

INTERNATIONAL STANDARD

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Steel — Determination of boron content — Curcumin spectrophotometric method

*Aciers — Dosage du bore — Méthode spectrophotométrique
à la curcumine*



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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10153 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This second edition cancels and replaces the first edition (ISO 10153:1991), of which it constitutes a technical revision.

Annexes A to D of this international Standard are for information only.

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Steel — Determination of boron content — Curcumin spectrophotometric method

1 Scope

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

The method is applicable to a boron content of between 0,000 1 % (*m/m*) and 0,000 5 % (*m/m*) only in unalloyed steel, and between 0,000 5 % (*m/m*) and 0,012 % (*m/m*) in steel.

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 :1997, *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.

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

Formation of a coloured complex between orthoboric acid and curcumin in a buffered acetic medium.

Spectrophotometric measurement at a wavelength of about 543 nm.

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

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and having a very low boron content 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 Sodium hypophosphite monohydrate, $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$

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

4.4 Nitric acid, ρ about 1,40 g/ml.

4.5 Sulfuric acid, ρ about 1,84 g/ml.

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

4.7 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.

4.8 Mixture of acetic and sulfuric acids.

While cooling under water and swirling, add in small portions 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 1 000 ml polypropylene volumetric flask. Dilute to the mark with water and mix.

4.10 Sodium fluoride, 40 g/l solution.

Store in a polypropylene bottle.

4.11 Boron, standard solution.

4.11.1 Stock solution, corresponding to 0,10 g of B 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.

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

4.11.2 Standard solution, corresponding to 0,002 g of B per litre.

Transfer 20,0 ml of the stock solution (4.11.1) to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix. Store in a polypropylene bottle.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 2 μg of B.

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 (4.7) 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.

5 Apparatus

Glassware containing boron shall not be used and shall be replaced by 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 Quartz beakers with quartz covers, of capacity 100 ml, external dimensions: 51 mm diameter and 70 mm height.

5.2 Polypropylene volumetric flasks, of capacities 50 ml and 100 ml.

5.3 Aluminium alloy block, with holes designed to allow the positioning of the 100 ml quartz beakers, heated by surface contact with a hot plate. Diagrams of such blocks are shown in annex A.

NOTE 1 The dimensions of the holes should be adapted to the dimensions of the quartz beakers available.

5.4 Spectrophotometer, suitable for measuring the absorbance of the solution at a wavelength of 543 nm with 2 cm optical cells.

6 Sampling

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

The size of the chip used shall be smaller than 1 mm.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,000 2 g, a test portion of mass m , as a function of the expected boron content as follows:

- a) content from 0,000 1 % (m/m) to 0,006 % (m/m), m is approximately 1,00 g;
- b) content from 0,006 % (m/m) to 0,012 % (m/m), m is approximately 0,50 g.

For grades of steel with nickel content plus cobalt content greater than 30 % (m/m), use a test portion of approximately 0,50 g.

7.2 Blank test

Carry out an analysis on a portion of pure iron (4.1), corresponding to the mass of test portion used (see 7.1), in parallel with the test portion analysis, 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 (5.1). Add 10 ml of hydrochloric acid (4.3) and 5 ml of nitric acid (4.4), cover the beaker with a quartz cover (5.1) and maintain the solution at the ambient temperature (see note 2).

NOTE 2 Maintaining the ambient temperature is most important, in order to avoid possible loss of boron at higher temperatures.

When dissolution is complete, carefully add 10 ml of orthophosphoric acid (4.6) and 5 ml of sulfuric acid (4.5).

Heat until white sulfuric acid fumes are generated. Place the reaction beaker in a hole in the aluminium alloy block (5.3) and place this on a heat source permitting a temperature of 290 °C (see note 3) to be obtained in the solution. Maintain heating for 30 min, taking care to replace the quartz cover after the appearance of white fumes, swirling from time to time in order to recover any particles attached to the side of the beaker.

NOTE 3 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 beaker containing the same amounts of dissolution reagents.

Remove the beaker from the heat source and allow to cool. Add 30 ml of water to the syrupy solution, warm and stir.

CAUTION — It is essential that care is taken after the addition of 30 ml of water, when the solution is warmed. A sudden eruption may occur resulting in loss of the test solution.

Then, carefully add 5 ml of hydrochloric acid (4.3) and bring to the boil. Add 3 g of sodium hypophosphite (4.2) and allow to boil very gently for 15 min.

Remove the beaker from the heat source and allow to cool. Transfer the solution quantitatively to a 50 ml polypropylene volumetric flask (5.2), dilute to the mark with water and mix.

7.3.2 Formation of the coloured complex

7.3.2.1 Take a 1,0 ml aliquot of the test solution (see 7.3.1) and transfer it to a 100 ml polypropylene volumetric flask (5.2) which has previously been cleaned and dried.

7.3.2.2 Add to the flask, while swirling 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. Then mix.
- 6,0 ml of curcumin acetic acid solution (4.12). Stopper the flask and mix. Leave 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 acetic buffer solution (4.9). The solution becomes orange. Stopper, shake and allow to stand for exactly 15 min.

7.3.3 Preparation of the compensating solution

Take a 1,0 ml aliquot of the test solution (see 7.3.1) and transfer it to a 100 ml polypropylene volumetric flask (5.2) which has previously been 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. Leave to stand for 1 h.

Proceed as specified in 7.3.2.2.