



# BUREAU OF ANALYSED SAMPLES LTD

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## BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

# CERTIFICATE OF ANALYSIS BCS-CRM No. 317 (ECRM 151-1) LOW CARBON, HIGH SILICON STEEL

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

### ANALYSES OF MAJOR ELEMENTS

Mean of 4 values - mass content in %.

Analyst No.	C	Si	Mn	P
1	0.029	3.50	0.081	0.017
2	0.028	3.49	0.086	0.015
3	0.027	3.50	0.085	0.015
4	0.028	3.49	0.087	0.015
5	0.029	3.47	0.082	0.014
6	0.028	3.49	0.087	0.016
7	0.027	3.49	0.087	0.016
8	0.026	3.50	0.091	0.016
9	0.027	3.48	0.088	0.013
<b>M<sub>M</sub></b>	<b>0.028</b>	<b>3.49</b>	<b>0.086</b>	<b>0.015</b>
<b>s<sub>M</sub></b>	0.001	0.01	0.004	0.002

The certification of sulphur was carried out as a subsequent separate exercise with the following results.

### ANALYSES OF SULPHUR

Mean of 4 values - mass content in %.

Analyst No.	S
1	0.022
2	0.022
3	0.024*
4	0.025
5	0.024
6	0.022
7	0.025*
8	0.023
<b>M<sub>M</sub></b>	<b>0.023</b>
<b>s<sub>M</sub></b>	0.002

\*original values determined in 1968

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 (Cv)

mass content in %

	C	Si	Mn	P	S
<b>Cv</b>	<b>0.028</b>	<b>3.49</b>	<b>0.086</b>	<b>0.015</b>	<b>0.023</b>
C(95%)	0.001	0.01	0.003	0.001	0.002

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-CRM No. 317 (ECRM 151-1)

## LOW CARBON, HIGH SILICON STEEL

### ANALYSES OF MAJOR ELEMENTS

#### CO-OPERATING ANALYSTS

##### INDEPENDENT ANALYST

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##### GOVERNMENT DEPARTMENT

- 2 EDWARDS, F. H., *BSc*, Bragg Laboratory, Chemical Inspectorate, Sheffield.

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#### NOTES ON METHODS USED

##### CARBON

Analysts Nos. 1, 4 and 6 determined carbon by electrical conductivity after combustion in oxygen. Nos. 2 and 7 used a non-aqueous titration method (Jones, et.al., Analyst, 1965, **90**, 623; 1966, **91**, 399). Nos. 3, 5, 8 and 9 used the low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144).

Analysts Nos. 1 and 9 also determined carbon by non-aqueous titration and found 0.028% in each case. No. 2 also used two alternative methods (a) a microchemical conductimetric method (b) the Standard gravimetric method B.S. 1121: Part 11: 1942; the results were 0.028% and 0.03% respectively. Nos. 3 and 5 also determined carbon by infrared absorption (Tipler, Analyst, 1963, **88**, 272) and found 0.026% and 0.029% respectively.

##### SILICON

All Analysts determined silicon gravimetrically by double dehydration with perchloric acid according to the Standard method B.S. 1121: Part 10: 1967.

##### MANGANESE

All Analysts except No. 5 determined manganese photometrically after oxidation to permanganic acid. Nos. 1, 2, 3, 4, 6, 7 and 9 oxidized with periodate according to the Standard method B.S. 1121: Part 23: 1951. No. 8 oxidized with persulphate/silver nitrate. No. 5 determined manganese titrimetrically by oxidation with persulphate/silver nitrate and titration with arsenite solution.

##### PHOSPHORUS

Analysts Nos. 1, 2 and 9 determined phosphorus gravimetrically according to the Standard method B.S. 1121: Part 9: 1948. Nos. 3, 6, 7 and 8 determined phosphorus photometrically as phosphovanadomolybdate; Nos. 3, 7 and 8 used the Standard method B.S. 1121 Part 45: 1966, whereas No. 6 used the Elwell-Wilson method (Analyst, 1956, **81**, 136; 1957, **82**, 453). Nos. 4 and 5 used titrimetric methods after precipitation of the phosphorus as phosphomolybdate.

Analysts Nos. 1, 2, 4 and 9 also used the Standard photometric method and found 0.013%, 0.015%, 0.014% and 0.013% respectively.

#### ANALYSES OF SULPHUR

#### CO-OPERATING ANALYSTS

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8 WOODMAN, G.L., British Steel Electricals, Orb Works, Newport.

#### NOTES ON METHODS USED

##### SULPHUR

Analyst No. 1 determined sulphur gravimetrically after chromatographic separation on alumina (Nydahl, Anal. Chem., 1954, **26**, 580). The other Analysts used combustion methods. Nos. 2, 4, 5 and 8 used high frequency combustion in oxygen/infrared absorption. No. 4 used a flux consisting of 80% tungsten / 20% tin and No. 8 used a 100% tungsten flux. Nos. 6 and 7 used methods based on combustion in air (Jones et al., J.Iron Steel Inst., 1966, **204**, 505). No. 7 determined sulphur titrimetrically and No. 8 coulometrically.

Analyst No. 1 also determined sulphur using a combustion method and obtained a mean value of 0.023%.

#### DESCRIPTION OF SAMPLE

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

#### INTENDED USE & STABILITY

The chip sample, BCS-CRM 317 (ECRM 151-1), 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.

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For BUREAU OF ANALYSED SAMPLES LTD

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Managing Director

Preliminary Edition ..... November 1966  
Main Edition ..... February 1968  
Main Edition (revised with certified value for Sulphur) ..... October 1989  
Main Edition (revised with C(95%) and  $s_M$  values for each element) ..... September 2011