SLOVENSKI STANDARD
SIST EN 993-17:2000

01-september-2000

Metode za preskušanje gostih oblikovanih ognjevzdržnih izdelkov - 17. del: Ugotavljanje prostorninske mase podrobljenih materialov z metodo z živim srebrom v vakuumu

Methods of test for dense shaped refractory products - Part 17: Determination of bulk density of granular materials by the mercury method with vacuum

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 17: Bestimmung der Rohdichte an körnigem Gut nach dem Quecksilber-Verdrängungsverfahren unter Vakuum

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 17: Détermination de la masse volumique apparente des matériaux en grains par la méthode au mercure sous vide

Ta slovenski standard je istoveten z: **EN 993-17:1998**

ICS:

81.080	Ognjevzdržni materiali	Refractories

SIST EN 993-17:2000 **en**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 993-17

December 1998

ICS

Descriptors: refractory materials, shaped refractories, dense shaped refractory products, tests, determination, measurements, density (mass/volume), bulk density, granular materials, pycnometric analysis, mercury, procedures

English version

Methods of test for dense shaped refractory products - Part 17: Determination of bulk density of granular materials by the mercury method with vacuum

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 17: Détermination de la masse volumique apparente des matériaux en grains par la méthode au mercure sous vide

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 17: Bestimmung der Rohdichte an körnigem Gut nach dem Quecksilber-Verdrängungsverfahren unter Vakuum

This European Standard was approved by CEN on 28 November 1998.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

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

Contents

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 June 1999, and conflicting national standards shall be withdrawn at the latest by June 1999.

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

Part 1 : Determination of bulk density, apparent porosity and true 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 of rupture at ambient temperature
Part 7 : Determination of modulus of 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 (refractoriness)
Part 13 : Specification for pyrometric reference cones for laboratory use
Part 14 : Determination of thermal conductivity by the hot wire (cross-array) method
Part 15 : Determination of thermal conductivity by the hot wire (parallel) method
Part 16 : Determination of resistance to sulfuric acids
Part 17 : Determination of bulk density of granular materials by the mercury method with vacuum
Part 18 : Determination of bulk density of granular materials by the water method with vacuum

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.

1 Scope

This European Standard specifies the determination of the bulk density of granular refractory materials (grain bulk density) having a grain size greater than 2 mm, by the mercury method with vacuum.

NOTE 1: This method is intended as the reference method because of its reproducibility and simplicity in use. However, mercury is known to be a hazardous substance, and therefore the determination of bulk density of granular materials can be carried out according to prEN 993-18. This standard, which defines a method by water with vacuum, is recommended for all routine purposes. Nevertheless, depending on the nature of the material tested, the two methods can give different results.

NOTE 2: Under test conditions, applying a mercury pressure of 26,5 kPa, round pores with a diameter ≥ 55 µm and elongated pores with a width ≥ 27,5 µm are penetrated by mercury.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the 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.

ISO 383	Laboratory glassware - Interchangeable conical ground joints.
ISO 565	Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings

3 Definitions

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

3.1 bulk density of a granular material (grain bulk density)

The ratio of the mass of a quantity of the material to the total volume of its grains, including the volume of any closed pores within the grains.

3.2 closed pores

Pores that are not penetrated under the conditions of this test when grains are immersed in mercury.

4 Principle

The measurement of the mass of a test sample previously crushed to the required grain size, and the determination of its volume by the mercury displacement method with vacuum below 3 000 Pa residual pressure and preferably with a residual pressure of 133 Pa.

5 Apparatus

5.1 Vacuum pycnometer, a vessel as shown in figure 1 (incorporating conical ground glass joint in accordance with ISO 383).

5.2 Test arrangement, for filling and emptying the pycnometer under a vacuum. A suitable test arrangement is shown in figure 2.

5.3 Vacuum pump, capable of reducing the pressure to a value not more than 3000 Pa.

5.4 Balance, with an accuracy of ± 0,1 g and a graduation of 0,01 g.

5.5 Dessicator

5.6 Oven, capable of being controlled to 110 °C ± 5 °C

5.7 Sieves, of nominal aperture of 2 mm and 5,6 mm in accordance with ISO 565.

6 Test samples

6.1 Preparation of test samples

The material to be tested shall consist of fractions or groups of fractions of grains. However, laboratory samples shall be produced from these fractions by sieving and comminution to obtain a grain size between 2 mm and 5,6 mm.

NOTE: Any dust or loose particles adhering to the grains should be removed before testing by washing or, with materials sensitive to moisture or humidity, by air blowing.

6.2 Number of test samples

Take at least three test samples from the laboratory sample and carry out one determination of bulk density on each test sample.

6.3 Mass of test samples

The mass of the test samples depends on the grain size and homogeneity of the material being tested. A mass of 100 g is recommended.

NOTE: For inhomogeneous samples, the mass of the test sample may be increased to 200 g.

Dry the test sample to constant mass in the drying oven maintained at 110 °C ± 5 °C and allow it to cool to ambient temperature in the desiccator. Weigh the test sample to the nearest 0,1 g on the balance. This gives the mass, in grams, of the dried test sample (m_P).

7 Procedure

7.1 Measurement of ambient temperature

The temperature at which the test is carried out shall be measured before and after the series of sample determinations.

NOTE: Operational details of the test arrangement described below refer to figure 2.

7.2 Determination of the volume of the test sample (V_R)

7.2.1 Determination of mass of empty vacuum pycnometer (m_L)

Clean and dry the empty vacuum pycnometer (see 5.1) and weigh it to the nearest 0,1 g. This gives the mass in grams of the empty pycnometer (m_L).

NOTE: This weighing can be omitted if all the determinations are carried out at temperature which does not vary by more than ± 2 °C.

7.2.2 Determination of mass of pycnometer filled with mercury (m_G)

Fill the mercury dish (1) with sufficient mercury from the reservoir (11) to fill the pycnometer. With stopcock (2) closed and stopcock (5) and tap (12) open, evacuate the pycnometer to a residual pressure of below 3000 Pa, as indicated by the manometer (13). Open stopcock (2) and allow the pycnometer to fill with mercury until mercury emerges from the capillary tube (7). Close stopcocks (2) and (5) in that order and remove the pycnometer from the test arrangement. Pour off the surplus of mercury that has come out of the capillary tube and remove the mercury remaining in the suction tube below stopcock (2) and in the capillary above stopcock (5) using a steel wire. Weigh the pycnometer to the nearest 0,1 g.

This gives the mass, in grams, of the pycnometer filled with mercury (m_G).

NOTE: If all the determinations are carried out at a temperature which does not vary by more than ± 2 °C, this weighing needs to be carried out only once per series of sample determinations.

7.2.3 Determination of mass of pycnometer containing test sample and filled with mercury (m_T)

Empty and dry the pycnometer.

NOTE 1: This can be achieved quickly by fitting the pycnometer to the mercury extraction bush (10) and evacuating the mercury reservoir (11).

Transfer the dried and weighed test sample (see 6.3), without loss, into the pycnometer and fill the pycnometer, under vacuum, as specified in 7.2.2. This will give an average pressure on the grains of about 26,5 kPa. Remove the pycnometer from the test arrangement, again as specified in 7.2.2 and weigh the pycnometer and contents to the nearest 0,1 g. This gives the mass of the pycnometer containing the test sample and filled with mercury (m_T).

Fit the pycnometer to the extraction bush (10), close stopcock (16) and evacuate the mercury reservoir (11). Open stopcock (2) and remove the mercury from the pycnometer while under a pressure not exceeding 3000 Pa. Close stopcock (2), remove the pycnometer and by gently shaking, remove any mercury adhering to the test sample. Open stopcock (5) and allow the pycnometer to return to atmospheric pressure. Remove the test sample from the pycnometer.

Determine the amount of mercury still remaining in the test sample by weighing it and finding the difference from its original mass. If the mass of mercury remaining in the test sample is over 5 % of the original mass of the sample, this amount, expressed as a percentage by mass, shall be stated in the test report.

NOTE 2: The stopcocks (2) and (5) should be closed in the order stated and while the pycnometer vessel is still connected to the vacuum system. Closing stopcock (5) after the apparatus has been disconnected from the vacuum system, or reopening stopcock (5) and thereby subjecting the contents to atmospheric pressure, will give an increased impregnation of the grain porosity with mercury and thus a high erroneous result. This situation can also occur if repeated evacuation, filling and release of the system to atmospheric pressure is carried out, and therefore this should also be avoided.