



BUREAU OF ANALYSED SAMPLES LTD

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BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS[†]/SS[‡]-CRM No. 290/2 (ECRM 253-1)

13% MANGANESE 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 SWINBURN, D. G., AIM,
 Steel Castings Research and Trade Association, Sheffield.

ANALYSTS representing MANUFACTURERS and USERS

- 3 BAGSHAWE, B., AMet, FIM, MInstF,
 Brown-Firth Research Laboratories, Sheffield.

ANALYSTS representing MANUFACTURERS and USERS (cont.)

- 4 BROWN, J., BSC, River Don and Associated Works, Sheffield.
 5 HOBSON, J. D., BSc, PhD, AMet, FRIC,
 Dunford Hadfields Ltd., Sheffield.
 6 JACKSON, L., BSc, BSC, Workington Works, Workington.
 7 McDONALD, R., CGIA, AIM,
 Head Wrightson and Co. Ltd., Thornaby.
 8 PRIESTLEY, I., Edgar Allen Foundry Ltd., Sheffield.

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Ni	Mo	Co	Cu	N	V
1	1.15	0.35	12.45	0.041	0.020	0.16	0.28	0.031	0.36	0.17	...	0.021
2	1.17	0.34	12.41	0.044	0.018	0.16	0.29
3	1.15	0.33	12.48	0.040	0.020	0.16	0.30
4	1.16	0.33	12.46	0.044	0.018	0.17	0.29
5	1.16	0.34	12.60	0.043	0.018	0.17	0.29	0.02
6	1.15	0.34	12.45	0.044	0.019	0.17	0.29	0.013	...
7	1.15	0.34	12.51	0.044	0.020	0.16	0.29	...	0.34	0.02
8	1.14	0.34	12.44	0.040	0.021	0.17	0.28
M_M	1.15	0.34	12.5	0.042	0.019	0.16	0.29
s_M	0.01	0.01	0.1	0.002	0.002	0.01	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 (C_v)

mass content in %

	C	Si	Mn	P	S	Cr	Ni
C_v	1.15	0.34	12.5	0.042	0.019	0.16	0.29
C(95%)	0.01	0.01	0.1	0.002	0.001	0.01	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.

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.

BCS/SS-CRM No. 290/2 (ECRM 253-1)

13% MANGANESE STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 7 and 8 determined carbon gravimetrically by the British Standard Carbon Method 1*. Nos. 2 and 4 used a non-aqueous titration method (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399). No. 3 used a low pressure method (Cook and Speight, Analyst, 1956, **81**, 144). No. 5 determined carbon coulometrically. No. 6 absorbed the carbon dioxide on a molecular sieve, released it by heating and carried out the final measurement by thermal conductivity.

Analysts Nos. 3 and 5 also determined carbon by both non-aqueous titration and infrared absorption. Results were as follows:- non-aqueous titration 1.15% (No. 3), 1.16% (No. 5); infrared absorption 1.15% (No. 3), 1.16% (No. 5).

SILICON

All Analysts determined silicon gravimetrically using the British Standard Silicon Method 1*.

Analysts Nos. 3, 4 and 7 also used photometric methods depending on the formation of molybdenum-blue and found 0.32%, 0.33% and 0.34% respectively.

MANGANESE

All Analysts determined manganese titrimetrically. Nos. 1, 2, 4, 5 and 8 used the British Standard Manganese Method 1* which includes a zinc oxide separation. No. 3 titrated with permanganate in fluoride solution (Headridge and Taylor, Analyst, 1962, **87**, 905). No. 6 used a bismuthate method. No. 7 titrated potentiometrically with permanganate in tetra-sodium pyrophosphate solution, the method being modified to eliminate chromium interference (Scribner, Anal. Chem., 1960, **32**, 970).

Analyst No. 3 used two alternative methods: (a) British Standard Manganese Method 3* (b) the same method modified to eliminate chromium interference (Scribner, *loc. cit.*); these gave results of 12.64% and 12.57% respectively. No. 5 also used a photometric periodate method in conjunction with an automatic analyser and found 12.52%.

PHOSPHORUS

All Analysts determined phosphorus photometrically as phosphovanadomolybdic acid according to the British Standard Phosphorus Method 2*.

Analyst No. 5 also used a photometric molybdenum-blue method in conjunction with an automatic analyser and found 0.044%.

SULPHUR

Analysts Nos. 1, 6 and 8 determined sulphur gravimetrically according to the British Standard Sulphur Method 1*. The remaining Analysts determined sulphur by combustion in oxygen and titration with sodium borate solution (No. 7 carried out a potentiometric titration).

Analyst No. 5 also used the British Standard Sulphur Method 1* and found 0.018%. No. 6 also determined sulphur by combustion and found 0.019%.

CHROMIUM

Analysts Nos. 1, 4, 5, 7 and 8 determined chromium titrimetrically after oxidation with persulphate/silver nitrate. Nos. 1 and 7 titrated with ammonium ferrous sulphate solution using sodium diphenylamine sulphonate indicator and applied corrections for the small amount of vanadium present. Nos. 4 and 5 titrated with ammonium ferrous sulphate/dichromate. No. 7 titrated with ammonium ferrous sulphate/permanagante according to the British Standard Chromium Method 1*. Nos. 2, 3 and 6 determined chromium photometrically with diphenylcarbazide.

Analysts Nos. 2 and 3 also determined chromium titrimetrically and found 0.18% and 0.17% respectively.

NICKEL

Analysts Nos. 1, 2, 4 and 7 determined nickel titrimetrically after precipitation with dimethylglyoxime. No. 1 dissolved the precipitate in sulphuric acid and boiled with excess of ferric sulphate: the ferrous salt thus formed was titrated with dichromate solution (Analoid Method No. 62). No. 2 titrated with EDTA. Nos. 4 and 7 titrated cyanometrically according to the British Standard Nickel Method 1*. Nos. 3, 5, 6 and 8 determined nickel photometrically with dimethylglyoxime/iodine. No. 3 used a modified version of the British Standard Nickel Method 3*. Nos. 5 and 6 first separated the nickel with dimethylglyoxime before determining it photometrically. No. 8 used the British Standard Nickel Method 3*.

Analyst No. 4 also used a modification of the British Standard Nickel Method 3* and found 0.28%. No. 5 also used a photometric method in conjunction with an automatic analyser (without previous separation of the nickel) and found 0.29%. No. 7 also determined nickel gravimetrically with dimethylglyoxime and found 0.29%.

MOLYBDENUM

Molybdenum was determined photometrically as the oxythiocyanate after extraction into butyl acetate (Analoid Method No. 63).

COBALT

Both Analysts determined cobalt photometrically with nitroso-R-Salt. No. 1 used the British Standard Cobalt Method 1* whereas No. 7 used a modification of the British Standard Cobalt Method 2*.

COPPER

Copper was determined photometrically with bis-cyclohexanone oxalyldihydrazone. (Analoid Method No. 65).

NITROGEN

The sample was fused in a current of helium and nitrogen determined by thermal conductivity.

VANADIUM

Analyst No. 1 separated iron by extraction into diethyl ether and determined vanadium photometrically as phosphovanadotungstate according to the Analoid Method No 59. Nos. 5 and 7 used the British Standard Vanadium Method 1*.

*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.

INTENDED USE & STABILITY

The finely divided sample, BCS-CRM 290/2 (ECRM 253-1), 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.

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For BUREAU OF ANALYSED SAMPLES LTD

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