MAIN EDITION

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 396
LOW SILICA MAGNESITE-CHROME

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

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ANALYSES

Mean of 4 values - mass content in %.

All results relate to the dried (105°C) sample.

Analyst No.	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	Cr_2O_3	B_2O_3	*LOI*
1	1.34	5.74	0.27	10.82	0.16	1.14	64.4	15.53	0.086	*0.04*
2	1.45	5.77	...	10.93	0.20	1.10	64.7	15.79	0.090	...
3	1.34	5.88	0.26	10.92	0.17	1.15	64.4	15.65	...	...
4	1.30	5.84	0.26	11.15	0.18	1.10	64.5	15.60	0.091	..
5	1.43	5.74	0.23	11.24	0.21	1.10	65.0	15.59	...	...
6	1.42	5.56	0.27	10.82	...	1.14	64.8	15.45	...	...
7	1.39	5.68	0.28	10.74	...	1.10	...	...	...	...
8	1.32	5.67	0.27	10.85	0.14	1.10	64.8	15.6	0.093	...
9	1.36	5.67	0.24	10.82	0.15	1.12	64.5	15.61	0.082	*0.04*
M_M	**1.37**	**5.73**	**0.26**	**10.9**	**0.17**	**1.12**	**64.6**	**15.6**	**0.09**	...
s_M	0.06	0.10	0.02	0.2	0.03	0.03	0.3	0.1	0.01	...

EQUIVALENT % ELEMENT CONTENT OF OXIDE CONSTITUENTS

Si	Al	Ti	Fe	Mn	Ca	Mg	Cr	B
0.64	**3.03**	**0.16**	**7.6**	**0.13**	**0.80**	**39.0**	**10.7**	**0.03**

The above figures are those which each Analyst has decided upon after careful verification.
Figures in bold type certified, figures in small italic type only approximate.
M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means.
The following information based on flame emission spectroscopy was supplied by Analyst No. 3:
Na_2O 0.06%, K_2O 0.03%, Li_2O 0.05%.

CERTIFIED VALUES (Cv)

mass content in %

	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MnO	CaO	MgO	Cr_2O_3	B_2O_3
Cv	**1.37**	**5.73**	**0.26**	**10.9**	**0.17**	**1.12**	**64.6**	**15.6**	**0.09**
C(95%)	0.05	0.08	0.02	0.2	0.03	0.02	0.2	0.1	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. 396 LOW SILICA MAGNESITE-CHROME

NOTES ON METHODS USED

SILICA

Analysts Nos. 1, 3, 4, 6, 8 and 9 determined silica gravimetrically as quinoline molybdosilicate according to the Standard method B.S. 1902: Part 2C: 1974. Analyst No. 2 used atomic absorption spectroscopy. No. 5 fused the sample with lithium metaborate and completed photometrically as molybdenum blue. Analyst No. 7 treated the sample with perchloric acid and completed gravimetrically after dehydration.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence and found 1.28%, 1.29% and 1.25% respectively. Analyst No. 9 also used solution optical emission spectroscopy and found 1.25%.

ALUMINA

Analysts Nos. 1, 3, 4, 6, 8 and 9 determined alumina by titration with CDTA after removal of chromium by extraction with liquid ion exchange resin and other interfering elements by extraction with diethyldithiocarbamate/cupferron/chloroform. All followed the procedure of the Standard method B.S. 1902: Part 2C: 1974. No. 7 used a similar method but titrated with EDTA. Analyst No. 2 used atomic absorption spectroscopy and No. 5 used inductively coupled plasma emission spectroscopy.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence and found 5.82%, 5.77% and 5.54% respectively. No. 9 also used solution optical emission spectroscopy and found 5.75%.

TITANIA

Analysts Nos. 1, 3, 4, 6, 7, 8 and 9 determined titania photometrically with hydrogen peroxide. All except No. 7 used the Standard method B.S. 1902: Part 2C: 1974 in which chromium is removed by extraction with liquid ion exchange resin. No. 7 removed chromium by volatilization as chromyl chloride and separated Group III elements by precipitation with ammonia. Analyst No. 5 used a diantipyrylmethane photometric method.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence and found 0.26%, 0.24% and 0.25% respectively. No. 5 also used inductively coupled plasma emission spectroscopy and found 0.25%. No. 9 also used solution optical emission spectroscopy and found 0.27%.

FERRIC OXIDE

Analysts Nos. 1, 3, 6, 8 and 9 determined ferric oxide photometrically with 1:10 phenanthroline after removal of chromium by extraction with liquid ion exchange resin according to the Standard method B.S. 1902: Part 2C: 1974. No. 2 used atomic absorption spectroscopy. Analysts Nos. 4 and 5 separated iron by ammonia precipitation, reduced with stannous chloride and completed by titration with dichromate. No. 7 used a thioglycollic acid photometric method.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence and found 10.95%, 10.88% and 11.05% respectively. No. 5 also used inductively coupled plasma emission spectroscopy and found 11.24%.

MANGANOUS OXIDE

Analysts Nos. 1, 3, 4, 5, 8 and 9 determined manganous oxide photometrically after oxidation with periodate. All except No. 5 followed the procedure of the Standard method B.S. 1902: Part 2C: 1974 in which chromium is removed by extraction with liquid ion exchange resin. Analyst No. 2 used atomic absorption spectroscopy.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence spectroscopy and found 0.18%, 0.22% and 0.18% respectively. No. 5 also used inductively coupled plasma emission spectroscopy and found 0.18%. No. 9 also used solution optical emission spectroscopy and found 0.19%.

LIME

Analysts Nos. 1, 3, 4, 6, 8 and 9 determined lime by the Standard Method B.S. 1902: Part 2C: 1974 in which chromium is removed by extraction with liquid ion exchange resin and magnesium separated by precipitation as hydroxide before titration with EGTA. Analyst No. 2 used atomic absorption spectroscopy. Analyst No. 7 removed chromium by volatilization as chromyl chloride, separated Group III hydroxides by precipitation with ammonia and precipitated calcium with oxalate. The determination was completed gravimetrically.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence spectroscopy and found 1.10%, 1.06% and 1.09% respectively. No. 9 also used solution optical emission spectroscopy and found 1.12%.

MAGNESIA

Analysts Nos. 1, 3, 4, 6, 8 and 9 determined magnesia titrimetrically with CDTA according to the Standard method B.S. 1902: Part 2C: 1974 in which chromium is removed by extraction with liquid ion exchange resin and other interfering elements by extraction with diethyldithiocarbamate/cupferron/chloroform. No. 2 used atomic absorption spectroscopy. Analyst No. 5 used a gravimetric method depending upon precipitation as magnesium ammonium phosphate and ignition to pyrophosphate.

Analysts Nos. 1, 3 and 9 also determined magnesia by X-ray fluorescence and found 64.5%, 64.8% and 64.7% respectively. No. 5 also used inductively coupled plasma emission spectroscopy and found 64.7%.

CHROMIC OXIDE

Analysts Nos. 1, 3, 4, 6, 8 and 9 determined chromic oxide by the Standard method B.S. 1902: Part 2C: 1974 which involves fusion of the sample with fusion mixture/boric acid and titration with ammonium ferrous sulphate/ potassium dichromate. No. 2 used atomic absorption spectroscopy. No. 5 used a titrimetric method involving oxidation with persulphate/silver nitrate.

Analysts Nos. 1, 3 and 9 also used X-ray fluorescence and found 15.58%, 15.63% and 15.73% respectively. No. 5 also used inductively coupled plasma emission spectroscopy and found 15.63%.

BORIC OXIDE

All Analysts except No. 2 determined boric oxide photometrically with carminic acid. No. 4 carried out a preliminary separation by distillation as methyl borate whereas the other Analysts used direct methods and corrected for titanium.

Analysts Nos. 1, 4 and 8 also used a direct curcumin photometric method and found 0.082%, 0.090% and 0.100% respectively.

Analyst No. 2 used arc emission spectroscopy calibrated with synthetic standards and found 0.09%.

LOSS ON IGNITION

Loss on ignition was determined by heating to constant weight at 1000°C.

NOTE: The additional X-ray fluorescent methods of analysis used by laboratories 1, 3 & 9 were carried out by the fused bead technique, with fundamental calibration against synthetic standards prepared from pure oxides by the same technique.

DESCRIPTION OF SAMPLE

Bottles of 100g powder graded <75µm (200 mesh).

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.

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For BUREAU OF ANALYSED SAMPLES LTD

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