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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Steel — Determination of low carbon contents — Part IV : Coulometric method after combustion

Acier — Dosage du carbone en faibles teneurs — Partie IV : Méthode coulométrique 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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UDC 669.1 : 546.26 : 543.258

Ref. No. ISO/TR 4830/IV-1978 (E)

Descriptors : steels, low carbon steels, chemical analysis, determination of content, carbon, coulometric analysis.

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Printed in Switzerland



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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 coulometric method for the determination of low carbon contents in steels after combustion in a current of oxygen.

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 at a high temperature ($> 1\,250\text{ }^{\circ}\text{C}$; see note below) in a current of pure oxygen, converting the carbon to carbon dioxide. Entrainment of the carbon dioxide by the current of oxygen, and separation from the other volatile oxides. Absorption of the carbon dioxide in a barium hydroxide solution containing barium perchlorate : during the absorption, re-establishment of the initial barium hydroxide concentration by electrolysis of the barium perchlorate, the variation in the pH of the solution being followed by a pH meter. Measurement of the quantity of electricity required for re-establishment of the initial pH, in order to deduce the quantity of barium hydroxide formed and hence the quantity of carbon dioxide neutralized.

The pH of the absorbing solution must always be equal to or greater than 9,0.

NOTE — Certain types of apparatus are incapable of obtaining the temperature of $1\,250\text{ }^{\circ}\text{C}$ necessary to comply with the requirements of this Technical Report.

4 REAGENTS

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

- 4.1 **Industrial compressed oxygen** in cylinders.
- 4.2 **Platinized asbestos**, with 10 % platinum.
- 4.3 **Soda asbestos**, granulated.
- 4.4 **Hydrogen peroxide fixed on urea**.
- 4.5 **Magnesium perchlorate**, anhydrous, granulated, or **calcium sulphate**, anhydrous, granulated.
- 4.6 **Silica wool**.
- 4.7 **Barium carbonate**.
- 4.8 **Iron**, with a very low carbon content ($< 0,005\text{ }\%$), in the form of chips.

4.9 Tin, free from carbon, in thin long chips.

4.10 Barium perchlorate, 100 g/l solution.

5 APPARATUS (See figure 1)

5.1 For purification of the oxygen

5.1.1 Furnace, allowing a temperature of 625 °C to be obtained inside the tube (5.1.2) over a length of 250 mm.

5.1.2 Tube, with an inside diameter 20 mm and a length of 500 mm, the ends of which are fitted with standard male joints allowing connections to be made without contact of the hot gas with organic materials that are liable to volatilize.

This tube is filled with platinized asbestos (4.2).

5.1.3 Condenser, allowing the hot gas coming from the furnace (5.1.1) to be cooled to ambient temperature.

5.1.4 Gas conditioning column containing a layer of magnesium perchlorate or calcium sulphate (4.5), then a layer of soda asbestos (4.3), and finally a layer of magnesium perchlorate or calcium sulphate (4.5).

Between layers of reagents and at the entry and exit of the column, the reagents are retained by a layer of silica wool (4.6).

5.1.5 Flowmeter, allowing a flow of oxygen from 12 to 180 l/h to be measured (free from liquid).

5.2 For combustion of the test portion

5.2.1 System for injection of the purified oxygen into the entry to the laboratory tube (5.2.3).

This system should allow the circulation pumps to carry out the selection of the gases necessary for the measurement, without the pressure inside the tube differing from the ambient atmospheric pressure. A waiting chamber should be provided, allowing the air contained in the boat and cartridge (5.2.4) to be eliminated. (See note 9.3.)

5.2.2 Furnace, allowing a temperature of 1 400 °C (see note in clause 3) to be obtained inside and along the axis of the laboratory tube (5.2.3) over a length of 180 mm.

The temperature of this furnace may be stabilized either with the aid of a regulator or by its thermal inertia. Its temperature is checked with the aid of a platinum/platinum-rhodium thermocouple.

5.2.3 Laboratory tube arranged horizontally.

The length (720 mm) is such that its ends remain at a temperature acceptable for the connections with the measuring device (5.4). The composition of the refractory material should allow continuous heating at 1 400 °C (see note in clause 3).

A laboratory tube formed of two cylinders (27 mm/33 mm diameter by 450 mm, and 3 mm/11 mm diameter by 250 mm) connected by a cone is preferable to a tube consisting of a single cylinder.

5.2.4 Test portion support (cartridge, refractory filter); this is a cylindrical tube plugged at one end (14 mm/16 mm diameter by 50 mm). Made with a paste rich in aluminium oxide (85 to 90 %) with a minimum alkaline oxide or alkaline earth content, it should resist thermal shock and molten metallic oxides.

The porosity of the cartridge is defined by the gas flow across the entire surface at a given pressure : 4 to 5 litres of gas per minute at a pressure of 2 450 Pa*. (See figure 2.)

The cartridge rests on a boat, made of refractory material, with a wide flat base, the usual dimensions of which are : length 80 to 100 mm, height 8 to 9 mm, width 15 to 16 mm.

When a boat is used without a cartridge it is recommended that a lid be used to protect the laboratory tube (5.2.3) from projected oxides, and that a silica wool plug be used at the exit of the tube to retain oxide powder.

Boats and cartridges should be calcined at 1 000 °C in an oxidizing atmosphere before they are used.

* 2 450 Pa $\approx$ 250 mmH₂O