



# BUREAU OF ANALYSED SAMPLES LTD

Directors:-

R. P. MEERES, *BA (Oxon), MRSC* (Managing)G. C. FLINTOFT, *ACMA*

J. C. MEERES

M. S. TAYLOR, *PhD, CChem, MRSC*

**BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL**

## CERTIFICATE OF ANALYSIS BCS-CRM No. 270 (ECRM 054-1) 0.09% PHOSPHORUS STEEL

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

### CO-OPERATING ANALYSTS

#### INDEPENDENT ANALYSTS

1 SPEIGHT, K., *AMet, AIM*,  
British Iron & Steel Research Association, Sheffield.

2 COPPINS, W. C., *MSc, ARIC*, Ridsdale & Co. Ltd., Middlesbrough.

#### ANALYSTS representing MANUFACTURERS and USERS

3 BAGSHAWE, B., *AMet, FIM, MInstF*,  
Brown Firth Research Laboratories, Sheffield.

4 CHARTERIS, W. F., Colvilles Ltd., Motherwell.

5 CLYDE, C., Lanarkshire Steel Co., Ltd., Motherwell.

6 ELWELL, W. T., *FRIC*,  
Imperial Chemical Industries Ltd., Metals Division, Birmingham.

#### ANALYSTS representing MANUFACTURERS and USERS (cont.)

7 GARDNER, L. E., *AMet, FIM*, Edgar Allen & Co., Ltd., Sheffield.

8 HARRISON, J. M.,  
Lancashire Steel Corporation Ltd., Irlam, Manchester.

9 LUNT, A. P., *AIM*, Park Gate Iron & Steel Co., Ltd., Rotherham.

10 MANTERFIELD, D., *FIM*,  
Steel, Peech & Tozer (branch of United Steel Co's, Ltd.), Sheffield.

11 SHORT, C. W., J. Lysaght's Scunthorpe Works Ltd., Scunthorpe.

12 WILLIAMS, T. H., *BSc*,  
Stewarts & Lloyds Ltd., Corby.

### ANALYSES

Mean of 4 values - mass content in %.

Analyst No.	C	P	Si	Mn	S*	Cr	Mo	Ni	As	Cu	V
1	0.21	0.091	...	...	...	...	...	...	...	...	...
2	0.22	0.092	<i>0.05</i>	<i>0.88</i>	<i>0.11</i>	<i>0.17</i>	<i>0.02</i>	<i>0.14</i>	<i>0.03</i>	<i>0.21</i>	<i>&lt;0.01</i>
3	0.22	0.091	...	...	...	...	...	...	...	...	...
4	0.22	0.093	...	...	...	...	...	...	...	...	...
5	0.22	0.092	...	...	...	...	...	...	...	...	...
6	0.21	0.092	...	...	<i>0.10</i>	...	...	...	...	...	...
7	0.22	0.091	...	...	...	...	...	...	...	...	...
8	0.22	0.092	...	...	...	...	...	...	...	...	...
9	0.22	0.092	...	...	...	...	...	...	...	...	...
10	0.22	0.090	...	...	...	...	...	...	...	...	...
11	0.22	0.092	...	...	...	...	...	...	...	...	...
12	0.21	0.091	...	...	...	...	...	...	...	...	...
<b>M<sub>M</sub></b>	<b>0.22</b>	<b>0.092</b>	...	...	...	...	...	...	...	...	...
<b>S<sub>M</sub></b>	<b>0.01</b>	<b>0.001</b>	...	...	...	...	...	...	...	...	...

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

\* This material was also examined by 70 laboratories in the E.U. and the Sulphur content was found to have a mean value of 0.109% with a standard deviation of 0.005%.

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

mass content in %

	C	P
<b>C<sub>v</sub></b>	<b>0.22</b>	<b>0.092</b>
<b>C(95%)</b>	<b>0.01</b>	<b>0.001</b>

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. 270 (ECRM 054-1)

## 0.09% PHOSPHORUS STEEL

### NOTES ON METHODS USED

#### CARBON

All Analysts determined carbon gravimetrically by combustion. Nos. 1, 2, 3, 4, 8, 9 and 12 used the Standard method B.S. 1121: Part 11: 1948. On account of the high sulphur content of this steel additional chromic-sulphuric acid traps were used by Nos. 1, 2, 3 and 12 (see Section Two of Part 11). Other Analysts used similar methods but did not follow the Standard procedure. Those who followed the Standard procedure, with the exception of No. 2, used lead or red lead flux; No. 2 used no flux. Lead was also used by No. 5 and red lead by No. 11; No. 8 used lead chromate and No. 12 tin.

Analyst No. 11 also determined carbon by a Ströhlein method and found 0.21%.

#### PHOSPHORUS

Analysts Nos. 1, 2, 3, 4, 5, 7, 8, 9, 11 and 12 used the Standard gravimetric method for phosphorus in high chromium-nickel steels B.S. 1121: Part 9 : 1948. No. 10 used the Standard gravimetric method B.S. 1121 Part 1: 1943. No. 6 determined phosphorus photometrically as phosphovanadomolybdic acid after a preliminary treatment with hydrochloric/hydrobromic acid (Elwell and Wilson, Analyst, 1956, **81**, 136; 1957, **82**, 453).

A number of Analysts also used alternative methods as follows. Nos. 2, 5 and 11 used the Analoid Method No. 21 and found 0.092%, 0.094% and 0.095% respectively. No. 4 used B.S. 1121: Part 1: 1943 and a phosphomolybdate titrimetric method and found 0.093% in each case. No. 6 obtained a result of 0.091% by B.S. 1121: Part 9: 1948. No. 7 found 0.090% by a phosphovanadomolybdate photometric method. Nos. 9 and 10 used phosphomolybdate titrimetric methods and found 0.095% and 0.091% respectively.

#### SILICON

*Standard gravimetric method B.S. 1121: Part 10: 1948.*

#### MANGANESE.

*Oxidation to permanganic acid with persulphate /silver nitrate followed by titration with arsenite/nitrite solution.*

#### SULPHUR

*Standard gravimetric method B.S. 1121: Part 1A: 1957.*

#### CHROMIUM.

*Diphenylcarbazide photometric method (Analoid Method No. 51).*

#### MOLYBDENUM.

*Thiocyanate photometric method (Analoid Method No. 42).*

#### NICKEL.

*Dimethylglyoxime photometric method (Analoid Method No. 44).*

#### ARSENIC.

*Hypophosphite titrimetric method.*

#### COPPER.

*Standard photometric method B.S. 1121: Part 36: 1956.*

#### VANADIUM.

*Standard titrimetric method B.S. 1121: Part 25: 1956.*

### DESCRIPTION OF SAMPLE

British Chemical Standard - bottles of 100g chips graded 1700 - 250 $\mu$ m (10 - 60 mesh) for chemical analysis.

### INTENDED USE & STABILITY

The chip sample, BCS-CRM 270, 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,  
MIDDLESBROUGH, ENGLAND, TS8 9EA  
Email: enquiries@basrid.co.uk  
Website: www.basrid.co.uk

For BUREAU OF ANALYSED SAMPLES LTD

R.P. MEERES,  
Managing Director

*Preliminary Edition* ..... May 1957  
*Main Edition* ..... July 1959  
*Main Edition (additional information for sulphur)* ..... November 1975  
*Main Edition (revised with C(95%) and s<sub>M</sub> values for each element)* ..... November 2010