MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

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4004

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS
BCS-CRM No. 114
LOW ALLOY STEEL

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

ANALYSES

Mean of 4 values - mass content in %.

Lab No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	B	Co
1	0.4012	0.2990	0.4212	0.0043	0.0044	0.1975	0.1851	1.4939	0.0806	0.0022	0.0006	0.0179
2	0.4168	0.2847	0.4143	0.0051	0.0044	0.1867	0.1866	1.5197	0.0818	0.0023	0.0008	0.0178
3	0.4004	0.3031	0.4216	0.0041	0.0047	0.1889	0.1807	1.5045	0.0746	…	0.0007	0.0168
4	0.4023	0.3004	0.4155	0.0040	0.0047	0.1848	0.1787	1.4993	0.0785	0.0030	0.0008	0.0167
5	0.3970	…	…	…	0.0052	…	…	…	…	…	…	…
6	…	0.2910	0.3995	0.0041	0.0050	0.1844	0.1878	1.4888	0.0745	0.0026	0.0009	0.0160
7	0.4025	0.2917	0.4218	0.0050	0.0041	0.1800	0.1836	1.5055	0.0771	0.0024	0.0007	0.0172
M_M	**0.4034**	**0.2950**	**0.4157**	**0.0044**	**0.0046**	**0.1871**	**0.1838**	**1.5020**	**0.0779**	**0.0025**	**0.0008**	**0.0171**
s_M	0.0069	0.0070	0.0086	0.0005	0.0004	0.0059	0.0035	0.0108	0.0031	0.0003	0.0001	0.0007
s_w	0.0077	0.0020	0.0026	0.0003	0.0003	0.0022	0.0019	0.0071	0.0010	0.0003	0.0001	0.0003

Lab No.	Cu	N	Nb	Sn	Ti	V	Zr	*Pb*	*W*	*Ca*	*Sb*
1	0.3639	0.0044	0.0043	0.0458	0.0099	0.0096	0.0055	…	*<0.001*	*<0.0001*	*<0.0025*
2	0.3656	0.0040	0.0043	0.0381	0.0098	0.0078	0.0043	*0.0002*	…	…	…
3	0.3537	0.0042	0.0039	0.0406	0.0092	0.0087	0.0053	*<0.0001*	*0.0004*	*<0.0001*	*0.0002*
4	0.3589	0.0045	0.0039	0.0409	0.0093	0.0082	0.0053	*<0.0004*	*<0.0006*	*<0.001*	*0.0022*
5	…	0.0041	…	…	…	…	…	…	…	…	…
6	0.3633	…	0.0043	0.0402	0.0097	0.0088	0.0055	*<0.001*	…	*<0.0001*	*<0.001*
7	0.3546	0.0046	0.0045	0.0420	0.0094	0.0082	0.0049	*<0.0001*	…	*<0.0001*	*0.0002*
M_M	**0.3600**	**0.0043**	**0.0042**	**0.0413**	**0.0096**	**0.0086**	**0.0051**	…	…	…	…
s_M	0.0051	0.0003	0.0003	0.0026	0.0003	0.0007	0.0005	…	…	…	…
s_w	0.0018	0.0002	0.0004	0.0008	0.0005	0.0004	0.0004	…	…	…	…

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

M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means. s_w: intralaboratory standard deviation.
Values given above in small italic type are for information only.

CERTIFIED VALUES

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	B	Co	Cu
M_M	**0.403**	**0.295**	**0.416**	**0.0044**	**0.0046**	**0.187**	**0.184**	**1.502**	**0.078**	**0.0025**	**0.0008**	**0.0171**	**0.360**
C(95%)	0.007	0.008	0.009	0.0005	0.0004	0.006	0.004	0.012	0.004	0.0004	0.0001	0.0008	0.005

	N	Nb	Sn	Ti	V	Zr
M_M	**0.0043**	**0.0042**	**0.041**	**0.0096**	**0.0086**	**0.0051**
C(95%)	0.0003	0.0003	0.003	0.0003	0.0008	0.0005

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

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

BCS-CRM No. 114
LOW ALLOY STEEL
NOTES ON METHODS USED

CARBON: Analyst No. 1 determined carbon using non-aqueous titration according to the Standard Method BS 6200:3.8.2:1991. The other Analysts used high frequency combustion-infrared absorption**.**

SILICON: Analysts Nos. 1, 2, 3 and 7 determined silicon gravimetrically, Nos. 1, 2 and 7 after dehydration with perchloric acid and No. 3 after a sulphuric acid dehydration. Analyst No. 4 used a molybdenum blue photometric method, whilst Analyst No. 6 used Inductively Coupled Plasma Optical-Emission Spectrometry (ICP-OES).

MANGANESE: Analysts Nos. 1, 4 and 7 determined manganese photometrically after oxidation with potassium periodate. Analysts Nos. 2, 3 and 6 used ICP-OES

PHOSPHORUS: Analyst No 1 determined phosphorus photometrically as phosphovanadomolybdate with extraction according to the Standard Method BS EN 10184. Analysts Nos. 2, 3 and 6 used ICP-OES whilst Analysts Nos. 4 and 7 determined phosphorus photometrically as phosphovanadomolybdate without extraction.

SULPHUR: Analyst No.1 determined sulphur gravimetrically according to the Standard Method BS 1 6200:3.26. Analyst No.6 determined sulphur by combustion, according BS 7020:7.2. The other analysts used high frequency combustion infrared absorption.

CHROMIUM: All Analysts, except No. 7, determined chromium by ICP-OES. Analyst No. 7 used Flame Atomic Absorption Spectrometry (FAAS).

MOLYBDENUM: All Analysts, except No. 7, determined molybdenum by ICP-OES. Analyst No. 7 used a photometric method with thiocyanate in the presence of Sn (II).

NICKEL: All Analysts, except No. 7, used ICP-OES. Analyst No.7 titrated with EDTA.

ALUMINIUM: Analysts Nos. 1, 2, 3, 4 and 6 used ICP-OES. Analyst No. 7 used FAAS.

ARSENIC: Analyst No. 1 determined arsenic photometrically with silver diethyldithiocarbamate after separation as arsine, according to BS EN 10212:1996. Analysts Nos. 2 and 4 used ICP-OES and Analyst No.6 used hydride generation atomic absorption spectrometry. Analyst No. 7 determined arsenic photometrically as molybdenum blue.

BORON: All Analysts, except Nos. 2 and 7, determined boron photometrically with curcumin, No.1 according to BS EN 10200. Analyst No. 2 determined boron by ICP-OES and No. 7 used dianthrimide to determine the boron photometrically.

COBALT: All Analysts, except No. 7, determined cobalt by ICP-OES. No.7 used FAAS.

COPPER: All Analysts, except No. 7, determined copper by ICP-OES. No.7 used FAAS.

NITROGEN: Analyst No. 1 determined nitrogen using an acidimetric titration after distillation, according to the Standard Method BS 6200:3.22.1:1992. The remaining Analysts used thermal conductivity after decomposition in a graphite crucible.

NIOBIUM: All Analysts, except No. 7, determined niobium using ICP-OES. Analyst No. 7 determined niobium photometrically with 4-(2 pyridylazo)-resorcinol.

TIN: All analysts, except No.7, used ICP-OES. Analyst No.7 determined tin by FAAS.

TITANIUM: Analysts Nos. 1, 2, 3, and 6 determined titanium by ICP-OES. Analysts Nos. 4 and 7 determined titanium photometrically using diantipyrylmethane.

VANADIUM: All Analysts, except No. 7, determined vanadium by ICP-OES. Analyst No. 7 used FAAS.

ZIRCONIUM: All Analysts, except No. 7 determined zirconium by ICP-OES. Analyst No. 7 used a xylenol orange photometric method.

LEAD: *Analysts Nos. 2 and 6 determined lead by ICP-OES. Nos. 3 and 4 used FAAS and Analyst No. 7 used electrothermal atomic absorption spectrometry (ETAAS)*

TUNGSTEN: *All Analysts used ICP-OES*

CALCIUM: *Analysts Nos. 1, 3 and 4 used FAAS, Analyst No.6 used ICP-OES and Analyst No. 7 used ETAAS*

ANTIMONY: *Analyst No. 1 used FAAS, Analysts Nos. 3 and 7 used hydride generation atomic absorption spectrometry and Analysts Nos. 4 and 6 used ICP-OES*

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LOW ALLOY STEEL

CO-OPERATING ANALYSTS

INDEPENDENT ANALYST

1 PAGE-GIBSON, J.E., *BSc, CChem, MRSC* Ridsdale & Co. Ltd., Middlesbrough.

ANALYSTS representing MANUFACTURERS and USERS

2 CROOK, D., Corus Strip Products, Llanwern.
3 FOX, G., Corus Engineering Steels, Stocksbridge.
4 WEERDT, Miss J A., BRAS, P.W. ten & GULDEMOND, Dr D., Corus Staal BV, IJmuiden.
5 RICHMOND, Mrs H., & RAW, M., Corus Construction and Industrial, Redcar.
6 SNOWDEN, Miss Y.A. and RAW, M., Corus Construction and Industrial, Scunthorpe.
7 WILSON, J., Allvac Ltd., Sheffield.

DESCRIPTION OF SAMPLE

BCS-CRM 114 is a low alloy steel supplied in bottles of 100g chips nominally graded 1700 – 250µm (10 – 60 mesh) for chemical analysis. The sample is also available in pieces of 44mm diameter as SS-CRM 114.

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

BAS is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS-CRM 114 as defined in ISO Guide 34:2009 section 3.1, is fully responsible for assigning the certified values and their uncertainties in accordance with ISO Guides 34:2009 and 35:2006.

This material was originally certified in 2004, before BAS achieved accreditation to ISO Guide 34 in 2006. However, following an extensive review of the certification of this BCS-CRM, it has been established that the procedures used were exactly the same as those which have since been accredited and that therefore BCS-CRM 114 can be issued under the ISO Guide 34 Accreditation.

INTENDED USE

This sample is intended for uses such as the verification of the accuracy and repeatability 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 a minimum sub-sample size of 1.0g. Users of this material should be aware that the use of a smaller sub-sample size will invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

STABILITY

BCS-CRM 114 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.

MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS-CRM 114 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 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 114 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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For BUREAU OF ANALYSED SAMPLES LTD.
R P MEERES,
Managing Director

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