MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 370
MAGNESITE - CHROME

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, issued by the Bureau of Analysed Samples Ltd.

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

1 BENNETT, H., *BSc, ARIC, FI Ceram,* British Ceramic Research Association Limited, Stoke-on-Trent.

2 COPPINS, W. C., *MSc, FRIC,* Ridsdale and Co. Ltd., Middlesbrough.

ANALYSTS representing MANUFACTURERS and USERS

3 DAVEY, J., *AIM,* BSC Research Centre (Teesside), Middlesbrough.

ANALYSTS representing MANUFACTURERS and USERS (cont.)

4 FLETCHER, B., *LRIC, LI Ceram,* Pickford, Holland & Co. Ltd., Sheffield.

5 SANDERSON, J., *ARIC,* BSC, Consett Works, Consett.

6 SHELTON, N. F. C., *LRIC,* General Refractories Ltd., Worksop.

7 WILBURN, P., *ARIC,* Simon Engineering Ltd., Stockport.

ANALYSES

Mean of 4 values - mass content in %. All results relate to the ignited (1000°C) sample.

Analyst No.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	Cr_2O_3	Na_2O	K_2O	Li_2O
1	2.96	12.23	0.12	7.16	0.10	1.57	62.0	13.58	0.04	0.04	0.02
2	3.02	12.28	0.12	7.19	0.13	1.56	61.6	13.52	0.06	0.03	0.03
3	3.06	12.17	0.16	7.22	0.12	1.55	61.7	13.40	0.07	0.04	0.04
4	3.02	12.24	0.12	7.33	0.10	1.54	61.8	13.52	0.06	0.03	0.02
5	3.03	12.50	0.11	7.30	0.08	1.52	62.0	13.10	0.05	0.03	0.03
6	2.97	12.26	0.11	7.25	0.12	1.54	61.8	13.44	0.05	0.02	0.02
7	3.01	12.30	0.15	7.16	0.09	1.52	62.0	13.28	0.07	0.03	0.03
M_M	**3.01**	**12.3**	**0.13**	**7.23**	**0.11**	**1.54**	**61.8**	**13.4**	**0.06**	**0.03**	**0.03**
s_M	0.04	0.2	0.02	0.07	0.02	0.02	0.2	0.2	0.02	0.01	0.01

The above figures are those which each Analyst has decided upon after careful verification.
M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means.

Analyst No. 1 supplied the following additional information obtained by spectrographic analysis:
SrO <*0.01*%, BaO <*0.01*%, NiO *0.1*%, Co_3O_4 *0.02*%, CuO <*0.01*%, V_2O_5 *0.15*%, MnO *0.1*%.

CERTIFIED VALUES (Cv)

mass content in %

	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	Cr_2O_3	Na_2O	K_2O	Li_2O
Cv	**3.01**	**12.3**	**0.13**	**7.23**	**0.11**	**1.54**	**61.8**	**13.4**	**0.06**	**0.03**	**0.03**
C(95%)	0.04	0.1	0.02	0.07	0.02	0.02	0.2	0.2	0.02	0.01	0.01

The half width confidence interval **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate two sided Student's t value at the 95% confidence level for "n" acceptable mean values.

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 sections 6.1 and 10.5.2.

BCS-CRM No. 370 MAGNESITE - CHROME

NOTES ON METHODS USED

SILICA

Analysts Nos. 1, 5 and 7 fused the sample with alkali carbonate/boric acid and dissolved in sulphuric acid. Ammonium molybdate was added and the silica precipitated with quinoline; the determinations were completed gravimetrically. Nos. 2, 4 and 6 determined silica gravimetrically after decomposition by acid attack with sulphuric/perchloric acid (No. 2), nitric/perchloric acid (No. 4) or perchloric acid only (No. 6). Nos. 2 and 4 volatilized the bulk of the chromium as chromyl chloride before dehydrating the silica. No. 2 recovered the residual silica in the filtrate by means of a second dehydration whereas No. 4 determined it photometrically as molybdenum-blue. No. 3 fused the sample with alkali carbonate/boric acid and dissolved in sulphuric acid; chromium was removed with a liquid ion-exchange resin and silica determined photometrically as silicomolybdate yellow.[1]

Analyst No. 6 also determined silica by direct-reading spectroscopy* and found 3.01%.

ALUMINA

Analysts Nos. 1, 2, 3 and 7 fused the sample with alkali carbonate/boric acid and dissolved in sulphuric acid. Chromium was separated with a liquid ion-exchange resin and other interfering elements were removed by solvent extraction with diethyldithiocarbamate and cupferron. The determinations were completed titrimetrically with CDTA. Nos. 4, 5 and 6 used similar methods except that EDTA was used instead of CDTA and corrections were applied for residual chromium.

TITANIA

All Analysts except No. 6 determined titania photometrically with hydrogen peroxide. Nos. 2 and 7 carried out preliminary separation of the titanium with cupferron. No. 3 carried out a preliminary separation of the chromium by solvent extraction with hexone.

Analyst No. 6 also determined titania by direct-reading spectroscopy* and found 0.11%.

FERRIC OXIDE

Analysts Nos. 1, 4, 6 and 7 determined iron photometrically. No. 1 fused the sample with alkali carbonate/boric acid, dissolved in sulphuric acid and separated chromium with a liquid ion-exchange resin; iron was then determined photometrically with 1,10 phenanthroline. Nos. 6 and 7 used a similar method without separation of chromium.[1] No. 4 determined iron photometrically with thioglycollic acid. Nos. 2 and 3 determined the iron titrimetrically by titration with dichromate after reduction with hydrogen sulphide. No. 5 determined iron titrimetrically with dichromate after reduction with stannous chloride.

MANGANOUS OXIDE

All Analysts determined manganous oxide photometrically by oxidation with periodate.

Analyst No. 6 also determined manganous oxide by direct-reading spectroscopy* and found 0.12%.

LIME

Analyst No. 1 separated chromium with a liquid ion-exchange resin and after addition of triethanolamine to complex interfering elements, excess of EGTA was added and the solution made alkaline. The magnesium hydroxide was filtered off after addition of a flocculating agent and the excess EGTA back-titrated with standard calcium solution. Nos. 2 and 6 separated chromium with liquid ion-exchange resin, removed other interfering elements by solvent extraction with diethyldithiocarbamate and cupferron and completed titrimetrically in the same way as No. 1. Nos. 3 and 4 used a similar method but titrated with EDTA/calcium solution without filtration.[1] No. 7 used a similar method to Nos. 3 and 4 but titrated directly with EDTA after making alkaline and adding a flocculating agent. No. 5 took the sample into solution by decomposition with perchloric acid, separated Group III elements with ammonia and determined lime gravimetically via the oxalate.

Analyst No. 6 also used atomic-absorption spectroscopy and found 1.67%.

MAGNESIA

All Analysts except No. 5 determined magnesia titrimetrically. Nos. 1, 2, 3, 6 and 7 separated chromium with a liquid ion-exchange resin and other interfering elements by solvent extraction with diethyldithiocarbamate and cupferron. The determinations were completed by titration with CDTA. No. 4 used a similar method but titrated with EDTA. In all cases corrections were made for lime. No. 5 separated Group III elements with ammonia and lime as oxalate and determined magnesia gravimetrically after precipitation as magnesium ammonium phosphate.

CHROMIC OXIDE

All Analysts determined chromic oxide titrimetrically. Nos. 1 and 4 fused the sample with alkali carbonate/boric acid, extracted with dilute sulphuric acid, oxidized with permanganate and titrated with ammonium ferrous sulphate/dichromate. Nos. 3, 5, 6 and 7 used a similar method but fused with a mixture of sodium hydroxide and sodium peroxide.[1] No. 2 dissolved the sample by heating with sulphuric/phosphoric acid mixture, oxidized with persulphate/silver nitrate and titrated directly with ammonium ferrous sulphate.

ALKALIS

All Analysts treated the sample with a mixture of hydrofluoric, nitric and perchloric acids and determined the alkalis with a flame photometer.[2]

* Where reference has been made to "direct-reading spectroscopy", solution methods were used in all cases and calibration was carried out with accurately made up synthetic standards.

REFERENCES

1. BS 1902: Part 2C: 1967
2. Bennett and Hawley, Methods of Silicate Analysis, Academic Press, 1965, Chapter 32.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded <75 micron (200 mesh) for chemical analysis.

INTENDED USE & STABILITY

The sample is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

It will remain stable provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

TRACEABILITY

The traceability of this BCS-CRM to the SI is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

NEWHAM HALL, NEWBY,
MIDDLESBROUGH, ENGLAND, TS8 9EA
Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk

For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

Preliminary Edition ..... June 1969
Main Edition ..... October 1971
Main Edition (revised with C(95%)and s_M values for each certified constituent) ..... August 2015