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**Kemiska analysmetoder för järnmetaller —
Bestämning av bor i stål — Spektrofotometrisk metod**

Orientering

Denna standard utgörs av den engelska versionen av Euro-
pastandarden EN 10 200:1991. Motsvarande ISO-standard
är ISO 10 153:1991

**Chemical analysis of ferrous materials —
Determination of boron in steel —
Spectrophotometric method**

Introduction

This Swedish standard consists of the English version of the
European standard EN 10 200:1991. The corresponding
ISO-standard is ISO 10 153:1991

UDK 669.14:543.42:546.27

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English version

Chemical analysis of ferrous materials —
Determination of boron in steel —
Spectrophotometric method

Analyse chimique des matériaux
sidérurgiques —
Dosage du bore dans les aciers —
Méthode spectrophotométrique

Chemische Analyse des Werkstoffes in der
Eisen- und Stahlindustrie —
Ermittlung des Borgehaltes in Stahl —
Spektrophotometrisches Verfahren

This European Standard was approved by CEN on 1991-12-20. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard was prepared by Technical Committee, ECISS/TC 20, the Secretariat of which is held by Dansk Standardiseringsraad. The development of the method was carried out by a working group under French convenership.

The results of the inter-laboratory tests indicated that the lower limit of application of the method should be 0,0004 % (*m/m*) boron, based on a relative deviation not exceeding 10 % within 66 % confidence limits. However, further work has shown that the method may be used for lower boron contents where a higher relative deviation is acceptable or where the reported result is based on the mean of replicate determinations.

The Coordinating Commission (COCOR) of ECISS agreed on 1991-05-27/28 to submit this draft European Standard to the CEN formal vote.

This European Standard EN 10200 was approved by CEN on 1991-12-20.

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

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

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1 Scope

This European Standard specifies a spectrophotometric method for the determination of boron in steel.

The method is applicable to non-alloyed and alloyed steels with boron contents of 0,0004 to 0,0120 % (*m/m*).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- EURONORM 18 : 1979 - Selection and preparation of samples and test pieces for steel and iron and steel products.
- ISO 5725 : 1986 - Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.

3 Principle

Dissolution of the test portion in hydrochloric and nitric acids.

Decomposition of boron compounds (nitrides etc.) with orthophosphoric and sulfuric acids at 290 °C.

Spectrophotometric measurement at a wavelength of 543 nm of the complex formed between boric acid and curcumin in buffered acetic medium.

4 Reagents

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

4.1 Pure iron, free of boron or of known low boron content.

4.2 Crystalline sodium hypophosphite monohydrate ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$).

4.3 Hydrochloric acid, ρ approximately 1,19 g/ml.

4.4 Nitric acid, ρ approximately 1,40 g/ml.

4.5 Sulfuric acid, ρ approximately 1,84 g/ml.

4.6 Orthophosphoric acid, ρ approximately 1,71 g/ml.

4.7 Acetic acid free of aldehyde, ρ approximately 1,05 g/ml.

Test for absence of aldehyde. Place 20 ml of the acetic acid (4.7) to be tested and 1 ml of a 1 g/l solution of potassium permanganate (KMnO_4) into a 50 ml beaker. In the absence of aldehyde, the initial violet colour of the potassium permanganate will persist; otherwise the solution will become brown, easily identifiable after 15 min.

4.8 Mixture of acetic and sulfuric acids

Add in small portions, cooling under water and swirling, a volume of sulfuric acid (4.5) to an equal volume of acetic acid (4.7).

4.9 Acetic buffer solution

Dissolve 225 g of ammonium acetate in 400 ml of water. Add 300 ml of acetic acid (4.7). Filter the solution obtained into a 1000 ml polyethylene volumetric flask. Dilute to the mark with water and mix.

4.10 Sodium fluoride, 40 g/l solution.

4.11 Boron, standard solution corresponding to 0,1 mg of boron per ml.

Dissolve 0,2858 g of boric acid in water in a 500 ml volumetric flask. Dilute to the mark with water and mix. Store in a polyethylene flask.

4.12 Boron, standard solution corresponding to 0,002 mg of boron per ml.

Take exactly 20 ml of the boron standard solution (4.11). Place in a 1000 ml volumetric flask. Dilute to the mark with water and mix. Store in a polyethylene flask.

4.13 Curcumin, 1,25 g/l acetic solution (prepared immediately before use).

Place 0,125 g of curcumin in 60 ml of acetic acid (4.7) into a polyethylene or quartz vessel. Heat on a water bath at 40 °C and stir using a magnetic stirrer. After dissolution, cool and transfer to a 100 ml polyethylene volumetric flask. Dilute to the mark with acetic acid (4.7) and mix.

5 Apparatus

Glassware containing boron shall not be used and shall be replaced by polyethylene and quartz equipment rinsed with acetic acid (4.7), then with water and finally dried.

5.1 100 ml quartz beakers with quartz covers (outside dimensions ϕ 51 mm and height 70 mm).

5.2 Aluminium alloy block with holes designed to allow the location of the 100 ml quartz beakers and heated by surface contact with a hotplate. (Diagrams of such blocks are shown in figures 1 and 2. The dimensions of the holes should be adapted to the dimensions of the quartz beakers available).

5.3 50 ml polyethylene volumetric flasks.

5.4 100 ml polyethylene flasks.

5.5 Spectrophotometer suitable for measuring the absorbance of the solution at a wavelength of 543 nm with 20 mm optical cells.

6 Sampling

Sampling shall be carried out in accordance with EURONORM 18.

The chips used shall be smaller than 1 mm.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, a test portion (m) as indicated below:

$m = 1 \text{ g} \pm 0,05 \text{ g}$ for boron contents less than 0,006 %;

$m = 0,5 \text{ g} \pm 0,03 \text{ g}$ for boron contents from 0,006 % to 0,012 %.

NOTE. For grades of steel with nickel and cobalt total content greater than 30 % do not use a test portion larger than 0,5 g.

7.2 Blank test

With each analytical run, carry out an analysis on a portion of pure iron (4.1) of mass corresponding to that of the test portion (see 7.1), in parallel with the analysis of the test portion, using identical reagents, conditions, analytical procedure and dilutions throughout. The absorbance of the blank test solution (A_b) and that of the blank compensating solution (A_{bc}) are thus obtained.

7.3 Determination

7.3.1 Preparation of the test solution

Place the test portion (7.1) in a 100 ml quartz beaker with 10 ml of hydrochloric acid (4.3) and 5 ml of nitric acid (4.4). Put the quartz cover on the beaker and, most important, leave at ambient temperature in order to avoid possible loss of boron at higher temperatures.

Wait until dissolution is complete or until the end of the effervescence for samples which are difficult to dissolve. Then add carefully 10 ml of orthophosphoric acid (4.6) and 5 ml of sulfuric acid (4.5).

Heat to copious white sulfuric fumes, swirling from time to time in order to recover any particles attached to the sides of the beaker. Place the reaction beaker in a hole in the aluminium alloy block (5.2) and place this on a heat source which allows a temperature of 290 °C to be obtained in the solutions. Maintain the heating for 30 min, taking care to replace the quartz cover after the appearance of white fumes.

NOTE. The temperature of 290 °C \pm 5 °C is obtained by calibration of the heat source by means of a thermometer, graduated from 0 to 350 °C, immersed in a beaker identical to those for the test and containing the same amounts of dissolution reagents.

Remove the beaker from the heat source and allow to cool. Take up the syrupy solution in 30 ml water. Warm and stir. Remove from the heat source. Then add carefully 5 ml of hydrochloric acid (4.3) and bring to the boil. Add 3 g of sodium hypophosphite (4.2). Allow to boil gently for 15 min.

Remove from the heat source and allow to cool. Transfer quantitatively to 50 ml polyethylene volumetric flask. Dilute to the mark with water and mix.

7.3.2 Formation of the coloured complex

Take 1,0 ml of test solution obtained in accordance with 7.3.1 and place in a 100 ml polyethylene flask previously cleaned and dried.

Add to the flask, swirling in a circular movement to avoid contact with the stopper, the following quantities of reagents:

- 6,0 ml of the mixture of acetic and sulfuric acids (4.8), avoiding contact of the pipette with the neck and sides of the flask; mix;
- 6,0 ml of the curcumin acetic solution (4.13). Stopper the flask and mix;

Allow to stand for 2 h 30 min for complete development of the colour;

- 1,0 ml of orthophosphoric acid (4.6) to stabilize the colour. Shake and allow to stand for 30 min;
- 30,0 ml of the acetic buffer solution (4.9). The solution turns orange. Stopper and shake. Allow to stand for exactly 15 min.

7.3.3 Compensating solution

Take 1,0 ml of the test solution (7.3.1) and place in a 100 ml polyethylene flask, previously cleaned and dried. Add 0,2 ml of sodium fluoride solution (4.10) to the bottom of the flask.

Carefully swirl the small volume of solution. Allow to stand for 1 h.

Continue as in 7.3.2 from '.... add to the flask, swirling in a circular movement'.