BRITISH STANDARD

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Methods of test for dense shaped refractory products

Part 10. Determination of permanent change in dimensions on heating

The European Standard EN 993-10 : 1997 has the status of a British Standard

ICS 81.080

National foreword

This British Standard is the English language version of EN 993-10 : 1997 published by the European Committee for Standardization (CEN). It supersedes BS 1902 : Part 5 : Section 5.10 : 1986 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/1/1, Sampling and physical testing of refractory materials, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 7, and a back cover.

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Methods of test for dense shaped refractory products — Part 10: Determination of permanent change in dimensions on heating

Méthodes d'essai pour produits réfractaires façonnés denses — Partie 10: Détermination de la variation permanente de dimensions sous l'action de la chaleur

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse — Teil 10: Bestimmung der bleibenden Längenänderung nach Temperatureinwirkung

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN 993-10 : 1997 E

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 187, Refractory Products and materials, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 1998, and conflicting national standards shall be withdrawn at the latest by May 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

It is closely based on the corresponding International Standard, ISO 2478, *Dense shaped refractory products — Determination of permanent change in dimensions on heating*, published by the International Organization for Standardization (ISO).

Reproducibility and repeatability data are not available at present but may be included in a subsequent edition.

EN 993, *Methods of test for dense shaped refractory products* consists of 18 Parts:

Part 1: *Determination of bulk density and porosity of dense shaped products*
Part 2: *Determination of true density*
Part 3: *Test methods for carbon-containing refractories*
Part 4: *Determination of permeability to gases*
Part 5: *Determination of cold crushing strength*
Part 6: *Determination of modulus rupture at ambient temperature*
Part 7: *Determination of modulus rupture at elevated temperatures*
Part 8: *Determination of refractoriness-under-load*
Part 9: *Determination of creep in compression*
Part 10: *Determination of permanent change in dimensions on heating*
Part 11: *Determination of resistance to thermal shock (ENV)*
Part 12: *Determination of pyrometric cone equivalent*
Part 13: *Specification for pyrometric cones*
Part 14: *Determination of thermal conductivity (hot wire, cross-array)*
Part 15: *Determination of thermal conductivity (hot wire, parallel)*
Part 16: *Determination of resistance to acids*
Part 17: *Determination of bulk density of granular material (mercury method)*
Part 18: *Determination of bulk density of granular material (water method)*

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1 Scope

This European Standard describes three methods for the determination of the permanent change in dimensions on heating of dense shaped refractory products.

NOTE. The method can be applied to materials sensitive to oxidation. However, some of these materials can be affected during the test in such a way as to make the measurement of the dimensional changes impossible to carry out to the required accuracy.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 993-1: *Methods of test for dense shaped refractory products — Part 1: Determination of bulk density, apparent porosity and true porosity.*

ISO 3611: *Micrometer callipers for external measurement.*

3 Definitions

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

3.1 permanent change in dimensions on heating

The expansion or contraction that remains in a shaped refractory product that is heated to a specified temperature for a specified time and then cooled to ambient temperature.

3.2 dense shaped refractory products

A refractory product having a true porosity of less than 45 % (*V/V*) when measured in accordance with EN 993-1.

4 Principle

Test pieces in the shape of rectangular prisms or cylinders are cut from each brick or item, then dried, and their linear dimensions (Methods 1 and 2) or volume (Method 3) measured. The test pieces are heated in a furnace having an oxidizing atmosphere at a prescribed rate to a specified temperature, which is maintained for a specified time. After cooling to ambient temperature, the measurements on the test pieces are repeated, and the permanent change in dimensions or volume is calculated.

5 Apparatus

5.1 *Furnace*, either electric or gas-fired, capable of heating the test pieces described in clause **6**, in a continuously oxidizing atmosphere, at the specified rate (see **7.6**), and of maintaining the test temperature for the required time.

NOTE. The use of an electric furnace is recommended, but a gas-fired furnace may be used provided that the furnace atmosphere is continuously oxidizing and there is provision for monitoring this condition.

5.2 *Thermocouples*, a minimum of three, to measure the temperature and the temperature distribution over the space occupied by the test pieces.

5.3 *Temperature/time registration device*, for use in conjunction with the thermocouples (**5.2**), so that a continuous record of the temperature is obtained.

5.4 *Length measuring device*

5.4.1 *General.* Two measuring devices may be used, either the dial gauge apparatus (Method 1) or vernier callipers (Method 2).

Method 1 shall be used as a reference method.

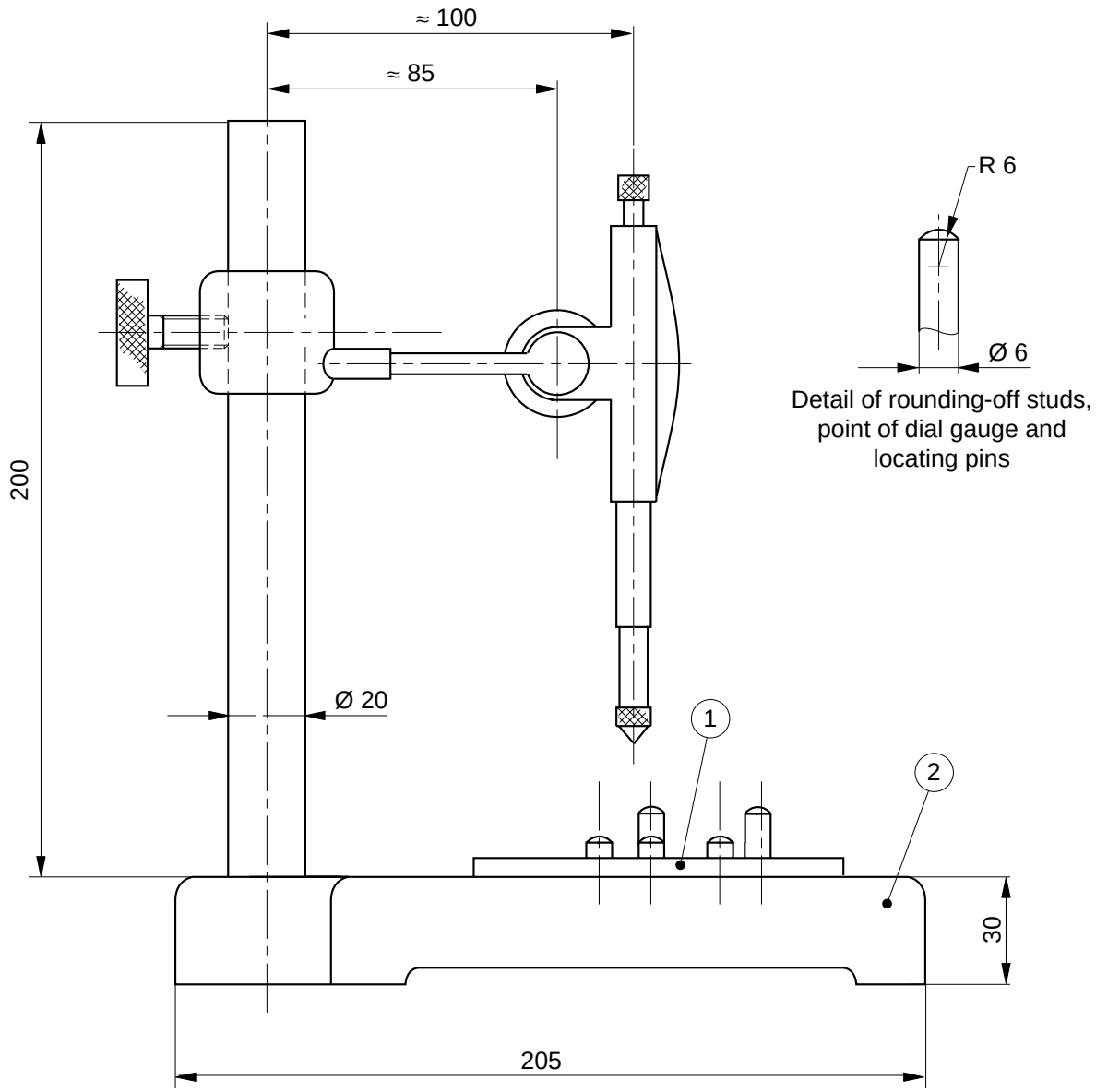
5.4.2 *Dial gauge apparatus* (Method 1), consisting of a dial gauge or micrometer with an accuracy of 0,01 mm, mounted on a stand which has a surface ground base plate (see figure 1), and a test piece carrier (see figure 2), with three studs to support the test piece and two pins to locate it. The dimensions of the locating pins shall be as shown in figure 2. The under surface of the carrier shall be ground flat. A diagonal mark shall be inscribed at one corner to enable a rectangular test piece to be placed symmetrically on the studs. A cylinder of known length shall be used to calibrate the device.

The carrier shall be used to support and locate the test piece so that measurements with the dial gauge or micrometer before and after firing are made at the same points on the test piece surface.

5.4.3 *Vernier callipers* (Method 2), in accordance with ISO 3611.

5.5 *Volume measuring device* (Method 3), of the water displacement type, the bulk volume being determined in accordance with the method specified in EN 993-1.

5.6 *Drying oven*, capable of being controlled at (110 ± 5) °C, which shall be fan-assisted and shall have openings which permit efficient ventilation.



Dimensions in millimetres

① Carrier (see figure 2)

② Base plate

Figure 1. Length-measuring device (Method 1) (Plan view)

6 Test pieces

The number of items tested and the number of test pieces per item, shall be by agreement between the parties.

NOTE 1. It is recommended that only one test piece should be taken from each item.

Test pieces shall be in the form of either:

a) rectangular prisms, 50 mm × 50 mm × 60 mm; or

b) cylinders, 50 mm diameter and 60 mm long.

Tolerances on all dimensions shall be ± 2mm.

NOTE 2. The 60 mm dimension should coincide with the direction of the forming pressure during manufacture if this direction is known.

The position of each test piece in the brick shall be recorded. The 50 mm × 50 mm faces of the prism, or the ends of the cylinder, shall be ground plane and parallel before the test. Each test piece shall be identified by appropriate marking.

7 Procedure

7.1 Drying of the test pieces

Dry each test piece in the drying oven (**5.6**) at (110 ± 5) °C to constant mass.

7.2 Measurement of test pieces

7.2.1 *Linear measurement by dial gauge apparatus (Method 1)*

Calibrate the length-measuring device (**5.4**) using the cylinder of known length. Place the test piece on the carrier, with the 60 mm dimension vertical. For rectangular test pieces, align one corner with the diagonal mark on the carrier, and mark this corner so that the test piece may be placed in the same position for measurement after firing. Mark cylindrical test pieces adjacent to the diagonal mark.

Measure the length of the test piece in four positions, to an accuracy of 0,01 mm, by moving the carrier with the test piece over the base plate. For rectangular test pieces, the four positions are located on the diagonals, between 20 mm and 25 mm from each corner. For cylindrical test pieces, the positions are 10 mm to 15 mm from the perimeter, on two diameters at right angles.

Record each measuring point.

7.2.2 *Linear measurement by vernier callipers (Method 2)*

Measure the length of the test piece using the vernier callipers (**5.4.3**) to an accuracy of ± 0,02 mm. Measure the length at three positions using the measuring points shown in figure 3. Mark the positions at which the measurements are made, either with refractory paint or by cutting small grooves across the edges of the test pieces (see figure 3).

NOTE. Sliding test pieces in and out of the jaws of callipers can lead to wear of the surface of the jaws, although such an effect will be reduced because the length change is calculated by the difference of two measurements made with the same callipers. To minimize such wear, the test pieces should be placed carefully in the open jaws in contact with the upper fixed jaw, and the lower jaw moved up until contact is made.

7.2.3 *Volume measurement (Method 3)*

Determine the bulk density of the test piece in accordance with EN 993-1.

Calculate the bulk volume, V_B, in cubic centimetres, from the measurements taken, using the following equation:

$$V_B = \frac{m_2 - m_1}{\rho_{liq}}$$

where

m_1 is the apparent mass of the immersed test piece in grams;

m_2 is the mass of the soaked test piece in grams;

ρ_{liq} is the density of the immersion liquid in grams per cubic centimetre.

7.3 Mounting of test pieces in the furnace

Place the test pieces in the furnace (**5.1**), each one resting on one of its 50 mm × 50 mm faces (for prisms) or on end (for cylinders), and protected from direct radiation in an electrically heated furnace or from the flame of the gas burner in a gas-fired furnace. Do not superimpose test pieces one on another. To allow free circulation of the hot gases, the test pieces shall be separated from each other by a distance of not less than 20 mm, and shall be not nearer than 50 mm to the walls of the furnace.

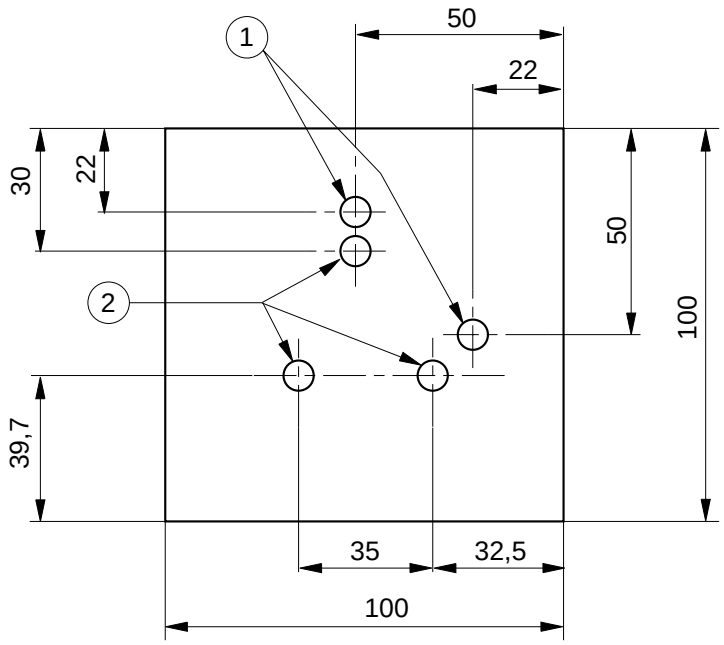
The test pieces shall be placed in the furnace on bricks, 30 mm to 65 mm thick, of the same material as the test pieces, the bricks being laid flat on the apices of two supports of triangular cross-section, 20 mm to 50 mm in height and about 80 mm apart.

7.4 Test temperature

The test temperature shall be 800 °C or a higher temperature, which is a multiple of 50 °C.

7.5 Temperature measurement and distribution

Using at least three thermocouples placed away from the walls of the furnace, away from the heaters and so as not to be in contact with any flames, measure and record the temperature distribution over the limits of the space occupied by the test pieces. The variation in temperature shown between the thermocouples shall not be greater than 10 °C.



Dimensions in millimetres

① Two locating pins ∅ 6

② Three studs ∅ 6

Figure 2. Test piece carrier (Method 1)

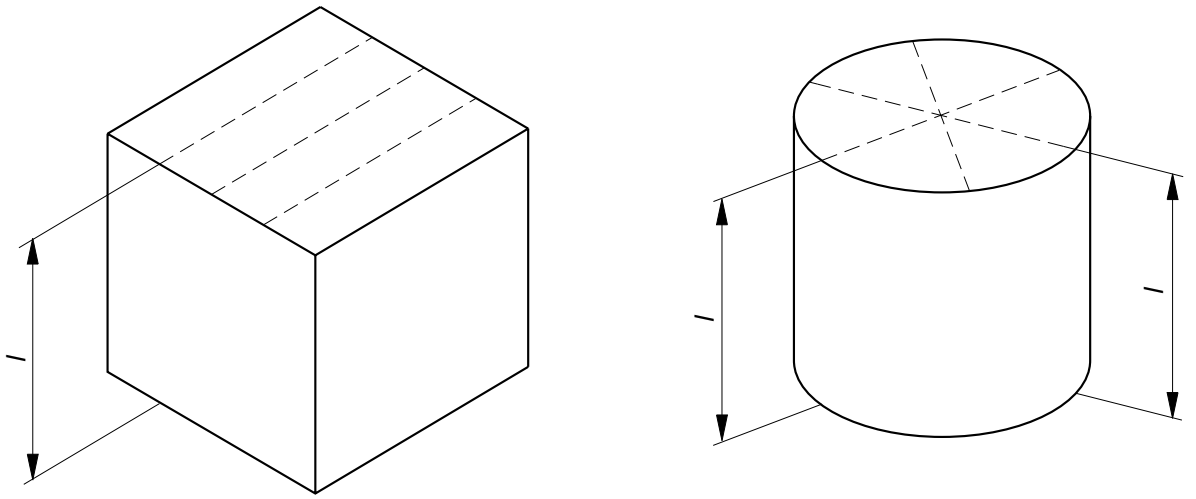


Figure 3. Measuring points when using vernier callipers (Method 2)

7.6 Heating

Raise the temperature in the furnace at one of the following rates:

a) for test temperatures up to 1 250 °C:

– from ambient temperature up to 50 °C below the test temperature: between 5 °C/min and 10 °C/min;

– for the last 50 °C: between 1 °C/min and 5 °C/min.

NOTE. For the last 50 °C, rates of temperature increase between 1 °C/min and 2 °C/min are preferred.

b) for test temperatures above 1 250 °C (electric furnace) or from 1 250 °C to 1 500 °C (gas furnace)

– from ambient temperature up to 1 200 °C: between 5 °C/min and 10 °C/min;

– from 1 200 °C up to 50 °C below the test temperature: between 2 °C/min and 5 °C/min;

– for the last 50 °C: between 1 °C/min and 5 °C/min (see preceding Note).

c) for temperatures greater than or equal to 1500 °C (gas furnace)

– from ambient temperature up to 1200 °C: between 5 °C/min and 20 °C/min;

– from 1200 °C up to 50 °C below the test temperature: between 2 °C/min and 5 °C/min;

– for the last 50 °C: between 1 °C/min and 2 °C/min.

7.7 Maintenance of test temperature

Maintain the temperature recorded on each of the three thermocouples (**5.2**) to within $\pm$ 10 °C of the test temperature for a period of 5 h. Record the mean of these three temperatures as the actual test temperature.

NOTE. If required, further tests may be carried out for periods of 12 h or 24 h.

7.8 Sampling of furnace atmosphere

Sample the atmosphere of gas-fired furnaces in the vicinity of the test pieces at some time during the heating period specified in **7.7**, and determine its oxygen content.

7.9 Cooling

Switch off the furnace and allow it to cool at its natural rate, the test pieces being allowed to cool in the furnace.

7.10 Measurement of test pieces after firing

7.10.1 *Linear measurement (Methods 1 and 2)*

Examine the test pieces, noting particularly any blisters or accretions products during firing. If any of the measurement points might be affected by such a defect, measure instead at the nearest point unaffected. If necessary, rotate the test piece to avoid contact between defects and any of the three supports of the measuring device.

Subject to these restrictions, measure the length of the test piece at each of the four positions specified in **7.2.1** and each of the positions in **7.2.2**, using the same method and measuring device (**5.4**) as for the original measurement.

7.10.2 *Volume measurement (Method 3)*

Measure the volume of the test piece in accordance with **7.2.3**.

8 Expression of results

Express the permanent change in dimensions either as the change in length (Methods 1 and 2), or as the change in volume (Method 3). Calculate the change in the relevant quantity, ΔL, or ΔV, as a percentage of the original value L_o or V_o, i.e. 100 $\Delta L/L_o$ or 100 $\Delta V/V_o$. Calculate the change in length for each measuring point.

Report increases in length or volume as positive (+), and decreases as negative (−).

Report the individual values calculated for each test piece, together with the mean value.

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 European Standard, i.e. in accordance with EN 993-10.

d) a reference to the method used (Method 1, 2 or 3);

e) description of the material tested (manufacturer and type, batch number);

f) the number of items tested (see clause **6**);

g) the number of test pieces per item or brick (see clause **6**);

h) the dimensions of the test pieces and their positions in the brick (see clause **6**);

i) the type of furnace used (see **5.1**);

j) the oxygen content of the furnace atmosphere, if required (see **7.8**);

k) the heating schedule used (see **7.6**);

l) the nominal test temperature (see **7.4**);

m) the actual mean temperature (see **7.7**);

n) the period at the actual mean temperature (see **7.7**);

o) the appearance of the test pieces after heating (see **7.10.1**);

p) the individual values and the mean value of the percentage linear or volume change (and whether positive or negative) for each test piece.

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