MAIN EDITION

BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS†/SS‡-CRM No. 465/1

AUSTENITIC STAINLESS STEEL

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

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ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P	S	Cr	Mo	Ni	Al (Total)	B	Co	Cu	N	Ti	V	*Pb*
1	0.068	0.394	1.355	0.018	0.012	…	0.099	…	…	0.0005	0.051	0.096	0.010	0.39	0.090	*0.0005*
2	0.065	0.412	1.374	0.022	0.012	17.38	0.094	9.28	…	…	0.052	0.100	…	0.42	0.114	…
3	0.065	0.406	1.400	0.021	0.010	17.31	0.093	9.16	0.025	0.0004	0.053	0.100	0.010	0.38	0.099	*0.0004*
4	0.065	0.409	1.390	0.024	0.014	17.18	0.083	9.25	0.027	…	0.054	0.095	…	0.38	0.102	*0.0001*
5	0.066	0.409	1.395	0.020	0.009	17.22	0.091	9.28	0.025	0.0008	0.054	0.098	0.010	0.41	0.103	*0.0007*
6	0.067	0.408	1.379	0.022	0.013	17.40	0.103	9.23	0.029	…	0.056	0.096	…	0.42	0.092	*<0.0001*
7	0.066	0.401	1.371	0.021	0.012	17.32	0.085	9.25	0.026	0.0006	0.055	0.097	0.009	0.40	0.108	…
8	0.064	…	1.376	…	0.011	17.28	0.083	9.22	0.025	…	0.054	0.097	0.010	0.39	0.107	…
9	0.064	0.404	1.380	0.021	0.012	17.38	0.095	9.27	0.025	0.0006	0.051	0.101	0.011	0.40	0.105	…
10	…	…	…	…	…	…	…	…	…	0.0006	…	…	…	…	…	…
M_M	**0.066**	**0.405**	**1.380**	**0.021**	**0.012**	**17.31**	**0.092**	**9.24**	**0.026**	**0.0006**	**0.053**	**0.098**	**0.010**	**0.40**	**0.102**	…
s_M	0.002	0.006	0.014	0.002	0.002	0.08	0.008	0.04	0.002	0.0002	0.002	0.003	0.001	0.02	0.008	…

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

Figures in bold type certified, figures in small italic type only approximate.

M_M: Mean of the intralaboratory means. s_M: standard deviation of the intralaboratory means.

CERTIFIED VALUES (Cv)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Al (Total)	B	Co	Cu	N	Ti	V
Cv	**0.066**	**0.405**	**1.380**	**0.021**	**0.012**	**17.31**	**0.092**	**9.24**	**0.026**	**0.0006**	**0.053**	**0.098**	**0.010**	**0.40**	**0.102**
C(95%)	0.002	0.005	0.011	0.002	0.002	0.07	0.006	0.04	0.002	0.0002	0.002	0.002	0.001	0.02	0.006

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.

N.B. Due to slight segregation of certain elements an area 6mm in diameter in the centre of the disc samples should be avoided for emission spectroscopy.

BCS/SS-CRM No. 465/1 AUSTENITIC STAINLESS STEEL

NOTES ON METHODS USED

CARBON

Analysts Nos. 1, 2, 4, 6, 7, 8 and 9 determined carbon by high frequency combustion/infrared absorption. Nos. 3 and 5 used non-aqueous titration according to the British Standard Carbon Method 4*.

SILICON

All analysts determined silicon gravimetrically after dehydration with perchloric acid, according to the British Standard Silicon Method 1*.

MANGANESE

Analyst No. 1 determined manganese using FAAS. Nos. 2, 3, 4, 5, 6, 8 and 9 used photometric methods, all, except No. 9, oxidised manganese with periodate, Nos. 2, 4, 6 and 8 according to the British Standard Manganese Method 2*, Nos. 3 and 5 according to BS 6200: 3.18.2§. No. 9 oxidised with ammonium persulphate. No. 7 used ICP-OES.

PHOSPHORUS

All analysts, except No. 2, determined phosphorus photometrically as phosphovanadomolybdate, according to BS 6200: 3.24.1§. No. 2 used a titrimetric method after separation as phosphomolybdate.

SULPHUR

Nos. 1, 2, 4, 6, 7, 8 and 9 determined sulphur using high frequency combustion/infrared absorption. No. 3 determined sulphur gravimetrically according to BS 6200: 3.28.1§ and No. 5 also used a gravimetric method involving a preliminary chromatographic separation of sulphur on an alumina column (Nydahl, Anal. Chem., 1954, 26, 580).

CHROMIUM

All analysts determined chromium titrimetrically after oxidation with ammonium persulphate, Nos. 2, 4, 6, 7, 8 and 9 according to the British Standard Chromium Method 1*, Nos. 3 and 5 according to BS 6200: 3.10.1§.

MOLYBDENUM

Analysts Nos. 1, 2, 4 and 8 determined molybdenum using FAAS. Nos. 3, 5, 6 and 9 determined molybdenum photometrically as oxythiocyanate, Nos. 3 and 5 according to BS 6200: 3.19.1§, Nos. 6 and 9 according to the British Standard Molybdenum Method 1*. No. 7 used ICP-OES.

NICKEL

Analyst No. 2 determined nickel cyanometrically according to the British Standard Nickel Method 1*. Nos. 3, 5, 6, 7, 8 and 9 determined nickel titrimetrically after separation with dimethylglyoxime. All, except No. 5, titrated with EDTA, according to BS 6200: 3.20.1§, No. 5 dissolved the precipitate in dilute sulphuric acid, boiled with excess ferric sulphate, and titrated with dichromate (Analoid Method No. 62). No. 4 determined nickel gravimetrically.

ALUMINIUM (total)

Analysts Nos. 3, 4, 5, 7, 8 and 9 determined aluminium using FAAS according to BS 6200: 3,4,1§. No. 6 determined aluminium photometrically with eriochrome cyanine R according to the British Standard Aluminium Method 3*.

Analyst No. 6 also determined aluminium using ICP-MS and obtained a mean value of 0.029%.

BORON

All analysts determined boron photometrically with curcumin, Nos. 1 and 3 according to the British Standard Boron Method 1*, Nos. 5, 7, 9 and 10 according to method EN 10200.

COBALT

Analysts Nos. 1, 3, 4, 5, 8 and 9 determined cobalt using FAAS. Nos. 2 and 6 determined cobalt photometrically with Nitroso-R-Salt according to the British Standard Cobalt Method 1*, No. 7 used ICP-OES.

COPPER

Analysts Nos. 1, 2, 3, 4, 5, 8 and 9 determined copper using FAAS. No. 6 determined copper photometrically with bis-cyclohexanone oxalyldihydrazone. No. 7 used ICP-OES.

NITROGEN

Analysts Nos. 1, 5, 7 and 8 determined nitrogen using thermal conductivity after fusion in graphite crucibles. Nos. 3 and 9 used acidimetric titration after conversion to ammonia and steam distillation according to the British Standard Nitrogen Method 1*.

TITANIUM

Analyst Nos. 1 and 8 determined titanium using FAAS. No. 2 determined titanium photometrically with hydrogen peroxide after cupferron separation according to the British Standard Titanium Method 1*. Nos. 3, 5, 6 and 9 also determined titanium photometrically with diantipyrylmethane according to BS 6200: 3.32.1§. Nos. 4 and 7 used ICP-OES.

VANADIUM

Analysts Nos. 1, 8 and 9 determined vanadium using FAAS. Nos. 2 and 6 determined vanadium titrimetrically according to the British Standard Vanadium Method 1*. No. 2 first separated chromium by volatilisation as chromyl chloride and No. 6 by mercury cathode separation. Analysts Nos. 3 and 5 determined vanadium photometrically according to BS 6200: 3.34.2§. Nos. 4 and 7 used ICP-OES. Analysts Nos. 3 and 5 also determined vanadium titrimetrically and obtained mean values of 0.118% and 0.115% respectively. Analyst No. 6 also used ICP-MS and obtained a mean value of 0.088%.

LEAD

Analysts Nos. 1, 3, 5 and 7 determined lead using FAAS, No. 5 separated lead by extraction using trioctyl phosphine oxide. No. 6 used ICP-MS.

*Methods for Sampling and Analysis of Iron, Steel and Other Ferrous Metals, B.S. Handbook No. 19, first published in 1970 by the BSI, 389 Chiswick High Road, London. W4 4AL.

§BS 6200: Sampling and Analysis of Iron, Steel and Other Ferrous Metals: Part 3, Method of Analysis, published by the BSI, 389 Chiswick High Road, London. W4 4AL.

Abbreviations

FAAS:	Flame Atomic Absorption Spectrometry.
ICP-OES:	Inductively Coupled Plasma - Optical Emission Spectrometry.
ICP-MS:	Inductively Coupled Plasma - Mass Spectrometry.

DESCRIPTION OF SAMPLE

† British Chemical Standard – bottles of 100g chips graded 1700 - 250µm (10 - 60 mesh) for chemical analysis.

‡ Spectroscopic Standard – 38 mm diameter x 19 mm thick discs for spectroscopic analysis.

INTENDED USE & STABILITY

The chip sample, BCS-CRM 465/1, is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

It will remain stable provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

The disc sample, SS-CRM 465/1, 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. It will remain stable provided that it is not subject to excessive heat (e.g., during preparation of the working surface). An area 6mm in diameter in the centre of the disc should be avoided for optical emission spectrometry.

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

R.P. MEERES,
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