XRF					
Manganese, Mn (wt.%)	45.06	44.79	45.32	44.87	45.24
Manganese oxide, MnO (wt.%)	58.17	57.83	58.51	57.93	58.41
Iron oxide, Fe ₂ O ₃ (wt.%)	6.49	6.41	6.56	6.46	6.51
Silicon dioxide, SiO ₂ (wt.%)	11.34	11.30	11.39	11.30	11.38
Aluminium oxide, Al ₂ O ₃ (wt.%)	4.44	4.41	4.47	4.40	4.48
Magnesium oxide, MgO (wt.%)	0.222	0.207	0.237	0.207	0.237
Calcium oxide, CaO (wt.%)	0.090	0.088	0.092	IND	IND
Titanium dioxide, TiO ₂ (wt.%)	0.179	0.176	0.181	IND	IND
Potassium oxide, K ₂ O (wt.%)	1.70	1.67	1.72	1.68	1.72
Phosphorus oxide, P ₂ O ₅ (wt.%)	0.152	0.149	0.155	0.148	0.155
Sodium oxide, Na ₂ O (wt.%)	0.276	0.251	0.302	0.264	0.289
LOI, (wt.%)	12.14	12.09	12.19	12.07	12.21

SI unit equivalents: ppm, parts per million ≡ mg/kg ≡ µg/g ≡ 0.0001 wt.% ≡ 1000 ppb, parts per billion.

Note 1: intervals may appear asymmetric due to rounding.

Note 2: the number of decimal places quoted does not imply accuracy of the certified value to this level but are given to minimise rounding errors when calculating 2SD and 3SD windows.

Table 2. Indicative Values for OREAS 170a.

Constituent	Unit	Value	Constituent	Unit	Value	Constituent	Unit	Value
Laser Ablation ICP-MS								
Ag	ppm	0.250	Hf	ppb	1435	Sn	ppm	1.10
As	ppm	37.4	Ho	ppb	3140	Sr	ppm	421
Ba	ppm	2925	In	ppm	< 0.05	Ta	ppb	170
Be	ppm	4.50	La	ppm	86	Tb	ppb	3125
Bi	ppm	0.34	Lu	ppb	985	Te	ppb	500
Cd	ppm	0.50	Mo	ppm	31.6	Th	ppm	4.86
Ce	ppm	145	Nb	ppm	1.74	Tl	ppm	1.80
Co	ppm	168	Nd	ppm	99	Tm	ppb	1175
Cr	ppm	36.5	Ni	ppm	116	U	ppm	3.11
Cs	ppm	2.02	Pb	wt.%	0.005	V	ppm	393
Cu	ppm	71	Pr	ppm	26.1	W	ppm	6.83
Dy	ppm	16.3	Rb	ppm	35.2	Y	ppm	56
Er	ppm	8.09	Re	ppb	20.0	Yb	ppb	7415
Eu	ppb	5605	Sb	ppm	1.90	Zn	ppm	140
Ga	ppm	< 10	Sc	ppm	12.7	Zr	ppm	53
Gd	ppm	20.1	Se	ppm	< 5			
Ge	ppb	1025	Sm	ppm	23.7			

SI unit equivalents: ppm, parts per million \equiv mg/kg \equiv μ g/g \equiv 0.0001 wt.% \equiv 1000 ppb, parts per billion.

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.

OREAS reference materials enable users to successfully achieve process control of these tasks because the observed variance from repeated analysis has its origin almost exclusively in the analytical process rather than the reference material itself.

SOURCE MATERIAL

OREAS 170a is a high grade supergene manganese ore matrix-matched certified reference material (MMCRM) prepared and certified by Ore Research & Exploration. 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. OREAS 170a is one of two MMCRMs characterised for MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI.

COMMUNITION AND HOMOGENISATION PROCEDURES

The material was prepared in the following manner:

- Drying at 105°C to constant mass;
- Crushing and multi-stage milling to 100% minus 40 microns;
- Homogenisation;
- Packaging into 1.0 kg units using wide mouth plastic jars.

ANALYTICAL PROGRAM

Ten commercial laboratories participated in the analytical program to characterise MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI. Each laboratory received 5 samples of 20g each. Each set of subsamples submitted to each laboratory was taken at regular intervals during packaging of the standard in order to maximise their representation. All laboratories were instructed to either dry the samples for a minimum of 6 hours (or until constant mass was achieved), then place the samples in a desiccator with fresh desiccant until weighed or alternatively, to equilibrate the samples to the lab atmosphere and correct the results to a dry basis after moisture analysis at 105°C. All labs employed a lithium borate fusion technique with XRF finish to determine the requested oxides. LOI was determined at 1000°C using thermogravimetric methods.

The results together with uncorrected means, medians, one sigma standard deviations, relative standard deviations and percent deviation of lab means from the corrected mean of means (PDM³) are presented in Tables A2 to A12 (Appendix). The analytical methods employed by each laboratory are explained, together with other abbreviations used, in Table A1 (Appendix).

Table 1 presents the certified values together with their associated 95% confidence and tolerance limits and Table 2 provides the approximate trace element composition.

An Excel spreadsheet containing the certified values, 95% confidence and tolerance intervals and performance gates is also available (**see OREAS 170a DataPack.xlsx**). The parameter PDM³ 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.

STATISTICAL EVALUATION

Certified Value and Confidence Intervals

The certified value is the mean of means of accepted replicate values of accepted participating laboratories computed according to the formulae

$$\bar{x}_i = \frac{1}{n_i} \sum_{j=1}^{n_i} x_{ij} \qquad \bar{\bar{x}} = \frac{1}{p} \sum_{i=1}^p \bar{x}_i$$

where,

- x_{ij} is the j th result reported by laboratory i ;
- p is the number of participating laboratories;
- n_i is the number of results reported by laboratory i ;
- \bar{x}_i is the mean for laboratory i ;
- \bar{x} is the mean of means.

The confidence intervals are obtained by calculation of the variance (\hat{V}) of the consensus value (\bar{x}) (mean of means) and reference to Student's- t distribution with degrees of freedom ($p-1$).

$$\hat{V}(\bar{x}) = \frac{1}{p(p-1)} \sum_{i=1}^p (\bar{x}_i - \bar{x})^2$$

$$\text{Confidence Interval} = \bar{x} \pm t_{1-x/2}(p-1)(\hat{V}(\bar{x}))^{1/2}$$

where,

$t_{1-x/2}(p-1)$ is the $1-x/2$ fractile of the t -distribution with $(p-1)$ degrees of freedom.

The distribution of the values is assumed to be symmetrical about the mean in the calculation of the confidence interval. The test for rejection of individual outliers from each laboratory data set is based on z scores (rejected if $|z| > 2.5$) computed from the robust estimators of location and scale, T and S , respectively, according to the formulae

$$S = 1.483 \frac{\text{median} |x_j - \text{median}(x_i)|}{j=1, \dots, n \quad i=1, \dots, n}$$

$$z_i = \frac{x_i - T}{S}$$

where,

T is the median value in a data set;

S is the median of all absolute deviations from the sample median multiplied by 1.483, a correction factor to make the estimator consistent with the usual parameter of a normal distribution.

The z -score test is used in combination with a second method of individual outlier detection that determines the percent deviation of the individual value from the median. Outliers in general are selected on the basis of z -scores > 2.5 and with percent deviations $> 1.5\%$. In certain instances statistician's prerogative has been employed in discriminating outliers.

Each laboratory data set is tested for outlying status based on z -score discrimination and rejected if $|z| > 2.5$. After individual and lab data set 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. Individual outliers and, more rarely, laboratory means deemed to be outlying are shown left justified and in bold in the tabulated results (see Appendix) and have been omitted in the determination of certified values.

The magnitude of the confidence interval is inversely proportional to the number of participating laboratories and interlaboratory agreement. It 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. A 95% confidence interval indicates a 95% probability that the interval includes the true value of the analyte under consideration.

Indicative (uncertified) values (Table 2) are provided for trace elements determined by laser ablation with ICP-MS (Ag to Zr) and are the means of duplicate assays from Bureau Veritas, Perth.

Statement of Homogeneity

The standard deviation of each laboratory data set includes error due to both the imprecision of the analytical method employed and to possible inhomogeneity of the material analysed. The standard deviation of the pooled individual analyses of all participating laboratories includes error due to the imprecision of each analytical method, to possible inhomogeneity of the material analysed and, in particular, to deficiencies in accuracy of each analytical method.

In determining tolerance intervals that component of error attributable to measurement inaccuracy was eliminated by transformation of the individual results of each data set to a common mean (the uncorrected grand mean) according to the formula

$$x'_{ij} = x_{ij} - \bar{x}_i + \frac{\sum_{i=1}^p \sum_{j=1}^{n_i} x_{ij}}{\sum_{i=1}^p n_i}$$

where,

x_{ij} is the j th raw result reported by laboratory i ;

x'_{ij} is the j th transformed result reported by laboratory i ;

n_i is the number of results reported by laboratory i ;

p is the number of participating laboratories;

\bar{x}_i is the raw mean for laboratory i .

The homogeneity of each constituent was determined from tables of factors for two-sided tolerance limits for normal distributions (ISO 3207) in which

$$\text{Lower limit is } \bar{x} - k'_2(n, p, 1 - \alpha) s''_g$$

$$\text{Upper limit is } \bar{x} + k'_2(n, p, 1 - \alpha) s''_g$$

where,

n is the number of results;

$1 - \alpha$ is the confidence level;

p is the proportion of results expected within the tolerance limits;

k'_2 is the factor for two – sided tolerance limits (m, α unknown);

s''_g is the corrected grand standard deviation

The meaning of these tolerance limits may be illustrated for Cu by 4-acid digest, where 99% of the time at least 95% of subsamples will have concentrations lying between 0.156 and 0.168 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).

The corrected grand standard deviation, s_g'' , used to compute the tolerance intervals is the weighted means of standard deviations of all data sets for a particular constituent according to the formula:

$$s_g'' = \frac{\sum_{i=1}^p (s_i (1 - \frac{s_i}{s_g'}))}{\sum_{i=1}^p (1 - \frac{s_i}{s_g'})}$$

where,

$1 - (\frac{s_i}{2s_g'})$ is the weighting factor for laboratory i ;

s_g' is the grand standard deviation computed from the transformed (i.e. means -adjusted) results

according to the formula

$$s_g' = \left[\frac{\sum_{i=1}^p \sum_{j=1}^{n_i} (x'_{ij} - \bar{x}'_i)^2}{\sum_{i=1}^p n_i - 1} \right]^{1/2}$$

where \bar{x}'_i is the transformed mean for laboratory i

The weighting factors were applied to compensate for the considerable variation in analytical precision amongst participating laboratories. Hence, weighting factors for each data set have been constructed so as to be inversely proportional to the standard deviation of that data set. 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.

Performance Gates

Performance gates 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 and CRM variability. For an effective CRM the contribution of the latter should be negligible in comparison to measurement errors. Sources of measurement error include inter-lab bias, analytical precision (repeatability) and inter-batch bias (reproducibility).

Two methods have been employed to calculate performance gates. The first method uses the same filtered data set used to determine the certified value, i.e. after removal of all individual, lab dataset (batch) and 3SD outliers. 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 individual analyses generated from the certification program.

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. Performance Gates for OREAS 170a.

Constituent	Certified Value	1SD	2SD window		3SD window		Relative Standard Deviations			5% window	
			Low	High	Low	High	1RSD	2RSD	3RSD	Low	High
Borate Fusion XRF											
Manganese, Mn (wt.%)	45.06	0.36	44.32	45.79	43.96	46.16	0.81%	1.62%	2.44%	42.80	47.31
Manganese oxide, MnO (wt.%)	58.17	0.47	57.22	59.11	56.75	59.59	0.81%	1.62%	2.44%	55.26	61.08
Iron oxide, Fe ₂ O ₃ (wt.%)	6.49	0.10	6.28	6.69	6.17	6.80	1.61%	3.22%	4.82%	6.16	6.81
Silicon dioxide, SiO ₂ (wt.%)	11.34	0.07	11.19	11.49	11.12	11.57	0.66%	1.32%	1.98%	10.78	11.91
Aluminium oxide, Al ₂ O ₃ (wt.%)	4.44	0.05	4.34	4.54	4.29	4.59	1.13%	2.25%	3.38%	4.22	4.66
Magnesium oxide, MgO (wt.%)	0.222	0.021	0.179	0.264	0.158	0.285	9.52%	19.05%	28.57%	0.211	0.233
Calcium oxide, CaO (wt.%)	0.090	0.004	0.082	0.097	0.079	0.101	4.14%	8.28%	12.41%	0.085	0.094
Titanium dioxide, TiO ₂ (wt.%)	0.179	0.005	0.169	0.188	0.165	0.192	2.60%	5.21%	7.81%	0.170	0.187
Potassium oxide, K ₂ O (wt.%)	1.70	0.03	1.63	1.76	1.60	1.80	1.95%	3.91%	5.86%	1.61	1.78
Phosphorus oxide, P ₂ O ₅ (wt.%)	0.152	0.005	0.143	0.161	0.138	0.165	2.99%	5.99%	8.98%	0.144	0.159
Sodium oxide, Na ₂ O (wt.%)	0.276	0.034	0.209	0.344	0.175	0.378	12.23%	24.47%	36.70%	0.263	0.290
LOI, (wt.%)	12.14	0.07	12.00	12.27	11.94	12.34	0.55%	1.10%	1.65%	11.53	12.75

SI unit equivalents: ppm, parts per million \equiv mg/kg \equiv μ g/g \equiv 0.0001 wt.% \equiv 1000 ppb, parts per billion.

Note: intervals may appear asymmetric due to rounding

PARTICIPATING LABORATORIES

1. Acme Analytical Laboratories Ltd, Vancouver, BC, Canada
2. Activation Laboratories, Ancaster, ONtario, Canada
3. Activation Laboratories, Perth, WA, Australia
4. ALS Chemex, Brisbane, QLD, Australia
5. ALS Chemex, Vancouver, BC, Canada
6. Amdel Laboratories, Perth, WA, Australia
7. Bureau Veritas (Ultra Trace) Geoanalytical, Perth, WA, Australia
8. Genalysis Laboratory Services Pty Ltd, Perth, WA, Australia
9. SGS Mineral Services Australia, Perth, WA, Australia
10. SGS Mineral Services, Lakefield, ON, Canada

PREPARER AND SUPPLIER OF THE REFERENCE MATERIAL

Reference material OREAS 170a has been prepared and certified and is supplied by:



ORE Research & Exploration Pty Ltd
37A Hosie Street
Bayswater North VIC 3153
AUSTRALIA

Tel: +613-9729 0333
Fax: +613-9729 8338
Web: www.ore.com.au
Email: info@ore.com.au

OREAS 170a is available in 10g units packaged in laminated foil pouches or 1kg units in wide mouth plastic jars.

INTENDED USE

OREAS 170a is a reference material intended for the following:

- i) For the monitoring of laboratory performance in the analysis of MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI in geological samples;
- ii) For the calibration of instruments used in the determination of the concentration of MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI;
- iii) For the verification of analytical methods for MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI.

STABILITY AND STORAGE INSTRUCTIONS

OREAS 170a is a reference material made from high grade manganese oxide ore sourced from the Groote Eylandt Mining Company Pty Ltd (GEMCO) Mn deposit. Under normal conditions of storage it has a shelf life beyond five years.

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.

INSTRUCTIONS FOR THE CORRECT USE

The certified values for OREAS 170a refer to the concentration level of MnO, Fe₂O₃, SiO₂, Al₂O₃, MgO, CaO, TiO₂, K₂O, P₂O₅, Na₂O and LOI after removal of hygroscopic moisture by drying in air to constant mass at 105° C. If the reference material is not dried by the user prior to analysis, the certified values should be corrected to the moisture-bearing basis.

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

Guide ISO/TR 16476:2016, section 5.3.1 describes metrological traceability in reference materials as it pertains to the transformation of the measurand. In this section it states, *“Although the determination of the property value itself can be made traceable to appropriate units through, for example, calibration of the measurement equipment used, steps like the transformation of the sample from one physical (chemical) state to another cannot. Such transformations may only be compared with a reference (when available), or among themselves. For some transformations, reference methods have been defined and may be used in certification projects to evaluate the uncertainty associated with such a transformation. In other cases, **only a comparison among different laboratories using the same method is possible. In this case, certification takes place on the basis of agreement among independent measurement results** (see ISO Guide 35:2006, Clause 10).”*

COMMUTABILITY

The measurements of the results that underlie the certified values contained in this report were undertaken by methods involving pre-treatment (digestion/fusion) of the sample. This served to reduce the sample to a simple and well understood form permitting calibration using simple solutions of the CRM. Due to these methods being well understood and highly effective, commutability is not an issue for this CRM. All OREAS CRMs are sourced from natural ore minerals meaning they will display similar behaviour as routine ‘field’ samples in the relevant measurement process. Care should be taken to ensure ‘matrix matching’ as close as practically achievable. The matrix and mineralisation style of the CRM is described in the ‘Source Material’ section and users should select appropriate CRMs matching these attributes to their field samples.

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

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.



DOCUMENT HISTORY

Revision No	Date	Changes applied
1	3 rd Sep, 2018	Added major and trace element characterisation.
0	7 th Aug, 2012	First publication.

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.

APPENDIX

Analytical Data for OREAS 170a

Table A1. Explanation of abbreviations used in Tables A2 – A12.

Abbreviation	Explanation
Std.Dev.	one standard deviation
Rel.Std.Dev.	one relative standard deviation (%)
PDM ³	percent deviation of lab mean from corrected mean of means
NR	not reported
BF	lithium metaborate fusion
LOI	loss on ignition at 1000° C
XRF	x-ray fluorescence

Table A2. Results for MnO in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	61.10	58.36	58.00	57.40	58.10	58.20	58.52	57.90	58.28	57.80
2	60.96	58.36	57.79	57.28	58.50	58.10	58.40	57.80	58.25	58.10
3	61.20	59.40	57.91	57.40	58.50	58.10	58.31	58.00	58.34	58.20
4	61.11	59.27	57.95	57.42	58.40	58.10	58.62	57.90	58.60	57.80
5	61.05	60.04	57.93	57.21	59.30	58.10	58.49	57.90	58.39	58.10
Mean	61.08	59.09	57.92	57.34	58.56	58.12	58.47	57.90	58.37	58.00
Median	61.10	59.27	57.93	57.40	58.50	58.10	58.49	57.90	58.34	58.10
Std.Dev.	0.09	0.72	0.08	0.09	0.44	0.04	0.12	0.07	0.14	0.19
Rel.Std.Dev.	0.14%	1.22%	0.14%	0.16%	0.76%	0.08%	0.20%	0.12%	0.24%	0.32%
PDM ³	5.01%	1.57%	-0.44%	-1.42%	0.67%	-0.08%	0.51%	-0.46%	0.35%	-0.29%

Table A3. Results for Fe₂O₃ in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	6.65	6.22	6.43	6.41	6.48	6.44	6.47	6.65	6.54	6.42
2	6.64	6.21	6.40	6.42	6.50	6.45	6.45	6.64	6.53	6.49
3	6.63	6.35	6.41	6.42	6.53	6.45	6.47	6.66	6.54	6.48
4	6.62	6.36	6.42	6.42	6.51	6.46	6.47	6.65	6.57	6.43
5	6.61	6.40	6.42	6.41	6.60	6.45	6.46	6.65	6.56	6.47
Mean	6.63	6.31	6.41	6.42	6.52	6.45	6.46	6.65	6.55	6.46
Median	6.63	6.35	6.42	6.42	6.51	6.45	6.47	6.65	6.54	6.47
Std.Dev.	0.02	0.09	0.01	0.01	0.05	0.01	0.01	0.01	0.02	0.03
Rel.Std.Dev.	0.24%	1.38%	0.18%	0.09%	0.71%	0.11%	0.10%	0.11%	0.25%	0.48%
PDM ³	2.22%	-2.75%	-1.12%	-1.08%	0.58%	-0.56%	-0.36%	2.53%	0.96%	-0.43%

Table A4. Results for SiO₂ in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	11.37	11.00	11.38	11.25	11.20	11.40	11.49	11.30	11.35	11.45
2	11.32	11.00	11.27	11.23	11.30	11.40	11.41	11.30	11.35	11.30
3	11.34	11.15	11.31	11.24	11.30	11.40	11.40	11.30	11.36	11.35
4	11.30	11.20	11.34	11.31	11.30	11.40	11.44	11.30	11.39	11.50
5	11.31	11.25	11.27	11.18	11.50	11.40	11.45	11.30	11.39	11.35
Mean	11.33	11.12	11.31	11.24	11.32	11.40	11.44	11.30	11.37	11.39
Median	11.32	11.15	11.31	11.24	11.30	11.40	11.44	11.30	11.36	11.35
Std.Dev.	0.03	0.12	0.05	0.05	0.11	0.00	0.04	0.00	0.02	0.08
Rel.Std.Dev.	0.24%	1.04%	0.43%	0.41%	0.97%	0.00%	0.31%	0.00%	0.18%	0.72%
PDM ³	-0.14%	-1.98%	-0.27%	-0.90%	-0.21%	0.49%	0.82%	-0.39%	0.21%	0.40%

Table A5. Results for Al₂O₃ in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	4.53	4.43	4.37	4.42	4.44	4.40	4.51	4.45	4.42	4.42
2	4.50	4.43	4.29	4.42	4.50	4.42	4.47	4.47	4.40	4.42
3	4.51	4.50	4.38	4.41	4.49	4.42	4.51	4.48	4.43	4.42
4	4.52	4.52	4.37	4.37	4.48	4.40	4.43	4.44	4.43	4.40
5	4.50	4.53	4.32	4.35	4.58	4.45	4.44	4.45	4.42	4.44
Mean	4.51	4.48	4.35	4.39	4.50	4.42	4.47	4.46	4.42	4.42
Median	4.51	4.50	4.37	4.41	4.49	4.42	4.47	4.45	4.42	4.42
Std.Dev.	0.01	0.05	0.04	0.03	0.05	0.02	0.04	0.02	0.01	0.01
Rel.Std.Dev.	0.29%	1.09%	0.89%	0.73%	1.14%	0.46%	0.84%	0.37%	0.28%	0.32%
PDM ³	1.59%	0.91%	-2.13%	-1.07%	1.27%	-0.53%	0.71%	0.37%	-0.48%	-0.48%

Table A6. Results for MgO in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	0.250	0.240	0.186	0.200	0.210	0.200	0.229	0.220	0.230	0.230
2	0.240	0.250	0.177	0.200	0.220	0.210	0.220	0.220	0.240	0.240
3	0.250	0.250	0.184	0.200	0.210	0.220	0.237	0.210	0.220	0.280
4	0.240	0.250	0.191	0.210	0.210	0.220	0.246	0.220	0.220	0.230
5	0.250	0.260	0.172	0.200	0.210	0.210	0.237	0.210	0.230	0.240
Mean	0.246	0.250	0.182	0.202	0.212	0.212	0.234	0.216	0.228	0.244
Median	0.250	0.250	0.184	0.200	0.210	0.210	0.237	0.220	0.230	0.240
Std.Dev.	0.005	0.007	0.007	0.004	0.004	0.008	0.010	0.005	0.008	0.021
Rel.Std.Dev.	2.23%	2.83%	4.05%	2.21%	2.11%	3.95%	4.26%	2.54%	3.67%	8.50%
PDM ³	10.97%	12.8%	-17.9%	-8.9%	-4.37%	-4.37%	5.48%	-2.56%	2.85%	10.07%

Table A7. Results for CaO in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	0.080	0.090	0.095	0.070	0.080	0.090	0.106	0.090	0.090	0.090
2	0.080	0.090	0.095	0.080	0.080	0.090	0.106	0.090	0.090	0.090
3	0.090	0.090	0.089	0.070	0.090	0.090	0.105	0.090	0.090	0.100
4	0.080	0.090	0.093	0.070	0.090	0.090	0.105	0.090	0.090	0.080
5	0.080	0.090	0.094	0.070	0.090	0.090	0.105	0.090	0.090	0.090
Mean	0.082	0.090	0.093	0.072	0.086	0.090	0.105	0.090	0.090	0.090
Median	0.080	0.090	0.094	0.070	0.090	0.090	0.105	0.090	0.090	0.090
Std.Dev.	0.004	0.000	0.002	0.004	0.005	0.000	0.000	0.000	0.000	0.007
Rel.Std.Dev.	5.45%	0.00%	2.49%	6.21%	6.37%	0.00%	0.04%	0.00%	0.00%	7.86%
PDM ³	-8.76%	0.14%	3.63%	-19.9%	-4.31%	0.14%	17.4%	0.14%	0.14%	0.14%

Table A8. Results for TiO₂ in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	0.170	0.170	0.175	0.180	0.180	0.180	0.176	0.180	0.180	0.180
2	0.170	0.180	0.179	0.180	0.170	0.180	0.176	0.180	0.190	0.180
3	0.170	0.180	0.177	0.180	0.170	0.180	0.176	0.180	0.180	0.180
4	0.170	0.180	0.175	0.180	0.170	0.180	0.176	0.190	0.190	0.170
5	0.170	0.180	0.178	0.180	0.170	0.180	0.176	0.180	0.180	0.180
Mean	0.170	0.178	0.177	0.180	0.172	0.180	0.176	0.182	0.184	0.178
Median	0.170	0.180	0.177	0.180	0.170	0.180	0.176	0.180	0.180	0.180
Std.Dev.	0.000	0.004	0.002	0.000	0.004	0.000	0.000	0.004	0.005	0.004
Rel.Std.Dev.	0.00%	2.51%	1.06%	0.00%	2.60%	0.00%	0.04%	2.46%	2.98%	2.51%
PDM ³	-4.78%	-0.30%	-0.89%	0.82%	-3.66%	0.82%	-1.52%	1.94%	3.07%	-0.30%

Table A9. Results for K₂O in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	1.65	1.64	1.77	1.72	1.67	1.72	1.28	1.71	1.69	1.67
2	1.67	1.64	1.76	1.71	1.67	1.73	1.27	1.72	1.68	1.68
3	1.67	1.68	1.76	1.72	1.67	1.73	1.26	1.72	1.69	1.69
4	1.64	1.67	1.76	1.73	1.69	1.71	1.27	1.71	1.71	1.68
5	1.67	1.68	1.74	1.71	1.69	1.72	1.26	1.71	1.66	1.69
Mean	1.66	1.66	1.76	1.72	1.68	1.72	1.26	1.71	1.69	1.68
Median	1.67	1.67	1.76	1.72	1.67	1.72	1.27	1.71	1.69	1.68
Std.Dev.	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.01
Rel.Std.Dev.	0.85%	1.07%	0.56%	0.49%	0.65%	0.49%	0.61%	0.32%	1.08%	0.50%
PDM ³	-2.21%	-2.27%	3.59%	1.21%	-1.15%	1.44%	-25.5%	0.97%	-0.68%	-0.91%

Table A10. Results for P₂O₅ in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	0.151	0.151	0.157	0.150	0.150	0.160	0.070	0.153	0.146	0.150
2	0.146	0.152	0.151	0.160	0.150	0.160	0.070	0.154	0.147	0.150
3	0.147	0.154	0.152	0.160	0.140	0.150	0.062	0.155	0.146	0.150
4	0.148	0.154	0.152	0.160	0.150	0.160	0.062	0.152	0.148	0.150
5	0.150	0.153	0.153	0.160	0.150	0.150	0.062	0.154	0.147	0.150
Mean	0.148	0.153	0.153	0.158	0.148	0.156	0.065	0.154	0.147	0.150
Median	0.148	0.153	0.152	0.160	0.150	0.160	0.062	0.154	0.147	0.150
Std.Dev.	0.002	0.001	0.002	0.004	0.004	0.005	0.005	0.001	0.001	0.000
Rel.Std.Dev.	1.40%	0.85%	1.50%	2.83%	3.02%	3.51%	7.44%	0.74%	0.57%	0.00%
PDM ³	-2.19%	0.71%	0.79%	4.13%	-2.46%	2.82%	-57.1%	1.23%	-3.25%	-1.14%

Table A11. Results for Na₂O in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A BF*XRF	Lab B BF*XRF	Lab C BF*XRF	Lab D BF*XRF	Lab E BF*XRF	Lab F BF*XRF	Lab G BF*XRF	Lab H BF*XRF	Lab I BF*XRF	Lab J BF*XRF
1	0.330	0.340	0.276	0.280	0.270	0.290	0.237	0.290	0.280	0.210
2	0.340	0.340	0.260	0.280	0.270	0.280	0.255	0.300	0.280	0.240
3	0.330	0.350	0.273	0.270	0.260	0.290	0.237	0.310	0.300	0.240
4	0.330	0.360	0.274	0.290	0.260	0.280	0.193	0.300	0.300	0.230
5	0.330	0.360	0.305	0.290	0.270	0.290	0.202	0.300	0.290	0.240
Mean	0.332	0.350	0.278	0.282	0.266	0.286	0.225	0.300	0.290	0.232
Median	0.330	0.350	0.274	0.280	0.270	0.290	0.237	0.300	0.290	0.240
Std.Dev.	0.004	0.010	0.017	0.008	0.005	0.005	0.026	0.007	0.010	0.013
Rel.Std.Dev.	1.35%	2.86%	5.99%	2.97%	2.06%	1.92%	11.61%	2.36%	3.45%	5.62%
PDM ³	20.1%	26.6%	0.44%	2.04%	-3.75%	3.49%	-18.6%	8.56%	4.94%	-16.0%

Table A12. Results for LOI in OREAS 170a (abbreviations as in Table A1; values in wt.%).

Replicate No.	Lab A LOI	Lab B LOI	Lab C LOI	Lab D LOI	Lab E LOI	Lab F LOI	Lab G LOI	Lab H LOI	Lab I LOI	Lab J LOI
1	12.05	12.15	12.14	13.48	12.00	12.10	12.04	12.10	12.23	12.39
2	12.05	12.25	12.19	13.42	12.00	12.00	12.08	12.20	12.23	12.41
3	12.10	12.70	12.18	13.46	11.70	12.10	12.11	12.10	12.22	12.42
4	12.10	12.20	12.20	13.47	11.80	12.00	12.13	12.10	12.21	12.45
5	12.15	12.20	12.15	13.47	11.90	12.20	12.11	12.10	12.19	12.38
Mean	12.09	12.30	12.17	13.46	11.88	12.08	12.09	12.12	12.22	12.41
Median	12.10	12.20	12.18	13.47	11.90	12.10	12.11	12.10	12.22	12.41
Std.Dev.	0.04	0.23	0.03	0.02	0.13	0.08	0.04	0.04	0.02	0.03
Rel.Std.Dev.	0.35%	1.84%	0.21%	0.17%	1.10%	0.69%	0.29%	0.37%	0.14%	0.22%
PDM ³	-0.40%	1.33%	0.25%	10.9%	-2.13%	-0.48%	-0.37%	-0.15%	0.64%	2.24%