

Sampling and analysis of iron, steel and other ferrous metals —

Part 3: Methods of analysis — Section 3.8 Determination of carbon —

Subsection 3.8.2 Steel and cast iron: non-aqueous titrimetric method after combustion

Confirmed November 2008

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Iron and Steel Standards Policy Committee (ISM/-) to Technical Committee ISM/18, upon which the following bodies were represented:

- BCIRA
- British Steel Industry
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Ferro Alloys and Metals Producers' Association
- Ministry of Defence

This British Standard, having been prepared under the direction of the Iron and Steel Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 29 November 1991

© BSI 09-1999

The following BSI references relate to the work on this standard:
Committee reference ISM/18
Draft for comment 91/35212 DC

ISBN 0 580 19948 7

Amendments issued since publication

Amd. No.	Date	Comments

Contents

	Page
Committees responsible	Inside front cover
Foreword	ii
1 Scope	1
2 Principle	1
3 Reagents	1
4 Apparatus	1
5 Sampling	4
6 Procedure	4
7 Calculation and expression of results	7
8 Test report	8
Figure 1 — Apparatus for the determination of carbon: non-aqueous titration	2
Figure 2 — Detail of tuyère for rapid insertion of the sample	3
Figure 3 — Detail of heat-resisting glass prolong for manganese dioxide	3
Figure 4 — Standardization using carbon dioxide injection	6
Table 1 — Mass of test portion and titrant concentration	4
Table 2 — Precision data	8
Table 3 — Predicted values of r and R	8
Publications(s) referred to	Inside back cover

Foreword

This Subsection of BS 6200 has been prepared under the direction of the Iron and Steel Standards Policy Committee and supersedes method 4 for the determination of carbon in BSI Handbook No. 19, to which it is technically equivalent.

BS 6200 is a multipart British Standard covering all aspects of the sampling and analysis of iron, steel and other ferrous metals. A list of contents, together with general information, is given in Part 1.

Other methods for the determination of carbon in ferrous metals have been published in BS 6200-3.8.3 and in BS EN 10036. A method for the determination of non-combined carbon (graphite) has been published as Subsection 3.8.5 for use in conjunction with BS EN 10036.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Subsection of BS 6200 describes a non-aqueous titrimetric method for the determination of carbon in steel and cast iron.

The method covers the range of carbon contents found in all types of steel, pig and cast iron and commercially pure iron.

NOTE The titles of the publications referred to in this Subsection of BS 6200 are listed on the inside back cover.

2 Principle

The test portion is burnt in pure oxygen at a high temperature (up to 1 350 °C) in the presence of a flux. Oxides of sulfur are removed with manganese dioxide and carbon dioxide is absorbed in a mixture of dimethylformamide-ethanolamine. It is then titrated with tetra-*n*-butylammonium hydroxide.

3 Reagents

During analysis use only reagents of recognized analytical grade.

3.1 Benzoic acid, standard solution, 0.1 mol/L, in toluene, equivalent to 1.2 mg carbon per millilitre. Dissolve 1.221 g of high-purity benzoic acid C_6H_5COOH , in toluene which has been neutralized previously with tetra-*n*-butylammonium hydroxide (**3.14**) to the change point of thymolphthalein (**3.15**). Transfer to a 100 mL volumetric flask, dilute to the mark with neutralized toluene and mix.

3.2 Benzoic acid, standard solution, 0.02 mol/L, in toluene. Transfer by means of a safety pipette, calibrated for delivery of toluene, 20 mL of benzoic acid solution (**3.1**) to a 100 mL volumetric flask, dilute to the mark with neutralized toluene and mix.

3.3 Carbon dioxide absorbent. Replacement for soda asbestos manufactured from synthetic silicates; granular, particle size from 0.71 mm to 1.18 mm, (14 mesh to 22 mesh).

3.4 Carbon dioxide, gas, not less than 99.8 % purity.

3.5 Charcoal, activated for gas absorption; granular, particle size from 0.85 mm to 1.70 mm, (10 mesh to 18 mesh).

3.6 Dimethylformamide absorption solution. Add 2 mL of thymolphthalein (**3.15**) and 5 mL of monoethanolamine to 150 mL of dimethylformamide and mix.

WARNING. Suitable safety precautions should be taken when making and using this solution. Dimethylformamide is toxic if inhaled, ingested or absorbed through the skin.

3.7 Fluxes. Tin powder, low in carbon, or alternatively use freshly calcined carbon-free *trilead* tetraoxide instead of tin powder. When *trilead* tetraoxide is used keep it in a hot muffle furnace, at 450 °C to 480 °C.

Pure iron of known carbon content less than 0.005 % (*m/m*) and in a similar state of division to the test sample may be required as a supplementary flux for highly alloyed steels.

3.8 Magnesium perchlorate, anhydrous, $Mg(ClO_4)_2$; granular, particle size from 0.71 mm to 1.18 mm, (14 mesh to 22 mesh).

3.9 Manganese dioxide, chemically-reactive precipitated grade; granular, particle size from 0.50 mm to 1.70 mm, (10 mesh to 30 mesh).

3.10 Oxygen, at least 99.5 % purity.

3.11 Platinized ceramic fibre, 5 % platinum.

3.12 Synthetic ceramic fibre.

3.13 Tetra-*n*-butylammonium hydroxide, 0.1 mol/L, (0.1 N) approximately, in toluene/methanol.

NOTE This reagent is best obtained from commercial suppliers.

3.14 Tetra-*n*-butylammonium hydroxide, 0.02 mol/L, (0.02 N), approximately. Transfer 200 mL of tetra-*n*-butylammonium hydroxide (**3.13**) to a 1 L volumetric flask, dilute to the mark with toluene and mix. Allow to stand for 24 h before standardization and use.

NOTE If this reagent is stored in a cold room two phases may separate. To avoid this it is recommended that the titrant is stored at a room temperature of 20 °C or above. If separation still occurs it can be overcome by making small additions of anhydrous methanol and shaking until the solution is clear.

3.15 Thymolphthalein, 1 g/L. Dissolve 0.1 g of thymolphthalein in 100 mL anhydrous methanol.

4 Apparatus

4.1 Ordinary laboratory apparatus

4.2 Volumetric glassware, in accordance with class A of BS 846, BS 1583 or BS 1792, as appropriate.

4.3 Apparatus for carbon determination

NOTE The apparatus for the determination of carbon by combustion and non-aqueous titration is shown diagrammatically in Figure 1.