SLOVENSKI STANDARD
oSIST prEN 993-10:2019

01-december-2019

Metode za preskušanje gostih oblikovanih ognjevzdržnih izdelkov - 10. del: Ugotavljanje trajnih sprememb mer pri segrevanju

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

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

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

Ta slovenski standard je istoveten z: **prEN 993-10**

ICS:

81.080	Ognjevzdržni materiali	Refractories

oSIST prEN 993-10:2019 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 993-10

September 2019

ICS

Will supersede EN 993-10:1997

English Version

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

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 187.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. prEN 993-10:2019 E

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European foreword

This document (prEN 993-10:2019) has been prepared by Technical Committee CEN/TC 187 “Refractory products and materials”, the secretariat of which is held by BSI.

This document is currently submitted to the enquiry.

This document will supersede EN 993-10:1997.

Reproducibility and repeatability data are available only for a limited number of testing methods and materials, but may be complemented in subsequent edition.

The series of standards EN 993 'Methods of test for dense shaped refractory products' consists of 20 Parts, some of which have been withdrawn and replaced by equivalent standards:

— *Part 1: Determination of bulk density and porosity*

— *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, ambient temperatures*

— *Part 7: Determination of modulus of rupture, elevated temperatures*

— *Part 8: Determination of refractoriness-under-load – withdrawn – replaced by EN ISO 1893*

— *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) – withdrawn – replaced by EN ISO 8894-1*

— *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)*

— *Part 19: Determination of thermal expansion by a differential method*

— *Part 20: Determination of resistance to abrasion at ambient temperature – withdrawn – replaced by EN ISO 16282*

1 Scope

This document specifies 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

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.

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

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

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

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
permanent change in dimensions on heating
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 product
product with specific dimensions, having a true porosity of less than 45 % by volume, when measured in accordance with EN 993-1

3.3
sample
representative collection of items that can be obtained by sampling in accordance with ISO 5022

3.4
item
refractory brick or shape

3.5
test piece
piece of material extracted from an item (3.12) and suitably shaped and prepared for the test

4 Principle

Test pieces in the shape of rectangular prisms or cylinders are cut from each 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.

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 (see 5.2), so that a continuous record of the temperature is obtained. Temperature shall be recorded with a minimum accuracy of +- 5°C.

5.4 Length measuring device.

5.4.1 General

Two measuring devices may be used, either the dial gauge apparatus (method 1) or callipers (method 2).

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 Callipers, (Method 2), in accordance with ISO 13385-1, with a minimum accuracy of ± 0,02 mm.

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

6 Test pieces

At least four items form a sample. In the case of acceptance testing, the number of items forming the sample to be tested shall be determined according to ISO 5022.

The number of test pieces to be cut from each item is a matter of agreement between the interested parties and shall be stated in the test report (see Clause 9). In order to facilitate statistical analysis, the same number of test pieces shall be cut from each item.

It is recommended that only one test piece located near the geometrical centre of the item should be taken from each item.

Test pieces shall be in the form of either:

a) cylinders, 50 mm diameter and 60 mm length.

b) rectangular prisms, 50 mm x 50 mm x 60 mm.

Tolerances on all dimensions shall be ± 2 mm.

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 item shall be recorded. The 50 mm x 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 the test piece in the drying chamber (see 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 (see 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 the nearest 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 (see 5.4.3) to the nearest 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).

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:

$$V_b = \frac{m_2 - m_1}{\rho_{liq}} \quad (1)$$

where

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

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

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

7.3 Determination of apparent mass of immersed test piece (m_2)

Place the test pieces in the furnace (see 5.1), each one resting on one of its 50 mm x 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.

7.6 Heating

Depending on the type of refractory tested, variation in the heating schedule of the test pieces might statistically affect permanent linear change values obtained. In case of dispute the minimum heating rate at each heating step shall be followed.

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

a) for test temperatures up to 1250 °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 1250 °C (electric furnace)

— from ambient temperature up to 1 200 °C: between 5 °C/min and 10 °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 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 (see 5.2) to within ± 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 three positions in 7.2.2, using the same method and measuring device (see 5.4) as for the original measurement.