SVENSK STANDARD
SS-EN 10200:2012

Fastställd/Approved: 2012-11-08
Publicerad/Published: 2012-11-12
Utgåva/Edition: 2
Språk/Language: engelska/English
ICS: 77.040.30; 77.080

Kemiska analysmetoder för järn och stål – Bestämning av bor i stål – Spektrofotometrisk metod

Chemical analysis of ferrous materials – Determination of boron in steels – Spectrophotometric method

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Denna standard ersätter SS-EN 10200, utgåva 1.

The European Standard EN 10200:2012 has the status of a Swedish Standard. This document contains the official version of EN 10200:2012.

This standard supersedes the Swedish Standard SS-EN 10200, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemiska analysmetoder för metaller, SIS/TK 122.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 10200

October 2012

ICS 77.040.30

Supersedes EN 10200:1991

English Version

Chemical analysis of ferrous materials - Determination of boron in steels - Spectrophotometric method

Analyse chimique des matériaux ferreux - Détermination du bore dans les aciers - Méthode spectrophotométrique

Chemische Analyse von Eisenwerkstoffen - Bestimmung von Bor in Stahl - Spektrophotometrisches Verfahren

This European Standard was approved by CEN on 17 August 2012.

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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 CEN-CENELEC Management Centre has the same status as the official versions.

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Ref. No. EN 10200:2012: E

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Foreword

This document (EN 10200:2012) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2013, and conflicting national standards shall be withdrawn at the latest by April 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights other than those identified above. CEN [and/or] CENELEC shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 10200:1991.

Since the previous edition, no technical changes have been made, but the text has been editorially revised.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The development of the method was carried out by a working group under French convenorship. The results of the inter-laboratory tests have shown that the determination of lower limit application of the method should be 0,000 4 % (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 if a higher relative deviation is acceptable.

1 Scope

This European Standard specifies a spectrophotometric method for the determination of boron in steels. The method is applicable to non-alloyed and alloyed steels with boron contents of 0,000 4 to 0,012 0 % (m/m).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition (ISO 14284)*

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Decomposition of boron compounds (nitrides, etc.) with orthophosphoric and sulphuric 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 recognised analytical grade 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** ($NaH_2PO_2.H_2O$).

4.3 **Hydrochloric acid,** HCl (ρ_{20} = 1,19 g/ml).

4.4 **Nitric acid,** HNO_3 (ρ_{20} = 1,40 g/ml).

4.5 **Sulphuric acid,** H_2SO_4 (ρ_{20}= 1,84 g/ml).

4.6 **Orthophosphoric acid,** H_3PO_4 (ρ_{20}= 1,71g/ml).

4.7 **Acetic acid free of aldehyde,** CH_3COOH (ρ_{20}= 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 sulphuric acids.

Add in small portions whilst cooling under water and swirling, a volume of sulphuric 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 1 000 ml polyethylene one-mark volumetric flask. Dilute to the mark with water and mix.

4.10 Sodium fluoride, 40 g/l solution.

4.11 Boron, 100 mg/l standard solution.

Dissolve 0,285 8 g of boric acid in water in a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Store this solution in a polyethylene flask.

1 ml of this solution contains 0,1 mg of boron.

4.12 Boron, 2 mg/l standard solution.

Transfer 20 ml of the boron standard solution (4.11) into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Store this solution in a polyethylene flask.

1 ml of this solution contains 2 µg of boron.

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

Dissolve 0,125 g of curcumin in 60 ml of acetic acid (4.7) in a polyethylene or quartz vessel. Heat at 40 °C in a water bath and stir with a magnetic stirrer. After dissolution, cool and transfer into a 100 ml polyethylene one-mark 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 vessel rinsed with acetic acid (4.7), then with water and finally dried.

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

5.2 Aluminium alloy blocks, allowing a temperature of 290 °C to be achieved and maintained in the test solutions throughout the fuming period.

The block has holes designed to allow the location of the 100 ml quartz beakers and is heated by surface contact with a hotplate.

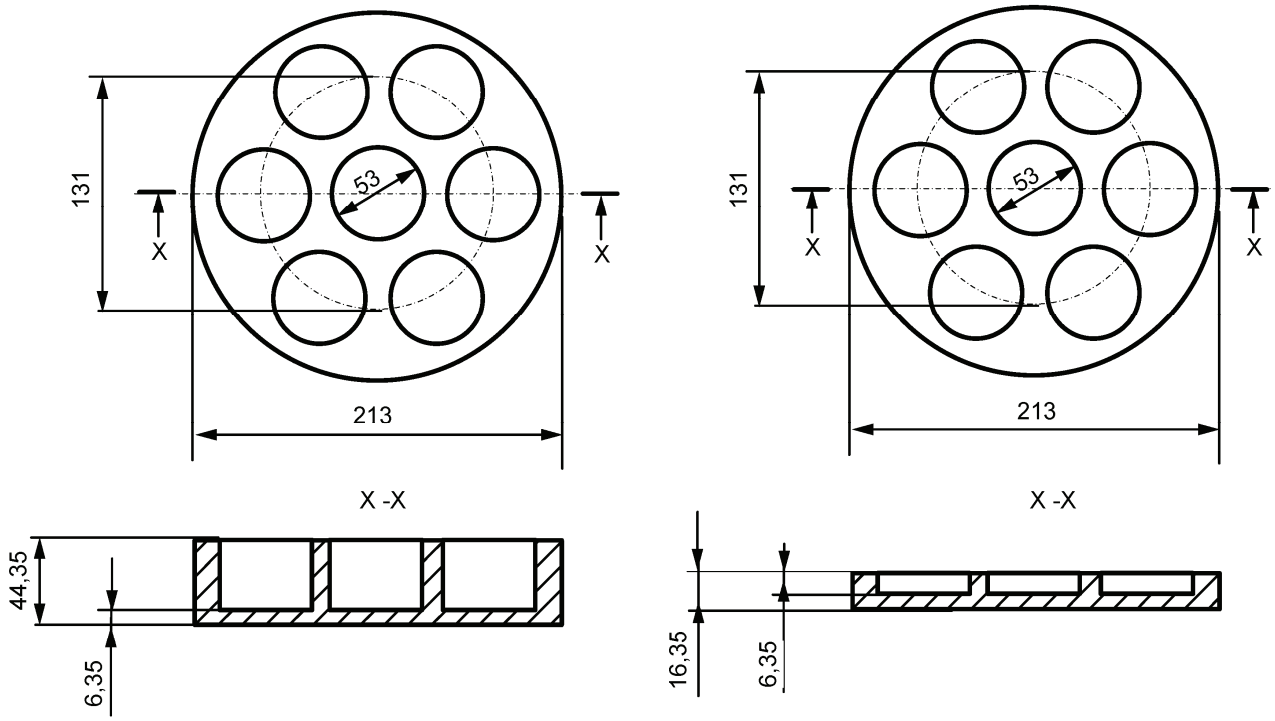
NOTE Diagrams of these blocks are shown in Figures 1 and 2. The dimensions of the holes should be adapted to the dimensions of the quartz beakers available.

5.3 50 ml polyethylene one-mark volumetric flasks.

5.4 100 ml polyethylene flasks.

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

Dimensions in millimetres



NOTE Adapt the dimensions of the holes according to the dimensions of the beakers available.

Figure 1 — Example of circular aluminium alloy heating block