BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

4004

CERTIFICATE OF ANALYSIS

BCS/SS-CRM No. 111/1

LOW CARBON STEEL

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

ANALYSES

Mean of 4 values - mass content in %

Analyst	C	Si	Mn	P	S	Cr	Mo	Ni	As	Co	Cu	N	Sn	Ti	V	Al
1	0.0064	0.0030	0.1639	0.0050	0.0047	0.0061	0.0004	0.0159	0.0012	0.0052	0.0089	0.0027	0.0005	0.0003	0.0002	*<0.001*
2	0.0075	0.0038	0.1587	0.0056	0.0045	0.0059	0.0007	0.0153	0.0011	0.0056	0.0086	0.0028	0.0005	…	0.0003	*<0.0005*
3	0.0061	0.0045	0.1679	0.0033	0.0050	0.0056	0.0003	0.0164	0.0014	0.0055	0.0096	0.0026	0.0007	0.0005	0.0002	*0.0005*
4	0.0071	…	0.1633	…	0.0060	0.0055	0.0006	0.0160	…	0.0055	0.0086	…	0.0006	0.0002	0.0002	*0.0013*
5	0.0073	0.0037	0.1611	0.0049	0.0046	0.0051	…	0.0170	…	0.0052	0.0090	0.0020	…	…	…	*<0.002*
6	0.0069	...	…	…	0.0048	…	…	…	…	…	…	0.0026	…	…	…	…
7	0.0074	0.0049	0.1621	0.0041	0.0052	0.0050	0.0004	0.0148	0.0010	…	0.0083	0.0022	0.0005	0.0004	0.0002	*0.0008*
8	…	…	0.1618	0.0043	…	0.0050	…	0.0150	0.0009	0.0052	0.0092	…	…	0.0004	0.0002	*0.0003*
9	0.0060	0.0035	0.1608	0.0037	0.0046	0.0056	…	0.0163	…	0.0050	0.0086	0.0025	…	…	…	…
10	…	…	0.1623	0.0041	0.0049	0.0053	…	0.0176	…	…	…	0.0025	…	…	…	…
11	…	…	0.1627	0.0050	…	…	…	…	…	…	…	…	…	…	…	…
12	0.0068	…	0.1610	0.0047	0.0051	0.0058	…	0.0167	…	…	…	0.0022	…	…	…	…
13	0.0083	…	…	…	…	…	…	…	…	…	…	…	…	…	…	…
M_M	**0.0070**	**0.0039**	**0.1623**	**0.0045**	**0.0049**	**0.0055**	**0.0005**	**0.0161**	**0.0011**	**0.0053**	**0.0089**	**0.0025**	**0.0006**	**0.0004**	**0.0002**	
s_M	0.0008	0.0007	0.0024	0.0007	0.0005	0.0004	0.0002	0.0009	0.0002	0.0003	0.0005	0.0003	0.0001	0.0002	0.0001	
s_w	0.0002	0.0003	0.0014	0.0002	0.0003	0.0002	0.0001	0.0003	0.0001	0.0001	0.0002	0.0002	0.0001	0.0001	0.0001	

M_M: Mean of the laboratory mean values. **s_M**: standard deviation of the laboratory mean values. **s_W**: average within laboratory standard deviation.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	As	Co	Cu	N	Sn	Ti	V
Cv	**0.0070**	**0.0039**	**0.1623**	**0.0045**	**0.0049**	**0.0055**	**0.0005**	**0.0161**	**0.0011**	**0.0053**	**0.0089**	**0.0025**	**0.0006**	**0.0004**	**0.0002**
C(95%)	0.0006	0.0008	0.0016	0.0005	0.0004	0.0003	0.0003	0.0007	0.0003	0.0003	0.0004	0.0003	0.0002	0.0002	0.0001
Minimum weight (g)	0.5	0.6	0.1	0.1	0.17	0.1	0.5	0.1	0.5	0.5	0.5	0.5	0.5	0.25	0.25

The half width confidence interval, C(95%), is an expression of the uncertainty of the certified value.

$C(95\%) = \frac{t \times s_M}{\sqrt{n}}$ 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.

NB: Although widely accepted within the industry "mass content in %" is neither an SI nor an IUPAC supported quantity. Multiplication of the certified value (Cv) by 10^4 will yield the value in µg/g.

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(The current edition is available at www.basrid.co.uk)

BCS/SS-CRM 111/1
LOW CARBON STEEL
NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 2 determined carbon using a non-aqueous titration following combustion in a stream of oxygen according to BS 6200.8.2: 1981. Analysts Nos. 3, 4, 5, 6, and 9 used combustion/infra-red methods calibrated with carefully selected CRMs (see below for details). Analysts Nos. 7, 12 and 13 used combustion infrared methods, Nos. 7 and 12 calibrating with Na_2CO_3 and No. 13 with sucrose.

SILICON

Analysts Nos. 1 and 2 determined silicon photometrically as molybdenum blue and without extraction. The remaining Analysts used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES), No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

MANGANESE

With the exception of Analysts Nos. 2 and 10 all Analysts determined manganese using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O. Analysts Nos. 2 and 10 each used a photometric method in which the manganese is oxidised with periodate.

PHOSPHORUS

Analysts Nos. 1 and 2 determined phosphorus using a photometric method, extracting the yellow phosphovanadomolybdate complex. Analyst No. 10 also used a photometric method, this time as molybdenum blue and without extraction. The remaining Analysts determined phosphorus using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

SULPHUR

Analyst No 1 determined sulphur by an acid-base titration following combustion in a stream of oxygen. Analyst No. 2 used ICP-OES whilst Nos. 3, 4, 5, 6 and 9 used combustion/infra-red methods calibrated with carefully selected CRMs (see below for details). Nos. 7, 8 and 12 used combustion infrared methods calibrated with pure chemicals or stoichiometric compound. Analyst No. 10 used a methylene blue photometric method, having evolved sulphur as H_2S from the sample solution using hypophosphoric and formic acids.

CHROMIUM

With the exception of Analyst No. 10, who used a photometric method with diphenylcarbazide, all Analysts determined chromium by ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

MOLYBDENUM

All Analysts determined molybdenum using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

NICKEL

With the exception of Analyst No 10, who used a photometric method involving dimethylglyoxime with extraction, all of the Analysts determined nickel using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

ARSENIC

With the exception of Analysts Nos.1 and 2, who each used a photometric method in which AsH_3 is bubbled through a solution of silver diethyldithiocarbamate, all of the Analysts determined arsenic using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

COBALT

All Analysts determined cobalt using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O

COPPER

All Analysts determined copper using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O

NITROGEN

Analysts Nos. 1 and 10 determined nitrogen photometrically with Nessler's reagent, No. 1 after a Kjeldahl digestion and steam distillation. No. 2 also used a Kjeldahl digestion and steam distillation but with an acid-base titration. Analysts Nos. 3, 5, 6 and 9 determined nitrogen by thermal conductivity after decomposition in a graphite crucible and calibrated with carefully selected CRMs (see below for details). Analysts Nos. 7 and 12 used thermal conductivity after decomposition in a graphite crucible and calibrated with pure chemicals or stoichiometric compounds.

TIN

All Analysts determined tin using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O

TITANIUM

All Analysts determined titanium using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O

VANADIUM

All Analysts determined vanadium using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O

ALUMINIUM

All Analysts determined aluminium using ICP-OES, No. 3 after acid dissolution and using standard addition and No. 7 after dissolution with a 1:3:16 (v/v/v) mixture of HCl, HNO_3 and H_2O.

BCS/SS-CRM 111/1
LOW CARBON STEEL
CO-OPERATING ANALYSTS

1	CROCKER, F. H.,	Pattinson & Stead (2005) Ltd., Middlesbrough.
2 & 11	JONES, S.J,	Ridsdale & Co Ltd., Middlesbrough.
3	HURDITCH, P.,	AMG Analytical Services, Rotherham.
4	JAMIESON S.N.,	NSG Technical Centre, Lathom.
5	COFFEY, R. N.,	Exova Teesside, Middlesbrough.
6	RUGG, P.,	Liberty Speciality Steels, Rotherham.
7	KUMAR, A.,	Tata Steel, Jamshedpur, India.
8	KONDOROSI, G.,	Dunaferr Labor Nonprofit Kft, Dunaújváros, Hungary.
9	CARDY, J.,	British Steel Testing Solutions, Scunthorpe.
10 & 12	STEPANOVSKIKH, V.,	ICRM, Yekaterinburg, Russia.
13	WICHARDT, C.,	Sandvik Materials Technology, Sandviken, Sweden.

DESCRIPTION OF SAMPLE

British Chemical Standard BCS-CRM 111/1 is sold in the form of chips passing a nominal 1700µm aperture sieve from which the fines passing a nominal 250µm aperture sieve have been removed. It is supplied in bottles containing 100g.

Spectroscopic Standard SS-CRM 111/1 is sold in the form of 44mm diameter discs.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS/SS-CRM 111/1 as defined in BS EN ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with both BS EN ISO 17034 and ISO Guide 35.

INTENDED USE

BCS-CRM 111/1 is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments, for establishing values for secondary reference materials and for training purposes.

In order to ensure that a fully representative sample is taken users should take not less than the minimum weight stated on the certificate for that element, this being the lowest sample mass used by any of the Co-operating Analysts contributing to the certified value. Users of this material should be aware that the use of a smaller sub-sample size may invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

SS-CRM 111/1 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. An area 6mm in diameter in the centre of the disc should be avoided for optical emission spectrometry. Users of optical emission spectrometry should also be aware that a minimum of three sparks should be made before calculating mean values.

STABILITY

BCS-CRM 111/1 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. Once the bottle has been opened the lid should be secured immediately after use.

SS-CRM 111/1 will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

TRACEABILITY

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 of the analytical methods used in the characterisation of this CRM were either international or national standard methods or methods which are technically equivalent. With the exception of C, S and N all laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

For C, S and N results have been obtained using stoichiometric analytical techniques, methods which were calibrated against pure stoichiometric compounds or by calibration using 5 certified reference materials carefully selected to be similar in composition to BCS/SS-CRM 111/1 and all of which demonstrate full unbroken traceability to the SI. Different Analysts used different combinations of CRMs. The CRMs used were as follows: BCS-CRMs 111, 112, 114, 115, 161/4, 404/2 and 432/1 and ECRMs 056-2, 057-2, 059-2, 064-1, 083-1, 088-2, 097-1, 097-2, 191-2 and 196-2.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS/SS-CRM 111/1 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's t value at the 95% confidence level for the number of results. Homogeneity has been assessed in accordance with ASTM E826 – 85 and found to be acceptable. Homogeneity has not, therefore, been included in the calculated measurement uncertainty. The stability of this CRM and its transportation also make negligible contributions to the overall uncertainty of the certified values.

COMMUTABILTY

BCS-CRM 111/1 is intended to be used in the same physical form as that used by the participating analysts and therefore commutability is not of relevance in respect of this CRM.

In the case of SS-CRM 111/1 it has been established that, when using optical emission spectrometers, materials of similar composition from different sources may respond differently. The user should be aware that the metallurgical history of this SS-CRM may not accurately reflect the metallurgical history of the user's own materials.

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

R P MEERES,
Managing Director
January 2018