MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS-CRM No. 225/2 (ECRM 155-1)

Ni-Cr-Mo 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 SCHOLES, P. H., *AMet*, British Iron and Steel Research Association, Sheffield.
2 COPPINS, W. C., *MSc, ARIC*, Ridsdale & Co. Ltd., Middlesbrough.
3 ATKINSON, C. D., *AMet*, Sheffield Testing Works Ltd., Sheffield.

ANALYSTS representing MANUFACTURERS and USERS

4 DICKINSON, E. A., *AMet, AIM*, Catton & Co. Ltd., Leeds.
5 HESLOP, S., *FIM, ACT*, The Rover Co. Ltd., Solihull.
6 KIDMAN, L., *AMet, AIM, AMInstF*, English Steel Corporation Ltd., Sheffield.
7 MATHIESON, V., Clyde Alloy Steel Co. Ltd., Motherwell.
8 MURRAY, G., Brown Bayley Steels Ltd., Sheffield.

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	*Cu*	*V*
1	0.40	0.24	0.57	0.021	0.012	1.08	0.35	1.42	…	…
2	0.41	0.23	0.56	0.018	0.012	1.08	0.35	1.43	*0.17*	*<0.01*
3	0.41	0.23	0.57	0.021	0.012	1.08	0.34	1.43	…	…
4	0.40	0.22	0.56	0.020	0.013	1.07	0.35	1.44	*0.17*	…
5	0.39	0.24	0.56	0.019	0.011	1.08	0.34	1.43	…	…
6	0.40	0.23	0.57	0.020	0.013	1.09	0.35	1.44	*0.16*	…
7	0.40	0.22	0.56	0.017	0.013	1.08	0.35	1.45	…	…
8	0.41	0.24	0.55	0.018	0.012	1.07	0.33	1.43	…	…
M_M	**0.40**	**0.23**	**0.56**	**0.019**	**0.012**	**1.08**	**0.34**	**1.43**	…	…
s_M	0.01	0.01	0.01	0.002	0.001	0.01	0.01	0.01	…	…

The above figures are those which each Analyst has decided upon after careful verification.

Figures in bold type certified, figures in small italic type only approximate.

M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means.

The following additional information was supplied by Analyst No. 6:- Sn 0.017%, As 0.035%, Sb 0.006%, Co 0.018%, Al 0.009%, Zr <0.01%, Nb 0.003%, Ta 0.001%, B 0.0007%, N 0.012%.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni
Cv	**0.40**	**0.23**	**0.56**	**0.019**	**0.012**	**1.08**	**0.34**	**1.43**
C(95%)	0.01	0.01	0.01	0.002	0.001	0.01	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.

BCS-CRM No. 225/2 (ECRM 155-1) Ni-Cr-Mo STEEL

NOTES ON METHODS USED

CARBON

Analyst No. 1 determined carbon both conductimetrically using the Wösthoff apparatus and gravimetrically; the results were 0.394% and 0.40% respectively. The other Analysts used the Standard gravimetric method B.S. 1121: Part 11 1948.

SILICON

Analyst No. 1 determined silicon photometrically as molybdenum-blue using the Standard photometric method B.S. 1121: Part 15: 1949. The other Analysts determined silicon gravimetrically. Nos. 2, 4, 5, 6, 7 and 8 used the Standard method B.S. 1121: Part 10: 1948. No. 3 used a nitro-sulphuric acid method.

Analysts Nos. 4 and 6 also determined silicon photometrically as molybdenum-blue and found 0.23% in each case. No. 8 also determined silicon gravimetrically by evaporation with hydrochloric acid and found 0.22%.

PHOSPHORUS

Analysts Nos. 1, 3, 4, 7 and 8 determined phosphorus titrimetrically after precipitation as ammonium phosphomolybdate. No. 4 precipitated the phosphomolybdate according to the Standard gravimetric method B.S. 1121: Part 9: 1948, No. 8 according to the original Standard method B.S. 1121: Part 1: 1943. Nos. 2, 5 and 6 determined phosphorus gravimetrically by precipitation of phosphomolybdate and conversion to lead molybdate according to the Standard method B.S. 1121: Part 9: 1948.

Analysts Nos. 1 and 6 also determined phosphorus photometrically as phosphovanadomolybdic acid (Elwell and Wilson, Analyst, 1956, **81**, 136; 1957, **82**, 453) and both found 0.020%.

SULPHUR

Analysts Nos. 1, 2, 3, 5, 6 and 8 determined sulphur gravimetrically as barium sulphate. All except No. 3 followed the procedure of the Standard method B.S. 1121: Part 1A: 1957. Nos. 4 and 7 determined sulphur by combustion. No. 4 absorbed the sulphur gases in hydrogen peroxide and titrated with sodium borate solution. No. 7 absorbed in dilute hydrochloric acid and titrated with iodide/iodate solution.

Analysts Nos. 1, 6 and 8 also used combustion methods and found 0.012%, 0.013% and 0.012% respectively. No. 5 also used a combustion method on a 10g. sample, but after absorbing the sulphur gases in hydrogen peroxide the sulphur was precipitated as barium sulphate and determined gravimetrically; the result by this method was 0.010%.

CHROMIUM

All Analysts dissolved the sample in sulphuric-phosphoric acid mixture and oxidized the chromium with persulphate/silver nitrate. Nos. 1, 4, 5, 7 and 8 titrated with ammonium ferrous sulphate/permanganate according to the Standard titrimetric method B.S. 1121: Part 13: 1954, No. 2 titrated directly with ammonium ferrous sulphate (Analoid method No. 37), No. 6 titrated with ferrous sulphate/dichromate.

Analyst No. 4 also used a method similar to the Analoid method and found 1.06%. No. 5 also used a method similar to the Standard titrimetric method but with N-phenylanthranilic acid as indicator and found 1.08%.

MOLYBDENUM

Analysts Nos. 1, 2, 4, 5, 6, 7 and 8 determined molybdenum photometrically as molybdenum oxythiocyanate. Nos. 1, 4 and 6 used methods involving extraction of the coloured complex with butyl acetate. No. 2 used the Analoid Method No. 42. Nos. 5, 7 and 8 used the Standard method B.S. 1121: Part 34: 1955. No. 3 determined molybdenum both photometrically and gravimetrically by means of benzoin α-oxime; the results were 0.34% and 0.33% respectively.

NICKEL

All Analysts precipitated nickel with dimethylglyoxime and completed titrimetrically. Nos. 1 and 5 used the Standard method B.S. 1121: Part 37: 1961 in which the nickel is titrated with cyanide solution. Nos. 3, 6 and 8 used similar methods. No. 2 dissolved the dimethylglyoxime precipitate in dilute sulphuric acid and boiled with excess of ferric sulphate; the ferrous salt thus formed was titrated with dichromate solution. No. 4 precipitated the nickel according to the Standard method, but completed by titration with EDTA. No. 7 used both the Standard titrimetric method and a photometric method depending on the use of dimethylglyoxime; the result was 1.45% in each case.

Analysts Nos. 4 and 5 also used photometric dimethylglyoxime methods and found 1.45% and 1.44% respectively.

MANGANESE

Analyst No. 1 used both the Standard titrimetric method B.S. 1121: Part 16: 1949 which involves a zinc oxide separation and the Standard photometric method B.S. 1121: Part 23: 1951 which depends on oxidation with periodate. Nos. 2, 3 and 7 determined manganese titrimetrically by oxidation with persulphate/silver nitrate and titration with arsenite/nitrite solution (Nos. 2 and 3) or arsenite solution (No. 7). No. 4 used the Standard titrimetric method. Nos. 5 and 6 used the Standard photometric method. No. 8 used a photometric method depending on oxidation with persulphate/silver nitrate.

Analysts Nos. 3 and 8 also used titrimetric bismuthate methods and found 0.57% and 0.55% respectively. No. 4 also used the Standard photometric method and found 0.55%.

COPPER

*No. 2 used the Standard photometric method B.S. 1121: Part 36: 1956. Nos. 4 and 6 used the bis-cyclohexanone oxalyldihydrazone method (Haywood and Sutcliffe, Analyst, 1956, **81**, 651).*

VANADIUM

Titrimetric Analoid Method No. 34.

TIN

Reduction with aluminium and titration with iodate.

ARSENIC

Standard titrimetric method B.S. 1121: Part 38: 1958.

ANTIMONY

*Photometric rhodamine B method (Kidman and Waite, Metallurgia, 1962, **66**, 143).*

COBALT

Standard photometric method B.S. 1121: Part 42: 1961.

ALUMINIUM

*Photometric solochrome cyanine R method (Scholes and Smith, Analyst, 1958, **83**, 615).*

ZIRCONIUM

Gravimetric mandelic acid method

NIOBIUM

*Chromatographic method (Hunt and Wells, Analyst, 1954, **79**, 351).*

TANTALUM

*Photometric pyrogallol method (Kidman and White, Metallurgia, 1961, **64**, 153).*

BORON

*Photometric dianthrimide method (BISRA Methods of Analysis Committee, J. Iron Steel Inst., 1958, **189**, 227).*

NITROGEN

Standard titrimetric method, B.S. 1121: Part 39: 1959

INTENDED USE & STABILITY

The chip sample, BCS-CRM 225/2 (ECRM 155-1), 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.

NEWHAM HALL, NEWBY,
MIDDLESBROUGH, ENGLAND, TS8 9EA
Email: enquiries@basrid.co.uk
Website: www.basrid.co.uk

For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

Preliminary Edition ..... January 1963
Main Edition ..... April 1964
Main Edition (revised with C(95%) and s_M values for each element) ..... December 2010