INTERNATIONAL STANDARD

ISO 21078-2

First edition
2006-12-01

Determination of boron(III) oxide in refractory products —

Part 2: Acid extraction method for the determination of boron(III) oxide in binder components

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Dosage de l'oxyde de bore(III) dans les produits réfractaires —

Partie 2: Méthode d'extraction acide pour le dosage de l'oxyde de bore(III) dans les composants de liant

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Reference number
ISO 21078-2:2006(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21078-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21078 consists of the following parts, under the general title *Determination of boron(III) oxide in refractory products*:

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— *Part 1: Determination of total boric oxide in oxidic materials for ceramics, glass and glazes*

— *Part 2: Acid extraction method for the determination of boron(III) oxide in binder components*

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Determination of boron(III) oxide in refractory products —

Part 2:
Acid extraction method for the determination of boron(III) oxide in binder components

1 Scope

This part of ISO 21078 specifies procedures of chemical analysis for the determination of boron(III) oxide used as a binder component added to aluminosilicate refractories, using an acid extraction method.

It is applicable for refractories containing less than 1 % (mass fraction) of boron(III) oxide.

2 Normative references

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

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ISO 31-0, *Quantities and units — Part 0: General principles*

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ISO 836, *Terminology for refractories*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 836 and the following apply.

3.1
dry unshaped refractories
dry particles and/or powder of unshaped refractories

4 Preparation of the test sample

4.1 Refractory brick or its raw material

Sampling is carried out in accordance with the contract between the user and producer. A specified quantity taken from a lot of the sample is crushed to pass through a 6,7 mm sieve (see ISO 3310-1) and reduced to about 100 g by riffling or quartering. Then all the reduced sample is ground to pass through a 300 μm sieve (see ISO 3310-1).

4.2 Unshaped refractories

Prepare two approximately 100 g portions of the sample for analysis (both dry and wet), and crush to pass through a 300 μm sieve (see ISO 3310-1), in accordance with the following procedure in 4.3 to 4.5.

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4.3 Dry unshaped refractories

Take one bag or 50 kg of the sample from a lot and reduce to about 100 g as above, and crush to pass through a 300 µm sieve.

4.4 Laboratory sample

The laboratory sample from 4.1 or 4.2 is reduced to approximately 25 g by quartering, and ground to pass through a 106 µm sieve. This is the test sample for analysis. It is transferred into a container [e.g. a flat weighing bottle (50 mm × 30 mm) or sample tube (50 mm × 25 mm)], and dried in a desiccator with silica gel desiccant for a minimum of 8 h.

4.5 Weighing of test portion

The specified quantity of the test portion for chemical analysis shall be weighed to the nearest 0,1 mg using an analytical balance and recorded.

Carry out the extraction process in duplicate.

5 Reagents

Use only reagents of known analytical purity and water conforming to the requirements of grade 2 of ISO 3696 (e.g. double-distilled water).

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The boron and borate ion concentrations in the water and the reagents shall be negligible compared with the lowest concentration to be determined. All solutions are aqueous unless otherwise specified.

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NOTE The reagents below are for the extraction process only. Reagents for the determination of B_2O_3 are listed in the appropriate clauses.

5.1 Dilute hydrochloric acid (1 + 1).

Add 1 volume of hydrochloric acid (concentrated, 35 % by mass) to 1 volume of water, then mix and allow to cool.

5.2 Dilute hydrochloric acid (1 + 50).

Add 1 volume of hydrochloric acid (concentrated, 35 % by mass) to 50 volumes of water.

6 Mass of test portion for extraction

Weigh out 5,0 g of the test sample.

7 Preparation of the test solution

Weigh and transfer the dry sample into a 200 ml plastics beaker. Using a measuring cylinder, add 20 ml of hydrochloric acid solution (1 + 1) (5.1) and 50 ml of warm water, and mix intimately. Allow to stand for approximately 30 min ± 2 min while stirring, to dissolve the soluble binder components.

Filter with a fine-textured filter paper and wash with warm dilute hydrochloric acid (1 + 50) (5.2). Transfer the filtrate and washings into a 250 ml volumetric flask, and dilute to the mark with water. Transfer into a plastics bottle. This solution is referred to as the “test solution” for the determination of boron(III) oxide.

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8 Blank solution

Carry out the procedure given in Clause 7 without the sample. This solution is referred to as the “blank solution”.

9 Classification of determination methods

The determination of boron(III) oxide is carried out using one of the following three methods.

a) Sodium hydroxide titrimetry.

NOTE This method is applied to the sample containing more than 0,5 % (mass fraction) of boron(III) oxide.

b) Inductively coupled plasma atomic emission spectrometry (ICP/AES).

c) Curcumin absorption spectrophotometry (Rothocyanine method).

10 Determination of boron(III) oxide by sodium hydroxide titrimetry

10.1 Principle

A specified volume of test solution is taken and adjusted to pH approximately 5,0. The precipitate containing silicic acid is filtered off. The filtrate's pH is adjusted to 6,3 and D(–)-mannitol is added to the filtrate. Then, the H^+ ions liberated by the mannitol in the solution are titrated with the sodium hydroxide standard solution until the solution's pH becomes 6,3 again.

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10.2 Reagents

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10.2.1 Dilute hydrochloric acid.

Prepare dilute hydrochloric acid (1 + 50) as described in 5.2.

10.2.2 D(–)-Mannitol ($C_6H_{14}O_6$).

10.2.3 Bromocresol purple ($C_{21}H_{16}Br_2O_5S$) solution, 1 g/l.

Dissolve 0,1 g of bromocresol purple in water and dilute to approximately 100 ml. Keep this solution in the dark at a low temperature for a maximum of 7 days.

10.2.4 Boron(III) oxide standard solution, 0,1 mg B_2O_3/ml.

Transfer approximately 0,5 g of boric acid into a 100 ml beaker, spread it as a thin layer and dry for 24 h in a desiccator with silica gel desiccant. Weigh 0,177 6 g of this dry boric acid and transfer into a 200 ml plastics beaker. Dissolve in approximately 100 ml of water and dilute precisely to 1 000 ml in a volumetric flask.

10.2.5 Sodium hydroxide standard volumetric solution.

Transfer 50 g of sodium hydroxide into a polyethylene bottle, add 50 ml of water, dissolve while cooling, and put a seal on it. Keep the sealed bottle for several days, pipette 4,0 ml of the supernatant fraction, and dilute to 2 000 ml in a volumetric flask. Transfer this solution into a polyethylene bottle, using a 25 ml automatic filling burette with soda lime tubes to absorb the carbon dioxide in the air.

Pipette precisely 100 ml of boron(III) oxide standard solution (0,1 mg/ml) into a 200 ml beaker and stir using a magnetic stirrer, put a pH electrode in the solution, and add drops of the sodium hydroxide standard volumetric solution until the pH is 6,5. Remove the electrode, add 10 g of D(–)-mannitol (10.2.2), set the electrode again, and titrate with the sodium hydroxide standard volumetric solution until the pH is 6,8.

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Calculate the equivalent factor, F, i.e. the mass, in g, of boron(III) oxide equivalent to 1 ml of sodium hydroxide standard volumetric solution, using the equation:

$$F = \frac{0{,}01}{V} \tag{1}$$

where

V is the volume, in ml, of titration of sodium hydroxide standard volumetric solution after addition of D(–)-mannitol.

0,01 is the concentration of the NaOH solution, in mol/l.

10.3 Procedure

Pipette precisely 50 ml of both test solutions (see 4.5 and Clause 7) into two 200 ml beakers and add 2 or 3 drops of bromocresol purple (10.2.3) solution as an indicator. Add the standard volumetric sodium hydroxide solution (10.2.5) until the colour of the solution becomes blue, and add drops of dilute hydrochloric acid (1 + 50) (5.2) until the colour becomes yellow.

Boil for 20 min with a watch glass as a cover, wash the watch glass with water and remove it, filter with a closed-pore filter paper, and wash several times with hot water.

NOTE If the yellow-green colour appears during the boiling period, keep the colour yellow by adding drops of dilute hydrochloric acid (1 + 50) (5.2).

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Cool and dilute to 100 ml with water. Stir by using a magnetic stirrer, put a pH electrode in the solution, and add drops of the sodium hydroxide standard volumetric solution (10.2.5) until the pH is 6,3. Remove the electrode, add 10 g of D(–)-mannitol (10.2.2), set the electrode again, and titrate with the sodium hydroxide standard volumetric solution until the pH is 6,3.

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10.4 Blank test

Repeat the process described in 10.3 using the blank solution (see Clause 8).

10.5 Calculation and expression of results

Calculate the mass fraction of boron(III) oxide in the sample, w_B, as a percentage, using Equation (2):

$$w_B = \frac{(V_1 - V_2) \times F}{m} \times \frac{250}{50} \times 100 \tag{2}$$

where

V_1 is the volume, in ml, of titration of sodium hydroxide standard volumetric solution for test solution, after addition of D(–)-mannitol;

V_2 is the volume, in ml, of titration of sodium hydroxide standard volumetric solution for blank solution, after addition of D(–)-mannitol;

F is the equivalent factor, in g, of boron(III) oxide equivalent to 1 ml of sodium hydroxide standard volumetric solution;

m is the mass, in g, of the test portion in Clause 6.

Express the result as the mean of three determinations, rounded off in accordance with ISO 31-0 (see Clause 14).

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11 Determination of boron(III) oxide by inductived coupled plasma/atomic emission spectrometry

11.1 Principle

The sample solution is injected into the argon plasma of an inductively coupled plasma/atomic emission (ICP/AE) spectrometer and the B emission is determined at a wavelength of 249,678 nm, or at another suitable wavelength, against reference solutions.

The wavelength 249,678 nm can suffer from a Fe overlap. If so, 208,959 nm should be used.

11.2 Reagent

11.2.1 Boron(III) oxide standard solution, 0,1 mg B_2O_3/ml.

Transfer approximately 0,5 g of boric acid into a 100 ml beaker, spread it as a thin layer and dry for 24 h in a desiccator with silica gel desiccant. Weigh 0,177 6 g of this dry boric acid and transfer into a 200 ml plastics beaker. Dissolve in approximately 100 ml of water and dilute precisely to 1 000 ml in a volumetric flask.

11.3 Calibration

Transfer a range from 0 ml to 50,0 ml of the boron(III) oxide standard solution (10.2.4) [0 mg to 5 mg of boron(III) oxide] precisely into several 100 ml volumetric flasks. Add 25 ml of the blank solution obtained in Clause 8 to each flask and dilute to the mark with water.

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Run these solutions on the ICP/AE spectrometer, ensuring that the correlation coefficient of the regression is $\geqslant$ 0,999.

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After initial calibration, the instrument can be drift corrected on a zero and top calibration point, unless there has been a significant change in the instrument such as servicing, repair or change in sensitivity. In any event, a mid-point should be made using a different set of volumetric ware from the drift correction standards, preferably from a separate stock boron(III) oxide solution (11.2). This mid-point shall be run as an unknown to validate the calibration, and the difference between its actual and theoretical value must be less than twice the limits set for duplicates in Table 3. If not, the mid-point should be repeated and, if this fails to meet the criteria, the instrument shall be recalibrated.

An example of the preparation of calibration solutions is shown in Table 1. In accordance with the compositions of the samples, and the types and capabilities of instrument used, an appropriate solution series for calibration shall be prepared.

Table 1 — Example of preparation of calibration solutions

Calibration solution	Boron(III) oxide standard solution (10.2.4)	Blank solution	Mass of boron(III) oxide
No.	ml	ml	mg
1	0	25	0,0
2	5	25	0,5
3	10	25	1,0
4	20	25	2,0
5	30	25	3,0
6	40	25	4,0
7	50	25	5,0

To use this approach to calibration, it is essential that line interferences of any of these oxides on each other are checked for, and either found to be absent or, if present, appropriate corrections are applied.

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