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**Refractory mortars —**

**Part 7:**

**Determination of permanent change  
in dimensions on heating**

*Mortiers réfractaires —*

*Partie 7: Détermination des variations dimensionnelles permanentes  
lors du chauffage*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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

A list of all parts in the ISO 13765 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

# Refractory mortars —

## Part 7:

# Determination of permanent change in dimensions on heating

## 1 Scope

This document describes the method for determining the permanent change in dimensions on heating (drying and/or firing) of refractory mortars.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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

ISO 13765-1, *Refractory mortars — Part 1: Determination of consistency using the penetrating cone method*

ISO 13765-2, *Refractory mortars — Part 2: Determination of consistency using the reciprocating flow table method*

ISO 13765-3, *Refractory mortars — Part 3: Determination of joint stability*

## 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

## 4 Principle

A mortar sample is heated and the dimensional change after heating is determined. Place the test specimens of refractory mortars, whose length between two marks has been determined, in a drying oven or furnace. Heat (drying and/or firing) to a specified temperature for a specified time and then cool to room temperature. Measure the length between two marks again. Calculate the permanent change in dimensions on heating.

## 5 Apparatus

**5.1 Mixer**, as specified in ISO 13765-1 or ISO 13765-2.

**5.2 Balance**, with a maximum weighing capacity of 10 kg and capable of weighing to the nearest 1 g.

5.3 **Cutting device**, a trowel.

5.4 **Nylon filter cloth**, single layer nylon mesh filter cloth, 80 mesh~200 mesh.

5.5 **Electrical drying oven**, fitted with a temperature controller and capable of operating at  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ .

5.6 **Furnace**, the maximum temperature for the furnace should not lower than  $1\ 600\text{ }^{\circ}\text{C}$ . And the temperature difference shall not exceed  $\pm 10\text{ }^{\circ}\text{C}$  in the furnace.

5.7 **Refractory powders**, the particle size shall less than 0,1 mm.

5.8 **Refractory bricks**,  $(114 \pm 1)\text{ mm} \times (65 \pm 1)\text{ mm} \times (32 \pm 1)\text{ mm}$ , which should be the same as the bricks used in the actual construction of refractory mortars.

5.9 **Length determining device**, capable of measuring to the nearest 0,05 mm.

5.10 **Rectangular frame**, 2 mm thick, with an inner length of  $100_0^{+2}\text{ mm}$  , width of  $50_0^{+2}\text{ mm}$  .

## 6 Test preparation

### 6.1 Sampling

For dry mortar, sample the mortar in accordance with ISO 8656-1 or as agreed between parties. Reduce the sample to 5 kg by quartering or with a riffle sampler.

For ready-mixed mortars, sample the mortar after mixing thoroughly. It is important that any supernatant liquid not be discarded. Ensure that a representative sample of the wet mixture is obtained.

### 6.2 Preparation of the refractory bricks

The size of the test specimens and the refractory bricks recommended to use is shown in [Table 1](#). The initial distance between two marks referred in [Table 1](#) is the spacing of the two marks on each diagonal of the rectangular test specimens.

At least six refractory bricks which are similar to the ones to be used in service are required. These are used for making three test specimens. Each refractory mortar test specimen is formed by two refractory bricks. The surfaces of refractory bricks to be bonded shall be clean and have no visible craters, open cracks, fins, protrusions and indentations in the surface. The bricks used in the test shall be compatible with the mortar being tested. Before testing, place the bricks in the drying oven, raise the temperature to  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  and hold for at least 2 h or until constant weight is achieved. Then cool to room temperature naturally.

**Table 1 — Size of the test specimens and the refractory bricks**

Test specimen size $l \times b$ mm	Initial distance between two marks $l_0$ mm	Refractory brick size recommended $l \times b \times t$ mm
$(100_0^{+2}) \times (50_0^{+2})$	90~100	$(114 \pm 1) \times (65 \pm 1) \times (32 \pm 1)$

## 7 Procedure

### 7.1 Preparation of the test specimens

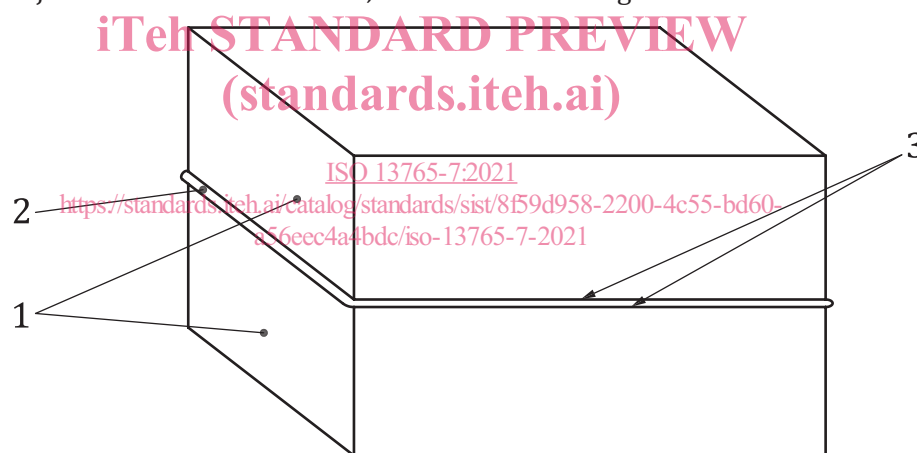
**7.1.1** For dry mortars, sample 2 kg using the balance and mix with water (or a specified mixing liquid) in accordance with ISO 13765-1 or ISO 13765-2 to attain the desired consistency. Determine the consistency in accordance with ISO 13765-1 and ISO 13765-2 and allow to rest for 30 min or in accordance with the manufacturer's instructions.

For ready-mixed mortars, sample 2,5 kg after mixing thoroughly.

After determining the consistency, carry out the procedures described in [7.1.2](#).

**7.1.2** Take a pair of dried refractory bricks and lay nylon filter cloth on both of the uncut faces for bonding. Take out one brick and put a 2 mm thick rectangular frame with an inner length of  $100^{+2}_0$  mm, width of  $50^{+2}_0$  mm on the nylon filter cloth, which is used to ensure the 2 mm~4 mm thick mortar joint, then apply a layer of the well-mixed mortar to the nylon filter cloth. Place the other refractory brick with the nylon filter cloth on the mortars as shown in [Figure 1](#). Press the top brick and move it laterally at the same time according to ISO 13765-3.

The faces of each brick shall be parallel to each other within a tolerance of 1mm. Force out the excess mortar to form a joint of 2 mm~4 mm thick, unless otherwise agreed.



#### Key

- 1 brick
- 2 mortar
- 3 nylon filter cloth

**Figure 1 — Placement of bricks**

**7.1.3** Leave the specimen at room temperature for  $4 \text{ h} \pm 0,5 \text{ h}$  to become stable (according to ISO 13765-3), remove the top refractory brick and peel off the top nylon filter cloth on the mortar samples. Sprinkle a layer of dry, fine refractory powders ( $\leq 0,1 \text{ mm}$ ), which does not react with the refractory mortars and bricks when heating, on the exposed surface, and place the refractory brick on the mortars again. Reverse the two refractory bricks and mortars as a whole and remove the top refractory brick and peel off the nylon filter cloth on the mortars just like the former step.

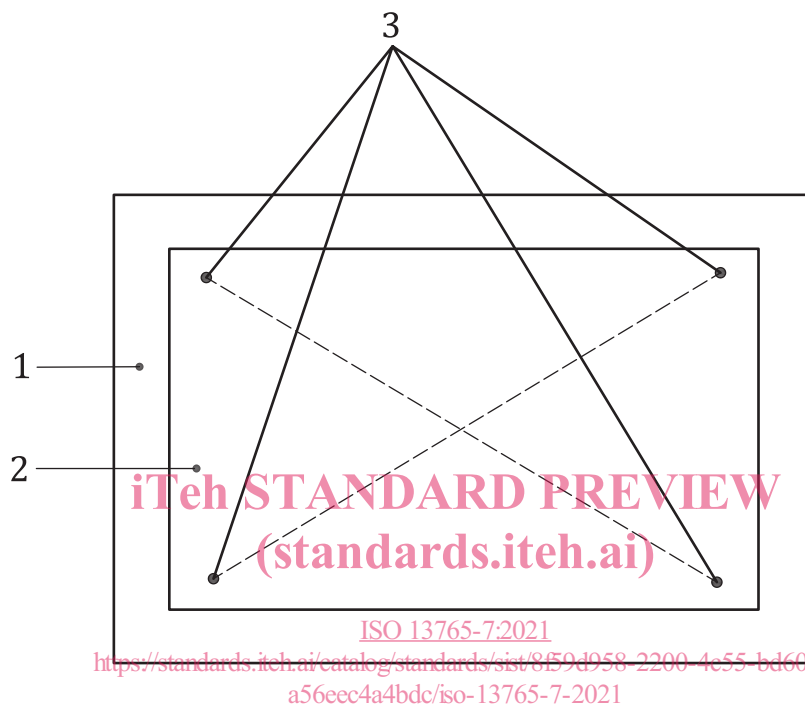
Do not break the mortar layer when peeling off the nylon filter cloth. Clearly visible bubbles and cavity are not permitted. Otherwise the specimens should be prepared again.

7.1.4 Separate the refractory mortar from the rectangular frame. Remove the rectangular frame and remove excess mortar (The mortar sample is stationary relative to the firebrick).

Repeat the above operations, using the six refractory bricks to make three specimens.

## 7.2 Measurement of test specimens

Mark 2 points on one diagonal of the mortar samples in the range of 90 mm~100 mm. Record the actual distance, mark as  $l_0$ . Repeat the above operations on the other diagonal. As shown in Figure 2.



### Key

- 1 brick
- 2 mortar specimen
- 3 marks

Figure 2 — Test specimen

## 7.3 Drying the test specimens

Air dry the test specimens and bricks naturally at room temperature for  $24\text{ h} \pm 1\text{ h}$ , and then carefully place the air-dried specimens and bricks in the drying oven, raise the temperature to  $65\text{ °C} \pm 5\text{ °C}$  for  $4\text{ h} \pm 0,5\text{ h}$ . Then raise the temperature to  $110\text{ °C} \pm 5\text{ °C}$  and hold for  $24\text{ h} \pm 1\text{ h}$ .

## 7.4 Firing the test specimens

Place the dried specimens and bricks in the soaking zone of the furnace. The distance between the specimens themselves shall not be less than 10 mm. The distance between the specimens and the heating element shall not be less than 20 mm.

When heating, maintain an oxidizing atmosphere in the furnace. Use the following heating rate:

- Normally  $5\text{ °C/min}$ ,
- Silica mortar  $3\text{ °C/min}$ .



When the specified temperature (The specified temperature varies from the type of mortars and shall be negotiated by both parties) is reached, hold for  $3 \text{ h} \pm 0,5 \text{ h}$  ( $5 \text{ h} \pm 0,5 \text{ h}$  for silica mortar), over which the fluctuations of the temperature shall not exceed  $\pm 10 \text{ }^\circ\text{C}$ .

After the holding period, cool the specimens in the furnace to room temperature naturally.

## 7.5 Measurement of test specimens after heating (drying or firing)

For each brick measure the distance (with an accuracy of 0,05 mm) between marks of both sets of two, marks  $l_1$  after drying and  $l_2$  after firing.

## 8 Calculations

Calculate the permanent change in dimensions for each of the two diagonals, in percentage. Report the permanent change in dimensions on heating of each test specimen as the mean value of the two results.

### 8.1 The permanent change in dimensions on drying

The permanent change in dimensions on drying,  $L_d$ , using [Formula \(1\)](#):

$$L_d = \frac{l_1 - l_0}{l_0} \times 100 \quad (1)$$

where

$l_0$  is distance between the two marks along the same diagonal of the specimens before drying, in millimetres;

$l_1$  is distance between the two marks along the same diagonal of the specimens after drying, in millimetres.

### 8.2 The permanent change in dimensions on firing

The permanent change in dimensions on firing,  $L_f$ , using the [Formula \(2\)](#):

$$L_f = \frac{l_2 - l_1}{l_1} \times 100 \quad (2)$$

where

$l_1$  is distance between the two marks along the same diagonal of the specimens after drying, in millimetres;

$l_2$  is distance between the two marks along the same diagonal of the specimens after firing, in millimetres.

### 8.3 The permanent change in dimensions on heating

The permanent change in dimensions on heating,  $L_c$ , using the [Formula \(3\)](#):

$$L_c = \frac{l_2 - l_0}{l_0} \times 100 \quad (3)$$

where

- $l_0$  is distance between the two marks along the same diagonal of the specimens before drying, in millimetres;
- $l_2$  is distance between the two marks along the same diagonal of the specimens after firing, in millimetres.

The permanent change in dimensions of a specimen on heating is the average value of three specimens respectively. Report the individual values calculated for each test specimen, together with the mean value, to the nearest 0,1 %. "+" stands for expansion and "-" stands for shrinkage.

The conditions of cracking of the specimens after drying and firing shall be recorded if any cracks occur.

## 9 Precision

### 9.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are summarized in [Annex A](#). The values derived from these interlaboratory tests may not be applicable to matrices other than those given.

### 9.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not in more than 5 % of the cases exceed the value of the repeatability limit,  $r$ , given in [Table A.1](#).

### 9.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will not in more than 5 % of cases exceed the value of the reproducibility limit,  $R$ , given in [Table A.1](#).

## 10 Reporting

The test report shall include the following information:

- a) all information necessary for identification of the material tested, including a description of the material, manufacturer, type, brand, batch number, etc.;
- b) a reference to this document, i.e., ISO 13765-7:2021;
- c) the name of the testing establishment;
- d) in the case of a dry mortar, the mass fraction of water (or specified liquid) added;
- e) the results of the test, i.e. including the results of the individual determinations and their mean;
- f) the consistency of the mortar tested and the method used to determine the consistency, i.e. ISO 13765-1 or ISO 13765-2;
- g) the firing temperature and holding time;
- h) any deviation from the procedure specified;
- i) any unusual features (anomalies) observed during the test;
- j) the date of the test.