BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

CERTIFICATE OF ANALYSIS

BCS/SS-CRM No. 458/2

CARBON STEEL (Residual Series)

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 %.

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

Analyst No.	C	Si	Mn	P	S	Al (Total)	Al (Acid Sol.)	B	Co	Nb	Pb	V	Sb	*Zr*
1	0.2047	0.4978	0.4829	0.0271	0.0315	...	...	...	...	...	...	...	...	*0.0568*
2	0.1970	0.5005	0.4825	0.0294	0.0320	...	...	...	...	...	...	...	...	...
3	0.2004	0.5058	0.4813	0.0288	0.0307	0.0531	0.0519	0.0068	0.1997	0.0493	0.0127	0.1033	0.0901	*0.0671*
4	0.1988	0.5016	0.4720	0.0263	0.0318	...	...	...	...	...	...	...	...	...
5	0.1953	0.5031	0.4806	0.0279	...	0.0550	0.0534	0.0073	0.1930	0.0528	0.0140	0.1032	...	...
6	...	...	...	...	...	...	...	0.0068	0.2011	0.0522	0.0134	0.1065	0.0901	*0.0634*
7	0.1938	0.5071	0.4814	0.0285	0.0312	0.0551	0.0509	0.0073	0.2029	0.0499	0.0158	0.1069	0.0891	...
8	0.2010	0.4948	0.4793	0.0291	0.0317	0.0591	0.0537	...	0.1951	0.0502	...	...	...	...
9	0.1945	0.5208	0.4735	0.0273	0.0311	0.0525	0.0508	0.0064	0.1990	0.0518	...	0.1055	0.0850	*0.0615*
M_M	**0.1982**	**0.5039**	**0.4792**	**0.0281**	**0.0314**	**0.0550**	**0.0521**	**0.0069**	**0.1985**	**0.0510**	**0.0140**	**0.1051**	**0.0886**	...
s_M	0.0038	0.0080	0.0042	0.0011	0.0005	0.0026	0.0014	0.0004	0.0038	0.0015	0.0014	0.0018	0.0025	...
s_W	0.0024	0.0042	0.0052	0.0006	0.0008	0.0014	0.0007	0.0001	0.0009	0.0006	0.0007	0.0009	0.0019	...

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 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	Al (Total)	Al (Acid Sol.)	B	Co	Nb	Pb	V	Sb
Cv	**0.198**	**0.504**	**0.479**	**0.0281**	**0.0314**	**0.055**	**0.052**	**0.0069**	**0.198**	**0.0510**	**0.0140**	**0.105**	**0.089**
C(95%)	0.004	0.007	0.004	0.0010	0.0005	0.004	0.002	0.0005	0.004	0.0015	0.0022	0.003	0.004

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

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

DESCRIPTION OF SAMPLE

British Chemical Standard, BCS-CRM 458/2, is sold in bottles containing 100g chips graded 1700 - 250µm (10 - 60 mesh) for chemical analysis.
Spectroscopic Standard, SS-CRM 458/2, is sold as 38 mm diameter x 19 mm thick discs for spectroscopic analysis.

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

BCS/SS-CRM 458/2 CARBON STEEL (Residual Series)

NOTES ON METHODS USED

CARBON

All Analysts, except Nos. 3 and 5 determined carbon using high frequency combustion and infrared absorption. Nos. 4 and 8 calibrated with pure chemicals. Analysts Nos. 3 and 5 used non-aqueous titration according to BS 6200:3.8.2:1991.

SILICON

Analysts Nos. 1, 2, 3, 4, 5 and 8 used gravimetric methods. All Analysts, except No. 4, after dehydration with perchloric acid according to BS 6200:3.26.1:1995 (ISO 439-1994). Analyst No. 4 after dehydration with nitrosulphuric acid. Analysts Nos. 7 and 9 determined silicon by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). No. 9 calibrated with pure metal.

MANGANESE

Analysts Nos. 1, 2, 7, 8 and 9 determined manganese using ICP-OES, Nos. 1 and 9 calibrated with pure chemicals. Analysts Nos. 3 and 5 determined manganese photometrically after oxidation with periodate according to BS 6200:3.18.2:1985 (ISO 629-1982). Analyst No. 4 used Flame Atomic Absorption Spectrometry (FAAS).

PHOSPHORUS

Analysts Nos. 1, 2, 7, 8 and 9 determined phosphorus using ICP-OES, Nos. 1 and 9 calibrated with pure chemicals. Analysts Nos. 3 and 5 determined phosphorus photometrically as phosphovanadomolybdate according to BS EN 10184:1992. Analyst No. 4 used a titrimetric method after precipitation as phosphomolybdate.

SULPHUR

All Analysts, except No. 3, determined sulphur using high frequency combustion and infrared absorption. Nos. 4, 8 and 9 calibrated with pure chemicals. Analyst No. 3 determined sulphur gravimetrically as barium sulphate.

ALUMINIUM (TOTAL)

All Analysts determined total aluminium using ICP-OES. Nos 3, 5 and 9 calibrated with pure metal.

ALUMINIUM (ACID SOLUBLE)

Analyst No. 3 determined acid soluble aluminium using ICP-OES, Nos. 3, 5 and 9 calibrated with pure metal. Analyst No. 3 after dissolution according to BS 6200:3.1.4:1990 (ISO 9658-1990). The other Analysts used ICP-OES after dissolution procedures as follows:-

Analyst No. 5, 1:1 hydrochloric acid and oxidation with nitric acid; Analyst No. 8, 1:1:2 hydrochloric and nitric acids and water; Analyst No. 8, 1:1 nitric acid and Analyst No. 9, a mixture of hydrochloric, nitric and hydrofluoric acids.

Analyst No. 3 also determined the **acid-insoluble aluminium** using ICP-OES and obtained a mean value of 0.0013%.

BORON

Analysts Nos.3, 5 and 6 determined boron photometrically with curcumin according to BS EN 10200:1992. Analysts Nos. 7 and 9 used ICP-OES.

COBALT

All Analysts, ex**cep**t No. 3, determined cobalt using ICP-OES. Nos. 5, 6 and 9 calibrated with pure metal. Analyst No. 3 used FAAS according to BS 6200:3.11.3:1997 (ISO 11652-1997).

NIOBIUM

All Analysts, except No. 3, determined niobium using ICP-OES. Nos. 5, 6 and 9 calibrated with pure metal or metal oxide. Analyst No. 3 used a photometric method with 4-(2-pyridylazo) resorcinol after separation with phenylarsonic acid according to BS EN 10178:1989.

LEAD

Analysts Nos. 3 and 6 determined lead using FAAS. Analysts Nos. 5, and 7 used ICP-OES. Analysts Nos. 3, 5, and 9 calibrated with pure metal.

VANADIUM

All Analysts, except No. 3, determined vanadium using ICP-OES. Nos. 5, 6 and 9 calibrated with pure chemicals. Analyst No. 3 used a photometric method with N-benzoylphenyl-hydroxylamine according to BS 6200:3.34.2:1989 (ISO 4942:1988).

ANTIMONY

Analyst No. 3 determined antimony using FAAS. Analyst No. 6 also used FAAS after separation of antimony with hydride generation. Analysts Nos. 7 and 9 used ICP-OES. Analysts Nos. 3, 6 and 9 calibrated with pure metal.

ZIRCONIUM

All Analysts, except No. 3, determined zirconium using ICP-OES. Analyst No. 3 used a photometric method with arsenazo III.

INTENDED USE

BCS-CRM 458/2 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. The sample should be mixed thoroughly before each use.

SS-CRM 458/2 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 should be linished before use to remove any protective coating. N.B. Due to slight segregation of certain elements an area 6mm in diameter in the centre of the disc sample should be avoided for 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 458/2 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. SS-CRM 456/2 will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surfaces).

TRACEABILITY

The characterisation of BCS/SS-CRM 458/2 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/SS-CRM 458/2 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.

COMMUTABILITY

BCS-CRM 458/2 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 458/2 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

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