

**Stål – Bestämning av borhalt – Spektrofotometrisk
metod med curcumin efter destillering**
(ISO 13900:1997)

**Steel – Determination of boron content – Curcumin
spectrophotometric method after distillation**
(ISO 13900:1997)

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English version

**Steel - Determination of boron content - Curcumin
spectrophotometric method after distillation (ISO 13900:1997)**

Aciers - Dosage du bore - Méthode spectrophotométrique à
la curcumine après distillation (ISO 13900:1997)

Stahl - Bestimmung des Borgehaltes - Curcumin-Verfahren
- Fotometrische Bestimmung nach Destillation (ISO
13900:1997)

This European Standard was approved by CEN on 26 May 2002.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

The text of the International Standard from Technical Committee ISO/TC 17 "Steel" of the International Organization for Standardization (ISO) has been taken over as a European Standard by Technical Committee ECISS/TC 20 "Methods of chemical analysis of ferrous products", 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of an identical text or by endorsement, at the latest by January 2003, and conflicting national standards shall be withdrawn at the latest by January 2003.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 13900:1997 has been approved by CEN as a European Standard without any modifications.

NOTE Normative references to International Standards are listed in annex ZA (normative).

Steel — Determination of boron content — Curcumin spectrophotometric method after distillation

1 Scope

This International Standard specifies a method for the determination of the boron content in steel using a curcumin spectrophotometric method after distillation.

The method is applicable to a boron content of between 0,000 05 % (*m/m*) and 0,001 0 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:—¹⁾, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*.

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*.

ISO 14284 :1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*.

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids.

1) To be published. (Revision of ISO 1042:1983)

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

Distillation of the solution after the addition of methanol and collection of methylborate in a receiver containing sodium hydroxide solution.

Evaporation of the solution to dryness. Formation of a coloured complex between orthoboric acid and curcumin in a methanol medium.

Spectrophotometric measurement at a wavelength of about 550 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

4.1 Pure iron, free from boron or with a known residual amount of boron.

4.2 Hydrochloric acid, ρ about 1,19 g/ml.

4.3 Nitric acid, ρ about 1,40 g/ml.

4.4 Sulfuric acid, ρ about 1,84 g/ml.

4.5 Orthophosphoric acid, ρ about 1,71 g/ml, boron content less than 0,02 $\mu\text{g/ml}$.

4.6 Acetic acid, free from aldehyde, ρ about 1,05 g/ml.

In order to test the acetic acid for the presence of aldehyde, pour 20 ml of acetic acid (ρ about 1,05 g/ml) and 1 ml of potassium permanganate solution (1 g/l) into a 50 ml beaker. In the absence of aldehyde, the initial violet colour of potassium permanganate will persist; otherwise the solution will become an easily identifiable brown colour after 15 min.

4.7 Acid mixture A

Add one volume of hydrochloric acid (4.2) to two volumes of acetic acid (4.6).

4.8 Acid mixture B

While cooling under water and swirling, add in small portions a volume of sulfuric acid (4.4) to an equal volume of acetic acid (4.6).

4.9 Sodium hydroxide, 8 g/l solution.

4.10 Methanol, minimum assay 99,6 % (V/V).

4.11 Boron, standard solution.

4.11.1 Stock solution, corresponding to 0,10 g of boron per litre.

Weigh, to the nearest 0,000 1 g, 0,286 0 g of orthoboric acid (H_3BO_3). Place in a 250 ml beaker and dissolve in about 200 ml of water. Transfer the solution quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix. Store in a polypropylene bottle (5.2).

1 ml of this stock solution contains 0,10 mg of boron.

4.11.2 Standard solution, corresponding to 0,001 g of boron per litre.

Transfer 1,0 ml of the stock solution (4.11.1) to a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix. Store in a polypropylene bottle (5.2).

Prepare this solution immediately before use.

1 ml of this standard solution contains 1 µg of boron.

4.12 Curcumin, acetic acid solution, 1,25 g/l.

Weigh 0,125 g of curcumin, $[\text{CH}_3\text{O}(\text{OH})\text{C}_6\text{H}_3\text{CH}=\text{CHCO}]_2\text{CH}_2$, in a polypropylene or quartz vessel, add 60 ml of acetic acid (4.6) and mix. Heat in a water bath at 40 °C and stir using a magnetic stirrer. After dissolution, cool and transfer to a 100 ml polypropylene volumetric flask. Dilute to the mark with acetic acid and mix.

4.13 Carrier gas, nitrogen, argon or dry air.

5 Apparatus

Glassware, polypropylene or quartz equipment rinsed with acetic acid (4.6) then with water and finally dried. All volumetric glassware shall be grade A, in accordance with ISO 385-1, ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus and the following shall be used.

5.1 Volumetric flask, of capacity 100 ml.

5.2 Polypropylene bottles, of capacities 100 ml and 500 ml.

5.3 Distillation apparatus (see annex A), comprising the following equipment.

- a) Distillation flask, of capacity 200 ml, made of quartz.
- b) Reservoir, made of quartz.
- c) Reflux condenser, made of quartz.
- d) Receiver, 100 ml quartz beaker marked to indicate a volume of 35 ml.
- e) Magnetic stirrer, with rotor covered by polytetrafluoroethene (PTFE).

Suitable forms of apparatus are illustrated in annex A.

5.4 Platinum dish or PTFE beaker, of capacity 100 ml.

5.5 Heater, for methanol evaporation.

6 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,000 1 g, about 0,50 g of the test sample.

7.2 Blank test and determination of boron in the pure iron (4.1)

7.2.1 Determination of boron in the pure iron (4.1)

Prepare two distillation flasks [5.3 a)]. Add 0,250 g of the pure iron (4.1) to one distillation flask and 0,500 g to the other.

Treat both as specified in 7.3.1, 7.3.2, 7.3.3 and 7.3.4.

Convert the absorbance readings obtained into micrograms of boron by means of the calibration graph (7.4).

The mass (m_3), expressed in micrograms, of boron in 0,500 g of the pure iron (4.1) is given by:

$$m_3 = 2 \times (m_6 - m_5)$$

where

m_6 is the mass, expressed in micrograms, of boron in the 0,500 g test;

m_5 is the mass, expressed in micrograms, of boron in the 0,250 g test.

7.2.2 Blank test and blank value

In parallel with the determination of the content and following the same procedure, carry out a blank test using 0,500 g of the pure iron (4.1) and the same quantities of all the reagents.

Obtain the absorbance of the blank test and convert it to micrograms of boron by means of the calibration graph (7.4).

The blank value (m_1) is obtained by subtracting the mass of boron (m_3) in the pure iron used (4.1) from the mass of boron in the blank test (m_4).

Blank value, $m_1 = m_4 - m_3$

7.3 Determination

7.3.1 Preparation of the test solution

Place the test portion (7.1) in a distillation flask [5.3 a)]. Add 10 ml of hydrochloric acid (4.2) and 5 ml of nitric acid (4.3). Maintain the solution at the ambient temperature (see note 1).

NOTE 1 Maintaining the ambient temperature is most important, in order to avoid possible loss of boron at higher temperatures.

Wait until dissolution is complete, or until the end of effervescence for samples which are difficult to dissolve. Then, carefully add 10 ml of orthophosphoric acid (4.5) and 5 ml of sulfuric acid (4.4).

Heat until white sulfuric acid fumes are generated, swirling from time to time in order to recover any particles attached to the side of the flask. Place the flask on a heat source permitting a temperature of 290 °C (see note 2) to be obtained in the solution. Maintain heating for 30 min.

NOTE 2 The temperature of (290 ± 5) °C is obtained by calibrating the heat source with a thermometer, graduated from 0 °C to 350 °C, immersed in a test flask containing the same amounts of dissolution reagents.