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EUROPEAN STANDARD

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English version

Methods of test for dense shaped refractory products - Part 12: Determination of pyrometric cone equivalent (refractoriness)

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 12: Détermination de la résistance pyroscopique (réfractarité)

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 12: Bestimmung des Kegelfallpunktes (Feuerfestigkeit)

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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

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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.

It is closely based on the text of the International Standard ISO 528 : 1983 'Refractory products - Determination of pyrometric cone equivalent (refractoriness).

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, 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
- Part 13 : Specification for pyrometric reference 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)

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 September 1997, and conflicting national standards shall be withdrawn at the latest by September 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, 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 a method for determining the pyrometric cone equivalent (refractoriness) for refractory materials including shaped and unshaped products. Its useful range of application depends on the availability of suitable pyrometric reference cones. In the temperature range from 1500 °C to 1800 °C, the pyrometric reference cones specified in EN 993-13 are suitable.

As pyrometric reference cones are primarily manufactured for estimating the effect of temperature on siliceous, low alumina fireclay and fireclay products, the results on materials other than these may not be as precise.

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.

EN 993-13:1995 Methods of test for dense shaped refractory products - Part 13 :
Specification for pyrometric reference cones for laboratory use

ISO 565 Test sieves - Woven metal wire cloth and perforated plate and
electroformed sheet - Nominal sizes of apertures.

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3 Definitions

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

3.1 refractoriness : The characteristic property of a material that allows it to withstand high temperature.

3.2 pyrometric reference cone : A blunt-tipped skew triangular pyramid with sharp edges, of specified shape and dimensions and of such composition that when mounted and heated under specified conditions, it bends in a known manner with reference to the temperature.

3.3 reference temperature, or temperature of collapse : The temperature at which the tip of a pyrometric reference cone reaches the level on which the base of the cone is mounted when the cone is heated at a specified rate under specified conditions.

4 Principle

Test pieces of refractory materials or products are raised in temperature, under specified conditions, alongside pyrometric reference cones of known refractoriness and their behaviour is compared with that of the reference cones.

5 Apparatus

5.1 Furnace

5.1.1 The furnace used for the determination may be cylindrical or rectangular, and either vertical or horizontal.

5.1.2 Under the test conditions, the difference in temperature between the coldest spot and the hottest spot of the space occupied by the stand, the test pieces and the pyrometric reference cones shall not exceed 10 °C (equivalent to approximately one-half of a reference cone number, see EN 993-13.)

NOTE 1 : In order that this requirement should be met when pyrometric reference cones of height 30 mm are used (the conventional height), a cylindrical furnace should have a chamber of at least 80 mm useful diameter and a rectangular furnace should have a chamber at least 60 mm in height and 100 mm in width.

NOTE 2 : In order to minimize the effects of any irregular temperature distribution in the furnace, it may be convenient to arrange for the stand to be kept in motion relative to the furnace during the determination, e.g. by rotating the stand about a vertical axis.

The uniformity of temperature shall be verified from time to time.

NOTE 3 : The uniformity can be measured by means of thermocouples or by the use of pyrometric reference cones.

5.1.3 The furnace shall be capable of reaching the required temperature at the rates of increase specified in 9.2 and 9.3.

5.1.4 The atmosphere in the furnace shall contain free oxygen at all times.

NOTE: Certain furnaces (for example, certain types of furnaces fired with hydrocarbon gas and oxygen) are not suitable for this method of determination because of the high content of reducing gases or water vapour in their atmospheres.

5.1.5 In the case of a gas-fired furnace, complying with 5.1.4, the pyrometric reference cones and the test pieces shall be protected from any direct action of the flame and from any turbulence of the hot gases.

5.2 Pyrometric reference cones

5.2.1 The pyrometric reference cones used should preferably conform to the requirements of EN 993-13.

5.2.2 Alternatively, other pyrometric cones may be used, provided that:

- a) the manufacturer of the cones has stated their respective reference temperatures;

- b) the cones conform to those reference temperatures within the tolerance permitted in EN 993-13;

NOTE : This condition may be deemed to be satisfied if the manufacturer of the cones has declared that they conform to this tolerance; otherwise, they should be tested in accordance with clause 7 of EN 993-13:1995.

- c) The manufacturer of the cones has specified the angle at which the leading edge is to be mounted (see 8.2) and the rates at which the cones are to be heated (see 9.3);
- d) if the authority carrying out the determination is distinct from that for whom it is carried out, the type of reference cone to be used shall be agreed between the parties concerned.

5.3 Stand for the pyrometric reference cones and the test pieces

5.3.1 The refractory stand for the pyrometric reference cones and the test pieces consists, depending upon the type of furnace used, of a rectangular plate or a disc of refractory material having adequately plane and parallel faces.

5.3.2 The stand and the refractory cement used for securing the pyrometric reference cones and the test pieces to the stand shall be such that up to the test temperature there is no reaction between these items and the pyrometric reference cones or the test pieces.

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6 Test pieces

6.1 Size and shape

Each test piece shall have a shape geometrically similar to that of the pyrometric reference cones being used. Each test piece shall have a height not less than 100 % and not more than 120 % of the height of the reference cones being used.

6.2 Preparation of test pieces

6.2.1 General

Where possible, test pieces from shaped and pre-fired unshaped products shall be cut in accordance with 6.2.2. If test pieces cannot be cut, they shall be moulded from ground material in accordance with 6.2.3. Test pieces from powder and granular materials shall always be moulded in accordance with 6.2.3.

6.2.2 Cut test pieces

6.2.2.1 Test pieces from bricks and shapes shall be cut with a saw and dressed with a grinding wheel. Any skin of fired materials shall be removed.

6.2.2.2 Samples of unshaped materials such as plastic refractories, ramming materials, refractory mortars and refractory castables shall be shaped and fired in a manner appropriate to the material and its condition of use; the firing temperature shall be stated in the test report. Test pieces shall then be cut from the fired material with a saw and dressed with a grinding wheel. Any skin of fired material shall be removed.

6.2.2.3 In preparing a cut test piece (see 6.2.2.1 and 6.2.2.2), it is advisable, as a first step, to cut a rectangular prism of a suitable size (15 mm x 15 mm x 40 mm for a test piece of 30 mm height) and, if the refractory material has a coarse or friable texture, to impregnate it with a suitable resin giving less than 0,5 % ash (e.g. Canada balsam). This rectangular prism is then cut and dressed.

6.2.3 Moulded test pieces

6.2.3.1 Test pieces for raw materials and prepared unshaped refractory materials, and for those shaped refractory products from which test pieces cannot be cut in accordance with 6.2.2, shall be prepared in accordance with 6.2.3.2 to 6.2.3.6.

6.2.3.2 Crush the sample or samples selected so that all the material passes through a test sieve with nominal aperture 2 mm and conforming to the requirements of ISO 565. Using a procedure agreed between the parties to the test, reduce the quantity of test material to that appropriate for the preparation of the number of test pieces required. Grind the reduced quantity of sample in a suitable mortar until it entirely passes through a test sieve of nominal aperture 180 µm conforming to the requirements of ISO 565. During this grinding, sieve the material frequently in order not to produce an excess of very fine powder.

NOTE : Less than 50 % of the ground sample should pass through a test sieve with nominal aperture size 90 µm, except in the case of raw materials containing, as received, a percentage of fines larger than 50 %.

6.2.3.3 At all stages, crushing and grinding should be carried out so as to avoid the introduction of extraneous material. At all stages, mixing should be carefully carried out so that the contents of the test pieces are truly representative of the samples.

6.2.3.4 Knead the powdered sample with water to which has been added, if the test material is lean, an organic binder having a maximum of 0,5 % ash content. If the test material reacts with water, use another suitable liquid instead of water.

6.2.3.5 Mould the test pieces in suitable moulds.

NOTE : A suitable mould is illustrated in the annex A.

6.2.3.6 Test pieces prepared from raw materials that are subject to considerable modification during reheating shall then be stabilized by heating before their refractoriness is determined. In particular, clays should be calcined at approximately 1 000 °C; after calcination, the test pieces shall comply with the requirements of 6.1.