BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

4004

CERTIFICATE OF ANALYSIS
BCS-CRM No. 231/5
FERRO-MOLYBDENUM

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

ANALYSES

Mean of 4 values - mass content in %.

Analyst	C	Si	P	S	Mo	Cu	*Mn*	*Cr*	*As*	*B*	*Nb*	*Pb*	*Sn*
1	0.2216	1.6703	0.0296	0.0449	61.9318	0.2675	...	...	...	...	...	...	...
2	0.2205	1.6566	0.0328	0.0434	61.6213	0.2696	...	...	*0.0100*	...	...	*0.00403*	*0.0036*
3	0.2131	1.7300	0.0293	0.0423	…	0.2750	...	...	...	...	...	...	...
4	…	…	0.0250	…	61.9825	0.2673	...	...	...	...	...	...	...
5	…	…	…	…	61.3025	0.2675	…	…	…	…	…	…	…
6	…	1.6400	0.0245	…	62.0350	…	...	...	...	...	...	...	...
7	0.2110	1.6273	0.0301	0.0423	61.3875	0.2733	*0.8748*	*0.7534*	...	...	*0.8575*	...	...
8	…	…	0.0267	0.0402	…	0.2610	*0.8320*	...	*0.0092*	*0.2116*	*0.6845*	*0.0035*	*0.0052*
9	0.2286	1.6400	0.0281	0.0396	61.4000	0.2773	...	...	...	...	...	...	...
10	…	1.6575	0.0284	…	61.3500	0.2690	...	...	...	...	...	...	...
11	…	1.7525	0.0300	…	61.4575	0.2810	...	...	...	...	...	...	...
12	…	1.6578	0.0315	…	62.2685	0.2775	...	...	...	...	...	...	...
13	0.2162	1.6596	0.0318	0.0426	61.6747	0.2632	...	...	...	...	...	...	...
14	0.2090	1.6751	0.0291	0.0463	61.8693	0.2673	...	...	...	...	...	...	...
15	0.1963	…	…	0.0428	…	…	...	...	...	...	...	...	...
16	…	…	…	…	61.7075	…	...	...	...	...	...	...	...
17	0.2097	…	…	0.0414	…	…	...	...	...	...	...	...	...
18	0.2018	…	…	0.0401	…	…	...	...	...	...	...	...	...
19	0.2060	…	…	0.0416	…	…	...	...	...	...	...	...	...
20	…	1.6160	…	…	…	0.2675	...	...	...	...	...	...	...
21	…	1.6703	…	..	61.5325	0.2613	...	...	...	...	...	...	...
22	0.2058	1.6325	0.0273	0.0404	62.1325	0.2648	*0.8000*	*0.8000*	*<0.005*	...	*0.9500*	*<0.005*	...
23	…	…	0.0298	0.0451	61.5978	0.2785	...	...	...	*0.1745*	...	...	...
24	0.2242	…	…	0.0435	…	…	...	...	...	...	...	...	...
25	0.2094	…	…	0.0421	…	…	...	...	...	...	...	...	...
M_M	**0.2124**	**1.6633**	**0.0289**	**0.0424**	**61.7032**	**0.2699**							
s_M	0.0090	0.0376	0.0024	0.0019	0.3008	0.0062							
s_W	0.0023	0.0183	0.0009	0.0011	0.0819	0.0034							

Values given in italics are for information only

Additional Information: Analyst No. 7 determined Fe by ICP-OES and found 32.0%.

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	P	S	Mo	Cu
Cv	**0.212**	**1.663**	**0.0289**	**0.0424**	**61.70**	**0.270**
C(95%)	0.006	0.022	0.0014	0.0011	0.17	0.004
Minimum Weight (g)	0.03	0.05	0.05	0.03	0.05	0.05

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.

This is an expression of the uncertainty of the certified value.

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.

Date of this edition: January 2021

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

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NOTES ON METHODS USED

CARBON

Analysts Nos. 1 and 24 determined carbon gravimetrically after combustion in a stream of oxygen. Analyst No 2 also used combustion in a stream of oxygen but determined the resulting CO_2 titrimetrically. The remaining Analysts determined carbon by combustion infra-red methods, Nos. 3, 7, 17, 18, 19, 22 and 25 calibrating with a carefully selected series of CRMs (see below for details) whilst Analysts Nos 9, 13, 14 and 15 calibrated with pure chemicals.

SILICON

Analysts Nos. 1, 2, 10, 13 and 14 determined silicon gravimetrically, Nos. 1, 10 and 13 dehydrating with nitric and sulphuric acids (No. 10 according to ISO 4158:1973), No. 2 using sulphuric acid and 14 using perchloric acid. Analysts Nos. 3, 6, 11, 12 and 22 used X-Ray Fluorescence Spectroscopy (XRF) whilst the remaining Analysts used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

PHOSPHORUS

Analysts Nos. 1, 2 and 9 determined phosphorus spectrophotometrically as phosphovanadomolybdate, extracting the complex into 4-methyl-pentan-2-one. Analyst No. 8 used Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), Nos. 12 and 22 used XRF and No 13 digested the sample with HNO_3/HCl and determined phosphorus volumetrically. The remaining Analysts used ICP-OES.

SULPHUR

Analysts Nos. 1, 23 and 24 determined sulphur by ICP-OES, No. 2 used an acid-base titration after combustion in a stream of oxygen and absorption of the gases in hydrogen peroxide and No. 8 used ICP-MS. The remaining Analysts determined sulphur by combustion infra-red methods, Nos. 3, 7, 17, 18, 19, 22 and 25 calibrating with a carefully selected series of CRMs (see below for details) whilst Nos. 9, 13, 14 and 15 calibrated with pure chemicals.

MOLYBDENUM

Analysts Nos. 4, 5, 7, 11, 12 and 22 determined molybdenum by XRF and No. 20 used ICP-OES. The remaining Analysts determined molybdenum gravimetrically, Analysts Nos. 1, 10 and 13 used 8-hydroxyquinoline, (Nos. 10 and 13 following ISO 4173:1980), No. 2 used benzoin oxime whilst Nos. 6, 9, 14, 16 and 20 precipitated molybdenum as lead molybdate.

Analyst No. 22 also determined molybdenum by ICP-OES and reported a value of 62.32% and gravimetrically according to ISO 4173 and reported 62.45%

COPPER

Analysts Nos. 1, 7, 9, 10, 11, 14, 20, 21 and 23 determined copper by ICP-OES. Analysts Nos. 2 and 13 used Flame Atomic Absorption Spectrometry (FAAS) and Nos. 3, 4, 5, 12 and 22 used XRF. No. 8 used ICP-MS.

MANGANESE

Analyst No. 7 determined manganese using ICP-OES, whilst No. 8 used ICP-MS and No. 22 XRF.

CHROMIUM

Analyst No. 7 determined chromium using ICP-OES, whilst No. 22 used XRF.

ARSENIC

Analyst No. 2 determined arsenic spectrophotometrically with diethyldithiocarbamate, No. 8 used ICP-MS and No. 22 used ICP-OES.

BORON

Analyst No 8 determined B using ICP-MS and No. 23 used ICP-OES.

NIOBIUM

Analyst No. 7 determined niobium using ICP-OES, whilst No. 8 used ICP-MS and No. 22 XRF.

LEAD

Analyst No.2 determined lead using ICP-OES, whilst No. 8 used ICP-MS and No. 22 ICP-OES.

TIN

Analyst No.2 determined tin using ICP-OES, whilst No. 8 used ICP-MS.

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CO-OPERATING ANALYSTS

1	Jones, S.J., *BSc, CChem, MRSC,*	Ridsdale & Co. Ltd., Middlesbrough.
2, 24	Crocker, F.H. & Atkinson, M J,	Pattinson & Stead (2005) Ltd., Middlesbrough.
3	Blundell, P.,	ALS Inspection UK, Prescot.
4, 16	Sutton, K.,	Climax Molybdenum (UK) Ltd., Stowmarket.
5	Hargreaves, C.,	Inspectorate International Ltd, Witham.
6	Hampson, N.,	Alfred H Knight International Ltd., St Helens.
7	Hurditch, P.,	AMG Superalloys UK. Ltd., Rotherham.
8	Rodushkin, I,	ALS Environmental AB, Lulea, Sweden.
9	Wichardt, C.,	Sandvik Materials Technology, Sandviken, Sweden.
10	Kondorosi, G.,	ISD Dunaferr, Dunaujvaros, Hungary.
11	Oosterhoff, A.,	Laboratory Services International, Rotterdam, The Netherlands.
12	van Croonenburg, F.,	SGS Nederland B.V, Spijkenisse, The Netherlands.
13	Chattopadhyay, S.,	Inspectorate Griffith India Pvt Ltd., Bhubaneswar, India.
14	Jiang, J. & Wu, M.,	Inspectorate (Shanghai) Ltd., Shanghai, China.
15	Lindkvist, L.,	Sandvik Heating Technology, Hallstahammar, Sweden.
17	Snowden, Y.,	Tata Steel Testing Solutions, Scunthorpe.
18	Jamieson, S. N., *MSc, CChem, MRSC*	NSG European Technical Centre, Lathom.
19	Scrimshire, P. J., *BSc,*	IncoTest, Hereford.
20	Chamarro, G.,	Inspectorate Services Peru, Callao, Peru.
21	Collins, P	Tata Steel, Swinden Labs, Rotherham.
22	Culley, N.,	LMA, Sleeuwijk, The Netherlands.
23	Granfors, M.	Swerea-Kimab, Stockholm, Sweden.
25	Rugg, P.	Liberty Speciality Steels, Stocksbridge.

DESCRIPTION OF SAMPLE

BCS-CRM 231/5 is sold in the form of finely divided material passing a nominal 150 micron aperture and packed into bottles of 100g.

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-CRM 231/5 as defined in ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO 17034 and ISO Guide 35.

INTENDED USE

This sample 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.

STABILITY

BCS-CRM 231/5 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. When the bottle has been opened the lid should be secured immediately after use.

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. 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 and S results have been obtained using stoichiometric analytical techniques, methods which were calibrated against pure stoichiometric compounds or by calibration using five carefully selected Certified Reference Materials (CRMs), each demonstrating full unbroken traceability to the SI. Different Analysts used different combinations of CRMs. At the time of certification there were insufficient good quality ferro-molybdenum CRMs to allow full matrix matching but theoretical considerations and practical experience showed that low alloy steels were an acceptable substitute. The CRMs used were as follows: BCS-CRM 111/1, BCS-CRM 232/2, BCS-CRM 405/1, BCS-CRM 408/1, BCS-CRM 432/2, BCS-CRM 435/2, ECRM 055-2, ECRM 057-2, ECRM 084-1, ECRM 085-1, ECRM 086-1, ECRM 087-1 and ECRM 578-1.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 231/5 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 on the bulk material using one way ANOVA and has been found to be acceptable. It 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 231/5 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.

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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First published January 2016 with uncertified values for C & S
Revised January 2021 with certified values for C and S