MAIN EDITION

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS†/SS‡-CRM No. 451/1

CARBON STEEL (Residual Series)

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	As	Cu	Sn	Ti	W	*Pb*
1	0.050	0.117	0.63	0.008	0.013	0.106	0.037	0.012	0.040	0.48	0.0014	0.108	0.095	*0.0005*
2	0.056	0.119	0.62	0.011	0.015	…	…	…	…	…	…	…	…	…
3	…	0.111	0.62	0.008	0.012	…	…	…	…	…	…	…	…	…
4	…	…	…	…	…	0.106	0.040	0.018	…	…	…	0.107	0.102	…
5	…	…	…	…	…	…	…	…	0.042	0.47	0.0023	0.106	…	…
6	0.047	0.119	…	…	0.014	…	…	0.015	…	…	0.0018	…	…	…
7	…	…	…	…	…	0.103	…	…	…	…	…	0.101	0.093	…
8	…	0.115	…	…	…	…	0.039	0.012	…	…	0.0014	…	…	…
9	0.053	…	0.61	0.008	…	…	…	0.016	…	0.47	0.0023	…	…	…
10	…	…	…	…	…	0.109	…	…	0.044	…	…	0.106	…	…
11	0.049	…	…	…	…	0.104	…	…	…	…	…	0.102	0.108	…
12	0.050	…	0.63	…	0.013	0.100	0.037	…	0.040	0.47	0.0013	0.102	0.099	…
13	…	…	…	…	…	0.100	0.041	0.021	…	…	…	…	…	…
14	…	…	0.61	0.007	0.015	…	0.038	…	0.040	0.48	…	…	…	…
15	…	0.114	…	0.010	0.015	…	…	…	…	…	…	…	…	…
M_M	**0.051**	**0.116**	**0.62**	**0.009**	**0.014**	**0.104**	**0.039**	**0.016**	**0.041**	**0.47**	**0.0018**	**0.105**	**0.099**	…
s_M	0.004	0.004	0.01	0.002	0.002	0.004	0.002	0.004	0.002	0.01	0.0005	0.003	0.006	…

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.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	As	Cu	Sn	Ti	W
Cv	**0.051**	**0.116**	**0.62**	**0.009**	**0.014**	**0.104**	**0.039**	**0.016**	**0.041**	**0.47**	**0.002**	**0.105**	**0.099**
C(95%)	0.004	0.004	0.01	0.002	0.002	0.004	0.002	0.004	0.003	0.01	0.001	0.003	0.008

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.

N.B. Due to slight segregation of certain elements an area 6mm in diameter in the centre of the disc samples should be avoided for emission spectroscopy.

BCS/SS-CRM No. 451/1 CARBON STEEL (Residual Series)

NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 2 and 6 determined carbon by non aqueous titration according to the British Standard Carbon Method 4*, Nos. 9 and 11 used coulometric methods according to Hobson and Leigh, Analyst, 1974, **99**, 93 and Boniface and Jenkins, Analyst, 1971, **96**, 37 respectively. No. 12 used the low pressure British Standard Carbon Method 3*

Analyst No. 9 also determined carbon by non aqueous titration and found 0.053%. No. 12 also used high frequency combustion/infrared absorption and found 0.050%.

SILICON

Analysts Nos. 1, 2, 6 and 15 determined silicon gravimetrically after dehydration with perchloric acid. Nos. 1, 2 and 6 followed the procedure of the British Standard Silicon Method 1*. Analyst No. 3 precipitated silicon as potassium fluorosilicate and completed by alkalimetric titration (Sneddon, Foundry Trade Journal 1966, **120**, 605). No. 8 used a molybdenum blue photometric method.

Analyst No, 6 also determined silicon photometrically as molybdenum blue and found 0.120%. No, 15 also used a potassium fluorosilicate titrimetric method and found 0.105%.

MANGANESE

All analysts except No, 2 determined manganese photometrically after oxidation with periodate according to the British Standard Manganese Method 2*. No. 2 used the British Standard Manganese Method 1* in which a zinc oxide separation is carried out before oxidation with persulphate/silver nitrate and titration with ammonium ferrous sulphate.

Analyst No. 9 also determined manganese by AAS and found 0.62%,

PHOSPHORUS

Analysts Nos. 1, 9 and 14 determined phosphorus photometrically as phosphovanadomolybdate according to the British Standard Phosphorus Method 2*, The remaining analysts used titrimetric methods after separation as ammonium phosphomolybdate (Nos. 2 and 3) or quinoline phosphomolybdate (No. 15).

SULPHUR

Analysts Nos. 1 and 6 determined sulphur gravimetrically according to the British Standard Sulphur Method 1*. The remaining analysts used combustion methods. Nos. 2 and 3 used air combustion (Green, BCIRA Journal, 1968, **16**, 244), Nos. 12 and 14 used high frequency combustion with infrared measurement and No, 15 used high frequency combustion with oxidation/reduction titration,

Analyst No. 6 also determined sulphur by high frequency combustion/infrared absorption and found 0.016%.

CHROMIUM

Analysts Nos. 1 and 13 determined chromium titrimetrically with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. No, 1 followed the procedure of the Analoid Method No. 37. No. 4 used a method involving removal of iron by solvent extraction and photometric measurement of chromium in the hexavalent state, Analysts Nos. 7, 11 and 12 used the British Standard Chromium Method 2* in which chromium is determined photometrically with diphenylcarbazide. No, 10 used ICP-OES after dissolution of the sample in phosphoric acid.

MOLYBDENUM

All analysts except No, 8 determined molybdenum photometrically as oxythiocyanate. Nos. 1, 4, 12 and 14 followed the procedure of the British Standard Molybdenum Method I * in which the oxythiocyanate complex is extracted into n-butyl acetate. Analyst No. 8 used AAS.

NICKEL

Analysts Nos. 1, 8 and 9 determined nickel by AAS. The remaining analysts used dimethylglyoxime photometric methods. No. 4 carried out a preliminary separation of iron by solvent extraction and No. 6 used the British Standard Nickel Method 3*.

Analyst No. 6 also determined nickel by AAS and found 0.012%.

ARSENIC

Analysts Nos. 1 and 14 determined arsenic photometrically as the molybdenum blue complex after extraction as iodide into chloroform (Fogg et al, Analyst 1972, **97**, 657). Nos. 5 and 12 separated arsenic as arsine by reduction with zinc and completed photometrically after absorption in a chloroform solution of silver diethyldithiocarbamate. Analyst No. 10 used ICP-OES after dissolution of the sample in phosphoric acid.

COPPER

Analyst No. 1 determined copper photometrically with biscyclohexanone oxalyldihydrazone according to the Analoid Method No. 65. No. 5 used AAS. Analysts Nos. 9, 12 and 14 used the British Standard Copper Method 3* which involves extraction of the 2, 2' diquinolyl complex into pentan-2-ol and subsequent photometric measurement.

Analyst No. 9 also determined copper by AAS and found 0.47%.

TIN

Analysts Nos. 1, 6, 8 and 12 determined tin by AAS. Nos. 1, 6 and 8 used electrothermal atomization and No. 12 extracted with tri-n-octylphosphine oxide in 4-methylpentan-2-one (Burke, Analyst 1972, **97**, 19; 1974, **99**, 469) before aspiration into a nitrous oxide/acetylene flame. No. 5 used a direct iodimetric method after dissolution in hydrochloric acid and reduction with aluminium.

TITANIUM

Analysts Nos. 1, 5 and 12 determined titanium photometrically with diantipyrylmethane. Nos. 4, 7 and 11 used the British Standard Titanium Method 1* in which titanium is separated by precipitation with cupferron and the determination completed photometrically with hydrogen peroxide. No. 10 used ICP-OES after dissolution of the sample in phosphoric acid.

TUNGSTEN

All analysts determined tungsten photometrically with thiocyanate according to the British Standard Tungsten Method 2*.

LEAD

AAS after solvent extraction of iron.

*Methods for Sampling and Analysis of Iron, Steel and Other Ferrous Metals, B.S. Handbook No. 19, first published in 1970 by the BSI, 389 Chiswick High Road, London. W4 4AL.

Abbreviations:-

AAS:	Atomic absorption spectrometry with synthetic calibration.
ICP-OES:	Inductively Coupled Plasma - Optical Emission Spectrometry with synthetic calibration.

DESCRIPTION OF SAMPLE

† British Chemical Standard – bottles of 100g chips graded 1700 - 250µm (10 - 60 mesh) for chemical analysis.

‡ Spectroscopic Standard – 38 mm diameter x 19 mm thick discs for spectroscopic analysis.

INTENDED USE & STABILITY

The chip sample, BCS-CRM 451/1, 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.

The disc sample, SS-CRM 451/1, is intended for establishing and checking the calibration of Optical Emission and X-Ray Spectrometers for the analysis of similar materials. The "as received" working surface of the sample should be linished before use to remove any protective coating. It will remain stable provided that it is not subject to excessive heat (e.g., during preparation of the working surface). An area 6mm in diameter in the centre of the disc should be avoided for optical emission spectrometry.

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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