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Steel — Determination of low carbon contents — Part II : Titrimetric method after combustion

Acier — Dosage du carbone en faibles teneurs — Partie II : Méthode titrimétrique après combustion

FOREWORD

Technical Report 4830 was drawn up by Technical Committee ISO/TC 17, *Steel*, and approved by a majority of its members. The reasons which led to the publication of this document in the form of a Technical Report are given in the Introduction.

In January 1978, this document was submitted to the ISO Council, which approved its publication as a Technical Report.

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0 INTRODUCTION

Four methods for the determination of low carbon contents in steels have been examined by ISO/TC 17, namely :

- manometric (low-pressure);
- titrimetric (titration in non-aqueous medium);
- conductimetric;
- coulometric.

The results of experiments carried out in relation to these four methods have not allowed a decision to be taken on the adoption of any one of them as a standard method at the international level.

Consequently, it has been generally agreed, as a preliminary measure, to make all four methods available in the form of parts of a Technical Report, i.e. ISO/TR 4830.

1 SCOPE AND FIELD OF APPLICATION

This part of this Technical Report describes a titrimetric method for the determination of the total carbon content in pure iron and in steels after combustion in an electric resistance furnace.

The method is applicable to carbon contents between 0,005 and 0,1 %.

2 REFERENCE

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel*.

3 PRINCIPLE

Combustion of a test portion in an electric resistance furnace in a current of oxygen, in the presence of a fluxing agent which encourages the combustion (see note 9.10), converting the carbon to carbon dioxide, which is absorbed in a mixture of pyridine and 2-aminoethanol or of formdimethylamide and 2-aminoethanol, and which is at the same time titrated using thymol blue as indicator.

Calculation of the carbon content from the consumption of the standard volumetric solution. This solution is standardized against calcium carbonate (primary standard), which is heated to a temperature between 1 250 and 1 400 °C in the same furnace.

4 REAGENTS

During the analysis, use only reagents of recognized analytical quality, and only distilled water or water of equivalent purity.

4.1 Oxygen, purified.

Pass the current of oxygen through a combustion tube, filled with platinized asbestos (4.2.1) and placed in a heated furnace so that a 25 cm portion of the tube has a temperature of 600 to 625 °C. Pass the oxygen leaving the tube through absorption columns, filled respectively with soda lime (4.2.3) and soda asbestos (4.2.2).

4.2 Oxygen-purifying agents :

4.2.1 Platinized asbestos, with 10 % platinum.

4.2.2 Soda asbestos, grain size about 2 to 4 mm.

4.2.3 Soda lime, grain size about 2 to 4 mm.

4.3 Agents for absorbing sulphur oxides :

4.3.1 Manganese(IV) oxide, grain size about 0,7 to 1,5 mm; or

4.3.2 Hydrogen peroxide fixed on urea, grain size about 4 mm.

4.4 Fluxing agents : tin, iron or steel, having the lowest possible carbon content determined in accordance with 7.4.2.

4.5 Iron having a low carbon content.

4.6 Active charcoal.

4.7 Pyridine, dried if necessary by means of an activated molecular sieve (water content of pyridine less than 0,25 %).

4.8 Formdimethylamide, dried if necessary.

4.9 2-Aminoethanol (ethanolamine).

Avoid contact with air.

1 ml of ethanolamine should consume not more than 1,25 ml of the standard volumetric solution (4.11).

4.10 Absorption mixture.

Add to 150 ml of the pyridine (4.7) or formdimethylamide (4.8), 2,5 ml of the ethanolamine (4.9) and 2 ml of the thymol blue solution (4.12).

This mixture should be freshly prepared each day (see note 9.13).

4.11 Standard volumetric solution.

4.11.1 Preparation

Prepare either of the two following solutions :

4.11.1.1 Sodium methanolate, about 0,02 M solution.

Dissolve 2,3 g of white sodium in 1 litre of methanol free from carbon dioxide (water content less than 0,02 %).

Transfer 200 ml of this solution to a 1 000 ml volumetric flask. Add 10 ml of the thymol blue solution (4.12), make up to volume with the pyridine (4.7) and mix.

NOTE — Prepare and store this solution in such a way as to exclude carbon dioxide.

4.11.1.2 Tetra-*n*-butylammonium hydroxide (TBAH), about 0,02 M solution.

Place 200 ml of a 0,1 M TBAH solution in a 1 000 ml volumetric flask. Add 10 ml of the thymol blue solution (4.12), make up to volume with the pyridine (4.7) and mix.

NOTE — Prepare and store this solution in such a way as to exclude carbon dioxide.

4.11.2 Standardization

Weigh 0,020 g of calcium carbonate (primary standard), previously dried for 2 h at 400 °C and kept on calcium sulphate free from water. Place in a calcined boat and treat as specified in 7.3.

4.11.3 Blank test

Carry out a blank test using an empty calcined boat and treating it as specified in 7.3.

4.11.4 Calculation of the concentration

The concentration T , expressed as a molarity, of the standard volumetric solution is given by the formula

$$T = \frac{m_1}{100,08 (V_1 - V_2)}$$

where

m_1 is the mass, in milligrams, of calcium carbonate taken for the standardization (4.11.2);

V_1 is the volume, in millilitres, of the standard volumetric solution consumed for the standardization (4.11.2);

V_2 is the volume, in millilitres, of the standard volumetric solution consumed for the blank test (4.11.3).

4.12 Thymol blue, 50 mg/25 ml solution in pyridine (4.7).

5 APPARATUS

The apparatus is divided into two parts :

- the first part includes a flowmeter, an electric resistance furnace with combustion tube, and an absorption tube, filled with absorbing agents (4.3) to retain the sulphur oxides;
- the second part is the apparatus for the titration of the carbon dioxide formed.

The two parts are connected by tubes and bungs to form an airtight unit.

A diagram of the complete apparatus is shown in figure 1. The titration vessel is shown in figure 2 (see also note 9.14), and details of the tuyere fitted to the combustion tube are given in figure 3.

The tuyere, which slides on to the combustion tube (see figure 3), may be considered as a T-connection : the oxygen on entering divides in two directions. One part is drawn into the combustion tube by means of a pump, at the end of the system, combined with a high-precision regulating valve and a flowmeter; the remaining part leaves the combustion tube at the point marked G. By adjusting the current of oxygen leaving at G to a sufficient rate, the penetration of air into the tube is prevented. Moreover, the combustion boat may be easily introduced into and withdrawn from the furnace.

The combustion gases leave the combustion tube together with the remaining oxygen via the tube for absorbing sulphur oxides, and are introduced into the titration vessel, where the carbon dioxide is absorbed. The residual gas leaves the titration vessel via two columns, filled with active charcoal (4.6), and is evacuated through a three-way tap and the high-precision regulating valve by means of the pump. The rate of flow of the gas passing through the titration vessel is controlled by the regulating valve. The three-way tap is used to put the current of gas "in" or "out" of the circuit. Account should be taken of the fact that the three-way tap has two incorrect positions.

Tap T₁ has no basic importance here but serves as a possible safety device in cases of emergency.

See also notes 9.4, 9.5, 9.6, 9.7 and 9.13.

6 SAMPLING

Sampling shall be carried out in accordance with ISO/R 377.

7 PROCEDURE

7.1 Preparation of the test portion

Degrease the test sample by washing (for example with freon or ether). Evaporate the last traces of the washing liquid by heating.

Weigh 1 g of the test sample to the nearest 0,001 g and transfer to a calcined boat (see note 9.6). Add 1,0 g of fluxing agent (4.4).

7.2 Preparation of apparatus

Heat the furnace to a temperature between 1 250 and 1 400 °C (see note 9.10). Adjust the current of oxygen (4.1) to at least 180 l/h, as shown by the flowmeter C. Set the three-way tap T₃ in the "out" position and set the pump working. Adjust the regulating valve for a very limited aspiration of air.

Open tap T₁ and set the three-way tap in the "in" position (see note 9.3). Now adjust the gas flow to about 18 l/h by means of the regulating valve. Introduce a suitable amount (see notes 9.1 and 9.14) of the absorption mixture (4.10) into the titration vessel (see figure 1 and note 9.8).

Owing to the carbon dioxide present in the apparatus, the absorption mixture turns yellow; add the standard volumetric solution (4.11) until a permanent blue colour is obtained (see note 9.11).