INTERNATIONAL STANDARD 2478

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Dense shaped refractory products — Determination of the permanent changes in dimensions on heating

First edition — 1973-11-01

UDC 666.76 : 531.7 : 620.179.13

Ref. No. ISO 2478-1973 (E)

Descriptors : refractory products, high temperature tests, dimensional stability, dimensional measurement.

Price based on 5 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 2478 was drawn up by Technical Committee ISO/TC 33, *Refractories,* and circulated to the Member Bodies in November 1971.

It has been approved by the Member Bodies of the following countries :

Austria	Netherlands	Sweden
Czechoslovakia	New Zealand	Turkey
Egypt, Arab Rep. of	Portugal	United Kingdom
Germany	Romania	U.S.S.R.
Hungary	South Africa, Rep, of	
India	Spain	

The Member Body of the following country expressed disapproval of the document on technical grounds :

France

Printed in Switzerland

Dense shaped refractory products – Determination of the permanent changes in dimensions on heating

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the permanent change in dimensions of dense shaped refractory products on heating to a predetermined temperature which is maintained for a specified period of time.

2 DEFINITION

permanent change in dimensions : The expansion or contraction remaining after cooling to ambient temperature of a refractory product heated for a fixed time at a given temperature.

It may be expressed as the change, ΔL, in one dimension of the sample, calculated as a percentage of the original dimension, L (i.e. $100\ \Delta L/L$), or as the change, ΔV, in the volume of the sample, calculated as a percentage of the original volume, V (i.e. $100\ \Delta V/V$).

3 PRINCIPLE

Cutting of test pieces, in the form of cylinders or rectangular prisms, from the sample and measurement of the linear dimensions and/or the volume of these test pieces.

Heating the test pieces at a prescribed rate in a furnace having an oxidizing atmosphere[1], to a predetermined temperature, and maintaining this temperature for a specified period of time.

Allowing the furnace containing the test pieces to cool to ambient temperature, then measuring the linear dimensions and/or the volume and calculating the changes which have taken place.

4 APPARATUS

The following apparatus is required :

4.1 Furnace

The use of an electric furnace is recommended for carrying out these tests but gas fired furnaces are acceptable.

The furnace shall be capable of heating the test piece to, and maintaining it at, the testing temperature specified in section 6.

4.2 Thermocouples and temperature recorder

4.3.1 *Dial gauge comparator* mounted on a carrier[2] which has a square steel base plate in accordance with Figure 1.

4.3.1 *Dial gauge comparator* mounted above a carrier[2], on which the test piece may be placed for measurement.

A diagonal should be marked on one corner of the plate to assist in locating rectangular test pieces symmetrically on the studs.

Calibration can be effected by means of a steel cylinder, 50 mm in diameter and of accurately known length (57 to 64 mm), placed vertically on the studs.

4.3.2 *Micrometer* (for rectangular test pieces only) mounted so that the test piece can be located in a fixed position where its length may be measured at one of the points specified in section 6. It shall be possible to rotate the test piece so that its length can be measured at the other three corresponding positions.

4.4 Volume measuring devices

A mercury volumenometer, a mercury balance (see Annex), or a water displacement apparatus may be used, following the normal method for measuring bulk volume.

5 TEST PIECES

Test pieces[3] shall be in the form of either

- rectangular prisms, 50 mm × 50 mm × 60 ± 2 mm, or
- cylinders, 50 mm diameter and 60 ± 2 mm long.

From standard squares and small special shapes, three test pieces shall be taken, with their long axes parallel respectively to the length, width and thickness of the brick.

1) For certain products other atmospheres may be required.

2) The carrier described is commercially available.

3) For shapes which do not permit the cutting of these sizes smaller test pieces may be used by agreement between the interested parties.

Approximate dimensions in millimetres

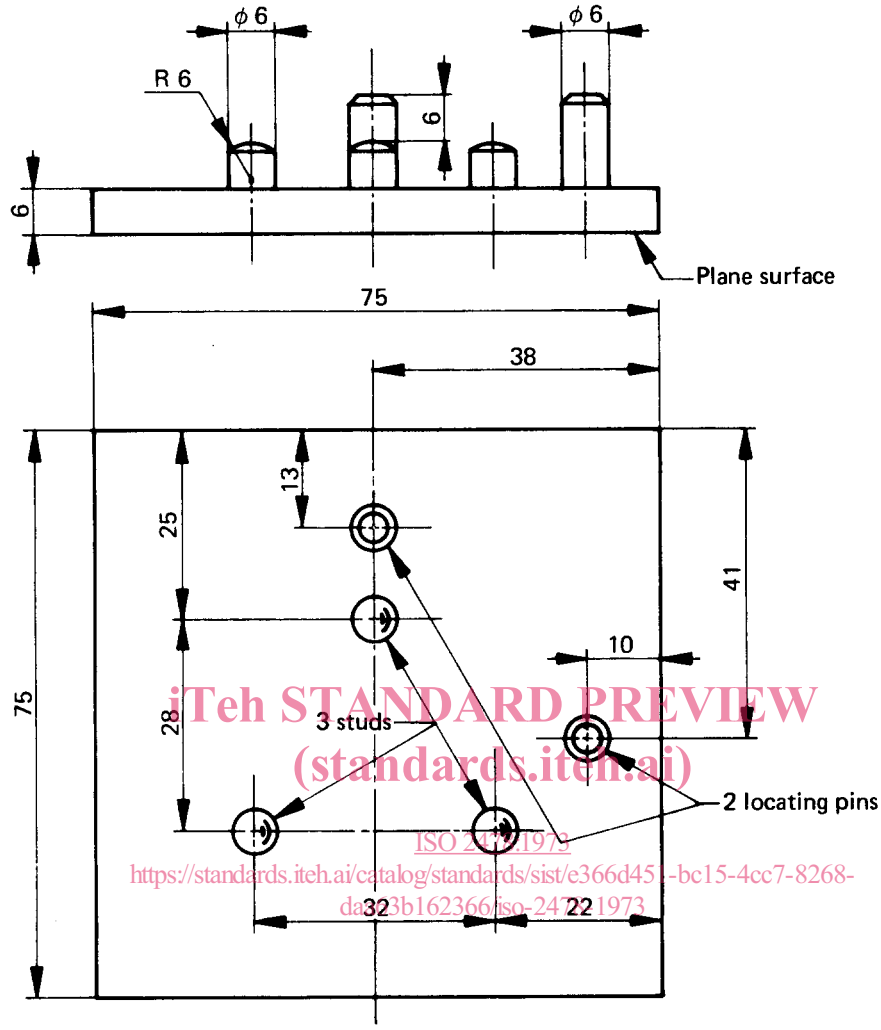

FIGURE 1 – **Carrier for the test piece**

From special shapes which have each dimension at least 150 mm, four test pieces shall be cut, two from the interior of the brick and two from near the exterior.

The 50 mm × 50 mm faces, or the ends of the cylinder, shall be ground plane and parallel before the determination of permanent linear changes.

6 PROCEDURE

6.1 Measurement of test pieces

6.1.1 *For permanent linear change*

Place each test piece on the carrier for measurement. If the test piece is rectangular, align one corner with the diagonal mark on the carrier and mark this corner so that the piece may be placed in the same position for measurement after firing. If the piece is cylindrical, mark it adjacent to the diagonal mark.

Measure the length of the test piece by means of the dial gauge comparator at four positions, located on the diagonals of a rectangular test piece, 13 to 19 mm from each corner, or on two diameters at right angles, and 10 to 13 mm from the periphery, on a cylindrical test piece.

Ring each measuring point with refractory paint.

When the micrometer is used, measure the length of the test piece near each corner, making all measurements to the nearest 0,05 mm. Take the mean of the four length measurements of each test piece as the original length in the calculation described in section 7.

6.1.2 *For permanent volume change*

Determine the volume of each test piece by a mercury volumenometer or mercury balance, by water displacement or by measurement and calculation.

6.2 Firing

6.2.1 *Location of test pieces*

Place the test pieces in the furnace, either horizontally or vertically, suitably shielded from direct flame impingement, on a layer of coarse, granular, closely-sized refractory material and at least 13 mm apart to allow free motion of the hot gases. Test pieces shall not be superimposed.

To comply with the requirements of 4.1, make provision for sampling the atmosphere in the vicinity of the test pieces at any time during the test and for the determination of oxygen in the gas samples.

6.2.2 *Temperature measurement and distribution*

Measure the temperature using at least three thermocouples placed away from the walls and heaters and out of contact with flames so as to record the temperature distribution over the limits of the space containing the test pieces. Use a temperature recorder, preferably a potentiometric recorder, to ensure that a record of the temperature control is available.

The temperature difference between any two of the thermocouples shall not exceed 10 K, and the average of the three readings shall be regarded as the test temperature.

6.2.3 *Temperature of the test*

The test shall be carried out at a temperature exceeding 800 °C by a multiple of 50 K.

6.2.4 *Heating schedule*

Raise the temperature of the furnace to 800 °C at a rate not exceeding 10 K/min, then to 1 200 °C at a rate of 3 ± 1 K/min, and finally, if required, to a temperature above 1 200 °C at a rate of 1 K/min.

Hold the temperature within ± 10 K of the test temperature for the specified period, after which cool the furnace through 200 K within 30 min and then at its natural rate overnight, the test pieces being allowed to cool in the furnace.

6.2.5 *Time at test temperature*

The specified period at the test temperature shall normally be 5 h, but in cases where adequate information is not obtained thereby, a period of 12 or 24 h may be chosen.

6.3 Measurement of test pieces after firing

6.3.1 *For permanent linear change*

Measure the length of the fired test pieces at each of the marked or fixed positions by the method previously used.

Do not remove blisters or accretions produced during firing but, if any of the measurements at a marked point might be affected by the presence of a blister or accretion which is not typical of the fired surface, the measurement made at that point shall not be included in the average value. Similarly, the test piece shall be rotated, if necessary, to avoid contact between such a blister or accretion and any of the three supports of the measuring device.

6.3.2 *For permanent volume change*

Measure the volume of the fired test pieces by the method previously used.

7 EXPRESSION OF RESULTS

7.1 Permanent linear change

Calculate the linear change at each measuring point as a percentage of the original length.

Report the individual values so calculated, together with the mean value, for each test piece, except that, where all the length changes in one test piece are not of the same sign, the mean value for that test piece shall not be given.

7.2 Permanent volume change

Calculate the volume change of each test piece as a percentage of the original volume.

8 TEST REPORT

The test report shall contain the following information :

a) type of furnace used;

b) direction in which the test pieces were cut, or their positions in the brick;

c) dimensions of the test pieces and their orientation in the furnace;

d) final test temperature;

e) period at test temperature;

f) appearance of the test pieces after heating;

g) oxygen content of the furnace atmosphere (if required) or nature of the atmosphere used;

h) one or both of the results described in section 7.

ANNEX

DETERMINATION OF VOLUME BY MERCURY BALANCE

A.1 APPARATUS

The mercury balance, which is illustrated in Figure 2, operates on the principle of a hydrometer but, since mercury is denser than the metal of which the balance is made, it is necessary to add mass at a position below the centre of buoyancy so that the balance will float in a vertical position.

This is achieved by placing the vessel containing the mercury on a bridge or shelf and providing a scale pan below the level of the support. Aluminium strip, which should be protected with a mercury-resistant coating, is recommended for the construction of the frame, this being light enough to require mass to be added to the scale pan to sink the hydrometer to the mark on the stem when the holder is empty.

If the balance is constructed of denser material and does not float in the mercury, it may be calibrated in the following manner :

A dry, machined steel block of accurately known volume and mass (m') is placed in the test piece holder so that the four prongs keep it vertical when immersed in the mercury.

Weights (of mass m'_1) are added to the scale pan until the hydrometer just sinks to the mark on the stem.

Approximate dimensions in millimetres

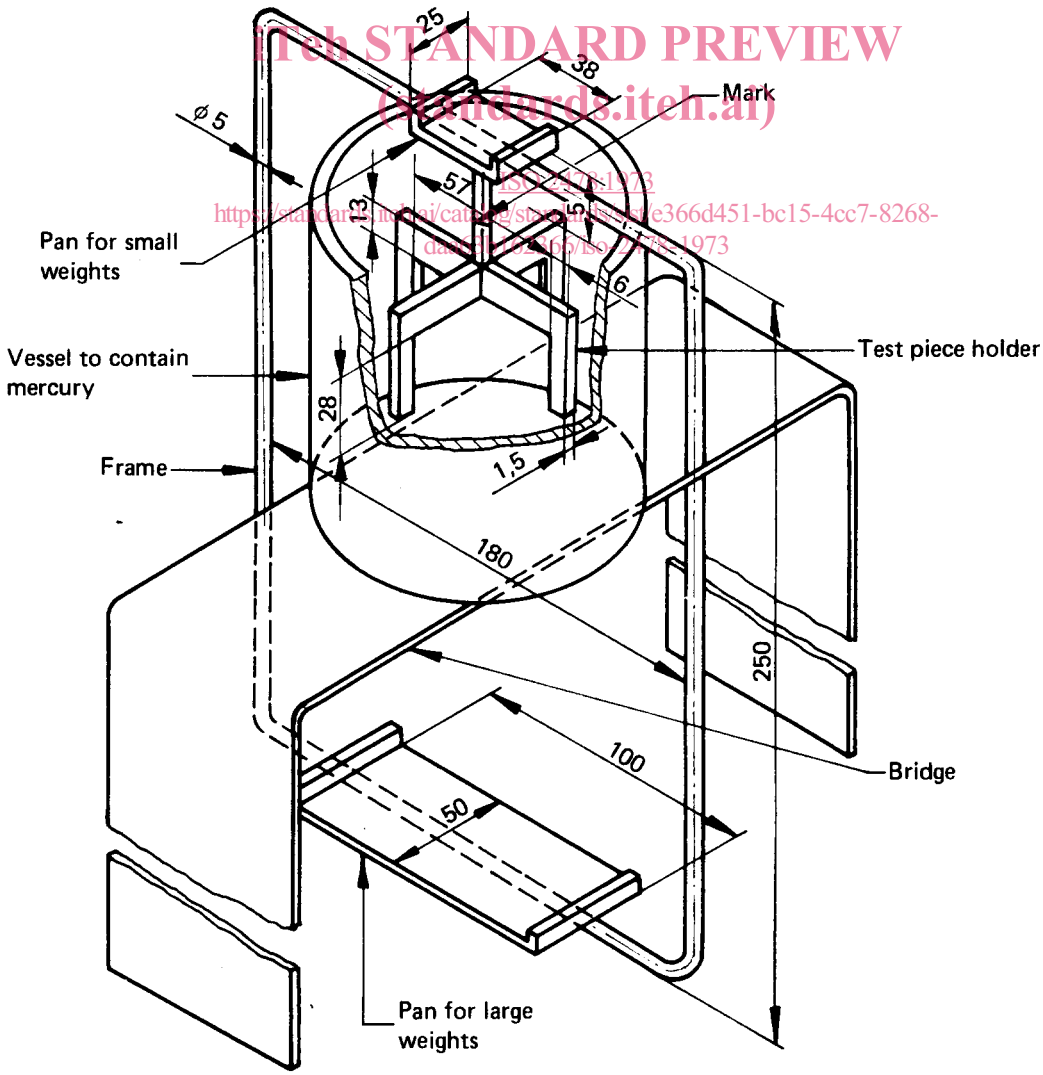

FIGURE 2 – **Mercury balance**

The apparent volume of the steel block is

$$\frac{m' + m'_1}{\rho}$$

where ρ is the density of mercury.

The difference between the value so obtained and the true volume is the volume calibration figure for the apparatus.

A.2 PROCEDURE

The test piece holder is immersed in the mercury and weights (of mass m_1) are added to the scale pan to sink the hydrometer to the mark.

Then the test piece holder is raised above the surface of the mercury, the dried test piece of known mass (m) is pushed under the surface of the mercury and the holder placed over it to prevent it from breaking the surface.

If this procedure is carefully followed, air should not be trapped between the holder and the test piece.

Additional weights (of mass m_2) are added to overcome the increased upthrust and sink the hydrometer to the mark.

If the test piece contains large pores, these may be penetrated by the mercury and the hydrometer will slowly sink. The error in bulk volume will be small if the procedure is carried out reasonably quickly.

A.3 EXPRESSION OF RESULTS

Calculate the bulk volume of the test piece as follows :

With no test piece in the holder and the hydrometer sunk to the mark,

$$m_0 + m_1 = V_0\rho + \chi \qquad \ldots (1)$$

where

m_0 is the mass, in grams, of the hydrometer;

m_1 is the mass, in grams, of the weights added to sink the hydrometer to the mark;

V_0 is the volume, in cubic millimetres, of mercury displaced by the hydrometer;

ρ is the density, in grams per cubic millimetre, of mercury at the temperature of the test;

χ is the small upthrust, in grams, due do the action of surface tension on the stem.

With the test piece in the holder and the hydrometer sunk to the mark,

$$m_0 + m_1 + m + m_2 = V_0\rho + \chi + V\rho \qquad \ldots . (2)$$

where

m is the mass, in grams, of the test piece;

m_2 is the mass, in grams, of the additional weights needed to overcome the upthrust of the test piece;

V is the bulk volume, in cubic millimetres, of the test piece.

Subtraction of (1) from (2) gives

$$m + m_2 = V\rho$$

whence

$$V = \frac{m + m_2}{\rho}$$