



CASTINGS TECHNOLOGY INTERNATIONAL

and

BUREAU OF ANALYSED SAMPLES LTD.



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SPECTROSCOPIC STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

SS-CRM No. 495/4

HIGH MANGANESE STEEL

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

The chill cast blocks from which this SS-CRM was prepared were specially cast by Castings Technology International, Sheffield (CTI).

The preparation of representative samples for chemical analysis and the certification by cooperative analysis was undertaken by Bureau of Analysed Samples Ltd. Bureau of Analysed Samples Ltd. has been accredited by The United Kingdom Accreditation Service (UKAS) in accordance with ISO Guide 34 and ISO/IEC 17025 as a Reference Material Producer No. 4004.

CO-OPERATING ANALYSTS AND FIRMS

INDEPENDENT ANALYSTS

1. CROCKER, F. H., Pattinson & Stead (2005) Ltd, Middlesbrough.
2. JONES, S. J., BSc, CChem, MRSC, Ridsdale & Co Ltd, Middlesbrough.
3. MCKENNA, H., Bodycote Materials Testing Teesside, Middlesbrough.

ANALYSTS REPRESENTING MANUFACTURERS and USERS

4. CROOK, D., Corus Strip Products, Llanwern.
5. HURDITCH, P., London & Scandinavian Analytical Services, Rotherham.
6. WILSON, J., Allvac Ltd., Sheffield.

ANALYSES

Mean of 4 values – mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co	Cu	N	V
1	0.8011	0.6777	13.1463	0.0933	0.0119	2.2342	0.2649	1.5994	0.0073	0.0121	0.0223	0.0409	0.0542
2	0.8019	0.6739	13.1880	0.0942	0.0132	2.2110	0.2647	1.6073	0.0075	0.0123	0.0231	0.0418	0.0555
3	0.7988	0.6684	13.0450	0.0923	0.0130	2.2260	0.2581	1.6178	0.0076	0.0112	0.0226	0.0410	0.0528
4	0.7871	0.6785	13.0993	0.0893	0.0118	2.1960	0.2630	1.6447	0.0094	0.0130	0.0217	0.0433	0.0499
5	0.7918	0.6706	13.1394	...	0.0128	2.2503	0.2726	1.6250	0.0095	0.0113	0.0231	0.0416	0.0514
6	0.7978	0.6742	13.0221	0.0935	0.0141	2.2211	0.2753	1.6243	0.0077	0.0119	0.0204	0.0412	0.0512
M_M	0.7964	0.6739	13.1067	0.0925	0.0128	2.2231	0.2664	1.6198	0.0082	0.0120	0.0222	0.0416	0.0525
s _M	0.0058	0.0040	0.0637	0.0020	0.0009	0.0188	0.0064	0.0158	0.0011	0.0007	0.0011	0.0009	0.0021
s _w	0.0035	0.0047	0.0445	0.0016	0.0004	0.0194	0.0015	0.0109	0.0004	0.0003	0.0002	0.0003	0.0006

As	Sn	Ti
...
0.0210	<0.0004	0.0010
...
0.0197	...	0.0016
...	<0.05	<0.001
...

Figures in italic print are for information only

Additional Information: - Analyst No. 5 determined W and Nb by XRF and O by thermal conductivity and found <0.05%, 0.0073% and 0.0206% respectively.

M_M: Mean of the intralaboratory means. **s_M**: Standard deviation of the intralaboratory means. **s_w**: Intralaboratory standard deviation.

CERTIFIED VALUES (C_v)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co	Cu	N	V
C_v	0.796	0.674	13.11	0.093	0.0128	2.223	0.266	1.620	0.0082	0.0120	0.0222	0.0416	0.0525
C(95%)	0.007	0.005	0.07	0.003	0.0010	0.020	0.007	0.017	0.0011	0.0008	0.0011	0.0010	0.0022

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 and 2 determined carbon by non-aqueous titration of the evolved carbon dioxide after combustion in a stream of oxygen. No. 2 used the standard method BS 6200.3.8.2:1991. The remaining Analysts determined carbon using high frequency combustion and infrared absorption.

SILICON

All Analysts, except for Nos. 3 and 5, determined silicon gravimetrically, dehydrating the silicon using perchloric acid. Analysts Nos. 3 and 5 determined silicon using Inductively Couple Plasma Optical Emission Spectroscopy (ICP-OES).

MANGANESE

Analyst No. 1 determined manganese by potentiometric titration with Mn (VII) in a pyrophosphate medium. Analysts Nos. 2 and 6 determined manganese photometrically after oxidation with periodate and the remaining Analysts used ICP-OES.

PHOSPHORUS

Analysts Nos. 1, 2 and 6 determined phosphorus photometrically as phosphovanadomolybdate, No. 1 after extraction with 4-methylpentan-2-one, No. 2 according to EN 10184:1992 Method 1 and No. 6 without extraction. Analysts Nos. 3 and 4 used ICP-OES.

Analyst No. 6 also determined phosphorus by ICP-OES after carefully matching the matrix of the calibration solutions with that of the sample solutions and found 0.0906%.

SULPHUR

Analyst No. 1 determined sulphur gravimetrically by acidimetric titration in a hydrogen peroxide. Analyst No. 2 determined sulphur by precipitation as barium sulphate. The remaining analysts determined sulphur using high frequency combustion and infrared absorption.

CHROMIUM

Analysts Nos. 1 and 6 determined chromium by titration with Fe (II) after oxidation with persulphate. The remaining Analysts used ICP-OES.

MOLYBDENUM

Analysts Nos. 1 and 6 determined molybdenum using Flame Atomic Absorption Spectrometry (FAAS). The remaining Analysts used ICP-OES.

NICKEL

Analyst No. 1 determined nickel using FAAS. All of the remaining Analysts, except for 6, determined nickel using ICP-OES. Analyst No. 6 precipitated nickel with dimethylglyoxime, dissolved the precipitate and titrated nickel with ethylene-diamine-tetra-acetic acid (EDTA).

ALUMINIUM

Analysts Nos. 1 and 6 determined aluminium by FAAS. The remaining Analysts used ICP-OES.

COBALT

Analyst No. 1 determined cobalt by FAAS. The remaining Analysts used ICP-OES.

COPPER

Analysts Nos. 1 and 6 determined copper by FAAS. The remaining Analysts used ICP-OES.

NITROGEN

Analyst No. 1 determined nitrogen photometrically with Nessler's reagent after a Kjeldahl digestion and distillation of the ammonia. Analyst No. 2 also used a Kjeldahl digestion and distilled the ammonia into boric acid before titrating with hydrochloric acid. The remaining Analysts used thermal conductivity methods after decomposition in a graphite crucible.

VANADIUM

Analysts Nos. 1 and 6 determined vanadium by FAAS. The remaining Analysts used ICP-OES.

Arsenic

Analyst No. 2 determined arsenic photometrically using silver diethylthiocarbamate after separation of arsine. Analyst No 4 used ICP-OES.

Tin

Analyst No. 2 determined tin by ICP-OES. Analyst No. 5 used XRF.

Titanium

Analyst Nos. 2 and 4 determined titanium by ICP-OES. Analyst No. 5 used XRF.

NOTE

The samples of this SS-CRM are in the form of chill cast rectangular blocks, each approximately 48mm x 42mm x 12mm thick with a single chilled working face. Spectroscopic reproducibility has been shown to be reliable to a depth of 5mm below the original surface of this block. Sparking must be made on the fully ground surface only and the sample should be discarded when this face has been ground back as far as the small shoulder around the edge of the sample.

Using vacuum direct reading optical emission spectrometers it has been established that materials of similar composition from different sources may respond differently. This SS-CRM is primarily intended for the construction of basic response curves which should be related to the response curves obtained from an identical examination of the user's own material. It will remain stable provided that the blocks are stored in a dry atmosphere. Provided that the material is suitably stored there will be no contribution to the uncertainty from the long term stability of this SS-CRM.

TRACEABILITY

The traceability of this SS-CRM has been established in accordance with principles of ISO Guides 30 – 35 and the International Vocabulary of Basic and General Terms in Metrology.

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above. Most methods used were either international or national standard methods or methods which are technically equivalent. All laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds. Four of the participating laboratories were accredited to ISO/IEC 17025 at the time of the analysis, although not necessarily for all of the constituents determined. It has been established statistically that there is no difference between the results of the accredited and the non-accredited laboratories.

Bureau of Analysed Samples Ltd is the reference material producer as defined in ISO Guide 34:2000 section 3.1 and is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2000 and 35:2006. Castings Technology International was responsible for the casting of the SS-CRM.

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd may be obtained from the address below:

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For BUREAU OF ANALYSED SAMPLES LTD.
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