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Gosti oblikovani ognjevzdržni izdelki - Preskusne metode - 16. del: Ugotavljanje odpornosti proti žvepleni kislini

Dense shaped refractory products - Methods of test - Part 16: Determination of resistance to sulphuric acid

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 16: Bestimmung der Beständigkeit gegen Schwefelsäure

Produits réfractaires façonnés denses - Méthode d'essai - Partie 16: Détermination de la résistance a l'acide sulfurique

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Dense shaped refractory products - Methods of test - Part 16: Determination of resistance to sulphuric acid

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CEN

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Contents

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 187 "Refractory products and materials", of which the secretariat 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of an identical text or by endorsement, at the latest by February 1996, and conflicting national standards shall be withdrawn at the latest by February 1996.

According to the CEN/CENELEC Internal Regulations, 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.

Reproducibility and repeatability data are not available, but may be given 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
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, ambient temperatures
Part 7 :	Determination of modulus of rupture, 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 of 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)

1 Scope

This Part of EN 993 specifies a method for determining the resistance of dense shaped refractory products to attack by sulfuric acid.

NOTE : Sulfuric acid is used since it gives results which are typical of the results of exposing refractory materials to many acids (other than hydrofluoric acid).

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 are 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 : 1976	Laboratory glassware - Interchangeable conical ground joints.
ISO 565 : 1990	Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings.
ISO 1770 : 1981	Solid-stem general purpose thermometers.
ISO 1773 : 1976	Laboratory glassware - Boiling flasks (narrow-necked).
ISO 4799 : 1978	Laboratory glassware - Condensers.

3 Principle

The test sample, crushed in a specified manner, is subjected for 6 h to attack by 70 % (*m/m*) boiling sulfuric acid, and the resultant mass loss is determined and expressed as a percentage of the initial mass of the dry material.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Suitable mechanical crushing device, preferably not steel.

4.2 Woven metal wire cloth sieve, 0,80 mm aperture, conforming to the requirements of ISO 565 : 1990.

4.3 Woven metal wire cloth sieve, 0,63 mm aperture, conforming to the requirements of ISO 565 : 1990.

4.4 Balance, capable of weighing 25 g the nearest 0,001 g.

4.5 Round-bottomed flasks, of capacity 500 ml (see ISO 1773 : 1976), each with a short, narrow neck, equipped with a ground glass stopper (see ISO 383 : 1976) for the insertion of a thermometer.

4.6 Coil condensers, of length 250 mm, having at least 16 turns in the coil (see ISO 4799 : 1978).

4.7 Immersion thermometers (length approximately 110 mm) (see ISO 1770 : 1981).

4.8 Sand bath, oil bath, or heating mantle.

4.9 Porcelain filter crucible, with a maximum pore size of 7 μm.

4.10 Thermostatically controlled drying oven, capable of maintaining a temperature of (110 ± 5) °C.

4.11 Desiccator.

5 Reagents

5.1 General. Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.2 Sulfuric acid, 70 % (*m/m*), ρ_{15} = 1,615 g/cm^3.

5.3 Barium chloride, 50 g/l solution.

6 Test samples

6.1 From each sample, take two pieces of a total mass of approximately 250 g, one from the centre and one from an edge.

6.2 Grind the two pieces together using the crushing device (see 4.1), sieving the material several times and re-grinding the residues until all the test material has passed through the 0,80 mm aperture sieve (see 4,2). If a steel mortar has been used, pass a magnet over the ground sample to remove any metallic contamination.

NOTE : The material of the mortar should be harder than the test material.

6.3 Sieve the material through the 0,63 mm aperture sieve (see 4.3). Clear the residue on the sieve of all the dust particles by washing with distilled water. This residue forms the test material.

NOTE : A systematic error will be introduced if grain sizes are used other than between the limits set in 6.2 and 6.3.

6.4 Dry the residue on the 0,63 mm aperture sieve in the drying oven (see 4.10), controlled at (110 ± 5) °C, until constant mass is reached. Before each weighing, allow the sieve with contents to cool to ambient temperature in the desiccator. Weigh to the nearest ± 0,001 g.

7 Procedure

7.1 Carry out at least two tests in parallel.

7.2 For each test, weigh, to the nearest 0,001 g, about 20 g of the dried material (mass m_1).

7.3 Place the weighed test material in a round-bottomed flask (see 4.5) and cover it with 200 ml of the sulfuric acid (see 5.1). Attach a coil condenser (see 4.6) and insert a thermometer (see 4.7) so that it is immersed at least 15 mm in the liquid.

7.4 Over a period of about 30 min, bring the contents of the flask to the boiling point in the sand or oil bath or heating mantle (see 4.8).

NOTE : The boiling point of 70 % (*m/m*) sulfuric acid is approximately 170 °C.

Record the temperature of the liquid after boiling has begun.

7.5 Keep the liquid boiling lightly for a period of 6 h. Note the temperature of the liquid at the end of this time.

7.6 After the liquid has boiled for 6 h, take the flask out of the bath or heating mantle and allow it to cool for 1 h. Decant the clear acid floating on top of the sample. Cautiously pour in approximately 500 ml of distilled water, and wash the whole contents of the flask into the porcelain filter crucible (see 4.9), previously dried and weighed to the nearest 0,001 g, using an aspirator to aid filtering. Wash the residue in the filter crucible with distilled water until the filtrate remains unclouded when a few drops of the barium chloride solution (see 5.2) are added.

7.7 Dry the crucible containing the residue in the oven, controlled at (110 ± 5) °C, until constant mass is reached. Before each weighing, allow the crucible and its contents to cool to ambient temperature in the desiccator.

Weigh the crucible and its contents to the nearest 0,001 g. Subtract the mass of the crucible to obtain the mass of the residue, M_2.

8 Expression of results

Calculate the loss of mass of the test material, L %, as a percentage of its initial mass, using the formula

$$L = \frac{m_1 - m_2}{m_1} \times 100$$

where

m_1 is the initial mass, in g, of the test material;

m_2 the mass, in g, of the residue.