SLOVENSKI STANDARD
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Metode za preskuġanje gostih oblikovanih ognjevzdrģnih izdelkov - 15. del: Ugotavljanje toplotne prevodnosti z metodo vroĮ e ģice (vzporedne ģice)

Methods of test for dense shaped refractory products - Part 15: Determination of thermal conductivity by the hot-wire (parallel) method

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse -Teil 15: Bestimmung der Wärmeleitfähigkeit nach dem Heißdraht-(Parallel-) Verfahren

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 15: Détermination de la conductivité thermique par la méthode du fil chaud (parallele)

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This European Standard was approved by CEN on 23 March 1998.

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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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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.

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of an identical text or by endorsement, at the latest by October 1998, and conflicting national standards shall be withdrawn at the latest by October 1998.

It is closely based on the corresponding International Standard, ISO 8894-2 "Refractory materials - Determination of thermal conductivity : Part 2 Hot-wire method (parallel)", published by the International Organization for Standardization (ISO).

The determination of thermal conductivity by the hot-wire (cross-array) method is given in EN 993-14.

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, 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, 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 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 acids
Part 17 : Determination of bulk density of granular material (mercury method)
Part 18 : Determination of bulk density of granular material (water method)

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 Part of EN 993 specifies a hot-wire (parallel) method for the determination of thermal conductivity of refractory products and materials. It is applicable to dense and insulating shaped products and to powdered or granular materials (see 7.2), for thermal conductivities of less than 25 W/m·K. The limits are imposed by the dimensions of the test pieces and higher thermal conductivities can be measured if larger pieces are used. Electrically conducting materials cannot be measured.

NOTE 1 : The thermal conductivity of bonded bricks can be affected by the appreciable amount of water that is retained after hardening or setting and is released on firing. These materials may therefore require pre-treatment; the nature and extent of such pre-treatment and the period for which the test piece is held at the measurement temperature, as a preliminary to carrying out the test, are details that are outside the scope of this Part of EN 993 and should be agreed between the parties concerned.

NOTE 2 : In general it is difficult to make measurements on anisotropic materials and the use of this method for such materials should also be agreed between the parties concerned.

2 Normative references

This European Standard incorporates by dated or undated references, 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.

EN 993-14	Methods of test for dense shaped refractory products - Determination of thermal conductivity by the hot wire (cross-array) method
ISO 31-4	Specification for quantities, units and symbols. Heat

3 Definitions

For the purposes of this standard, the following definitions, in accordance with EN 993-14, and ISO 31-4 apply:

3.1 thermal conductivity, λ: density of heat flow rate divided by the temperature gradient, in units of watt per metre kelvin (W/m·K).

3.2 thermal diffusivity, α: $\alpha = \dfrac{\lambda}{\rho . C_p}$

where:

λ is the thermal conductivity
ρ is the bulk density
C_p is the specific heat capacity at constant pressure

Thermal diffusivity is expressed in units of square metre per second ($m^2 \cdot s^{-1}$).

3.3 power, *P*: Rate of energy transfer, in units of watts (W).

4 Principle

The hot-wire method (parallel) is a dynamic measuring procedure based on the determination of the temperature increase against time at a certain location and at a specified distance from a linear heat source embedded between two test pieces.

The test pieces are heated in a furnace to a specified temperature and maintained at that temperature. Further local heating is provided by a linear electrical conductor (the hot wire) that is embedded in the test piece and carries an electrical current of known power that is constant in time and along the length of the test piece.

A thermocouple is fitted at a specified distance from the hot wire, the thermocouple leads running parallel to the wire (see figure 1). The increase in temperature as a function of time, measured from the moment the heating current is switched on, is a measure of the thermal conductivity of the material from which the test pieces are made.

5 Apparatus

5.1 Furnace, electrically heated, capable of taking one or more test assemblies (see 6.1) up to a maximum temperature of 1250 °C. The temperature at any two points in the region occupied by the test pieces shall not differ by more than 10 °C. The temperature measured on the outside of the test assembly during a test (of duration about 15 min) shall not vary by more than ± 0,5 °C, and shall be known with an accuracy of ± 5 °C.

5.2 Hot wire, preferably of platinum or platinum-rhodium, about 200 mm in length and not exceeding 0,5 mm in diameter, the actual length being known to within ± 0,5 mm.

NOTE : A hot wire made of base metal can be used at temperatures below 1000 °C.

Both ends of the hot wire are attached to the power source and the digital multimeter (see 5.5). The wires to the power source may also be a continuation of the hot wire itself and shall have the same diameter as the wire within the assembly. The wires to the digital multimeter shall be of a diameter not greater than that of the hot wire when within the assembly. Leads outside the assembly shall consist of two or more tightly twisted wires of 0,5 mm diameter. The current lead connections external to the furnace shall be made with heavy gauge cable.

5.3 Power supply, to the hot wire (see 5.2), which shall be stabilized a.c. or d.c. and shall not vary in power by more than 2 % during the period of measurement. A supply to the hot wire of at least 80 W is required.

NOTE : This is equivalent to 400 W/m for a 200 mm long wire.

5.4 Differential platinum/platinum-rhodium thermocouple (Type R: platinum 13 % rhodium/platinum thermocouple or Type S: platinum 10% rhodium/platinum thermocouple, see table 1) formed from a measurement thermocouple and a reference thermocouple connected in opposition (see figure 1). The leads of the measurement thermocouple shall run parallel to the hot wire at a distance of 15 mm ± 1 mm (see figure 2). The output of the reference thermocouple shall be kept stable by placing it between the top outer face of the upper test piece and a cover of the same material as the test piece (see figure 1). The diameter of the thermocouple wires shall be the same as that of the hot wire and the thermocouple wires shall be long enough to extend outside the furnace where connections to the measuring apparatus shall be made by wire of a different type. The external connections of the thermocouple shall be isothermal.

NOTE 1 : An insulating layer can be inserted between the cover and the upper test piece.

NOTE 2 : Base metal thermocouples can be used at temperatures below 1000 °C

5.5 Digital multimeter, for measuring the current in the hot wire and the voltage drop across it, and capable of measuring both to an accuracy of at least ± 0,5 %.

5.6 Data acquisition system, consisting of a temperature-time registration device with a sensitivity of at least 2 μV/cm or 0,05 μV/Digit, or a temperature measurement of 0,05 °C with a time resolution better than 0,5 s.

5.7 Containers (for use if the test is performed on powdered or granular material), having internal dimensions equal to those of the solid test assembly specified in clause 6, so that the test assembly shall consist of two sections as specified in 6.1. The bottom container shall have four sides and a base, and the top container shall have four sides only, plus a detachable cover (see figure 3).

NOTE : Containers should be of a material that will not react with the test piece at the temperature and should not be electrically conducting.

6 Test pieces

6.1 Dimensions

Each test assembly shall consist of two identical test pieces, not less than 200 mm x 100 mm x 50 mm in size.

NOTE 1 : It is recommended that the size of each test piece be 230 mm x 114 mm x 64 mm or 230 mm x 114 mm x 76 mm. Standard-size bricks can then be used as the pieces forming the test assembly, subject to the requirements of 6.2.

NOTE 2 : The limits of this method are imposed by the dimensions of the test pieces. With larger test pieces, higher values of thermal conductivity can be measured. The distance between hot wire and thermocouple should be extended to the same ratio as the test pieces. For example, with a test piece 230 mm x 180 mm x 95 mm, a thermal conductivity of 39,5 W/mK can be measured .

6.2 Surface flatness

The surfaces of the two test pieces forming the test assembly which are in contact with each other shall, if necessary, be ground so that the deviation from flatness between two points not less than 100 mm apart is not more than 0,2 mm.

6.3 Groove in dense materials

In dense materials, a groove to accommodate the hot wire and the thermocouple shall be machined in either or both of the contact faces or in the lower face only of the test assembly (see figure 4). The width and depth of the groove shall permit the arrangement shown in figure 4 to be achieved. Grooves shall be machined in both faces of materials of higher conductivity, e.g. greater than 5 W/mK.

7 Procedure

7.1 Arrange the test assembly ready for testing. Place the hot wire (see 5.2) and differential thermocouple (see 5.4) between the two test pieces, with the hot wire along the centreline of the brick faces in contact with each other and cement them into the grooves where appropriate, using a cement made from finely ground test material mixed with a small amount of a suitable binder (e.g. 2 % dextrin and water). Ensure that the wires are cemented evenly, to allow equal heat transfer to the two test pieces, as shown in figure 4.

7.2 If the test is being performed on powdered or granular material, fill the bottom container (see 5.7) with the test material up to its top, and place on it the hot wire and differential thermocouple as shown in figure 1. Place the top container (see 5.7) on the bottom one and fill with the test material. Cover the test assembly with a slab of the same material as that of the containers. Determine the apparent bulk density of the test material in the poured, untamped state.

NOTE : The container can be filled by vibration or by pressing to give a specific bulk density, where a figure has been agreed upon.

7.3 Place the test assembly in the furnace (see 5.1), resting each assembly (to ensure uniform heating) on three supports of a material similar to that being tested and having dimensions of 125 mm x 10 mm x 20 mm. The supports shall rest on a 125 mm x 10 mm face, and be placed parallel to the 114 mm x 76 mm (or 100 mm x 50 mm) faces of the test assembly about 20 mm from these faces.

7.4 Connect the test assembly to the digital multimeter (see 5.5). With the hot-wire circuit open, raise the temperature of the furnace, at not more than 10 °C/min, to the first test temperature required.

NOTE : Heating rates should be low enough to ensure that there is no risk of thermal shock damage.

7.5 Set the power input to a value that (from preliminary tests) is known to produce, for a chosen recorder sensitivity, an instrument deflection of at least 60 %, and preferably about 80 %, of full-scale deflection.

A guide to the choice of power input for a range of thermal conductivities and for a range of recorder sensitivities is given on table 1. The power levels are based on a recorder deflection of 0,8 x full-scale deflection for a given maximum duration of the test (t_{max}) and table 1 also shows the required accuracy for the measurement of time (accuracy t).

NOTE : The appropriate level of power input to the hot wire will differ between apparatus and needs to be evaluated in preliminary tests, but can eventually be based on experience.

7.6 When the furnace reaches the test temperature, verify that the temperature in the region occupied by the test assembly is uniform and constant. The differential thermocouple (see 5.4) shall not show a variation of more than 0,05 °C over a period of 10 min immediately prior to the test.

7.7 When the conditions of 7.6 are met, close the heating circuit and make a record of the output of the differential thermocouple with time. Mark the exact moment the power input to the hot wire was made. If not using an automatically controlled power supply, measure and record the voltage drop across the hot wire and the current in it, immediately after switching on the heating circuit and again at intervals during the test period.

7.8 After the appropriate test duration (see table 1), disconnect the heating circuit and discontinue recording the output of the differential thermocouple.

7.9 Allow time for the hot wire and test assembly to reach temperature equilibrium. Verify the uniformity and constancy of the temperature as specified in 7.6. Repeat the procedures of 7.7 and 7.8, so obtaining a further measurement of the rate of rise of temperature of the hot wire under the same conditions.

7.10 Raise the temperature of the furnace to the next higher test temperature at not more than 10 °C/min. Carry out again the procedure described in 7.5 to 7.9.

7.11 Repeat the procedure of 7.10 until at least two measurements have been obtained at each of the required test temperatures.

8 Assessment of results

8.1 If the current in the hot wire has varied by more than 2 % during the test, the results shall be disregarded and the test shall be carried out again with a current of a smaller value.

8.2 The rise of temperature of the hot wire with respect to time follows a logarithmic law. Recording the temperature rise as a function of time on semi-logarithmic paper should therefore produce a straight-line graph. If this is not the case, either the material does not fulfil the conditions necessary for this test and the results have no significance, or an operating error has been made and the test shall be repeated.

Table 1 : Recommended choice of scales and of power level (based on a deflection of 0,8 x full-scale)

Thermal conductivity λ W/m·K	Maximum duration of test t_{max} s	Accuracy of measurement of t s	Recommended power level (W/m)			
			0 to 20 μV scale	0 to 50 μV scale	0 to 100 μV scale	0 to 200 μV scale
0,1	2 500	4,0			7,5	15
0,4	1 260	2,0		15	30	60
1,0	900	2,0	15	40	75	150
2,0	450	1,0	30	75	150	-
4,0	350	1,0	60	150	300	-
8,0	190	0,4	120	300	-	-
16	100	0,2	240	-	-	-
25	65	0,2	375	-	-	-

Note : The figures in table 1 are based on the use of type 'S' thermocouples (see 5.4), and should be adjusted if a type 'R' thermocouple is used.