SLOVENSKI STANDARD
SIST EN 10036:1996

01-avgust-1996

Kemijska analiza železnih materialov - Ugotavljanje celotnega ogljika v jeklih in železu - Gravimetrična metoda po sežigu v toku kisika

Chemical analysis of ferrous materials - Determination of total carbon in steels and irons - Gravimetric method after combustion in a stream of oxygen

Chemische Analyse von Eisen- und Stahlwerkstoffen - Ermittlung des Gesamtkohlenstoffgehalts von Stahl und Roheisen - Gewichtsanalytische Ermittlung nach Verbrennung im Sauerstoffstrom

Analyse chimique des matériaux sidérurgiques - Dosage du carbone total dans les aciers et les fontes - Méthode gravimétrique apres combustion dans un courant d'oxygene

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English version

Chemical analysis of ferrous materials
Determination of total carbon in steels and irons
Gravimetric method after combustion in a stream of oxygen

Analyse chimique des matériaux sidérurgiques - Dosage du carbone total dans les aciers et les fontes Méthode gravimétrique après combustion dans un courant d'oxygène

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat : Rue Bréderode 2, B-1000 Brussels

Brief History

This European Standard takes over the content of EURONORM 36-83 "Chemical analysis of ferrous materials - Determination of total carbon in steels and irons - Gravimetric method after combustion in a stream of oxygen", prepared by ECISS/TC 20 "Methods of chemical analysis"; the Secretariat of which is allocated to the Dansk Standardiseringsrad (DS).

It has been submitted to the CEN Formal Vote following the decision of the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization on 1987-11-24/25.

It has been adopted and ratified by CEN BT on 1988-11-05.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard :

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Note in clauses 1 and 9 EURONORM shall read EUROPEAN STANDARD.

Chemical analysis of ferrous materials
Determination of total carbon in steels and irons
Gravimetric method after combustion in a stream of oxygen

CONTENTS

1 SCOPE AND FIELD OF APPLICATION

This EURONORM specifies a gravimetric method for the determination of total carbon in steels and irons after combustion in a stream of oxygen.

The method is applicable to carbon contents equal to or greater then 0.1% (*m/m*).

2 REFERENCE

EURONORM 18-79 Selection and preparation of samples and test pieces for steel and iron and steel products.

3 PRINCIPLE

Combustion of a test portion in a stream of oxygen in a high temperature furnace (1 200-1 400°C), with the addition of a fluxing agent to assist combustion.

Absorption of the evolved carbon dioxide in soda asbestos contained in a weighed absorption bulb.

Calculation of the carbon content from the increase in mass of the absorption bulb.

4 REAGENTS

During the analysis, use only reagents of recognized analytical quality.

4.1 Oxygen, cylinder oxygen, of at least 99% purity.

4.2 Magnesium perchlorate (Anhydrone) as a drying agent.[1]

4.3 Fluxes: lead oxide, copper (II) oxide, tin filings; high purity iron chips of certified very low carbon content.

4.4 Copper (II) oxide, granulated.

4.5 Soda asbestos, in granules of approximately 2 mm diameter. Avoid contact with air. Handling of this reagent should take place under a fume hood with sufficient suction to avoid inhalation of asbestos dust by the operator.

4.6 Sulphuric acid, ρ 1.84 g/ml approximately, (18 mol/l approximately).

4.7 Chromic-sulphuric acid; Saturate a sulphuric acid solution, ρ 1.23 g/ml approximately (4 mol/l approximately) with chromic acid anhydride (CrO_3). The solution remains effective only as long as it retains its red colour. Sulphuric acid solution ρ 1.23 approximately, is prepared by diluting sulphuric acid ρ = 1.84 g/ml, 1 + 4 with water.

[1] Contact with organic substances should be avoided because of the potential hazard of an explosion.

5 APPARATUS

The apparatus consists of three parts: The first part includes the oxygen cylinder and the oxygen-purifier, the second part comprises the furnace and combustion tube and the third part comprises the vessels for purification and absorption of the carbon dioxide produced by combustion of the carbon in the analysis sample.

The three parts, which are interconnected by tubing and hermetically sealed stoppers, are depicted in Fig. 1.

Fig. 1

A Oxygen cylinder (4.1) with a pressure regulating valve.

B Mercury valve.

C Furnace with non-porous porcelain tube, heated to 400°C, containing granules of copper (II) oxide (4.4).

D U tube containing magnesium perchlorate (4.2) and soda asbestos (4.5) separated by glass wool for drying and purification of oxygen (diameter of the tubes is 25 mm, height approximately 100 mm with connection). The U tube must be packed in such a manner that the oxygen passes first through the soda asbestos and afterwards through the magnesium perchlorate.

E Electric resistance furnace, capable of raising the temperature of the combustion tube to 1 400°C.

F Thermocouple for temperature measurement. The point of the thermocouple protected by its own sheath is placed near the external surface of the combustion tube.
It is recommended that the difference between the external and the internal temperature of the tube should be checked.

G Refractory combustion tube which is not porous at the test temperature, having an internal diameter of 20 to 30 mm and a length of at least 600 mm so that the ends of the tube remain cool during the combustion.
The tube should extend beyond each end of the furnace by not less than 150 mm.

H Refractory boat with eyelet (length 80 to 100 mm, width 12 to 14 mm, height 8 to 9 mm) calcined in a current of oxygen under the working conditions.

I,I' Small, porous refractory cylinders for protecting the end stoppers of the combustion tube against the heat. For subsequently cooling the tube, its ends can be enclosed by water-circulating lead or copper coils.

L Glass bulb tube filled with quartz wool to trap the dust carried along by the gas flow.

M Wash bottle containing the chromic-sulphuric acid (4.7).

M' Drier containing the magnesium perchlorate (4.2).

N Contact furnace with non-porous porcelain tube, heated to 400°C containing copper (II) oxide granules (4.4), in order to ensure complete oxidation to carbon dioxide.

O,O' Weighed absorption bulbs with turn off taps, for the absorption of the carbon dioxide, containing soda asbestos (4.5) and a layer (10-20 mm) of magnesium perchlorate (4.2) for retaining the water formed during the absorption of the carbon dioxide. The total mass of each absorption bulb, ready for use, should not exceed 100 g (see Fig. 2).

O'' Unweighed absorption bulb, facing the opposite way to O and O'.

P Bottle of sulphuric acid (4.6) for the protection of the absorption bulbs against the outside atmosphere and also for monitoring the rate of the oxygen flow.

6 SAMPLING

Sampling shall be carried out in accordance with EURONORM 18.- 79

The sample as prepared for analysis shall be in the form of very short chips having a thickness not exceeding a few tenths of a millimetre.

For white cast irons the analysis sample must be fine enough to pass through a sieve of 0.18 mm mesh size.

For grey cast irons the test piece shall be cut into plates 2 mm thick and broken up with pliers to avoid the possibility of errors arising from the segregation of graphite.

7 PROCEDURE

After checking the leak-tightness of the apparatus and having attained a temperature of 1 200-1 400°C in the combustion zone of the furnace (use highest temperature for the more highly alloyed steels), allow the oxygen (4.1) to flow into the apparatus for 10 to 15 minutes, at a rate of 300 to 500 ml per minute depending on the diameter of the tube used. Next disconnect the absorption bulbs and close the taps to avoid contact with ambient air. Weigh them after 10 minutes at ambient temperature and then reconnect them to the apparatus.

7.1 Test portion

Weigh the masses (m) given below as a function of presumed carbon content to the nearest 0.001 g remaining within the tolerance of ±10% of the specified mass:

(a) content from 0.1% to 1% m approximately 2 g;
(b) content above 1%, m approximately 1 g.

Transfer the weighed test portion to the calcined refractory boat. Add to the sample 1 to 2 g of carefully weighed flux (4.3).

7.2 Blank test

Determine the blank value of the apparatus, boat and flux by combustion of the boat containing the prescribed quantity of flux (4.3) exactly in accordance with the conditions specified for the analysis (7.3) and record the increase in mass (mass m_0) of the absorption bulb.

Where there is any doubt about the purity of the boat, this should be verified by the combustion in the boat of the exact amount of flux used in the analysis, together with 1 g of high purity iron of certified very low carbon content (4.3). In this case, the blank value (mass m_0) is obtained after correcting for the certified carbon content of the high purity iron.

The leak-tightness of the apparatus and the effectiveness of the purification system can be checked without submitting materials and fluxes to combustion.

7.3 Determination

Cut off the oxygen flow, then open the combustion tube at the end where the oxygen enters and introduce the boat with the sample into the middle of the heated zone of the tube by means of a rigid nickel rod. Close the tube immediately and after one minute allow an oxygen flow to pass through at a rate of 300 to 500 ml per minute depending on the diameter of the tube used.

Combustion should be complete after approximately 2 minutes but continue the oxygen flow for a further 15 to 20 minutes to ensure the complete expulsion of the carbon dioxide from the combustion tube and the purification bulbs.

Cut off the oxygen flow, close the taps of the weighed absorption bulbs and remove the refractory boat from the combustion tube. Check whether the combustion has been properly carried out by examining the fused mass in the boat withdrawn from the furnace. Complete combustion is indicated by a totally fused mass in which no traces of the original sample can be recognized.

Disconnect the absorption bulbs and after 10 minutes weigh them at ambient temperature.

The increase in mass represents the carbon dioxide absorbed (mass m_1).

8 EXPRESSION OF RESULTS

The percentage by mass of carbon (C) is given by the expression:

$$C\% = \frac{(m_1 - m_0) \times 27.29}{m}$$

where

m_1 is the mass of carbon dioxide from the test sample, in g;

m_0 is the mass of carbon dioxide from the blank test, in g;

m is the mass of the test portion, in g;

27.29 is the conversion factor of carbon dioxide to carbon, multiplied by 100.

9 TEST REPORT

The test report shall contain the following particulars:

(a) the method of analysis used by reference to this EURONORM;

(b) the results obtained, as well as the form in which they are expressed;

(c) any particular details which may have been noted during the determination;

(d) any operations not specified in this EURONORM or any optional operations which could have had an influence on the result.

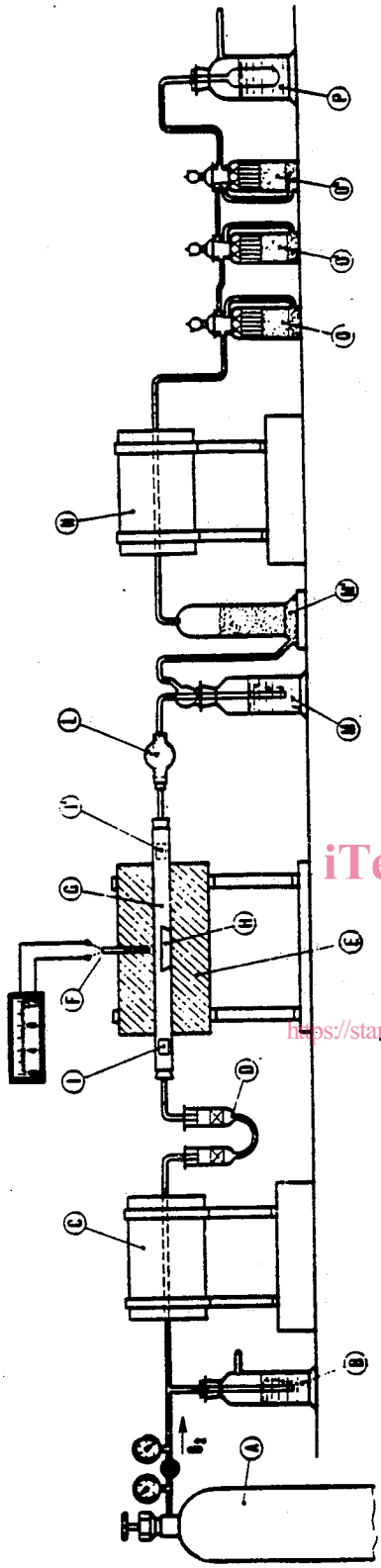

Fig. 1
Apparatus for the gravimetric determination of the total carbon after combustion in a stream of oxygen

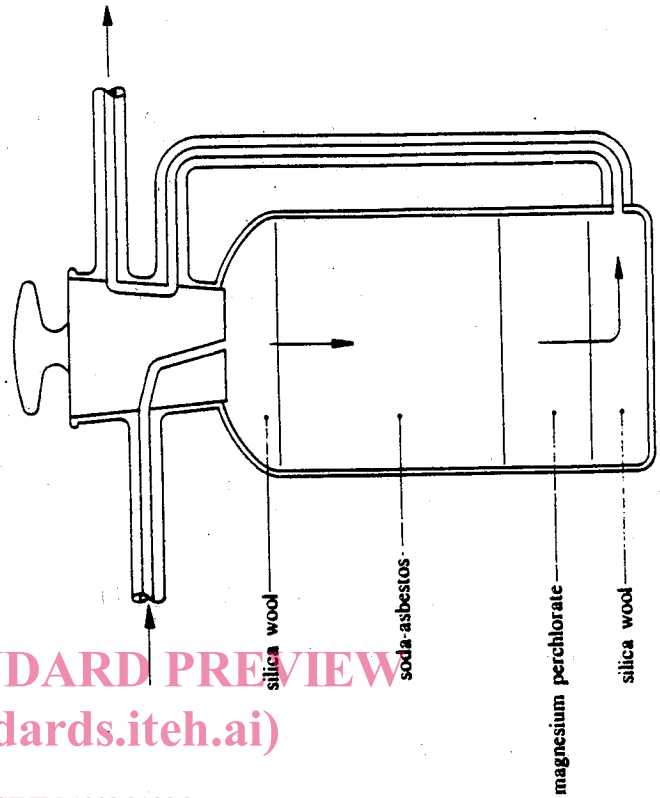

Fig. 2
Absorption bulb