INTERNATIONAL STANDARD

ISO 10060

First edition
1993-12-01

Dense, shaped refractory products — Test methods for products containing carbon

Produits réfractaires façonnés denses — Méthodes d'essai pour les produits contenant du carbone

Reference number
ISO 10060:1993(E)

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.

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 10060 was prepared by Technical Committee ISO/TC 33, *Refractories*.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Dense, shaped refractory products — Test methods for products containing carbon

1 Scope

This International Standard specifies test methods for refractory products containing residual carbon (see 7.3), the remainder being essentially sintered or fused doloma, magnesia or a mixture of these substances.

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 5014:1986, *Refractory products — Determination of modulus of rupture at ambient temperature.*

ISO 5017:1988, *Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity.*

ISO 8841:1991, *Dense, shaped refractory products — Determination of permeability to gases.*

ISO 10059-1:1992, *Dense, shaped refractory products — Determination of cold compressive strength — Part 1: Referee test without packing.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 pitch-bonded refractory: An unfired refractory shape which has been produced by pressing a mixture of graded aggregate and pitch.

NOTE 1 The term "tar" may be used as an alternative to pitch.

3.2 pitch-bonded tempered refractory: A pitch-bonded refractory shape which has been heated to a relatively low temperature (up to 800 °C).

3.3 resin-bonded refractory: An unfired refractory shape which has been produced by pressing a mixture of graded aggregate and resin.

3.4 resin-bonded tempered refractory: A resin-bonded refractory shape which has been heated to a relatively low temperature (up to 800 °C).

3.5 pitch-impregnated refractory: A refractory shape which has been impregnated by liquid pitch after forming. Such a shape may be either a fired product or one of the carbon-containing shapes defined in 3.1 to 3.4.

3.6 carbonization: The process of removing volatile components from test pieces of a refractory which has been either bonded or impregnated with carbonaceous material such as pitch (tar) or resin, so as to retain the residual carbon.

3.7 anti-oxidant: Metallic element or other substance added to the carbon-containing shapes defined in 3.1 to 3.4, in order to improve their resistance to oxidation.

4 Principle

Determination of physical properties of products containing carbon, both before and after the removal of volatile components by carbonization.

5 Test pieces

5.1 Size

Test pieces shall be of the size specified for each individual test method.

NOTE 2 Where irregularly shaped articles are tested, it may not be possible to obtain the appropriate size. In these cases, any variation of size should maintain a similar test piece volume or geometry and such variation should be stated in the test report.

5.2 Preparation

Test pieces shall be cut or drilled from the test brick or block, parallel to the direction of pressing.

NOTE 3 Materials containing graphite and carbon may exhibit marked anisotropy. For full characterization, samples may also be cut to be representative of the three axes at right angles.

Where wet cutting wheels or drills are used, test pieces shall be dried to a constant mass, using either a blast of warm air or a fan-assisted drying oven with free air flow to all surfaces.

NOTE 4 Where there is a possibility of softening or evaporation of volatile components, e.g. for pitch-bonded refractories, the temperature should not be greater than 40 °C.

Water-sensitive materials which are to be carbonized shall not be brought into contact with water.

Water-sensitive material which is to be tested at ambient temperature may only be prepared wet where it does not remain in contact with water for longer than 30 min, and if no hydration occurs during this time. If these criteria are not met (e.g. for untempered doloma), the material shall either be machined dry or by using a non-reacting liquid.

6 Removal of volatile components

6.1 Introduction

Some carbon-containing refractories, including the types defined in clause 3, contain volatile components. Carbonization is required for the determination of carbonization properties (see clause 7) and for some additional physical testing (see 8.2).

6.2 Apparatus

6.2.1 Furnace, gas or electric fired, capable of containing the carbonization box (6.2.3), and having a heat capacity such that, when it is maintained at 1 000 °C, the temperature at the centre of the carbonization box will rise from ambient temperature to 980 °C within 3 h. Ensure that the furnace is adequately ventilated.

6.2.2 Balance, capable of weighing to the nearest 0,2 g.

6.2.3 Carbonization box and lid, made from heat resisting steel 3 mm thick and suitable for use at 1 000 °C. The design and minimum dimensions shall be as shown in figure 1. Either the lid or a side shall contain a central hole allowing a sheathed thermocouple (6.2.4) to be inserted. The lid shall also contain a vent hole of diameter 3 mm. In order to avoid oxidation due to air draughts, only this vent hole should remain open.

NOTE 5 When there is deformation of the box and lid, or where an oxidizing furnace atmosphere is used, oxidation of the contents may occur. In such cases, it is recommended to seal the box, for example with air-setting mortar or by using a continuous sand seal into which the lid is placed. When using this configuration, a vent hole is not necessary.

6.2.4 Sheathed thermocouple, suitable for measurement up to 1 000 °C.

6.2.5 Desiccator, containing silica gel or phosphorus pentoxide.

6.2.6 Metallurgical coke, of grain size 0,5 mm to 2 mm, which shall be prefired in the carbonization box for 2 h at 1 000 °C ± 10 °C before use, and then stored under dry conditions.

6.3 Preparation of the carbonization box

Place a 25 mm layer of metallurgical coke (6.2.6) on the bottom of the carbonization box (6.2.3).

Where required for determination of carbonization properties (see clause 7), weigh each test piece to the nearest 0,2 g. The mass of the test piece is m_1.

Place the test pieces on the layer of coke, equidistantly from the sides of the box, in such a way that there is at least 25 mm thickness of coke between the test pieces and the walls of the box (see figure 1). Where required, insert blanks of the same size (see 5.2) and of similar chemical composition, to give uniform spacing within the box. Surround the test

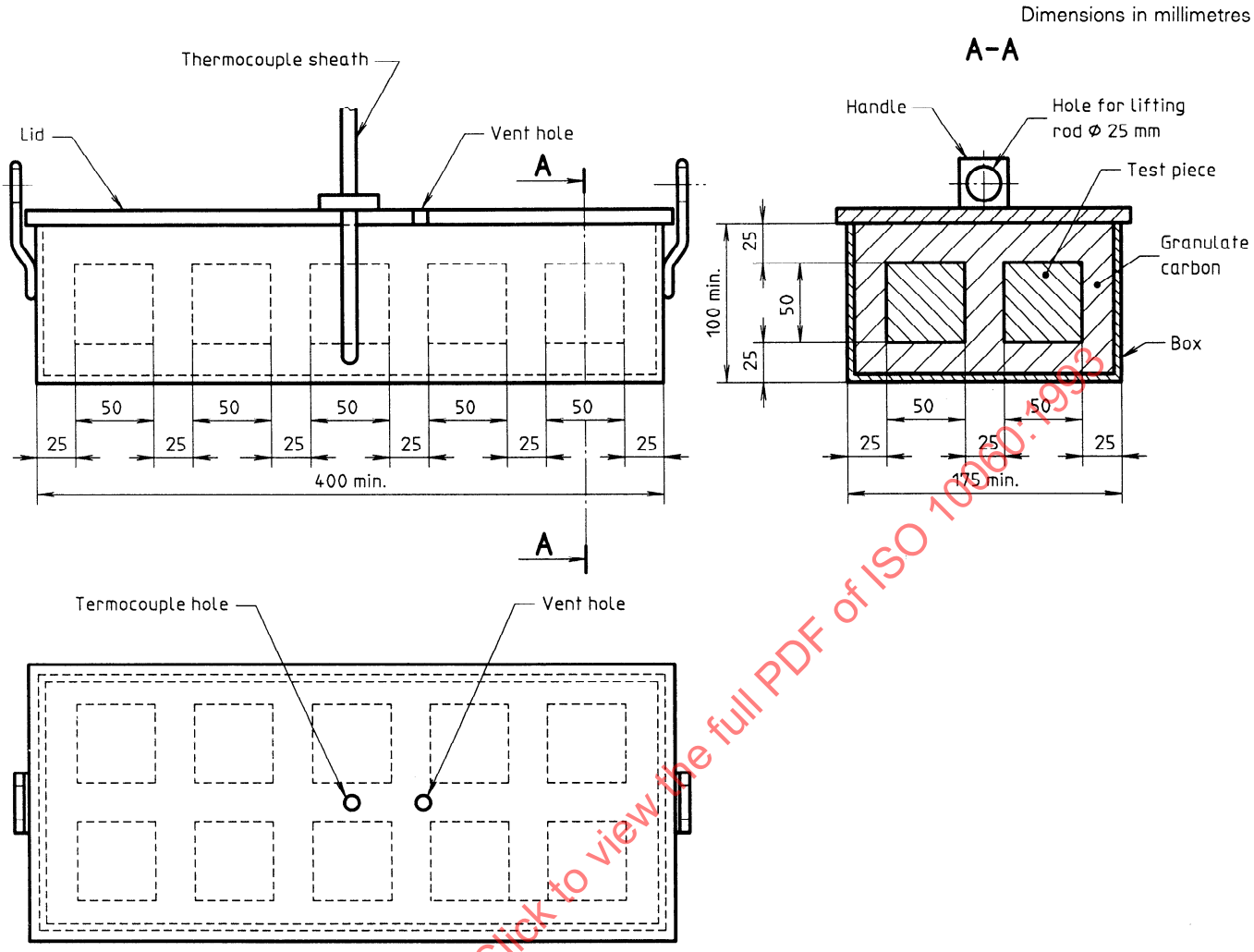

Figure 1 — Carbonization box and lid (example for 50 mm cubes)

pieces with the metallurgical coke, submerging them to a depth of 25 mm.

Position the lid as shown in figure 1, and insert the thermocouple (6.2.4), positioned centrally with respect to the test pieces.

6.4 Carbonization procedure

Heat the furnace (6.2.1) to 1 000 °C ± 10 °C and maintain it at this temperature for 2 h. Place the carbonization box (6.2.3) and contents in the hot furnace, whilst maintaining the furnace at 1 000 °C ± 10 °C. Note the time at which the temperature shown on the thermocouple (6.2.4) reaches 980 °C (see 6.2.1). Maintain the furnace at 1 000 °C ± 10 °C for 2 h after this time.

Remove the carbonization box from the hot furnace, allow to cool naturally to 100 °C, as shown by the thermocouple and then transfer the test pieces to the desiccator (6.2.5). Cool the test pieces to room temperature, and remove any coke adhering to them. If required, weigh each test piece to the nearest 0,2 g (see 6.3), and record this as the carbonized mass (m_2).

7 Determination of carbonization properties

7.1 Test pieces

Test pieces for the determination of the carbonization properties given in 7.2 to 7.4 shall be either cubes of side 50 mm ± 2 mm, or cylinders of diameter and height 50 mm ± 2 mm.

7.2 Carbonization mass loss

Calculate the carbonization mass loss CML, as a percentage of the original mass, using the following equation:

$$CML = \frac{m_1 - m_2}{m_1} \times 100 \quad \ldots (1)$$

where

m_1 is the original mass, in grams (see 6.3);

m_2 is the carbonized mass, in grams (see 6.4).

7.3 Residual carbon content

7.3.1 Direct analytical method

Grind the carbonized test pieces to a fine powder and take an appropriately sized sample. Determine the residual carbon content by chemical analysis.

NOTE 6 Commonly used analytical methods are coulometric titration and infrared detection of the carbon dioxide formed after combustion in a heated furnace under a flow of oxygen.

7.3.2 Loss on ignition method

This method is only suitable for products which do not contain any of the following:

a) anti-oxidants;

b) more than 1,5 % iron oxide;

c) materials (such as free lime) which react with the atmosphere.

Reweigh the carbonized test pieces m_2 and place them in a dry, tared, porcelain or fireclay crucible. Insert the crucible in the furnace (see 6.2.1), or any other furnace of suitable size, and heat it to 1 000 °C.

NOTE 7 The maximum rate of heating should be 250 °C/h.

Maintain an oxidizing atmosphere by passing a stream of air through the furnace, and maintain the temperature for at least 12 h. Cool the crucible in the furnace and then transfer it to the desiccator to cool to ambient temperature.

Remove the crucible from the desiccator and reweigh it to the nearest, 0,2 g. Subtract the mass of the crucible to give the mass after ignition (m_3).

Repeat the above procedure until constant mass is reached.

NOTE 8 Constant mass is normally reached after 12 h.

Calculate the residual carbon content (RC) as the difference in mass before and after ignition, expressed as a percentage of the carbonized mass, using the following equation:

$$RC = \frac{m_2 - m_3}{m_2} \times 100 \quad \ldots (2)$$

where

m_2 is as defined in 6.4;

m_3 is the mass after ignition, in grams.

7.3.3 Report

The test report (clause 9) shall indicate which procedure (7.3.1 or 7.3.2) was used.

7.4 Carbon yield

Calculate the carbon yield (CY) as the difference in mass between carbonized test piece before and after ignition, expressed as a percentage of the loss on ignition, using the following equation:

$$CY = \frac{m_2 - m_3}{m_1 - m_3} \times 100$$

where m_1, m_2, m_3 are as defined in 6.3, 6.4 and 7.3.2.

NOTE 9 This determination is not applicable to materials containing anti-oxidants or more than 1,5 % Fe_2O_3.

8 Physical testing

8.1 Tests on material as received

8.1.1 Test piece preparation

Prepare test pieces from the as-received material (see 5.2) of the sizes required for the tests described in 8.1.2 to 8.1.6.

8.1.2 Determination of bulk density and apparent porosity

Determine the bulk density and apparent porosity in accordance with ISO 5017.

If the sample contains free lime, either use a suitable organic liquid which does not dissolve pitch, or, if water is used, complete the test within 30 min.

NOTE 10 When water is used for the determination of bulk density and apparent porosity of test pieces which contain anti-oxidants reacting to form carbides sensitive to hydration, these test pieces should not be used for any further testing (such as strength tests).

8.1.3 Determination of geometric bulk density

As an alternative to 8.1.2, the bulk density only may be measured from the mass and dimensions as follows.

Measure the mass of the shaped product or any appropriate piece of it with at least one dimension greater than 100 mm, to an accuracy of 0,2 %.

Measure dimensions greater than 250 mm with a steel tape, graduated in millimetres, with a right-angled hook corresponding to the origin of measurement. Measure dimensions less than 250 mm with callipers. Make two measurements for the length, width and thickness, with distinct measurements for the outer and inner sides of the brick for the width and thickness. Do not place the callipers more than 10 mm from the edge, to avoid any error arising from tapering. To eliminate erroneous measurements likely to be obtained from imperfect bricks, determine the bulk density on bricks which do not bow more than 2 mm over their whole length.

8.1.4 Determination of cold compressive strength

Determine the cold compressive strength in accordance with ISO 10059-1. Pitch-bonded test pieces shall be stored at 22 °C ± 2 °C, and tested as close as possible to this temperature, with the test temperature being reported.

8.1.5 Determination of modulus of rupture at ambient temperature

Determine the modulus of rupture at ambient temperature in accordance with ISO 5014, using test pieces of 150 mm × 25 mm × 25 mm. For pitch-bonded products, the temperature requirements of 8.1.4 shall apply.

8.1.6 Change in mass on ignition

Remove all carbon from the test pieces in accordance with 8.3. Calculate the change in mass on ignition as a percentage of the original mass measured in 8.1.3.

NOTE 11 This test is less meaningful where anti-oxidants are present.

8.2 Tests after carbonization

NOTE 12 These tests are carried out after completion of the carbonization procedure (6.4).

8.2.1 Test piece preparation

Prepare test pieces (see 5.1) of the sizes required for the tests given in 8.2.2, and carbonize them in accordance with the procedure described in clause 6.

8.2.2 Ambient temperature properties

Determine the bulk density, apparent porosity, cold compressive strength and modulus of rupture at ambient temperature in accordance with the methods described in 8.1. In addition, determine the permeability to gases in accordance with ISO 8841.

If the test material contains anti-oxidants, carry out the test on freshly coked test pieces, in order to avoid reaction between the test piece and moisture in the air.

NOTE 13 When water is used for the determination of bulk density and apparent porosity of test pieces which contain anti-oxidants reacting to form carbides sensitive to hydration, these test pieces should not be used for any further testing (such as strength tests).

NOTE 14 For test pieces containing anti-oxidants which react to form carbides sensitive to hydration, it may be necessary to use a non-aqueous liquid for the determination of bulk density and apparent porosity.

8.3 Tests after total removal of carbon

Pitch-impregnated fired refractory products (3.5) may be tested after all the carbon has been removed.

Remove the carbon from the test pieces (see clause 5) by heating them in an oxidizing atmosphere with a maximum heating rate of 250 °C/h, to 1 000 °C ± 10 °C, and maintaining for at least 12 h or until constant mass is reached.

NOTE 15 For larger test pieces, a maximum heating rate of 60 °C/h would be appropriate.

Following the removal of carbon, the test pieces may be tested by any ISO method used for dense, shaped refractory products.

9 Test report

The test report shall include the following information:

a) the name of the testing establishment;

b) the date of the test;

c) a reference to this International Standard, i.e. "Determined in accordance with ISO 10060";

d) any reference to alternatives in the test procedure (see 7.3.3 and 8.1.3);

e) designation of bricks tested (manufacturer, type, shape, etc.);

f) the number of items (bricks) tested;

g) any variation in test piece size (see 5.1);

h) any use of liquids other than water in preparation and testing;

i) the results of each test carried out, including test temperatures where required.

This page intentionally left blank