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Certificate No. GB94/Q3993

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS^{*}/SS[§]-CRM No. 408/2

LOW ALLOY STEEL

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 %.

Lab No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al (Total)	As	Cu	N	Pb	V	<i>Sn</i>
1	0.107	...	4.11	...	0.0044	0.686	...	0.0008	0.070	...
2	0.294	0.248	0.554	0.056	0.150	0.0060
3	0.106	0.098	4.17	0.702	...	0.0004	0.068	0.003
4	0.290	0.234	0.564	0.055	0.029	0.116	0.102	4.16	0.151	0.0038	0.695	0.0073	0.0006	0.061	0.001
5	0.290	0.236	0.558	...	0.030	0.155	0.0049	...	0.0070
6	0.292	0.239	0.560	0.056	0.030	...	0.093	...	0.154	...	0.693	0.0075
7	0.290	0.235	0.547	0.058	0.032	0.0084
8	0.114	0.103	4.12	...	0.0047	0.696	...	0.0006	0.070	0.002
9	0.284	0.032	0.106	0.095	4.11	0.685	0.066	...
10	0.286	0.234	0.555	0.056	0.030	0.0089
11	0.286	0.236	0.560	0.056	0.030	0.115	0.087	4.16	0.160	...	0.702	0.0074	0.0004	0.065	0.001
12	0.110	0.110	4.08	...	0.0050	0.695	...	0.0010	0.070	0.002
M_M	0.289	0.237	0.557	0.056	0.030	0.111	0.098	4.13	0.154	0.0046	0.694	0.0075	0.0006	0.067	...
<i>s_M</i>	0.004	0.005	0.006	0.001	0.001	0.005	0.008	0.04	0.004	0.0005	0.006	0.0009	0.0002	0.004	...
<i>s_w</i>	0.002	0.004	0.005	0.002	0.001	0.005	0.005	0.03	0.003	0.0002	0.010	0.0002	0.0001	0.002	...

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. *s_w*: intralaboratory standard deviation.
 Values given above in small italic type are for information only.

CERTIFIED VALUES (C_v)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al (Total)	As	Cu	N	Pb	V
C_v	0.289	0.237	0.557	0.056	0.030	0.111	0.098	4.13	0.154	0.0046	0.694	0.0075	0.0006	0.067
C(95%)	0.003	0.005	0.005	0.001	0.001	0.004	0.007	0.04	0.005	0.0006	0.006	0.0009	0.0003	0.004

The half width confidence interval C(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:1989 section 4.

BCS/SS-CRM No. 408/2 LOW ALLOY STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 2 and 4 determined carbon by non-aqueous titration according to the British Standard Carbon Method 4*. Nos. 5, 6, 7, 10 and 11 used high frequency combustion/infrared absorption. No. 9 determined carbon coulometrically (Boniface and Jenkins, Analyst, 1971, **96**, 37).

SILICON

Analysts Nos. 2, 4, 6, 10 and 11 determined silicon gravimetrically by double dehydration with perchloric acid, according to the British Standard Silicon Method 1*. Nos. 5 and 7 used molybdenum blue photometric methods.

MANGANESE

Analysts Nos. 2 and 7 determined manganese by Flame Atomic Absorption Spectrometry (FAAS). Nos. 4, 5 and 10 determined manganese photometrically, Nos. 4 and 5 after oxidation with potassium periodate according to the British Standard Manganese Method 2*, No. 10 after oxidation with ammonium persulphate. Nos. 6 and 11 used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

PHOSPHORUS

All Analysts, except Nos. 6 and 11, determined phosphorus photometrically as phosphovanadomolybdate according to BS 6200:3.24.1:1985 (British Standard Phosphorus Method 2*). Nos. 6 and 11 used ICP-OES

SULPHUR

Analyst No. 4 determined sulphur gravimetrically according to the British Standard Sulphur Method 1*. Nos. 5, 6, 7, 10 and 11 used high frequency combustion/infrared absorption. No. 9 determined sulphur coulometrically.

CHROMIUM

Analysts Nos. 1, 8 and 11 determined chromium by FAAS. Analysts Nos. 3, 4, 9 and 12 determined chromium titrimetrically with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. No. 3 used the procedure in BCIRA Method A[‡] and Nos. 4, 9 and 12 followed the procedure of BS 6200:3.10.1:1985 (British Standard Chromium Method 1*).

Analyst No. 11 also used ICP-OES and obtained a mean value of 0.111%

MOLYBDENUM

Analysts Nos. 1, 3, 4, 8 and 12 determined molybdenum photometrically as oxythiocyanate, No. 3 according to the BCIRA Method B[‡] and Nos. 4, 8 and 12 according to the British Standard Molybdenum Method 1*. Nos. 6, 9, and 11 used ICP-OES.

Analyst Nos.6 and 8 also determined molybdenum by FAAS and obtained mean values of 0.096% and 0.094% respectively.

NICKEL

Analysts Nos. 1, 11 and 12 determined nickel using FAAS. Nos. 3, 4 and 8 determined nickel titrimetrically after separation with dimethylglyoxime. Nos. 3 and 8 titrated with EDTA. No. 4 dissolved the precipitate in dilute sulphuric acid, boiled with an excess of ferric sulphate and titrated with potassium dichromate (Analoid Method No. 62). No. 9 used ICP-OES.

Analyst No. 8 also determined nickel by FAAS and obtained a mean value of 4.11%.

ALUMINIUM (Total)

Analysts Nos. 2, 4, and 6 determined aluminium using FAAS. No. 6 used an injection technique. No.5 determined aluminium photometrically according to the British Standard Aluminium Method 3*. No. 11 used ICP-OES.

ARSENIC

Analysts Nos. 1 and 4 determined arsenic photometrically with silver diethyldithiocarbamate after separation of arsenic as arsine by reduction with zinc. Nos. 5 and 12 used Electrothermal Atomisation-Atomic Absorption Spectroscopy (ETA-AAS) and No. 8 used FAAS.

COPPER

Analysts Nos. 1, 8 and 12 determined copper by FAAS. Nos. 3 and 4 determined copper photometrically, No. 3 with 2, 2'-diquinolyly according to BCIRA Method C[‡], No. 4 with bis-cyclohexanone oxalyldihydrazone according to the Analoid Method No. 65. Nos. 6, 9 and 11 used ICP-OES.

Analyst No. 11 also used FAAS and obtained a mean value of 0.711%.

NITROGEN

Analyst No. 2 determined nitrogen photometrically with Nessler's reagent after distillation of ammonia. Nos. 4, 5 6 and 11 used thermal conductivity after decomposition in graphite crucibles. Nos. 7 and 10 determined nitrogen by acidimetric titration following distillation of ammonia, according to the British Standard Nitrogen Method 1*.

LEAD

All Analysts except Nos. 3 and 12 used FAAS, No.4 made a prior separation of lead with tri-n-octylphosphine oxide and 4-methylpetan-2-one. Analysts Nos. 3 and 12 used ETA-AAS.

VANADIUM

Analysts Nos. 1 and 8 determined vanadium by FAAS. Nos. 3, 9, 11 and 12 used ICP-OES. No.4 determined vanadium photometrically as vanadotungstate according to the Analoid method No. 59, after carrying out a prior separation of iron by ether extraction.

Tin

Analysts Nos. 3 and 12 determined tin by ETA-AAS. Nos. 4 and 8 used FAAS, No. 4 made a prior separation with of tin with tri-n-octylphosphine oxide and 4-methylpentan-2-one. No. 11 used ICP-OES.

*Methods for Sampling and Analysis of Iron, Steel and other Ferrous Metals, B.S. Handbook No. 19, published 1970 by the British Standards Institute, London.

[‡]Chemical Analysis for Iron Foundries, published 1976 by BCIRA.

DESCRIPTION OF SAMPLE

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

§ Spectroscopic Standard – 38 mm diameter discs for spectroscopic analysis

INTENDED USE & STABILITY

The chip sample, BCS-CRM 408/2, 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 408/2, 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 lished 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

TRACEABILITY

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

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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