

## QOTHO CERTIFIED REFERENCE MATERIAL (QCRM)

### QCRM-4-101

IRON ORE - LOW GRADE

### CERTIFICATE OF ANALYSIS

CERTIFIED VALUES			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
Al <sub>2</sub> O <sub>3</sub>	%	2.77	±0.04
CaO	%	0.028	±0.004
Fe	%	59.16	±0.13
K <sub>2</sub> O	%	0.42	±0.01
LOI (950°C)	%	1.05	±0.07
MgO	%	0.038	±0.006
Mn	%	0.54	±0.01
Na <sub>2</sub> O	%	0.024	±0.009
P	%	0.039	±0.002
S	%	0.014	±0.002
SiO <sub>2</sub>	%	9.86	±0.05
TiO <sub>2</sub>	%	0.14	±0.01
V <sub>2</sub> O <sub>5</sub>	%	0.010	±0.002
ASSIGNED VALUES (FOR INFORMATION ONLY)			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
Cr <sub>2</sub> O <sub>3</sub>	%	0.02	±0.01
SG	g/cm <sup>3</sup>	4.64	±0.34

**1. Use:**

QCRM-4-101 is a certified reference material which is suitable for use as random control samples in routine analytical laboratory quality control, when inserted within a batch of samples and measured in parallel to the unknown. The QCRM can also be used as a control sample in the analysis of samples of a similar type, verification of analytical methods for Iron Ore and as a calibration standard for the calibration of equipment used for analysing similar materials.

**2. Origin of Material:**

This standard was sponsored by Assmang Khumani Mine.

**3. Mineral and Chemical Composition:**

This Hematite iron ore deposit is classified as the Superior-Type Banded Iron Formation of the Transvaal Supergroup. It consists of ferruginous conglomerates of the Gamagara Formation which unconformably overlies supergene-enriched iron formation of the Asbestos Hill Subgroup. The Iron (Fe) containing assemblages of this material are made up of 92% Hematite and 2% Goethite with minor to trace amounts of limonite.

**4. Date of Initial Issue:**

10 February 2022.

**5. Packaging & Handling instructions:**

The material was packaged as 100g unit sizes, placed in geo-envelopes, within a vacuum sealed aluminum foil bag. Open the seal of the packaging with care and shake or otherwise agitate prior to use. Normal safety precautions for handling fine particulate matter are recommended, such as the use of safety glasses, breathing protection, gloves, and a laboratory coat. Once opened, material must be stored in a cool, dry environment. Results on page 1 is presented on dry basis. Analysis should therefore be done on dry basis, after drying to constant mass, at 105 °Celsius. Special packaging into 500g plastic tubs, where done for the client, following certification

**6. Method of Preparation:**

The material was sieved through a 75-micron screen and the oversize was re-milled to ensure 100% passing through the screen. It was then blended, systematically divided, and packaged into 100-gram zip-lock bags. Randomly selected samples, from the bags, were tested in-house via XRF, to confirm homogeneity. Once confirmed and certification completed, the items were placed in geo-envelopes and vacuum sealed in aluminium foil bags.

**7. Methods of Analysis used:**

- Sodium peroxide fusion with ICP-OES finish
- Multi-acid digestion with ICP-OES finish
- Iron by sodium peroxide fusion with Potentiometric Titration
- Iron by multi-acid digestion and Potentiometric Titration
- Silica by gravimetric analysis
- Phosphorous by multi-acid digestion and UV-VIS finish
- LOI by Thermographic analysis
- Fused beads with XRF finish
- Pressed pellet with XRF finish
- Sulphur by combustion analysis.

**8. Analysis required:**

An instruction letter was sent to all participants. The analysis required was noted in the instruction letter and reporting template, including but not limited to Al<sub>2</sub>O<sub>3</sub>, CaO, Cr<sub>2</sub>O<sub>3</sub>, Fe, K<sub>2</sub>O, LOI (950°C), MgO, Mn, Na<sub>2</sub>O, P, S, SiO<sub>2</sub>, TiO<sub>2</sub> and V<sub>2</sub>O<sub>5</sub>.

**9. Participating Laboratories:**

NO	LABORATORY	COUNTRY
1.	AHK Richards Bay	South Africa
2.	ALS Geochemistry Kempton Park	South Africa
3.	Anglo Technical Solutions	South Africa
4.	Assmang Beeshoek	South Africa
5.	Assmang King Laboratory	South Africa
6.	Assmang Parsons Laboratory	South Africa
7.	Bureau Veritas Minerals Perth	Australia
8.	Bushveld Vametco	South Africa
9.	Cawood Laboratories	South Africa
10.	Dorowa Minerals	Zimbabwe
11.	FLSmith South Africa	South Africa
12.	Intertek Genalysis Australia	Australia
13.	Intertek Kathu	South Africa
14.	Kolomela Laboratory	South Africa
15.	Metex Sishen	South Africa
16.	Mitra Sk South Africa	South Africa
17.	MSALABS Canada	Canada
18.	Palabora Mining Company	South Africa
19.	QLS Northern Cape	South Africa
20.	SABS PTA	South Africa
21.	Sishen Iron Ore Fusion	South Africa
22.	UIS Analytical Services GEO	South Africa
23.	UIS Analytical Services ICP	South Africa
24.	UIS Analytical Services XRF	South Africa

**10. Assay Data:**

Data used for Assigning Values and Certification.

LABORATORY	Al <sub>2</sub> O <sub>3</sub>	CaO	Cr <sub>2</sub> O <sub>3</sub>	Fe	K <sub>2</sub> O	LOI (950 °C)	MgO	Mn	Na <sub>2</sub> O	P	S	SG	SiO <sub>2</sub>	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>
UNIT	%	%	%	%	%	%	%	%	%	%	%	g/cm <sup>3</sup>	%	%	%
LAB001		0.004	0.028												
LAB002				58.985				0.58		0.04	0.019				
LAB003	2.862	0.029		59.344	0.428		0.029			0.042	0.013			0.153	0.016
LAB004	2.45	0.023			0.418		0.05	0.502	0.014		0.012			0.142	
LAB005	2.77	0.03	0.008	59.21	0.43	1.025	0.045	0.53	0.04	0.038	0.013		9.905	0.145	0.009
LAB006	2.75	0.03	0.008	59.175	0.423	1.03	0.035	0.535	0.01	0.039	0.014		9.935	0.145	0.01
LAB007	2.793	0.027		59.504	0.427		0.03	0.534		0.038	0.014			0.14	
LAB008				58.844	0.415	1.111				0.041			9.933		
LAB009				58.975											
LAB010		0.04	0.02	59.7			0.02			0.037				0.135	0.01
LAB011				59.38											
LAB012			0.028				0.034								0.011
LAB013									0.097						
LAB014			0.015												
LAB015		0.035		58.975			0.064	0.53		0.036	0.014			0.132	
LAB016	2.785	0.03		58.75	0.427	0.865	0.02	0.532		0.037	0.02		9.775	0.14	
LAB017	2.742			59.22	0.415	1.095	0.042	0.529	0.041	0.037	0.014	4.637	9.924	0.141	0.008
LAB018	2.78	0.045	0.046	59.175	0.419	1.04	0.05	0.534	0.02	0.039				0.16	0.01
LAB019	2.87	0.034		58.915			0.069	0.53		0.037	0.014			0.134	
LAB020	2.76	0.025	0.009	58.885	0.428		0.025	0.54	0.033	0.038	0.01		9.78	0.14	0.011
LAB021	2.784	< 0.022		59.34	0.411	1.198	< 0.027	0.532	< 0.032	0.038	0.016	4.62	9.816	0.143	0.01
LAB022		0.035	0.02	59.71			0.03		0.03					0.145	0.01
LAB023				58.355											
LAB024				59.23	0.43			0.58		0.038	0.018				
LAB025		0.027	0.037	59.127	0.412	1.168	0.034	0.538		0.039			9.966	0.141	

LABORATORY	Al <sub>2</sub> O <sub>3</sub>	CaO	Cr <sub>2</sub> O <sub>3</sub>	Fe	K <sub>2</sub> O	LOI (950 °C)	MgO	Mn	Na <sub>2</sub> O	P	S	SG	SiO <sub>2</sub>	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>
UNIT	%	%	%	%	%	%	%	%	%	%	%	g/cm <sup>3</sup>	%	%	%
LAB026				59.3											
LAB027	2.77	0.04	0.009	59.195	0.426	0.96	0.04	0.54	0.035	0.039	0.015	4.75	9.905	0.145	0.009
LAB028			0.034					0.835							
LAB029	2.624	0.028		59.303	0.411	1.375		0.513		0.042				0.14	
LAB030				57.535											
LAB031	2.808	0.026		59.101	0.43	1.051	0.05	0.529		0.039	0.015		9.938	0.136	0.019
LAB032	2.775	0.035	0.007	59.305	0.42	0.985	0.045	0.535	0.035	0.036	0.016		9.935	0.15	0.009
LAB033	2.71	0.02		59.045	0.42	1.01	0.05	0.571		0.038					
LAB034	2.821	0.028		59.285	0.432		0.024	0.397		0.041	0.015			0.148	0.007
LAB035	2.662	0.022		58.817	0.432		0.051	0.503	0.035		0.013			0.143	
LAB036			0.04				0.044	0.235							0.005
LAB037									0.009						
LAB038	2.698	0.02	0.008	59.309		1.05		0.521	< 0.010			4.3	9.625		< 0.010
LAB039				59.648						0.038					
LAB040				58.578	0.409			0.538				4.93		0.131	
LAB041	2.808	0.029		59.541	0.436		0.033	0.532		0.04	0.013			0.142	
LAB042	2.447	0.021		58.99	0.41	0.977	0.022	0.553	0.02		0.012		9.298	0.135	0.011
LAB043	2.745			58.935	0.45	0.977		0.53	0.022	0.037	0.014		9.543	0.141	
Lab044	2.768	0.031	0.016	59.069	0.428	1.153	0.048	0.554		0.037		4.606	9.792	0.144	0.009

## 11. Method of Certification:

### QM is a SANAS Accredited Proficiency Testing Scheme Provider, No. PTS0012

This material was distributed as test items, in the Qotho Iron PT Round 5 and 6 of 2021. The participating laboratories were each given 1 randomly selected sample from the batch, to analyse and report on in duplicate. Some laboratories reported results via more than one analytical method. Obvious blunders were resolved with the laboratories (if any), after which the data was processed using Robust Statistics, through PROLab Plus.

Not all the participating laboratories were accredited. Historical performance in Qotho PT Schemes, as well as an evaluation of the CRM QA/QC data generated by the laboratories, during the analysis of this QRM, were considered, to evaluate the competence of laboratories. Where competence could not be confirmed, the affected data was deselected from the dataset. Certification of analytes was then done, provided that a minimum of 10 qualifying datapoints remained available.

Where analytes could not be certified, estimate concentrations were assigned, using all the data in the dataset.

## 12. Measurement of Uncertainty:

Standard uncertainty,  $u_{CRM}$ , was calculated according to ISO 13528:2015 (equation 6), and it includes the effects of uncertainty due to inhomogeneity, transport, potential instability, and laboratory uncertainty. Because of all the uncertainties under consideration, QM further applies an expanded uncertainty, for certification purposes. The measurement uncertainty of the certified value is therefore calculated as follows:  $U_{CRM} = k u_{CRM}$ , where  $k$  is a coverage factor, which is determined from the Student's  $t$ -distribution, based on the degrees of freedom, per analyte.

This presents a certified value, as follows:  $x_{CRM} \pm U_{CRM}$ .

Measurement uncertainty for Assigned values, are calculated in the same manner.

For laboratories prefer to use the 95% measurement uncertainty, rather than the expanded uncertainty, all available information relating to measurement uncertainty of the certified/assigned values, are given below:

Analyte	Unit of measure	$s$ (Standard Deviation of Dataset)	$v$ (Degrees of Freedom)	$k$ (Coverage Factor)	$u$ (Standard Uncertainty)	95% Measurement Uncertainty	Expanded Uncertainty
Al <sub>2</sub> O <sub>3</sub>	%	0.069	22	2.074	0.016	± 0.03	± 0.04
CaO	%	0.008	25	2.060	0.002	± 0.004	± 0.004
Cr <sub>2</sub> O <sub>3</sub>	%	0.006	9	2.262	0.001	± 0.01	± 0.01
Fe	%	0.309	35	2.030	0.065	± 0.13	± 0.13
K <sub>2</sub> O	%	0.01	24	2.064	0.002	± 0.01	± 0.01
LOI (950 °C)	%	0.091	16	2.120	0.032	± 0.06	± 0.07
MgO	%	0.015	25	2.060	0.003	± 0.006	± 0.006
Mn	%	0.018	28	2.048	0.003	± 0.01	± 0.01
Na <sub>2</sub> O	%	0.017	15	2.131	0.004	± 0.008	± 0.009
P	%	0.002	25	2.060	0.001	± 0.002	± 0.002
S	%	0.002	20	2.086	0.001	± 0.002	± 0.002
SG	g/cm <sup>3</sup>	0.266	5	2.571	0.132	± 0.26	± 0.34
SiO <sub>2</sub>	%	0.117	14	2.145	0.022	± 0.04	± 0.05
TiO <sub>2</sub>	%	0.006	25	2.060	0.002	± 0.01	± 0.01
V <sub>2</sub> O <sub>5</sub>	%	0.002	17	2.110	0.001	± 0.002	± 0.002

**13. Metrological Traceability:**

The values quoted herein are based on the consensus values derived from statistical analysis of the data from an inter laboratory measurement program. Traceability to SI units is via the accredited laboratories, as ISO 17025 requires laboratories to use CRM's traceable to the SI units, during the calibration of their equipment. Not all laboratories were accredited.

Fortunately, most laboratories reported on the QA/QC CRMs used during the analysis of this QRM and reported the values obtained during the sample run. Evaluation of their QA/QC performance serves as further evidence of metrological traceability.

Equivalence tests were performed on all analytes to determine whether the metrologically traceable data and those for which traceability evidence was not available, could be treated as equal (at a level of significance of  $\alpha = 0.05$ ). Where equivalent, all the data was used. Where not equivalent, only the metrologically traceable data was considered.

**14. Minimum sample size:**

The recommended minimum sample size for the use of this material is as per the participants method validation criteria.

**15. Period of validity:**

The certified values are valid for this product, while still sealed in its original packaging, for a minimum period of 5 years from date of Initial Certification. Stability monitoring of inventory will be done at regular intervals. Any concerns regarding potential instability of the material, will immediately be communicated to all consumers.

**16. Legal:**

This certificate and the reference material described in it were prepared with due care and attention. The requirements of ISO Guide 31, ISO 17043 and ISO 17034 were followed in the preparation of this reference material and certificate of analysis.

Qotho Minerals, however, accepts no liability for any decisions or actions taken following the use of the reference material. The company has a complaints procedure, which will be made available upon request, should there be any dissatisfaction with either the product or the COA.

Certifying & Technical Signatory	
<b>Qotho Managing Director</b>	<b>10 February 2022</b>

*This Certificate of Analysis (CoA) has been electronically signed using an Advanced Electronic Signature (AES) in terms of the Electronic Communications and Transactions Act No. 15, 2002 (ECT Act). Any amendments to the CoA can be detected by reference to the Signature Panel displayed in the electronic pdf version of the CoA.*

END