

QOTHO CERTIFIED REFERENCE MATERIAL (QCRM)

QCRM-2-134

MANGANESE HIGH GRADE

CERTIFICATE OF ANALYSIS

CERTIFIED VALUES			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
CaO	%	4.99	±0.14
FeO	%	12.83	±0.18
K ₂ O	%	0.03	±0.02
MgO	%	1.27	±0.05
Mn	%	47.43	±0.23
P	%	0.029	±0.002
S	%	0.08	±0.03
SiO ₂	%	6.03	±0.07
ASSIGNED VALUES (FOR INFORMATION ONLY)			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
Al ₂ O ₃	%	0.35	±0.04
BaO	%	0.36	±0.02

1. Use:

QCRM-2-134 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 Manganese Ore and as a calibration standard for the calibration of equipment used for analyzing similar materials.

2. Origin of Material:

This material was sponsored by Assmang Blackrock Manganese Mine, located approximately 14 km north west of Hotazel, in the Northern Cape Province, South Africa.

3. Mineral and Chemical Composition:

The manganese ores of the Kalahari Manganese field are contained within sediments of the Hotazel Formation of the Griqualand West Sequence, a subdivision of the Proterozoic Transvaal Supergroup. The average thickness of the Hotazel Formation is approximately 40 meters. The manganese ore bodies exhibit a complex mineralogy and more than 200 mineral species have been identified to date. The hydrothermal upgrading has resulted in a zoning of the orebody with regard to fault positions. The ore is rich in Hausmannite, Bixbyite and Braunite II. It consists of shales, siltstones, quartzites, subordinate carbonates and basaltic to amygdaloidal lavas. This standard is a physically upgraded ore.

4. Date of Initial Issue:

9 September 2020.

5. Packaging & Handling instructions:

The material is packaged as 100g geo envelopes, within a vacuum sealed aluminum foil bag. Open the seal of the foil 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 degrees Celsius.

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
- 4-acid digestion with Potentiometric Titration
- Aqua Regia Digestion with Potentiometric Titration
- Hydrochloric Acid Digestion with Potentiometric Titration
- Fused beads/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_2O_3 , BaO , CaO , FeO , K_2O , MgO , Mn , P , S , SiO_2 .

9. Participating Laboratories:

No	LABORATORY	COUNTRY
1.	AHK Kitwe	Zambia
2.	AHK Richards Bay	South Africa
3.	ALS Geochemistry Kempton Park	South Africa
4.	ARM Machadodorp	South Africa
5.	Assmang Blackrock	South Africa
6.	Intertek JHB	South Africa
7.	Manganese Metal Company	South Africa
8.	Mitra SK South Africa	South Africa
9.	SGS Malaysia	Malaysia
10.	SGS Netherlands BV	Netherlands
11.	SGS Randfontein	South Africa
12.	South32 Mamatwan	South Africa
13.	South32 Wessels	South Africa
14.	UIS Analytical Services	South Africa

10. Assay Data:

Data used for Assigning Values and Certification.

Laboratory	Al ₂ O ₃	BaO	CaO	FeO	K ₂ O	MgO	Mn	P	S	SiO ₂
Unit	%	%	%	%	%	%	%	%	%	%
LAB001	0.395	0.379	5.056	12.898	0.032	1.258	47.798	0.029		6.127
LAB002	0.325	0.355	5.14	12.857	0.042	1.369	47.431	0.029		6.096
LAB003	0.351			12.809			47.66	0.005		6.655
LAB004							47.265			
LAB005	0.353	0.355	5.11	12.84	0.041	1.327	47.51	0.03	0.106	6.075
LAB006	0.395	0.38	5.02	12.98	0.04	1.28	47.335	0.029	0.077	6.095
LAB007	0.32	0.35	4.965	12.55	0.018	1.03	47.62	0.02	0.09	5.89
LAB008	0.21			14.59	0.01	0.945	47.92	0.029		
LAB009	0.235	0.48	5.275	13.08	0.018		48.02	0.031	0.053	
LAB010	0.405		5.075	12.635	0.028	1.175	47.51	0.029	0.045	6.025
LAB011							47.655			
LAB012	0.4		5.025	13.02		1.28	47.5	0.028	0.092	6.155
LAB013	0.257	0.363	4.706	12.615	0.046		47.25		0.099	
LAB014	0.372		4.801	12.747		1.252	48.065			5.864
LAB015							47.087			
LAB016							47.097			
LAB017	0.41	0.37	4.86	13.095	0.055	1.285	47.165	0.03	0.109	6.06
LAB018	0.345	0.36	4.98	12.71	0.03	1.255	47.127	0.026	0.09	6.065
LAB019							46.56			
LAB020	0.345		4.9		0.041	1.26	46.8		0.075	5.885

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 Manganese PT Round 2 of 2020. The participating laboratories were each given one randomly selected sample from the batch, to analyze and report on in duplicate. The data was collated, after which it was processed using Robust Statistics, through PROLab Plus.

Not all the participating laboratories were accredited. Equivalence tests were performed on all analytes to determine whether the data from the accredited and non-accredited laboratories, can be treated as equal (at a level of significance of $\alpha = 0.05$). Where equivalent, all the data was used. Where not, only the data from the accredited laboratories were considered. Certification of analytes were then done, provided that a minimum of 10 datapoints remained available.

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

12. Measurement of Uncertainty:

Measurement 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. $U_{CRM} = k u_{CRM}$, 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 in the following table:

Analyte	Unit of measure	ν (degrees of freedom)	k (coverage factor)	u (standard error)	95% measurement uncertainty	Expanded Uncertainty
Al ₂ O ₃	%	14	2.145	0.018	± 0.04	± 0.04
BaO	%	8	2.306	0.007	± 0.01	± 0.02
CaO	%	12	2.179	0.062	± 0.12	± 0.14
FeO	%	13	2.160	0.084	± 0.17	± 0.18
K ₂ O	%	11	2.201	0.007	± 0.01	± 0.02
MgO	%	11	2.201	0.021	± 0.04	± 0.05
Mn	%	19	2.093	0.111	± 0.22	± 0.23
P	%	11	2.201	0.001	± 0.002	± 0.002
S	%	9	2.262	0.01	± 0.02	± 0.03
SiO ₂	%	11	2.201	0.029	± 0.06	± 0.07

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. Most laboratories reported on the QA/QC CRM's used during the analysis of this QRM and reported the values obtained during the sample run. This provides additional evidence of measurement traceability.

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 have been 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/Analytical Report.

Certifying & Technical Signatory	
Qotho Managing Director	9 September 2020

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.

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