



# BUREAU OF ANALYSED SAMPLES LTD

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**BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL**

## CERTIFICATE OF ANALYSIS BCS<sup>†</sup>/SS<sup>‡</sup>-CRM No. 408/1 LOW ALLOY 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	Cu	V
1	0.278	0.23	0.52	0.038	0.027	0.105	0.092	4.46	0.66	0.031
2	...	...	...	...	...	0.105	0.10-	4.44	0.65	0.032
3	0.284	0.23	0.52	0.037	0.030	...	...	...	...	...
4	0.284	0.22	0.52	0.036	0.026	...	...	...	...	...
5	...	...	...	...	...	0.10-	0.11-	4.40	0.65	...
6	0.290	0.22	0.52	0.039	0.028	...	...	...	...	...
7	0.292	0.22	0.52	0.036	0.027	...	...	...	...	...
8	...	...	...	...	...	0.100	0.085	4.49	0.68	0.033
9	...	...	...	...	...	0.103	0.079	4.48	0.66	0.027
10	0.280	0.23	0.51	0.038	0.028	...	...	...	...	...
11	...	...	...	...	...	0.10-	0.10-	4.43	0.67	0.03-
12	0.287	0.23	0.49	0.035	0.028	...	...	...	...	...
13	...	...	...	...	...	0.102	0.089	4.48	0.67	0.033
<b>M<sub>M</sub></b>	<b>0.285</b>	<b>0.23</b>	<b>0.51</b>	<b>0.037</b>	<b>0.028</b>	<b>0.102</b>	<b>0.09-</b>	<b>4.45</b>	<b>0.66</b>	<b>0.031</b>
<b>s<sub>M</sub></b>	0.006	0.01	0.02	0.002	0.002	0.003	0.02-	0.04	0.02	0.003

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

**M<sub>M</sub>**: Mean of the intralaboratory means. **s<sub>M</sub>**: standard deviation of the intralaboratory means.

### CERTIFIED VALUES (C<sub>v</sub>)

mass content in %

	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	V
<b>C<sub>v</sub></b>	<b>0.285</b>	<b>0.23</b>	<b>0.51</b>	<b>0.037</b>	<b>0.028</b>	<b>0.102</b>	<b>0.09</b>	<b>4.45</b>	<b>0.66</b>	<b>0.031</b>
C(95%)	0.005	0.01	0.02	0.002	0.002	0.003	0.01	0.04	0.02	0.003

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.

# BCS/SS-CRM No. 408/1 LOW ALLOY STEEL

## NOTES ON METHODS USED

### CARBON

Analysts Nos. 1, 3, 4 and 10 determined carbon by non aqueous titration according to the British Standard Carbon Method 4\*. Nos. 6 and 12 used high frequency combustion with infrared measurement and No. 7 the gravimetric British Standard Carbon Method 1\*.

### SILICON

All analysts determined silicon by double dehydration with perchloric acid according to the British Standard Silicon Method 1\*.

### MANGANESE

Analysts Nos. 1, 3, 4, 6 and 10 determined manganese photometrically after oxidation with periodate and followed the procedure of the British Standard Manganese Method 2\*. No. 12 used a similar method but oxidized with persulphate/silver nitrate. Analyst No. 7 determined manganese titrimetrically after a zinc oxide separation and oxidation with persulphate/silver nitrate according to the British Standard Manganese Method 1\*.

Analysts Nos. 10 and 12 also determined manganese by atomic absorption spectrometry and both found 0.51%.

### PHOSPHORUS

Analysts Nos. 1, 3, 4, 7, 10 and 12 determined phosphorus photometrically as phosphovanadomolybdate according to the British Standard Phosphorus Method 2\*. Analyst No. 6 used a titrimetric method after precipitation as phosphomolybdate.

### SULPHUR

Analysts Nos. 1 and 3 determined sulphur gravimetrically according to the British Standard Sulphur Method 1\*. Nos. 4, 6, 7, 10 and 12 used combustion methods: No. 4 absorbed the evolved gases in water and titrated with iodate; No. 10 absorbed in hydrogen peroxide and titrated with borate; Nos. 6, 7 and 12 used high frequency combustion/infrared absorption.

### CHROMIUM

Analysts Nos. 1, 5 and 11 determined chromium by titration with ammonium ferrous sulphate after oxidation with persulphate/silver nitrate. No. 1 used the Analoid Method No. 37 and Nos. 5 and 11 followed the procedure of the British Standard Chromium Method 1\*. Analysts Nos. 2 and 9 used the atomic absorption spectrometry. Nos. 8 and 13 determined chromium photometrically with diphenylcarbazide according to the British Standard Chromium Method 2\*.

### MOLYBDENUM

Analysts Nos. 1, 5, 8, 11 and 13 determined molybdenum photometrically as the oxythiocyanate. No. 1 used the direct Analoid Method No. 42 whereas Nos. 5, 8, 11 and 13 followed the procedure of the British Standard Molybdenum Method 1\* which involves extraction of the oxythiocyanate complex into butyl acetate. Analysts Nos. 2 and 9 used atomic absorption spectrometry.

### NICKEL

Analysts Nos. 1, 2, 5, 8 and 13 determined nickel by methods involving precipitation with dimethylglyoxime. No. 1 dissolved the precipitate in sulphuric acid, boiled with excess of ferric sulphate and titrated with dichromate (Analoid Method No. 62). Nos. 2 and 13 dissolved in acid and titrated with EDTA. Nos. 5 and 8 completed cyanometrically according to the British Standard Nickel Method 1\*. Analyst No. 11 determined nickel photometrically with dimethylglyoxime and followed the procedure of the British Standard Nickel Method 3\*. No. 9 used atomic absorption spectrometry.

### COPPER

Analysts Nos. 1, 8, 11 and 13 determined copper by photometric methods. Nos. 1 and 13 used biscyclohexanone oxalyldihydrazone (Analoid Method No. 65) and Nos. 8 and 11 used 2,2'-diquinolyl with extraction into pentan-2-ol. No. 8 followed the procedure of the British Standard Copper Method 3\*. Analysts Nos. 2 and 9 used atomic absorption spectrometry. No. 5 used the British Standard Copper Method 2\* in which the copper is separated as sulphide and evaluated by iodimetric titration.

### VANADIUM

Nos. 1, 2 and 8 determined vanadium photometrically as phosphovanadotungstate. Nos. 1 and 8 followed the procedure of the Analoid Method No. 59. No. 2 carried out a preliminary mercury cathode separation. Nos. 5 and 11 used the titrimetric British Standard Vanadium Method 1\*. No. 9 used atomic absorption spectrometry. No. 13 used a photometric method with 3,3 dimethylnaphthidine.

\*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.

### DESCRIPTION OF SAMPLE

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

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

### INTENDED USE & STABILITY

The chip sample, BCS-CRM 408/1, 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 408/1, 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 finished 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

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Preliminary Edition ..... November 1978  
Main Edition ..... December 1980  
Main Edition (revised with C(95%) and s<sub>M</sub> values for each element) ..... December 2009