INTERNATIONAL STANDARD

ISO 12676

First edition
2000-11-15

Refractory products — Determination of resistance to carbon monoxide

Produits réfractaires — Détermination de la résistance au monoxyde de carbone

Reference number
ISO 12676:2000(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12676 was prepared by Technical Committee ISO/TC 33, *Refractories*.

Refractory products — Determination of resistance to carbon monoxide

1 Scope

This International Standard specifies a method for determining the comparative resistance of refractory materials to carbon monoxide disintegration.

The test is intended to be more severe than conditions encountered in service in order to enable probable behaviour of refractory materials to be assessed in a relatively short time.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5022:1979, *Shaped refractory products — Sampling and acceptance testing*.

ISO 8656-1:1988, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*.

3 Terms and definitions

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

3.1

carbon monoxide disintegration

breakdown of a refractory product caused by the deposition of carbon resulting from the dissociation of carbon monoxide

3.2

carbon monoxide resistance

resistance of a refractory product to carbon monoxide disintegration when exposed to carbon monoxide under specified conditions of atmosphere and temperature

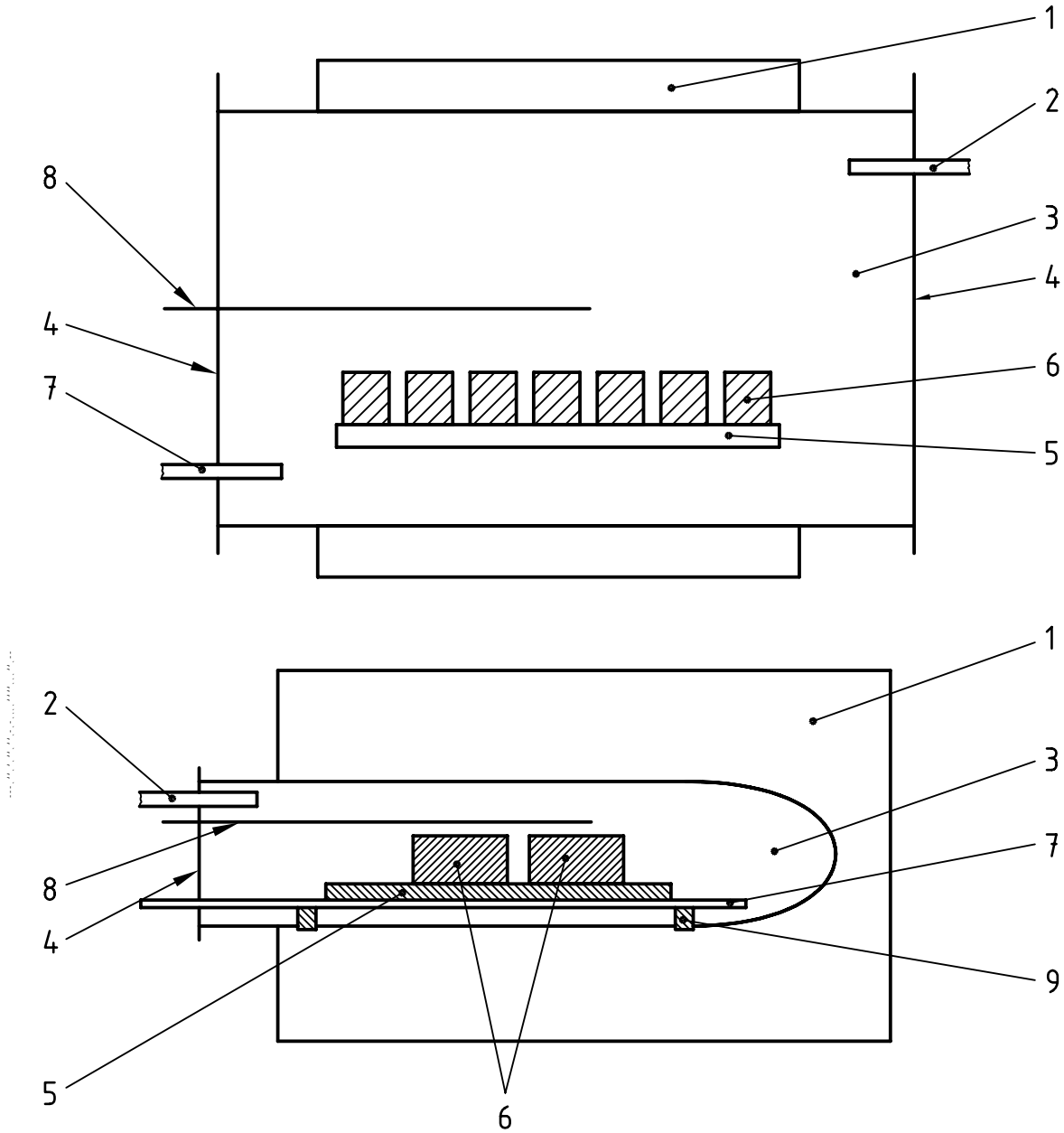
4 Principle

Test pieces are exposed to a specified carbon monoxide atmosphere at a controlled temperature for a specified time.

NOTE Carbon monoxide is toxic and suitable safety precautions should be observed when carrying out this test e.g. monitoring the atmosphere around the apparatus to detect leakage of carbon monoxide.

5 Apparatus

Schematic examples of test apparatus are shown in Figure 1.



Key

1	Furnace enclosure
2	Gas exit tube
3	Reaction chamber
4	Gas-tight end cap
5	Test piece support
6	Test pieces
7	Gas inlet tube
8	Temperature measuring device
9	Tube support

Figure 1 — Examples of test apparatus (schematic: not to scale)

5.1 Furnace, of sufficient size to be capable of maintaining the test zone within the reaction chamber (5.2) to within $\pm$ 10 °C at 500 °C.

NOTE The furnace may be fitted with one or more observation ports to enable direct observation of the test samples during the test when a glass reaction chamber is used.

5.2 Reaction chamber, comprising a gas-tight tube capable of withstanding a temperature of 500 °C and made of a material which does not react with carbon monoxide under the conditions of the test. The chamber shall be of a suitable size to accommodate the test pieces (see clause 7) within the test zone. It shall be fitted with gas inlet and outlet tubes so that the gas passes through the length of the test zone. Provision shall be made for the insertion of a temperature measuring and recording device (5.3) to be located in the test zone and adjacent to the test pieces. The chamber shall also contain supports on which to rest the test pieces and to prevent debris from falling into the chamber in cases of disintegration during the test. The tubes and the support shall also be of a material unreactive to carbon monoxide and capable of withstanding temperatures of up to 500 °C.

NOTE 1 Suitable materials for the reaction chamber are certain grades of stainless steel, brass, aluminium, unoxidised inconel metal, glass and ceramic.

NOTE 2 The reaction chamber may be closed at one end and one of the gas tubes used together with a second tube to carry the test piece support so long as the criteria above are maintained. Depending on the size of the reaction chamber and the number of test pieces, it may be necessary to provide support for the tubes themselves.

5.3 Temperature measuring and recording device, to enable the temperature of the test zone to be measured to the required accuracy (see 5.1) and to be continuously recorded.

5.4 Temperature controller, to control the furnace (5.1) to the required accuracy.

5.5 Carbon monoxide, of 99 % purity.

NOTE 1 The carbon monoxide may be supplied from gas bottles or a tank depending on the size of the reaction chamber and the gas flow required (see 8.6). If large quantities of CO are required then it may be purified to the above specification and recirculated.

NOTE 2 When carbon monoxide is supplied from a tank, iron carbonyl may be present in the gas and may cause clogging of the inlet tube [see 5.7 b)].

5.6 Nitrogen, of industrial grade.

5.7 Means of control of carbon monoxide atmosphere, operating as follows:

a) for gas supplied by tank or bottle, the pressure shall be reduced by a regulator made for that purpose and the flow of gas adjusted by means of a sensitive needle or regulating valve.

b) a purification train shall be situated after the fine control valve and consist of a drying tower (silica gel, magnesium perchlorate or phosphorus pentoxide may be used but not sulphuric acid or calcium chloride). If the gas is supplied from a tank follow the drying tower with a scrubbing tower to remove iron carbonyl (e.g. containing sodium hydroxide pellets).

c) the gas flow rate shall be measured using a suitable non-aqueous flowmeter fitted between the purification train and the reaction chamber inlet tube.

5.8 Reaction chamber pressure control, a bubbling bottle or sensitive pressure gauge shall be fitted to the outlet of the reaction chamber to ensure that a positive pressure, of at least 20 mm of water, is maintained throughout the test.

5.9 Gas analyser, capable of determining the carbon dioxide content of the exhaust gas to ensure that it contains a maximum of 5 % carbon dioxide.

5.10 Drying oven, capable of maintaining a temperature of 110 °C $\pm$ 5 °C.

5.11 Desiccator.

5.12 Timer.

5.13 Photographic equipment.

6 Sampling

The number of items to be tested shall be determined in accordance with ISO 5022 for shaped products or ISO 8656-1 for unshaped products or a sampling plan agreed between the interested parties.

7 Test pieces

7.1 General

Test pieces shall be rectangular prisms with a 50 mm ± 2 mm, 64 mm ± 2 mm or 76 mm ± 2 mm square cross-section. The length of the prisms shall be agreed between the interested parties but shall be a minimum of 76 mm.

NOTE 1 It is recommended that only test pieces from one material are included in each test to avoid the behaviour of one material affecting the behaviour of another.

NOTE 2 Care should be taken when cutting the test pieces from larger test items that the cut surfaces are not contaminated with iron or iron-containing alloy which may originate from the type of cutting equipment used.

7.2 Fired materials

Only one test piece shall be taken from each test item and cut so that a minimum of three faces are the original surfaces of the test item.

7.3 Unfired materials

Test pieces of unfired materials shall be prepared according to the manufacturers' instructions or in an appropriate manner according to the type of material. Alternatively, test pieces may be cut from larger prepared shapes.

The test pieces shall be fired in an oxidizing atmosphere at a rate not exceeding 5 °C/min to a temperature of 540 °C, maintained at this temperature for 5 h, then cooled and stored in the desiccator (5.11).

NOTE 1 Cylindrical test pieces of unfired materials cut before or after firing to 540 °C may be used. The test pieces shall be of a diameter of 50 mm ± 2 mm and a minimum length of 76 mm.

NOTE 2 In cases where large numbers of samples require testing or for quality control purposes, non-referee evaluations may be made on the basis of test results using small test pieces (e.g. 30 mm × 30 mm × 30 mm).

8 Procedure

8.1 Dry test pieces of fired materials at 110 °C ± 5 °C and allow to cool in the desiccator (5.11).

8.2 Place the cooled test pieces on the supports in the test zone of the reaction chamber so that they are separated by at least 20 mm in order to allow free circulation of gas.

8.3 Insert the temperature measuring device (5.3) and ensure it is located properly (see 5.2). Seal the reaction chamber.

8.4 Thoroughly flush the chamber with nitrogen (5.6) and heat the test pieces to 500 °C ± 10 °C whilst continuing to pass nitrogen through the chamber.

8.5 When the test pieces have attained the test temperature, cease the nitrogen flow and change the atmosphere to a minimum of 95 % in order carbon monoxide (5.5) as shown by analysis of the exhaust gas. Record the time and date.

NOTE This may be done by flushing the nitrogen with a high flow of carbon monoxide or by evacuating the chamber with a vacuum pump to a pressure of about 100 mmHg (1 mmHg = 133,322 4 Pa), then introducing carbon monoxide at a flow rate indicated in 8.6. The process is then repeated until the required exhaust gas analysis is obtained.

8.6 Regulate the flow of carbon monoxide (5.5) to a nominal rate of not less than 30 cm^3/h for each 15 cm^3 of total specimen volume. Analyse the exhaust gas after 1 h and if it is less than 95 % carbon monoxide, increase the gas flow. Repeat the analysis and flow rate adjustment until the exhaust gas is above 95 % carbon monoxide. Analyse the exhaust gas at least twice a day during the duration of the test and maintain the chamber atmosphere above 95 % carbon monoxide.

NOTE The initial flow rate of 30 cm^3/h for each 15 cm^3 of total specimen volume is considered to be a minimum rate with which to carry out the test. For materials suspected of reacting significantly with carbon monoxide, a flow rate 2 to 3 times greater may be required to maintain the chamber atmosphere. In these cases, it may be advisable to reduce size and number of test pieces in the chamber in order to avoid excessive use of carbon monoxide.

8.7 Continue the test for 200 h, at which time change to a flow of nitrogen to flush out the furnace chamber. Switch off the furnace and allow the chamber to cool. Record time and date at which the test was terminated, carefully remove the test pieces, inspect their condition in accordance with clause 9 and make a photographic record of their appearance.

NOTE 1 If the test apparatus has facilities for direct observation of the test pieces, then daily inspections can be made and recorded. The test can then be terminated when the test piece or half the number of test pieces show disintegration (see clause 9).

NOTE 2 A shorter or longer test duration may be agreed between interested parties. It must, however, be recognised that the clauses describing the test piece condition [see 9 a) 1) to 4)] will only be comparable for the same test duration.

9 Condition of test pieces

a) The condition of the test pieces shall be described according to the following classifications:

   1) unaffected: no carbon deposition, surface pop-outs or cracking observable;

   2) surface damage. surface pop-outs or spalls 10 mm or less in diameter visible;

   3) cracked. cracks visible within the test pieces, and/or spalls or surface pop-outs greater than 10 mm in diameter have occurred;

   4) disruption: test pieces have broken into two or more pieces, or when hand pressure can cause breaking.

b) If the condition of the test pieces is variable, the number of test pieces in each classification shall be stated.

10 Test report

The test report shall include the following information:

a) the name of the test establishment;

b) the time and date at which the test was commenced and terminated;

c) reference to this International Standard, i.e. ISO 12676;

d) any deviations in the test procedure;

e) designation of material tested (manufacture, type, brand, etc.);

f) the number of items tested;

g) the size of the test pieces;

h) the duration of the test;

i) the flow rate (expressed in cm^3/h per cm^3 of total test piece volume) used during the major part of the test duration;

j) the condition of the test pieces; according to the classifications in clause 9;

k) any observation in relation to the test pieces or the performance of the test, including those made during the test, if any, which may assist in the interpretation of the results;

l) the photographic record of the test pieces, if necessary.

ICS 81.080

Price based on 6 pages