



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS BCS-CRM No. 220/2 (ECRM 254-1) HIGH-SPEED STEEL

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

CO-OPERATING ANALYSTS

INDEPENDENT ANALYSTS

- 1 COPPINS, W. C., *MSc, FRIC*, Ridsdale & Co. Ltd., Middlesbrough.
2 KNAPP, G., Sheffield Testing Works Ltd., Sheffield.

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ANALYSTS representing MANUFACTURERS and USERS (cont.)

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6 KIDMAN, L., *AMet, AIM, AMInstF*,
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7 ROWLEY, E.,
James Neill and Co. (Magnets and Steel) Ltd., Sheffield.
8 SAXBY, A., *AMet*,
Osborn-Hadfields Steel Founders Ltd., Sheffield.
9 WISEMAN,
J., Swift Levick and Sons Ltd., Sheffield.

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	Sn	V	W
1	0.86	0.18	0.305	0.023	0.029	5.12	4.90	0.14	0.33	0.09	0.020	1.94	7.08
2	0.89	0.21	0.305	0.024	0.029	5.09	4.97	0.11	0.31	0.09	0.020	1.90	6.90
3	0.87	0.21	0.30	0.025	0.029	5.16	4.81	0.12	0.33	0.10	0.016	1.95	6.90
4	0.88	0.18	0.305	0.022	0.027	5.17	4.97	0.11	0.32	0.08	0.019	1.96	7.07
5	0.87	0.18	0.30	0.025	0.030	5.11	4.93	0.13	0.32	0.09	0.021	1.94	6.88
6	0.88	0.18	0.30	0.024	0.031	5.14	4.93	0.11	0.31	0.08	0.019	1.95	7.11
7	0.86	0.18	0.29	0.023	0.031	5.07	4.92	0.10	0.31	0.09	0.019	1.97	6.96
8	0.89	0.20	0.31	0.023	0.030	5.12	4.91	0.13	...	0.09	...	1.95	6.93
9	0.88	0.20	0.31	0.022	0.029	5.13	4.92	0.11	1.94	6.91
M_M	0.88	0.19	0.30	0.023	0.029	5.12	4.92	0.12	0.32	0.09	0.019	1.94	6.97
s_M	0.02	0.02	0.01	0.002	0.002	0.04	0.05	0.02	0.01	0.01	0.002	0.02	0.10

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 (C_v)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	Sn	V	W
C_v	0.88	0.19	0.30	0.023	0.029	5.12	4.92	0.12	0.32	0.09	0.019	1.94	6.97
C(95%)	0.01	0.02	0.01	0.001	0.001	0.03	0.04	0.02	0.01	0.01	0.002	0.02	0.07

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 1700 - 250µm (10 - 60 mesh) for chemical analysis.

BCS-CRM No. 220/2 (ECRM 254-1) HIGH-SPEED STEEL

NOTES ON METHODS USED

CARBON

All analysts determined carbon by combustion. Nos. 1, 3, 5, 7 and 9 completed gravimetrically according to the Standard method B.S. 1121: Part 11: 1967. Nos. 2 and 8 completed by measurement of infra-red absorption (Tipler, Analyst, 1963, **88**, 272). No. 4 used a low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144). No. 6 used a non-aqueous titration method (Jones et al., Analyst, 1965, **90**, 623: 1966, **91**, 399).

Analyst No. 4 also determined carbon by non-aqueous titration and by infra-red absorption; values of 0.88% were obtained in each case.

SILICON

Analysts Nos. 1, 2, 3, 4 and 9 determined silicon gravimetrically. Nos. 1, 3 and 9 carried out the determination on the same portion of sample that was used for the tungsten determination; after filtration and ignition, the combined tungstic oxide/silica precipitate was treated with hydrofluoric acid and ignited at 800°C. Nos. 2 and 4 determined silicon by dehydration with perchloric acid according to the Standard method B.S. 1121: Part 10: 1967. Nos. 5, 6, 7 and 8 used molybdenum-blue photometric methods.

MANGANESE

All analysts determined manganese photometrically after oxidation to permanganic acid. All except Nos. 5 and 7 oxidized with periodate according to the Standard method B.S. 1121: Part 23: 1951. Nos. 5 and 7 used persulphate

PHOSPHORUS

Analysts Nos. 1, 4, 5, 6 and 8 determined phosphorus photometrically as phosphovanadomolybdic acid; Nos. 1, 4, 5 and 6 used the Standard method B.S. 1121: Part 45: 1966 which includes a procedure for recovery of any phosphorus from the tungstic oxide residue. Nos. 2, 7 and 9 determined phosphorus titrimetrically after separation as ammonium phosphomolybdate; Nos. 2 and 7 included procedures for recovery of phosphorus from the tungstic oxide residue. No. 3 separated phosphorus as phosphomolybdate and completed gravimetrically as lead molybdate according to the Standard method B.S. 1121: Part 9: 1948.

SULPHUR

Analysts Nos. 1, 2 and 4 determined sulphur gravimetrically. Nos. 1 and 2 used the Standard method B.S. 1121: Part 1: 1966. No. 4 carried out a preliminary chromatographic separation of the sulphur, as sulphuric acid, on an alumina column (Nydahl, Anal. Chem., 1954, **26**, 580). Nos. 3, 5, 7, 8 and 9 determined sulphur titrimetrically after combustion in oxygen. No. 6 separated sulphur chromatographically and titrated with EGTA solution.

Analyst Nos. 4 and 6 also determined sulphur titrimetrically after combustion and found 0.028% and 0.030% respectively.

CHROMIUM

All analysts determined chromium titrimetrically after oxidation with persulphate/silver nitrate. No. 1 used the Analoid method No. 37 in which tungsten is held in solution with fluoride and the oxidized chromium titrated with ammonium ferrous sulphate; a correction was applied for vanadium. Nos. 2, 4, 5 and 9 carried out preliminary separations of tungsten as tungstic oxide and, after oxidizing the chromium, titrated with ammonium ferrous sulphate/permanganate according to the Standard method B.S. 1121: Part 13: 1954. No. 3 used a similar method but titrated with ammonium ferrous sulphate/dichromate and applied a correction for vanadium. No. 6 oxidized the chromium in presence of precipitated tungstic oxide, added excess of ferrous sulphate and back-titrated with dichromate using an external indicator; a correction was made for vanadium. No. 8 held tungsten in solution with fluoride and titrated the oxidized chromium with ammonium ferrous sulphate/permanganate according to the Analoid method No. 35.

MOLYBDENUM

Analysts Nos. 1, 2, 5, 6 and 9 determined molybdenum photometrically as oxythiocyanate. Nos. 1, 5 and 9 developed and measured the colour in aqueous solution; Nos. 1 and 8 used the Analoid method No. 34. No. 6 used the Standard method B.S. 1121: Part 48: 1966 in which the coloured complex is extracted with *n*-butyl acetate. No. 3 determined molybdenum gravimetrically by precipitation with benzoïn α -oxime, conversion to molybdenum sulphide and ignition to MoO₃. No. 4 determined molybdenum photometrically with toluene 3:4-dithiol (Wells and Pemberton, Analyst, 1947, **72**, 185).

Analyst No. 4 also used the British Standard photometric method and found 4.91%.

NICKEL

All analysts except No. 9 determined nickel photometrically with dimethylglyoxime. Nos. 3, 4, 5, 6 and 8 used the Standard method B.S. 1121: Part 6: 1967. No. 9 determined nickel titrimetrically according to the Standard method B.S. 1121: Part 37: 1961.

COBALT

All analysts except No. 2 determined cobalt photometrically with nitroso-R-salt according to the Standard method B.S. 1121: Part 30: 1954. No. 2 determined cobalt by atomic-absorption spectroscopy.

Analyst No. 3 also found 0.32% by atomic absorption spectroscopy.

COPPER

Analysts Nos. 1, 3, 4, 6, 7 and 8 determined copper photometrically. Nos. 1, 4, 7 and 8 used 2-2' diquinolyl according to the Standard method B.S. 1121: Part 36: 1956. Nos. 3 and 6 used *bis*-cyclohexanone oxalyldihydrazone. Nos. 2 and 5 determined copper titrimetrically; No. 5 used the Standard method B.S. 1121: Part 14: 1956.

TIN

All analysts determined tin titrimetrically according to the Standard method B.S. 1121: Part 20: 1961.

VANADIUM

All analysts except Nos. 3 and 6 determined vanadium titrimetrically by oxidation with permanganate and titration with ammonium ferrous sulphate. No. 1 used the Analoid method No. 34. Nos. 2, 4, 5 and 9 used the Standard method B.S. 1121: Part 25: 1956 (Method A). No. 8 used the Analoid method No. 35. No. 3 used a titrimetric method involving potentiometric titration with dichromate/ferrous sulphate. No. 6 determined vanadium photometrically as phosphovanadotungstic acid.

TUNGSTEN

All analysts except No. 8 determined tungsten gravimetrically as tungstic oxide according to the Standard Method B.S. 1121: Part 40: 1967; the impurities in the final residue were determined in all cases and the appropriate corrections made. No. 8 determined tungsten photometrically as the thiocyanate using a modification of the Standard method B.S. 1121: Part 32: 1954.

Analyst No. 7 also determined tungsten photometrically as the thiocyanate and found 7.00%.

INTENDED USE & STABILITY

The chip sample, BCS-CRM 220/2 (ECRM 254-1), 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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