MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS†/SS‡-CRM No. 405

LOW ALLOY STEEL

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN and the CZECH REPUBLIC, 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	Cu	V
1	0.057	1.38	1.27	0.015	0.057	0.22	0.017	0.14	0.017	0.32
2	0.060	1.40	1.28	0.017	0.062	0.22	0.015	0.12	0.018	0.32
3	0.059	1.38	1.28	0.018	0.060	…	…	…	…	…
4	…	…	…	…	…	0.22	0.018	0.12	0.012	0.32
5	0.056	…	…	0.018	…	0.21	0.017	0.11	0.012	…
6	…	…	…	…	…	0.21	0.016	0.12	0.013	0.32
7	0.057	1.39	1.27	0.018	0.058	…	…	…	…	…
8	0.059	1.38	1.28	0.018	0.060	…	…	…	…	…
9	…	…	…	…	…	0.21	0.016	0.12	0.017	0.32
10	0.058	1.38	1.29	0.015	0.060	0.20	0.020	0.12	0.012	0.31
11	0.060	1.38	1.26	0.014	0.060	…	…	…	…	…
12	…	…	…	…	…	0.22	0.020	0.11	0.016	0.33
M_M	**0.058**	**1.38**	**1.28**	**0.017**	**0.060**	**0.21**	**0.017**	**0.12**	**0.015**	**0.32**
s_M	0.002	0.01	0.01	0.002	0.002	0.01	0.002	0.01	0.003	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.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	V
Cv	**0.058**	**1.38**	**1.28**	**0.017**	**0.060**	**0.21**	**0.017**	**0.12**	**0.015**	**0.32**
C(95%)	0.002	0.01	0.01	0.002	0.002	0.01	0.002	0.01	0.003	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.

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NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 11 determined carbon by non-aqueous titration (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399). No. 2 determined carbon titrimetrically using the Strohlein apparatus. No. 3 used a conductimetric method with a balanced two-cell circuit. No. 5 used a low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144). Nos. 7, 8 and 10 used infra-red absorption methods.

Analyst No. 5 also determined carbon by infra-red absorption and by non-aqueous titration; these methods gave results of 0.055% and 0.058% respectively. No. 11 also used the Standard gravimetric method B.S. 1121: Part 11: 1967 and found 0.065%.

SILICON

All analysts determined silicon gravimetrically by double dehydration with perchloric acid; Nos. 1, 3, 7, 10 and 11 used the Standard method B.S. 1121: Part 10: 1967.

Analyst No. 2 also used a gravimetric method involving evaporation of a hydrochloric-nitric acid solution of the alloy and found 1.41%. No. 11 also determined silicon photometrically as molybdenum-blue and found 1.38%.

MANGANESE

Analysts Nos. 1 and 7 determined manganese titrimetrically. No. 1 used the direct Analoid method No. 53 whereas No. 7 used the Standard method B.S. 1121: Part 16: 1949 which includes a zinc oxide separation. The other analysts determined manganese photometrically after oxidation with periodate; Nos. 3, 10 and 11 used the Standard method B.S. 1121: Part 23: 1951.

Analyst No. 2 also used a direct titrimetric method and found 1.29%. No. 11 also used the British Standard titrimetric method and found 1.26%.

PHOSPHORUS

Analysts Nos. 1, 3, 5, 10 and 11 determined phosphorus photometrically as phosphovanadomolybdic acid according to the Standard method B.S. 1121: Part 45: 1966. Nos. 2 and 8 determined phosphorus gravimetrically by precipitation as phosphomolybdate and conversion to lead molybdate; No. 8 used the Standard method B.S. 1121: Part 9: 1948. No. 7 determined phosphorus titrimetrically after precipitation as phosphomolybdate.

Analyst No. 2 also determined phosphorus photometrically as phosphovanadomolybdic acid and found 0.016%. No. 11 also used the British Standard gravimetric method and found 0.014%.

SULPHUR

Analysts Nos. 1, 2, 7 and 11 determined sulphur gravimetrically. Nos. 1, 7 and 11 used the Standard method B. S. 1121: Part 1: 1966. No. 2 used a method involving preliminary separation of iron by shaking the sample with cupric potassium chloride solution. Nos. 3, 8 and 10 determined sulphur by combustion in oxygen (Nos. 3 and 8) or air (No. 10); No. 3 completed conductimetrically whereas Nos. 8 and 10 completed titrimetrically.

Analyst No. 11 also determined sulphur by combustion and found 0.061%.

CHROMIUM

All analysts except No. 10 determined chromium titrimetrically after oxidation with persulphate/silver nitrate. No. 1 used the Analoid method No. 37. Nos. 4, 5, 6, 9 and 12 used the Standard method B.S. 1121: Part 13: 1954. No. 10 determined chromium photometrically using diphenylcarbazide according to the Standard method B.S. 1121: Part 24: 1967.

Analyst No. 2 also determined chromium photometrically with diphenylcarbazide and found 0.22%.

MOLYBDENUM

All analysts except No. 5 determined molybdenum photometrically as oxythiocyanate; Nos. 1, 4, 6, 9, 10 and 12 followed the procedure of the Standard method B.S. 1121: Part 48: 1966. No. 5 determined molybdenum photometrically with toluene 3-4 dithiol (Wells and Pemberton, Analyst, 1947, **72**, 185).

NICKEL

Analysts Nos. 1, 4, 6 and 10 determined nickel photometrically with dimethylgloxime. No. 1 used the Analoid method No. 44. Nos. 4, 6 and 10 used the Standard method B.S. 1121: Part 6: 1967. No. 2 determined nickel gravimetrically by precipitation with dimethylglyoxime. Nos. 5, 9 and 12 precipitated nickel with dimethylglyoxime and completed cyanometrically according to the Standard titrimetric method B.S. 1121: Part 37: 1961.

Analysts Nos. 2 and 5 also determined nickel photometrically with dimethylglyoxime and found 0.11% in each case. No. 9 also separated nickel with dimethylglyoxime and completed by titration with EDTA; the result by this method was 0.12%.

COPPER

All analysts except No. 9 determined copper photometrically. Nos. 1, 4, 5, 6 and 10 used 2-2' diquinolyl according to the Standard method B.S. 1121: Part 36: 1956. No. 2 used tetraethylthiuram disulphide. No. 12 used bis-cyclohexanone oxalyldihydrazone (Haywood and Sutcliffe, Analyst, 1956, **81**, 651). No. 9 determined copper titrimetrically by iodometric titration using the Standard method B.S. 1121: Part 14: 1956.

Analysts Nos. 2, 4 and 5 also determined copper photometrically with bis-cyclohexonane oxalyldiyhdrazone and found 0.017%, 0.012% and 0.01% respectively.

VANADIUM

All analysts except No. 10 determined vanadium titrimetrically. Nos. 1, 4, 6, 9 and 12 used the Standard method B.S. 1121: Part 25: 1956. No. 2 titrated potentiometrically with permanganate. No. 10 determined vanadium photometrically as phosphovanadotungstate (Scholes, Analyst, 1957, **82**, 525).

Analysts Nos. 2 and 12 also determined vanadium photometrically as phosphovanadotungstate and found 0.32% in each case.

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 405, 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 405, 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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