MAIN EDITION

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 236/3 (ECRM 454-1)

HEMATITE IRON

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	Si	Mn	P	S	Ti	C_{TOTAL}	$C_{GRAPHITIC}$	*Ni*	*As*	*Cu*
1	1.99	1.17	0.044	0.068	0.049	...	...	...	...	...
2	2.00	1.15	0.044	0.067	0.050	*2.53*	*1.96*	*0.21*	*0.025*	*0.07*
3	2.00	1.15	0.046	0.069	0.054	...	...	...	...	...
4	2.00	1.16	0.049	0.066	0.057	...	...	...	...	...
5	2.01	1.17	0.045	0.070	0.050	...	...	...	...	...
6	2.00	1.17	0.048	0.071	0.054	...	...	...	...	...
7	2.03	1.15	0.047	0.068	0.049	...	...	...	...	...
8	2.00	1.15	0.046	0.069	0.055	...	...	...	...	...
M_M	**2.00**	**1.16**	**0.046**	**0.068**	**0.052**	...	...	...	...	...
s_M	0.02	0.01	0.002	0.002	0.004	...	...	...	...	...

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.

CERTIFIED VALUES (Cv)

mass content in %

	Si	Mn	P	S	Ti
Cv	**2.00**	**1.16**	**0.046**	**0.068**	**0.052**
C(95%)	0.01	0.01	0.002	0.002	0.003

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.

DESCRIPTION OF SAMPLE

British Chemical Standard – bottles of 100g chips graded 180µm (85 mesh) for chemical analysis.

BCS-CRM No. 236/3 (ECRM 454-1)
HEMATITE IRON

NOTES ON METHODS USED

SILICON

All Analysts except No. 3 determined silicon gravimetrically. No. 1 used a nitro-sulphuric acid method (Dicks and Clarke, B.C.I.R.A. Journ., 1962, 10, 303) whereas Nos. 2, 4, 5, 6, 7 and 8 used the Standard method B.S. 1121 Part 10: 1967 which depends on double dehydration with perchloric acid. No. 3 used a titrimetric method based on the precipitation of potassium fluorosilicate which was filtered off, dissolved in hot water and titrated with standard alkali (Sneddon, Foundry Trade Journ., 1966, **120**, 605).

MANGANESE

Analysts Nos. 1, 3, 4 and 5 determined manganese photometrically after oxidation with potassium periodate. No. 5 followed the procedure of the Standard method B.S. 1121: Part 23: 1951. Nos. 2, 6, 7 and 8 determined manganese titrimetrically by oxidation with persulphate/silver nitrate and titration with arsenite/nitrite solution (No. 2) or arsenite only (Nos. 6, 7 and 8).

Analyst No. 3 also determined manganese titrimetrically after a zinc oxide separation and found 1.14%.

PHOSPHORUS

Analysts Nos. 1, 5, 7 and 8 determined phosphorus photometrically as phosphovanadomolybdic acid according to the Standard method B.S. 1121: Part 45: 1966. No. 2 determined phosphorus gravimetrically by precipitation as ammonium phosphomolybdate and conversion to lead molybdate according to the Standard method B.S. 1121 Part 9: 1948. Nos. 3, 4 and 6 determined phosphorus titrimetrically after precipitation as phosphomolybdate.

Analysts Nos. 5 and 8 also determined phosphorus titrimetrically and found 0.047% and 0.046% respectively.

SULPHUR

Analysts Nos. 1, 3, 4, 7 and 8 determined sulphur by combustion, the sulphur gases being absorbed in hydrogen peroxide or silver nitrate solution and titrated with sodium borate ; Nos. 1, 3 and 7 used a stoichiometric method employing special porous cartridges for combustion of the samples (Green, B.C.I.R.A. Journ., 1963, **11**, 76). Nos. 2, 5 and 6 determined sulphur gravimetrically. No. 2 carried out a chromatographic separation of the sulphur, as sulphuric acid, on an alumina column before precipitation as barium sulphate (Nydahl, Anal. Chem., 1954, **26**, 580) whereas Nos. 5 and 6 used the Standard method B.S. 1121: Part 1: 1966.

Analyst No. 2 also used the British Standard gravimetric method and found 0.066%. No. 4 also used a similar gravimetric method and found 0064%. No. 5 also used a combustion method and found 0.068%.

TITANIUM

Analyst No. 1 determined titanium photometrically by extraction of its thiocyanate trioctylphosphine oxide complex in cyclohexane. The other Analysts separated the titanium by precipitation with cupferron and determined it photometrically with hydrogen peroxide. Nos. 2, 3, 5, 6 and 7 followed the procedure of the Standard method B.S. 1121: Part 46: 1966

TOTAL CARBON

Standard gravimetric method B.S. 1121: Part 11: 1967, Method 1.

GRAPHITIC CARBON

Standard gravimetric method B.S. 1121: Part 11: 1967, Method 2.

NICKEL

Photometric method using dimethylglyoxime (Analoid Method No. 44).

ARSENIC

Arsenic was precipitated with hypophosphite and determined iodimetrically.

COPPER

Photometric method using bis-cyclohexanone oxalyldihydrazone.

INTENDED USE & STABILITY

The chip sample, BCS-CRM 236/3 (ECRM 454-1), 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.

This Certified Reference Material has been prepared in accordance with the recommendations specified in ISO Guides 30 to 35, available from the International Standards Organisation in Geneva.

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For BUREAU OF ANALYSED SAMPLES LTD

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Preliminary Edition ..... October 1967
Main Edition ..... May 1968
Main Edition (revised with C(95%) and s_M values for each element) ..... May 2011