International Standard 528

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Refractory products — Determination of pyrometric cone equivalent (refractoriness)

Produits réfractaires — Détermination de la résistance pyroscopique (réfractarité)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 528 was developed by Technical Committee ISO/TC 33, *Refractories*, and was circulated to the member bodies in January 1982.

It has been approved by the member bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Italy	Spain
Brazil	Korea, Rep. of	Sweden
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Egypt, Arab Rep. of	Norway	USA
France	Poland	USSR
Germany, F. R.	Portugal	
Hungary	Romania	

The member body of the following country expressed disapproval of the document on technical grounds :

Canada

This International Standard cancels and replaces ISO Recommendation R 528-1966, of which it constitutes a technical revision.

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Refractory products — Determination of pyrometric cone equivalent (refractoriness)

1 Scope and field of application

1.1 This International Standard specifies a method for determining the pyrometric cone equivalent (refractoriness) of siliceous, low alumina fireclay and fireclay refractory materials and products.

1.2 Its useful range of application depends on the availability of suitable pyrometric reference cones. In the temperature range from 1 500 to 1 800 °C, the pyrometric reference cones specified in ISO/R 1146 are suitable.

1.3 Although pyrometric reference cones are primarily manufactured for estimating the effect of temperature on siliceous, low alumina fireclay and fireclay products with regard to their composition, the method may also be used for determining the refractoriness of other refractory materials and products, but the results may then be not so precise.

2 References

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO/R 836, *Vocabulary for the refractories industry.*

ISO/R 1146, *Pyrometric reference cones.*

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

3 Definitions

3.1 refractoriness : The characteristic property of a material of withstanding high temperature.[1)]

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; 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 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 an ISO reference cone number.[2)] The uniformity of temperature shall be verified from time to time.[3)]

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.[4)]

1) This definition is taken from ISO/R 836.

2) 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.

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

4) 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 flame-heated furnace, 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 ISO/R 1146.

5.2.2 Alternatively, the method of determination specified in this International Standard may be deemed to be satisfied if other pyrometric cones are used, provided that :

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

b) the cones conform[1] to those reference temperatures within the tolerance permitted in ISO/R 1146;

c) the manufacturer of the cones has specified the angle at which the leading edge or leading face is to be mounted (see 8.2) and the rate or 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, they have agreed on the type of reference cones to be used.

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.

5.3.3 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.

6 Test pieces

6.1 Sampling

The number of items (e.g. bricks) or the quantity of material to be tested shall be determined in accordance with a standard sampling procedure agreed between the parties concerned in the test.[2]

6.2 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.

6.3 Preparation of test pieces

6.3.1 General

Where possible, test pieces from shaped and pre-fired unshaped products shall be cut in accordance with 6.3.2. If test pieces cannot be cut, they shall be moulded from ground material in accordance with 6.3.3. Test pieces from powder and granular materials shall always be moulded in accordance with 6.3.3.

6.3.2 Cut test pieces

6.3.2.1 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.

6.3.2.2 Samples of unshaped materials such as plastic refractories, ramming materials, refractory cements and refractory concretes 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.

6.3.2.3 In preparing a cut test piece (see 6.3.2.1 and 6.3.2.2), it is advisable, as a first step, to cut a rectangular prism of a suitable size (15 mm × 15 mm × 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.

6.3.3 Moulded test pieces

6.3.3.1 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 6.3.2, shall be prepared in accordance with 6.3.3.2 to 6.3.3.6.

6.3.3.2 Crush the sample or samples selected in accordance with 6.1 so that all of the material passes through a test sieve with nominal aperture 2 mm and conforming to the requirements of ISO 565. Using a standard 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 an agate mortar until it entirely passes through a test sieve of

1) 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 6 of ISO/R 1146.

2) See, for example, ISO 5022.

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.[1)]

6.3.3.3 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.

6.3.3.4 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.

6.3.3.5 Mould the test pieces in suitable moulds.[2)]

6.3.3.6 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 6.2.

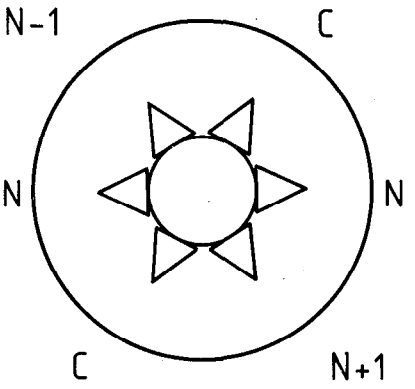
7 Selection of pyrometric reference cones

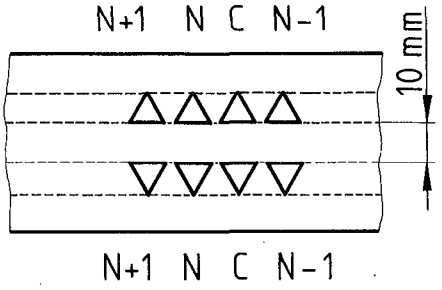
Select pyrometric reference cones in the following numbers :

	For a circular test stand	For a rectangular test stand
a) Corresponding to the estimated or anticipated refractoriness number of the material under test . . .	2	2
b) With a refractoriness number one lower than in a)	1	2
c) With a refractoriness number one higher than in a)	1	2

8 Preparation of test stand

8.1 Place two test pieces and the pyrometric reference cones selected in accordance with 7 on the test stand, arranging them in accordance with one of the diagrams in figure 1, depending upon whether the test stand is circular or rectangular. Allow sufficient room for the cones to be unobstructed when they bend. Fix the base of each cone to the stand with refractory cement.





Symbols	
C	Cone (test cone)
N − 1 N N + 1	Reference pyrometric cones expected to span the setting temperature of the test cone

Figure 1 — Examples of the arrangement of cones on the test stand

1) 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 %.

2) A suitable mould is illustrated in the annex.

8.2 The test pieces and the pyrometric reference cones shall be positioned so that an edge or a face[1] leans outwards at the angle to the vertical specified by the manufacturer of the reference cones (see figure 2).

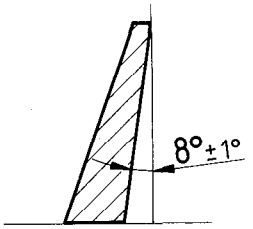


Figure 2 — Pyrometric reference cone showing typical angle of leaning outside edge or face

9 Procedure

9.1 Place the test stand with the test pieces and the pyrometric reference cones attached to it in the uniform temperature zone of the furnace.

9.2 Over a period of 1 1/2 h to 2 h, raise the temperature of the furnace to 200 °C below the estimated refractoriness temperature of the test material.

9.3 Then raise the temperature at a constant mean rate of 2,5 °C/min[2] or, if 5.2.2 applies, at the rate specified by the manufacturer of the cones. Maintain the rate of heating so that at any moment the deviation from the specified temperature rise curve is less than 10 °C.

9.4 Stop the heating as soon as the tip of one of the test pieces touches the stand or, if the test pieces cannot be observed during the test, at a temperature corresponding to the pyrometric resistance of the pyrometric reference cone most nearly corresponding to the estimated or expected refractoriness number of test material, as determined by means of an optical pyrometer or a thermocouple in a preliminary test on a pyrometric reference cone with that refractoriness number.

9.5 Remove the stand from the furnace and note the number of the pyrometric reference cone that has bent over to the same degree as each of the test pieces or, if appropriate, the numbers of the two pyrometric reference cones that have bent over, one a little more and one a little less than each of the test pieces.

9.6 The test shall be repeated if any one or more of the test pieces or pyrometric reference cones have not bent in the normal manner or if the difference in the bending of the two test pieces is greater than one-half of a pyrometric reference cone number.

10 Reporting of results

10.1 The pyrometric cone equivalent of the test material is expressed by the number or numbers of the pyrometric reference cone or cones as described in 9.5.

10.2 The report of the determination shall include :

a the name of the testing establishment;

b) the date of the test;

c) a reference to this International Standard, i.e. "Determination of refractoriness in accordance with ISO 528";

d) the product or material tested (manufacturer, type, batch number, etc.);

e) whether the test piece was cut or moulded;

f) if appropriate, the temperature of any preliminary firing of the test piece (see 6.3.3.1 and 6.3.3.6);

g) the pyrometric cone equivalent of the test piece and the type of pyrometric reference cones used, for example ISO 170 of ISO/R 1146.

10.3 In the case of repeated tests, all the results obtained shall be reported and not only the mean result.

1) Which of these alternatives is to be adopted will be apparent from the shape of the reference cones.

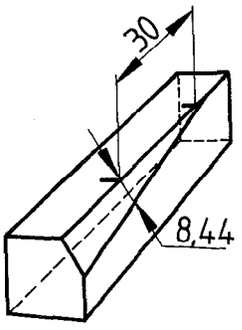
2) The rate of heating of 2,5 °C/min corresponds to a time interval of about 8 min between the collapse of two ISO pyrometric reference cones with consecutive numbers.

Annex

Typical mould for moulded test pieces

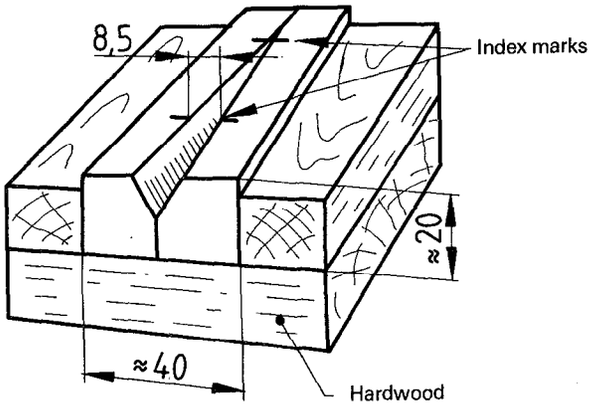
Figure 3[1] illustrates a typical mould which may be found suitable for the preparation of moulded test pieces (see 6.3.3.6).

Dimensions in millimetres



Left-hand half of split mould

NOTE — Make of copper or steel, preferably hardened, and grind important surfaces.



Mould assembly

NOTE — Index marks show the height of the cone.

Figure 3 — Typical mould for moulded test pieces

1) Based, with acknowledgements, on a figure in ASTM C 24.