MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD.

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 219/4 (ECRM 153-1)
Ni-Cr-Mo STEEL

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

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ANALYSES

Mean of 4 values – mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Sn
1	0.318	0.076	0.81	0.011	0.025	0.67	0.58	2.54	0.086	0.010
2	…	…	0.82	…	…	0.67	0.58	2.56	…	…
3	0.310	0.074	0.80	0.011	0.028	0.65	0.58	2.57	0.085	0.010
4	0.317	0.085	0.82	0.012	0.026	0.66	0.57	2.54	0.089	0.009
5	0.315	0.079	0.80	0.011	0.028	0.65	0.60	2.54	0.088	0.013
6	0.313	0.08-	0.81	0.011	0.027	0.65	0.57	2.56	0.082	0.011
7	0.308	0.074	0.82	0.011	0.027	0.65	0.57	2.57	0.085	0.010
8	0.312	0.089	0.83	0.012	0.026	0.66	0.58	2.54	0.093	0.012
9	0.317	0.076	0.81	0.012	0.026	0.66	0.57	2.57	0.093	0.010
M_M	**0.314**	**0.079**	**0.81**	**0.011**	**0.027**	**0.66**	**0.58**	**2.55**	**0.088**	**0.011**
s_M	0.004	0.006	0.01	0.001	0.001	0.01	0.01	0.02	0.004	0.001
s_W	0.003	0.002	0.01	0.001	0.001	0.01	0.01	0.01	0.002	0.001

(Additional Information:- Analyst No. 1:- Total Al 0.003%)

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

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

CERTIFIED VALUES

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Sn
M_M	**0.314**	**0.079**	**0.81**	**0.011**	**0.027**	**0.66**	**0.58**	**2.55**	**0.088**	**0.011**
C(95%)	0.004	0.005	0.01	0.001	0.001	0.01	0.01	0.02	0.004	0.001

The half width confidence interval **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:1989 section 4.

DESCRIPTION OF SAMPLE

Bottles of 100g chips graded 1700 – 250µm (10 – 60 mesh) for chemical analysis.

PTO

BCS-CRM 219/4 (ECRM 153-1)
Ni-Cr-Mo STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 8 determined carbon gravimetrically using the British Standard Carbon Method 1*. Nos. 3 and 4 determined carbon by infrared measurement of the evolved gases. Nos. 6 and 9 used coulometric methods, No. 6 according to Hobson and Leigh, Analyst, 1974, **99**, 93. No. 5 determined carbon by non-aqueous titration and No.7 used a low pressure method.

Analysts Nos. 6 and 8 also determined carbon by non-aqueous titration and found 0.31% and 0.311% respectively. No. 7 also used an infrared method and found 0.310%.

SILICON

Analysts 1, 7 and 9 used molybdenum-blue spectrophotometric methods, No. 1 used the Analoid Method No. 43. Nos. 3, 4, 5, 6 and 8 determined silicon gravimetrically after double dehydration with perchloric acid, according to the British Standard Silicon Method 1*.

Analysts Nos. 1, 7 and 9 also used the British Standard Silicon Method 1* and found 0.070%, 0.075% and 0.074% respectively.

MANGANESE

Analysts Nos. 1, 5 and 8 determined manganese titrimetrically after oxidation with persulphate/silver nitrate. No.1 titrated with arsenite/nitrite according to Analoid Method No.53 and Nos. 5 and 8 titrated with arsenite. Analyst No. 2 used ICP-OES, calibrated with pure manganese. Analysts Nos. 3, 4, 6, 7 and 9 determined manganese colorimetrically after periodate oxidation according to the British Standard Manganese Method 2*.

Analyst No. 6 also used a periodate colorimetric method with an automatic analyser and found 0.81%. No. 9 also used FAAS spectroscopy and found 0.80%

PHOSPHORUS

Analysts Nos. 1, 3, 4, 6 and 7 determined phosphorus colorimetrically as phosphovanadomolybdate according to the British Standard Phosphorus Method 2*. Nos. 5 and 9 determined phosphorus titrimetrically after precipitating as phosphomolybdate. No. 8 used the British Standard Phosphorus Method 1* in which phosphorus is determined gravimetrically after precipitation as phosphomolybdate and conversion to lead molybdate.

Analyst No. 6 also used a molybdenum blue colorimetric method with an automatic analyser and found 0.011%.

SULPHUR

Analysts Nos. 1 and 6 determined sulphur gravimetrically according to the British Standard Sulphur Method 1*. All other analysts determined sulphur by combustion methods. No.3 completed by borate titration, Nos. 4 and 7 by infrared absorption and Nos. 5 and 8 by iodate titration.

Analyst No. 6 also determined sulphur by combustion and found 0.027%.

CHROMIUM

All analysts except No.2 determined chromium titrimetrically after oxidation with persulphate/silver nitrate. No. 1 used the Analoid Method No. 37, Nos. 3, 5, 7, 8 and 9 used the British Standard Chromium Method 1*. Analyst No. 2 used ICP-OES, calibrated with pure chromium.

Analyst No. 9 also used FAAS and found 0.65%.

MOLYBDENUM

All analysts except No.2 determined molybdenum colorimetrically as oxythiocyanate. No. 1 measured the colour directly using Analoid Method No. 42. Nos. 4, 5, 6, 7 and 8 extracted the coloured complex into butyl acetate, Nos. 5, 6, 7 and 8 according to British Standard Molybdenum Method 1*. Analyst No. 2 used ICP-OES, calibrated with pure molybdenum.

Analysts Nos. 6 and 8 also used alternative oxythiocyanate methods, No. 6 with an automatic analyser, and each found 0.58%.

NICKEL

Analysts Nos. 1, 3, 4, 5, 6, 7 and 9 used methods involving precipitation with dimethylglyoxime. No. 1 dissolved the precipitate in dilute sulphuric acid, boiled with an excess of iron (III) ammonium sulphate and titrated with dichromate (Analoid Method No. 61). No.4 dissolved the precipitate and titrated with EDTA. Nos. 3, 5, 7 and 9 dissolved the precipitate and titrated with cyanide according to British Standard Nickel Method 1*. No.6 completed gravimetrically. Analyst No. 2 used ICP-OES, calibrated with pure nickel. Analyst No. 8 used a colorimetric dimethylglyoxime method.

Analyst No. 6 also determined nickel colorimetrically with dimethylglyoxime according to British Standard Nickel Method 3* and found 2.57%. Analyst No. 9 also used FAAS and found 2.55%.

COPPER

Analyst No. 1 determined copper by FAAS. No. 3 used a colorimetric *bis*-cyclohexanone oxalyldihydrazone method. Nos. 4 and 5 separated copper as sulphide and completed titrimetrically according to British Standard Copper Method 2*. Nos. 6, 7 and 8 determined copper colorimetrically as 2-2' diquinolyl according to British Standard Copper method 3*. No. 7 used a colorimetric diethyldithiocarbamate method.

Analyst No. 1 also used a direct *bis*-cyclohexanone oxalyldihydrazone colorimetric method and found 0.098%. No. 5 also used a diethyldithiocarbamate method and found 0.087%.

TIN

All analysts determined tin by reduction with aluminium and titration with iodate solution. Nos. 4, 5, 6, 7 and 9 used British Standard Tin Method 1*.

* Methods for Sampling and Analysis of Iron, Steel and Other Ferrous Metals, B.S. Handbook No. 19, first published 1970 by the British Standards Institution, London.

Traceability: The majority of Analysts calibrated using pure metals, metal oxides or primary chemicals.

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MIDDLESBROUGH
ENGLAND

For BUREAU OF ANALYSED SAMPLES LTD.
P.D. RIDSDALE,
Chairman

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