

QOTHO CERTIFIED REFERENCE MATERIAL (QCRM)

QCRM-1-204

FERROCHROME

CERTIFICATE OF ANALYSIS

CERTIFIED VALUES			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
Al	%	0.05	±0.01
C	%	6.10	±0.02
Co	%	0.059	±0.002
Cr	%	46.62	±0.09
Fe	%	37.56	±0.14
Mn	%	0.85	±0.02
Ni	%	0.184	±0.004
P	%	0.019	±0.002
S	%	0.074	±0.002
Si	%	5.96	±0.08
Ti	%	0.56	±0.01
V	%	0.40	±0.01
ASSIGNED VALUES (FOR INFORMATION ONLY)			
ANALYTES	UNITS	CONCENTRATIONS	EXPANDED UNCERTAINTY
Mg	%	0.04	±0.03

1. Use:

QCRM-1-204 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 Ferrochrome and as a calibration standard for the calibration of equipment used for analysing similar materials.

2. Origin of Material:

This standard was sponsored by Samancor Dikwena.

3. Mineral and Chemical Composition:

The material is not naturally occurring, but it is a product of Pyrometallurgical processing of the chromite ore or foundry sand to yield a ferrochrome alloy. From the molten alloy state, this sample is cast and crushed into a lump size product of <2mm in size. The alloy has main phases of (Cr,Fe)₇C₃, (Cr,Fe)₂₃C₆, traces (Fe,Cr)₃C carbides, Cr containing α-Fe, Cr-Si and traces of S, P, along with minor (<0,5%) amounts of Mn, V, Ti - all species are present in alloy form.

4. Date of Initial Issue:

25 October 2021.

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 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 °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
- Sodium peroxide fusion with Potentiometric Titration
- Phosphorous by perchloric digestion and UV-VIS finish
- Phosphorous by Aqua Regia digestion with ICP-OES finish
- 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, C, Co, Cr, Fe, Mg, Mn, Ni, P, S, Si, Ti and V.

9. Participating Laboratories:

NO	LABORATORY	COUNTRY
1.	Afarak Elektrowerk Weisweiler GmbH	Germany
2.	AHK North West	South Africa
3.	AHK Richards Bay	South Africa
4.	ALS Inspection Richards Bay	South Africa
5.	ARM Machadodorp	South Africa
6.	Columbus Analytical Lab	South Africa
7.	Glencore Boshhoek	South Africa
8.	Glencore Lion	South Africa
9.	Glencore Rustenburg Smelter	South Africa
10.	Glencore Wonderkop Smelter	South Africa
11.	Mintek	South Africa
12.	Mitra SK South Africa	South Africa
13.	PCL Rustenburg	South Africa
14.	PCL Steelpoort	South Africa
15.	Samancor Dikwena	South Africa
16.	Samancor Ferrometals	South Africa
17.	Samancor TCS Laboratory	South Africa
18.	Samancor Tubatse Alloy Smelter	South Africa
19.	Samancor Tubatse Chrome	South Africa
20.	SGS Netherlands BV	Netherlands
21.	SGS Richards Bay	South Africa
22.	Zimasco Kwekwe	Zimbabwe
23.	Zimbabwe Alloys Chrome	Zimbabwe

10. Assay Data:

Data used for Assigning Values and Certification.

LABORATORY	Al	C	Co	Cr	Fe	Mg	Mn	Ni	P	S	Si	Ti	V
UNIT	%	%	%	%	%	%	%	%	%	%	%	%	%
LAB001		6.065	0.06	46.665	37.57		0.864	0.185	0.02	0.074	6.035	0.554	0.397
LAB002				46.62									
LAB003	0.045	6.09	0.058		37.47		0.831	0.193	0.021	0.071	6.015	0.55	0.396
LAB004				46.895									
LAB005	0.025	6.088	0.06		37.655		0.823	0.199		0.079	6.175	0.565	0.404
LAB006				47.025									
LAB007		6.125			37.475		0.815	0.174	0.018	0.073	5.735	0.547	0.42
LAB008				46.48									
LAB009	0.068	6.14	0.056	46.795	38.495		0.845	0.185	0.023	0.073	5.945	0.575	0.41
LAB010				46.53									
LAB011		6.095	0.059	46.605	37.725		0.853	0.182	0.02	0.075	5.995	0.567	0.398
LAB012				46.465									
LAB013		6.104	0.058	46.68	37.603		0.848	0.175	0.017	0.071	5.857	0.555	0.397
LAB014				46.747									
LAB015		6.045	0.06	46.69	37.77	0.105	0.865	0.185	0.02	0.075	6.025	0.57	0.395
LAB016				46.74									
LAB017		5.91		46.55	37.55	0.06		0.18		0.065		0.55	0.36
LAB018	0.055				37.17	0.03	0.84				5.895		0.395
LAB019				45.97									
LAB020	0.067	6.105	0.06		37.73		0.715	0.18	0.018	0.071	5.825	0.555	0.39
LAB021				47.18									
LAB022	0.074	6.1	0.059	46.8	37.6		0.85	0.18	0.02	0.072	5.9	0.565	0.385

LABORATORY	Al	C	Co	Cr	Fe	Mg	Mn	Ni	P	S	Si	Ti	V
UNIT	%	%	%	%	%	%	%	%	%	%	%	%	%
LAB023		6.065		46.485	37.625				0.02	0.077			
LAB024				46.63									
LAB025		6.095	0.059	46.471	37.696	0.043	0.835	0.183	0.017	0.074	5.992		0.404
LAB026				46.775									
LAB027	0.042	6.125		46.47	37.88					0.069	5.825		
LAB028	0.043	6.08	0.054	46.938	37.026	0.033		0.182	0.021	0.08	5.925	0.554	0.419
LAB029				46.523									
LAB030	0.042	6.095	0.059	46.65	37.595		0.765		0.018	0.072	6	0.545	0.4
LAB031	0.011		0.064				0.835	0.198	0.02		5.935	0.61	0.421
LAB032				46.32									
LAB033		6.11			37.4		0.844		0.021	0.071	5.99		
LAB034				46.4									
LAB035				46.55	39.15				0.024				
LAB036	0.063	6.107	0.057	46.519	37.412	0.026	0.883	0.19	0.016	0.073	6.196	0.545	0.411
LAB037	0.044	6.117	0.061	46.451	37.327	0.013	0.873	0.186	0.015	0.08	6.102	0.561	0.391
LAB038	0.04	6.115		46.665	37.635	0.017	0.871	0.18	0.015	0.106	5.905	0.545	

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 Chrome PT Round 5 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:

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 below:

Analyte	Unit of measure	ν (degrees of freedom)	k (coverage factor)	u (standard error)	95% measurement uncertainty	Expanded Uncertainty
Al	%	12	2.179	0.005	± 0.01	± 0.01
C	%	19	2.093	0.008	± 0.02	± 0.02
Co	%	14	2.145	0.001	± 0.002	± 0.002
Cr	%	30	2.042	0.043	± 0.09	± 0.09
Fe	%	21	2.080	0.064	± 0.13	± 0.14
Mg	%	7	2.365	0.013	± 0.03	± 0.03
Mn	%	17	2.110	0.008	± 0.02	± 0.02
Ni	%	16	2.120	0.002	± 0.004	± 0.004
P	%	18	2.101	0.001	± 0.002	± 0.002
S	%	19	2.093	0.001	± 0.002	± 0.002
Si	%	19	2.093	0.035	± 0.07	± 0.08
Ti	%	16	2.120	0.003	± 0.01	± 0.01
V	%	17	2.110	0.004	± 0.01	± 0.01

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, 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	
Qotho Managing Director	25 October 2021

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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