MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 241/2 (ECRM 251-1)

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

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7 PRIESTLEY, I., Edgar Allen Foundry Ltd., Sheffield.

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	Sn	V	W
1	0.84	0.21	0.28	0.025	0.024	5.36	0.54	0.15	5.71	0.06	0.023	1.57	19.86
2	0.83	0.22	0.28	0.025	0.023	5.34	0.54	0.15	5.70	0.08	0.024	1.61	19.87
3	0.84	0.21	0.27	0.023	0.026	5.40	0.54	0.16	5.71	0.07	0.025	1.59	19.85
4	0.84	0.19	0.27	0.023	0.025	5.37	0.53	0.15	5.66	0.08	0.027	1.59	19.90
5	0.83	0.22	0.28	0.024	0.026	5.37	0.53	0.15	5.73	0.08	0.024	1.61	19.90
6	0.84	0.20	0.26	0.024	0.027	5.29	0.52	0.16	5.67	0.08	0.026	1.56	19.89
7	0.84	0.20	0.26	0.025	0.024	5.33	0.52	0.14	5.73	0.09	0.026	1.59	19.88
8	0.84	0.22	0.27	0.023	0.027	5.36	0.50	0.16	5.72	0.08	0.026	1.60	19.70
M_M	**0.84**	**0.21**	**0.27**	**0.024**	**0.025**	**5.35**	**0.53**	**0.15**	**5.70**	**0.08**	**0.025**	**1.59**	**19.9**
s_M	0.01	0.02	0.01	0.001	0.002	0.04	0.02	0.01	0.03	0.01	0.002	0.02	0.1

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.

This material was also examined by 13 laboratories in the E.U. and the Aluminium content was found to have a mean value of 0.009% with a standard deviation of 0.004%.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Cu	Sn	V	W
Cv	**0.84**	**0.21**	**0.27**	**0.024**	**0.025**	**5.35**	**0.53**	**0.15**	**5.70**	**0.08**	**0.025**	**1.59**	**19.9**
C(95%)	0.01	0.01	0.01	0.001	0.002	0.03	0.02	0.01	0.03	0.01	0.002	0.02	0.1

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, 3, 7 and 8 determined carbon gravimetrically according to the Standard method B.S. 1121: Part 11: 1967. No. 2 used a low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144). Nos. 4, 5 and 6 used a non-aqueous titration method (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399).

Analyst No. 2 also used a non-aqueous titration method and found 0.84%. Nos. 2, 4 and 8 also determined carbon by infrared absorption and found 0.84%, 0.84% and 0.85% respectively. No. 4 also used the British Standard gravimetric method and found 0.83%.

SILICON

All Analysts except Nos. 3 and 7 determined silicon gravimetrically. No. 1 used 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, 4, 5, and 8 determined silicon by dehydration with perchloric acid according to the Standard method B.S. 1121: Part 10: 1967. No. 6 dehydrated with hydrochloric acid before filtration and treatment of the combined tungstic oxide/silica precipitate. Nos. 3 and 7 determined silicon photometrically as molybdenum-blue.

Analysts Nos. 2, 5, and 8 also used photometric molybdenum-blue methods and found 0.22% in each case.

MANGANESE

All Analysts except No. 3 determined manganese photometrically after oxidation with periodate; Nos. 1, 2, 4, 5, 7 and 8 used the Standard method B.S. 1121: Part 23: 1951. No. 3 used a similar method but oxidized with persulphate.

PHOSPHORUS

All Analysts except No. 4 determined phosphorus photometrically as phosphovanadomolybdic acid; Nos. 1, 2, 5 and 7 used the Standard method B.S. 1121: Part 45: 1966 which includes a procedure for recovery of any phosphorus from the tungstic oxide residue. No. 4 determined phosphorus gravimetrically by the Standard method B.S. 1121: Part 9: 1948.

Analyst No. 4 also determined phosphorus photometrically as molybdenum-blue using an automatic method (Scholes and Thulbourne, Analyst, 1964, **89**, 466) and found 0.023%.

SULPHUR

Analysts Nos. 1, 2, 3, 5 and 8 determined sulphur titrimetrically after combustion in oxygen. No. 1 absorbed the sulphur gases in water and titrated with iodide/iodate solution whereas Nos. 2, 3, 5 and 8 absorbed in hydrogen peroxide solution and titrated with sodium borate solution. Nos. 4, 6 and 7 determined sulphur gravimetrically, Nos. 4 and 7 following the procedure of the Standard method B.S. 1121: Part 1: 1966.

Analyst No. 2 also determined sulphur gravimetrically after chromatographic separation (Nydahl, Anal. Chem., 1954, **26**, 580) and found 0.022%. Nos. 4 and 6 also used combustion methods and found 0.025% in each case.

CHROMIUM

All Analysts determined chromium titrimetrically after oxidation with persulphate/silver nitrate. Nos. 1 and 8 used methods in which the tungsten was held in solution with fluoride. After the oxidation No. 1 titrated directly with ammonium ferrous solution and made a correction for vanadium (Analoid Method No. 37). No. 8 added excess of ammonium ferrous sulphate and back-titrated with permanganate (Analoid Method No. 35). Nos. 2, 3, 4, 6 and 7 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. 5 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.

Analyst No. 2 also used Analoid method No. 35 and found 5.35%. No. 4 also used a method involving a preliminary separation of tungsten and titration of the oxidized solution with ferrous sulphate/dichromate; the result, after applying a correction for vanadium, was 5.36%. No. 8 also used a photometric method based on measurement of the dichromate colour produced by oxidation with persulphate and found 5.38%.

MOLYBDENUM

All Analysts determined molybdenum photometrically as oxythiocyanate. Nos. 1, 2, 4, 5 and 7 used the Standard method B.S. 1121: Part 48: 1966 in which the coloured complex is extracted with n-butyl acetate. Nos. 3, 6 and 8 measured the colour in aqueous solution.

Analyst No. 2 also determined molybdenum photometrically with toluene 3:4-dithiol (Wells and Pemberton, Analyst, 1947, **72**, 185) and found 0.54%. No. 4 also used an oxythiocyanate method in which the colour was measured in aqueous solution and found 0.53%. No. 8 used two alternative methods, viz. the Analoid photometric method No. 42 which gave a result of 0.51% and a gravimetric method depending on precipitation with benzoin α-oxime which gave a result of 0.48%.

NICKEL

All Analysts except No. 7 determined nickel photometrically with dimethylglyoxime. No. 1 used the Analoid method No. 44. Nos. 2, 3, 4 and 5 used the Standard method B.S. 1121: Part 6: 1967. No. 7 determined nickel titrimetrically after precipitation with dimethylglyoxime according to the Standard method B.S. 1121: Part 37: 1961

Analyst No. 4 also determined nickel gravimetrically by a modified Kirtchik method and found 0.14%.

COBALT

All Analysts except Nos. 6 and 7 determined cobalt photometrically with nitroso-R-salt according to the Standard method B.S. 1121: Part 30: 1954. No. 6 determined cobalt gravimetrically by double precipitation with 1-nitroso-2-naphthol. No. 7 determined cobalt by potentiometric titration with ferricyanide in ammoniacal citrate solution.

COPPER

Analysts Nos. 1, 5, 7 and 8 determined copper photometrically. Nos. 1, 5 and 8 used the Standard method B.S. 1121: Part 36: 1956 based on extraction of copper with 2-2' diquinolyl in amyl alcohol. No. 7 used a *bis*-cyclohexanone oxalyldihydrazone method after preliminary separation of copper as the sulphide. Nos. 2, 3, 4 and 6 determined copper iodometrically after separation of tungsten; Nos. 2, 3 and 4 used the Standard method B.S. 1121: Part 14: 1956.

TIN

All Analysts used the Standard titrimetric method B.S. 1121: Part 20: 1961 which includes a preliminary separation of the tin as sulphide.

VANADIUM

All Analysts except No. 5 determined vanadium titrimetrically by oxidation with permanganate and titration with ammonium ferrous sulphate. Nos. 1 and 8 used the Analoid Method No. 34. Nos. 2, 3, 4 and 7 used the Standard method B.S. 1121: Part 25: 1956 (Method A). No. 5 determined vanadium photometrically as phosphovanadotungstic acid.

TUNGSTEN

All Analysts determined tungsten gravimetrically after separation of tungstic oxide by hydrolysis. Nos. 1, 2, 3, 4, 5, 6 and 8 used the Standard method B.S. 1121: Part **40**: 1967. All except No. 7 determined the impurities in the final tungstic oxide residue and made the appropriate corrections. No. 7 used a method in which care was taken to avoid drying out of iron salts on the beaker during the evaporation and the solution was diluted to a large volume before adding cinchonine to complete the precipitation of the tungstic oxide; in this way contamination of the precipitate was prevented and the need for purification of the precipitate eliminated.

DESCRIPTION OF SAMPLE

British Chemical Standard - bottles of 100g chips graded 1700 - 180µm (10 - 85 mesh) for chemical analysis.

INTENDED USE & STABILITY

The chip sample, BCS-CRM 241/2 (ECRM 251-1), 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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