PRELIMINARY EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

Certificate No. Q3993

CERTIFICATE OF ANALYSIS
SS-CRM No. 113
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	0.8308	0.9345	1.1980	0.0583	0.0301	1.2487	0.0524	0.0772	0.0156	0.0019	0.0066	0.0420
2	0.8470	0.9350	1.2206	0.0611	0.0293	1.2418	0.0600	0.0815	0.0170	0.0022	0.0063	0.0436
3	0.8370	0.9329	1.1999	0.0604	0.0290	1.2448	0.0551	0.0792	0.0133	…	0.0065	0.0416
4	0.8305	0.9237	1.1880	0.0604	0.0301	1.2581	0.0553	0.0768	0.0147	…	0.0071	0.0412
5	0.8443	…	…	…	0.0284	…	…	…	…	…	…	…
6	…	0.9361	1.2142	0.0580	0.0302	1.2578	0.0558	0.0766	0.0133	0.0018	0.0073	0.0402
7	0.8313	0.9227	1.2186	0.0589	0.0286	1.2390	0.0580	0.0792	0.0167	0.0020	0.0057	0.0405
M_m	**0.8368**	**0.9308**	**1.2066**	**0.0595**	**0.0294**	**1.2484**	**0.0561**	**0.0784**	**0.0151**	**0.0020**	**0.0066**	**0.0415**
s_M	0.0073	0.0060	0.0132	0.0013	0.0008	0.0081	0.0026	0.0019	0.0016	0.0002	0.0006	0.0012
s_W	0.0059	0.0074	0.0024	0.0008	0.0006	0.0023	0.0010	0.0009	0.0004	0.0005	0.0001	0.0005

Lab No.	Cu	N	Nb	Sn	Ti	V	W	Zr	*Pb*	*Ca*	*Sb*
1	0.1788	0.0106	0.0495	0.0063	0.0398	0.2072	0.0131	0.0028	*0.0002*	*<0.0001*	*<0.0025*
2	0.1835	0.0111	0.0459	0.0075	0.0386	0.1938	0.0151	0.0018	*0.0007*	*...*	*0.0009*
3	0.1743	0.0106	0.0491	0.0061	0.0383	0.2061	0.0095	0.0036	*<0.001*	*0.0001*	*0.0003*
4	0.1774	0.0107	…	0.0061	0.0377	0.1993	0.0106	0.0031	*<0.0004*	*<0.0010*	*0.00287*
5	…	0.0108	…	…	…	…	…	…	*...*	*...*	*...*
6	0.1816	…	0.0493	0.0061	0.0390	0.2029	…	0.0034	*<0.0010*	*<0.0001*	*<0.0010*
7	0.1788	0.0116	0.0495	0.0080	0.0407	0.1984	0.0140	0.0029	*<0.0001*	*<0.0001*	*0.0003*
M_m	**0.1791**	**0.0109**	**0.0487**	**0.0067**	**0.0390**	**0.2013**	**0.0125**	**0.0029**	*...*	*...*	*...*
s_M	0.0032	0.0004	0.0016	0.0009	0.0011	0.0051	0.0024	0.0007	*...*	*...*	*...*
s_W	0.0008	0.0002	0.0008	0.0003	0.0005	0.0008	0.0008	0.0003	*...*	*...*	*...*

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	**0.837**	**0.931**	**1.207**	**0.0595**	**0.0294**	**1.248**	**0.056**	**0.0784**	**0.0151**	**0.0020**	**0.0066**	**0.0415**	**0.179**
C(95%)	0.008	0.007	0.014	0.0014	0.0008	0.009	0.003	0.0020	0.0017	0.0003	0.0007	0.0013	0.004

	N	Nb	Sn	Ti	V	W	Zr
M_m	**0.0109**	**0.0487**	**0.0067**	**0.0390**	**0.201**	**0.012**	**0.0029**
C(95%)	0.0004	0.0020	0.0010	0.0012	0.006	0.003	0.0008

The half width confidence interval **C**(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate Student's t value and "n" is the number of acceptable mean values

For further information regarding the confidence interval for the certified value see ISO Guide 35:1989 section 4.

THROUGHOUT BATCH COMPOSITIONAL VARIABILITY

	C	Si	Mn	P	S	Cr	Mo	Ni	Al	As	B	Co	Cu	N	Nb	Sn	Ti	V	W	Zr
µg/g	26.8	21.0	192.0	3.8	8.3	13.5	0.8	1.2	<0.3	<0.3	0.6	<0.3	16.7	3.3	<0.3	<0.3	10.7	2.7	1.0	1.2

SS-CRM No. 113

LOW ALLOY STEEL

NOTES ON METHODS USED

CHEMICAL ANALYSIS

CABON

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, 6 and 7 determined silicon gravimetrically after dehydration with perchloric acid, except for No. 3 who used a sulphuric acid dehydration. Analyst No. 4 used a molybdenum blue photometric method.

MANGANESE

Analysts Nos. 1 and 4 determined manganese photometrically after oxidation with potassium periodate. Analysts Nos. 2, 3 and 6 used Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). Analyst No 7 used Flame Atomic Absorption Spectrometry (FAAS).

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

Analysts Nos. 1 and 7 determined chromium titrimetrically, after oxidation with persulphate. The remaining Analysts used ICP-OES.

MOLYBDENUM

All Analysts, except No. 7, determined molybdenum by ICP-OES. Analyst No. 7 used FAAS

NICKEL

All Analysts, except No. 7, used ICP-OES. Analyst No.7 used FAAS.

ALUMINIUM

All Analysts, except No. 7, determined aluminium by 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. Analyst No. 2 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

Analysts Nos. 1 and 7 determined copper by FAAS. The remaining Analysts determined copper by ICP-OES.

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.

TUNGSTEN

Analysts Nos. 1 and 7 determined tungsten photometrically using thiocyanate. The other Analysts used ICP-OES

ZIRCONIUM

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

LEAD

Analysts Nos.1, 3 and 4 determined lead by FAAS. Analysts Nos. 2 and 6 determined lead by ICP-OES whilst Analyst No. 7 used electrothermal atomic absorption spectrometry (ETAAS)

CALCIUM

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

ANTIMONY

Analyst No. 1 used FAAS, Analysts Nos. 2, 4 and 6 used ICP-OES. No. 3 used XRF and Analyst No. 7 used hydride generation atomic absorption spectrometry

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

The sample is available in pieces 44mm in diameter and either 19mm or 50mm long.
It is also available in chip form as BCS-CRM 113

THROUGHOUT BATCH COMPOSITIONAL VARIABILITY

Samples taken from all the bars used in the preparation of this SS-CRM have been examined using optical emission spectrometry. The throughout batch compositional variability is obtained after the statistical elimination of instrumental and sample preparation variables. This is given in the table as the standard deviation, in µg/g, for each element certified in this CRM.

The results quantify the homogeneity of the material used to prepare this SS-CRM

INTENDED USE & STABILITY

This SS-CRM 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).

TRACEABILITY

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

This Certified Reference Material has been prepared in accordance with the recommendations specified in ISO Guides 30 to 35, available from the International Standards Organisation in Geneva.

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For BUREAU OF ANALYSED SAMPLES LTD.
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Chairman