MAIN EDITION

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SPECTROSCOPIC STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

SS-CRM No. 487/1

HIGH SPEED TOOL 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

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4 BATTY, G., Ross and Catherall Ltd., Sheffield.
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8 MATHIESON, V., BSC Craigneuk Works, Motherwell.
9 SAXBY, A., *AMet*, O.H. Steel Founders and Engineers Ltd., Sheffield.
10 SHAW, D., Howmet Alloys International Ltd., Exeter.
11 WILD, J. A., Eclipse Tools Ltd., Sheffield.
12 WILKINS, T. J. and BRADBURY, A., Edgar Allen Balfour Steels Ltd., Manchester.
13 WISE, R. A., Langley Alloys Ltd., Slough.

ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Al (Total)	Co	V	W	*Ni*	*As*	*Sn*
1	1.03	0.18	0.27	0.022	0.031	3.89	9.51	0.005	8.03	1.15	1.85	*0.14*	*0.014*	*0.005*
2	1.03	…	0.27	…	0.031	3.96	9.34	…	…	1.14	…	…	…	…
3	1.02	0.17	0.26	0.023	0.027	3.88	9.26	…	8.10	1.15	1.82	…	…	…
4	…	…	…	…	…	3.89	…	0.005	…	…	1.85	…	…	…
5	1.01	0.17	0.26	0.021	0.028	…	…	…	…	…	…	…	…	…
6	1.02	0.20	0.28	0.022	0.030	…	…	0.008	…	…	…	…	…	…
7	…	…	…	…	…	…	…	0.005	…	…	…	…	*0.011*	*0.007*
8	1.02	0.19	0.24	…	0.029	…	…	…	…	…	…	…	…	…
9	1.02	0.18	0.27	0.024	0.030	…	….	…	…	1.12	…	…	…	…
10	…	…	…	…	…	3.90	9.58	0.007	7.95	1.11	1.84	…	…	…
11	…	…	…	…	…	3.92	9.19	…	7.95	1.17	1.74	…	…	…
12	…	…	…	…	…	3.91	9.56	…	7.76	1.15	1.70	*0.15*	…	…
13	1.03	0.17	0.27	…	…	…	…	…	7.89	…	…	…	…	…
M_M	**1.02**	**0.18**	**0.26**	**0.022**	**0.029**	**3.91**	**9.41**	**0.006**	**7.95**	**1.14**	**1.80**	…	…	…
s_M	0.01	0.02	0.02	0.002	0.002	0.03	0.17	0.002	0.12	0.03	0.07	…	…	…

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.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Al (Total)	Co	V	W
Cv	**1.02**	**0.18**	**0.26**	**0.022**	**0.029**	**3.91**	**9.41**	**0.006**	**7.95**	**1.14**	**1.80**
C(95%)	0.01	0.02	0.01	0.002	0.002	0.03	0.18	0.002	0.13	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.

SS-CRM 487/1 HIGH SPEED TOOL STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 13 determined carbon by non-aqueous titration according to the British Standard Carbon Method 4*. Nos. 2, 5, 6, 8 and 9 used high frequency combustion with infrared measurement. No. 3 used a gravimetric method.

Analyst No. 8 also determined carbon by non-aqueous titration and found 1.02%.

SILICON

Analysts Nos. 1, 5 and 13 determined silicon gravimetrically after double dehydration with perchloric acid. Nos. 1 and 5 followed the procedure of the British Standard Silicon Method 1*. Nos. 3, 8 and 9 used photometric methods depending upon the formation of the molybdenum blue complex, Nos. 8 and 9 according to the British Standard Silicon Methods 4* and 3* respectively. No. 6 used a sulphuric acid gravimetric method (ASTM 350-72).

MANGANESE

Analysts Nos. 1, 2, 3, 6 and 9 determined manganese photometrically after oxidation with periodate according to the British Standard Manganese Method 2*. No. 5 used atomic-absorption spectrometry. Nos. 8 and 13 determined manganese titrimetrically after zinc oxide separation according to the British Standard Manganese Method 1*.

Analyst No. 8 also determined manganese titrimetrically with arsenite after oxidation with persulphate/silver nitrate and found 0.25%.

PHOSPHORUS

All Analysts determined phosphorus photometrically as phosphovanadomolybdate. Nos. 1, 5 and 9 followed the procedure of the British Standard Phosphorus Method 2*.

SULPHUR

Analysts Nos. 1, 3, 6, 8 and 9 determined sulphur by combustion. Nos. 1 and 3 absorbed the evolved gases in hydrogen peroxide solution and titrated with borate. Nos. 6, 8 and 9 used high frequency combustion with infrared measurement. Analysts Nos. 2 and 5 used gravimetric methods. No. 2 followed the procedure of the British Standard Sulphur Method 1* whereas No. 5 first separated sulphur chromatographically on an alumina column before precipitation as barium sulphate. (Nydahl, Anal. Chem., 1954, **26**, 580).

CHROMIUM

All Analysts determined chromium titrimetrically with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. No. 1 followed the procedure of the Analoid Method No. 37 and Nos. 3 and 11 that of the British Standard Chromium Method 1*.

MOLYBDENUM

All Analysts determined molybdenum photometrically as oxythiocyanate. No. 1 followed the procedure of the Analoid Method No. 42.

Analyst No. 1 also determined molybdenum by the British Standard Molybdenum Method 1* and found 9.50%.

ALUMINIUM (Total)

All Analysts except No. 6 determined aluminium by atomic-absorption spectrometry. No. 6 used a photometric method involving extraction of the aluminium complex with 8-hydroxyquinoline into chloroform (Anal. Chem., 1966, **38**, 1608).

COBALT

All Analysts except No. 13 determined cobalt photometrically with nitroso-R-salt according to the British Standard Cobalt Method 1*. Analyst No. 13 used -absorption spectrometry.

VANADIUM

All Analysts determined vanadium titrimetrically with ammonium ferrous sulphate after oxidation with permanganate. Analysts Nos. 1 and 9 followed the procedure of the Analoid Method No. 34 and Nos. 3, 10 and 11 that of the British Standard Vanadium Method 1*.

TUNGSTEN

Analyst No. 3 determined tungsten gravimetrically after separation of tungstic oxide by hydrolysis with corrections made for co-precipitated elements according to the British Standard Tungsten Method 1*. The remaining Analysts used thiocyanate photometric methods with corrections for molybdenum and vanadium.

NICKEL

Both Analysts determined nickel photometrically with dimethylglyoxime, No. 1 according to the Analoid Method No. 44.

ARSENIC

Analyst No. 1 determined arsenic titrimetrically with iodine according to the British Standard Arsenic Method 1. No. 7 used atomic-absorption spectrometry.*

TIN

Both Analysts used atomic-absorption spectrometry.

*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.

DESCRIPTION OF SAMPLE

Spectroscopic Standard - 38 mm diameter x 19 mm thick discs for spectroscopic analysis.

INTENDED USE & STABILITY

The disc sample, SS-CRM 487/1, is intended for establishing and checking the calibration of Optical Emission and X-Ray Spectrometers for the analysis of similar materials. The "as received" working surface of the sample should be linished before use to remove any protective coating. It will remain stable provided that it is not subject to excessive heat (e.g., during preparation of the working surface). Due to slight segregation of certain elements an area 6mm in diameter in the centre of the disc samples should be avoided for emission spectroscopy.

TRACEABILITY

The traceability of this SS-CRM to the SI is ensured by the use of either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds.

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For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,
Managing Director

Preliminary Edition ..... July 1980
Main Edition ..... May 1984
Main Edition (revised with C(95%) and s_M values for each certified element) ..... November 2013