

# BUREAU OF ANALYSED SAMPLES LTD

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**BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL** 

# CERTIFICATE OF ANALYSIS BCS-CRM No. 237/2 (ECRM 060-1) 0.1% CARBON STEEL

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

### **CO-OPERATING ANALYSTS**

| CO-OFERATING ANALISIS   |  |  |  |  |  |
|---|--|--|--|--|--|
| INDEPENDENT ANALYST   | ANALYSTS representing MANUFACTURERS and USERS (cont.)        |  |  |  |  |
| 1 COPPINS, W. C., MSc, FRIC, Ridsdale & Co. Ltd., Middlesbrough.      | 4 HARRISON, T. S., BSc, PhD, CChem., FRIC,                   |  |  |  |  |
| GOVERNMENT DEPARTMENT   | B.S.C. Scunthorpe Group, Scunthorpe.                         |  |  |  |  |
| 2 NICHOLLS,H.A., CChem., FRIC,  | 5 HUNTER, D. P., <i>FIM</i> , B.S.C. Orb Works, Newport.     |  |  |  |  |
| Bragg Laboratory, Materials Quality Assurance Directorate, Sheffield. | 6 KIDMAN, L., AMet, AIM, AMInstF,                            |  |  |  |  |
| ANALYSTS representing MANUFACTURERS and USERS                         | B.S.C. Rotherham Works, Rotherham.                           |  |  |  |  |
| 3 BAGSHAWE, B., AMet, FIM, MInstF,                                    | 7 MCARDLE, A. J., AIM, B.S.C. Lackenby Works, Middlesbrough. |  |  |  |  |
| Brown Firth Research Laboratories, Sheffield.                         | 8 MUIR, S., B.S.C. Corby Works, Corby.                       |  |  |  |  |

#### ANALYSES Mean of 4 values - mass content in %

| Mean of 4 values - mass content in 70. |       |      |        |      |       |       |       |        |       |       |       |       |
|--|-------|------|--------|------|-------|-------|-------|--------|-------|-------|-------|-------|
| Analyst<br>No.                         | С     | Mn   | Ν      | Si   | Р     | S     | Cr    | Мо     | Ni    | Al    | Си    | Sn    |
| 1                                      | 0.122 | 0.45 |        | 0.17 | 0.024 | 0.031 | 0.028 | <0.005 | 0.039 | 0.004 | 0.060 | 0.005 |
| 2                                      | 0.120 | 0.44 | 0.0040 |      |       |       |       |        |       |       |       |       |
| 3                                      | 0.120 | 0.45 | 0.0042 |      |       |       |       |        |       |       |       |       |
| 4                                      | 0.120 | 0.45 | 0.0047 |      |       |       |       |        |       |       |       |       |
| 5                                      | 0.124 | 0.46 | 0.0038 |      |       |       |       |        |       |       |       |       |
| 6                                      | 0.121 | 0.45 | 0.0040 |      |       |       |       |        |       |       |       |       |
| 7                                      | 0.125 | 0.46 | 0.0040 |      |       |       |       |        |       |       |       |       |
| 8                                      | 0.122 | 0.45 | 0.0048 |      |       |       |       |        |       |       |       |       |
| $\mathbf{M}_{\mathbf{M}}$              | 0.122 | 0.45 | 0.0042 |      |       |       |       |        |       |       |       |       |
| s <sub>M</sub>                         | 0.002 | 0.01 | 0.0004 |      |       |       |       |        |       |       |       |       |

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

Figures in bold type standardized, 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)

|        | С     | Mn   | Ν     |  |  |
|--------|-------|------|-------|--|--|
| Cv     | 0.122 | 0.45 | 0.004 |  |  |
| C(95%) | 0.002 | 0.01 | 0.001 |  |  |

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. 237/2 (ECRM 060-1) 0.1% CARBON STEEL

#### NOTES ON METHODS USED

#### CARBON

All analysts determined carbon by combustion in oxygen. Nos. 1, 2, 5 and 7 completed by non-aqueous titration (Jones et al., Analyst, 1965, **90**, 623; 1966, **91**, 399). Nos. 3 and 4 used the conventional low-pressure method (Cook and Speight, Analyst, 1956, **81**, 144) whereas No. 6 used a modification of this method (Dunnill and Kent, Metallurgia, 1970, **81**, 125). No. 8 completed coulometrically.

Analyst No. 3 also used an infrared absorption method and found 0.119%. Nos. 5 and 8 also determined carbon gravimetrically and found 0.121% and 0.125% respectively. Nos. 6 and 7 also used a thermal-conductivity method and found 0.122% and 0.126% respectively.

#### MANGANESE

Analyst No. 1 determined manganese by oxidation with persulphate/silver nitrate and titration with arsenite/nitrite solution (Analoid Method No. 53). Nos. 2, 3, 4, 6, 7 and 8 determined manganese photometrically by oxidation with periodate. Nos. 2, 4, 6, 7 and 8 used the British Standard Manganese Method 2\*; No. 3 used ISO Recommendation R629. No. 5 used atomic-absorption spectroscopy.

Analyst No. 5 also determined manganese photometrically according to the British Standard Manganese Method 2\* and found 0.47%. No. 7 also determined manganese by oxidation with persulphate/silver nitrate and titration with sodium arsenite solution and found 0.46%.

#### NITROGEN

All analysts used methods depending on conversion of the nitrides to ammonium salts, followed by distillation of the ammonia from a sodium hydroxide solution. Nos. 2, 4, 5 and 6 determined the nitrogen photometrically; Nos. 2, 4 and 6 used the Nessler Method whereas No. 5 used the indo-phenol blue reaction. Nos. 3, 7 and 8 titrated the ammonia with standard acid solution according to the British Standard Nitrogen Method 1\*.

Analyst No. 6 also used the British Standard Nitrogen Method 1\* and found 0.004%.

#### SILICON

British Standard Silicon Method 1\*.

#### PHOSPHORUS

British Standard Phosphorus Method 2\*.

#### SULPHUR

British Standard Sulphur Method 1\*.

#### CHROMIUM

Diphenylcarbazide photometric method (Analoid Method No. 51).

#### MOLYBDENUM

Photometric method depending on formation of oxythiocyanate (Analoid Method No. 63)

#### NICKEL

After separation of the bulk of the iron by extraction into diethyl ether the nickel was determined photometrically with dimethylglyoxime.

#### ALUMINIUM

British Standard Aluminium Method 3\*.

#### COPPER

bis-Cyclohexanone oxalyldihydrazone photometric method (Analoid Method No. 65).

#### TIN

Reduction with aluminium followed by iodimetric titration.

\*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.

#### **INTENDED USE & STABILITY**

The chip sample, BCS-CRM 237/2 (ECRM 060-1), 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.

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.

| NEWHAM HALL, NEWBY,  | For BUREAU OF ANALYSED SAMPLES LTD |
|--|------------------------------------|
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|  |                                    |
| Preliminary Edition  | April 1974                         |
| Preliminary Edition reprinted  | August 1974                        |
| Main Edition   | December 1975                      |
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|  |                                    |