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Refractory products — Methods of test for ceramic fibre products

*Produits réfractaires — Méthodes d'essai des produits à base de fibres
céramiques*



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Contents	Page
1 Scope	1
2 Normative references	1
3 Preparation of test pieces	1
4 Determination of thickness	2
5 Determination of bulk density	3
6 Determination of resilience	4
7 Determination of permanent linear change on heating	5
8 Determination of thermal conductivity	8
9 Determination of tensile strength	11
10 Determination of shot content	12
11 Test report	14
Annex A (informative) Calculation of the coefficient of thermal conductivity as a function of the actual temperature in each point of the test piece	19

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10635 was prepared by Technical Committee ISO/TC 33 *Refractories*.

Annex A of this International Standard is for information only.

Refractory products — Methods of test for ceramic fibre products

1 Scope

This International Standard specifies methods for determining the thickness, bulk density, resilience, permanent linear change on heating, thermal conductivity, tensile strength and shot content of ceramic fibre products. It applies to ceramic fibre bulk, blankets, felts, mats, boards, papers and pre-formed shapes with the exception of products delivered in a wet state.

The application of the individual test methods is given in table 1, with reference to the type of products.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tensile testing machines*.

3 Preparation of test pieces

The number of items to be tested shall be determined by agreement between the parties. The number of test pieces per item shall be determined in accordance with Table 1.

When the material to be tested is wound, any compressed material at the extreme ends shall be excluded. A strip shall be cut perpendicular to the length across the full material width, of sufficient size for the different tests planned.

The required number of test pieces of required dimensions shall be cut using a template, a sharp knife, a saw or other method which will not damage the test piece. Avoid excess pressure as this may crush the fibre.

Table 1 — Summary of test methods and designations, applicability to product types and number of test pieces per item required

Clause	Test	Material	Number of test pieces
4	Thickness: 725 Pa method or 350 Pa method	blanket, felt, mat, board, paper	3
5	Bulk density	blanket, felt, mat, board, paper	3
6	Resilience	blanket, felt, mat	3
7	Permanent linear change on heating by the: slow heat method hot furnace method	blanket, felt, mat, board, paper, pre-formed shapes	3
8	Thermal conductivity: calorimetric method up to hot face temperature of 1300 °C	blanket, felt, mat, board	1
9	Tensile strength	blanket, felt, paper	5
10	Shot content	bulk fibre, blanket, felt, mat, paper	3

4 Determination of thickness

4.1 Principle

Determination of the thickness of a product subjected to a compressive stress which depends on its nominal bulk density. There are two methods, of which the dial gauge comparator method (see 4.3.1) is the reference method and is the only method applicable to ceramic fibre paper.

4.2 Test piece dimensions

The size of the test piece shall be such that the disc rests on it entirely, and shall be at least 100 mm × 100 mm.

4.3 Methods

4.3.1 The dial gauge comparator method

4.3.1.1 Apparatus, consisting of a machined reference plate, a dial gauge comparator with a metallic disc, 75 ± 1 mm in diameter, fixed at right angles to the dial gauge measuring probe. The apparatus shall be capable of applying a 350 Pa ± 7 Pa compressive stress to products with a nominal bulk density < 96 kg/m³ and a 725 Pa ± 15 Pa compressive stress to products with a nominal bulk density ≥ 96 kg/m³.

4.3.1.2 Procedure. Brush the reference plate free of any residual material and check that the disc lies parallel to the reference plate and, when they are in contact, the dial gauge reads zero.

Gently raise the disc and slide the test piece underneath it. Slowly lower the disc on to the test piece until it supports the full pressure of the disc and weigh (see 4.3.2.1). When the reading is stable record the dial reading to an accuracy of ± 0,1 mm.

4.3.2 The needle method

4.3.2.1 Apparatus, consisting of a machined reference plate and a measuring device made up of a needle 150 mm \pm 1 mm in length and 3 mm \pm 0,2 mm in diameter, and a metallic disc 75 mm \pm 1 mm in diameter which slides along the needle and has a friction device to grip the needle unless purposely moved (see Figure 1).

The stress determined by the mass of the disc and its securing device shall not exceed 350 Pa \pm 7 Pa for products with a nominal bulk density $<$ 96 kg/m³ and 725 Pa \pm 15 Pa for products with a nominal bulk density \geq higher than 96 kg/m³.

4.3.2.2 Procedure. Place the test piece on the reference plate and force the penetrating needle of the depth gauge downward through the test piece, perpendicular to the reference plate. If necessary, to prevent compression of the test piece by the depth gauge needle, first pierce the test piece. When the point of the needle touches the reference plate, lower the sliding disc to the point of contact with the top surface of the test piece until it supports the full pressure of the disc and gripping device. Secure the disc in position and withdraw the gauge. Measure with a steel rule the distance from the point of the needle to the sliding disc within an accuracy of \pm 0,5 mm.

4.4 Test report

Report the data required by clause 11, the dimensions of each test piece, the individual values for each test piece and the mean value for each item.

5 Determination of bulk density

5.1 Principle

Determination of the bulk density by calculation of the ratio between the mass of the product and its volume geometrically determined, thickness having been first determined according to clause 4.

5.2 Apparatus

5.2.1 Thickness measurement device, in accordance with 4.3.1 or 4.3.2.

5.2.2 Steel rule, capable of being read to 0,5 mm, possibly with a square angle at the readings origin, or alternatively, callipers.

5.2.3 Ventilated oven, capable of being maintained at 110 °C \pm 5 °C;

5.2.4 Balance, accurate to \pm 0,1 g.

5.3 Test pieces

The dimensions of the test pieces shall be in accordance with 4.2.

The test pieces shall be dried in the oven at 110 °C \pm 5 °C to constant mass. Constant mass can be considered as achieved when the mass variation between two weighings, carried out within an interval of 1 h, does not exceed 0,1 %.

Reject any test piece where the loss of mass exceeds 5 % after drying.

5.4 Procedure

Measure the two other dimensions of the test piece with the steel rule or the callipers to an accuracy of 0,5 mm, and calculate the area of the test piece, the thickness being determined according to clause 4.

Carry out the measurements along the middle of each face of the test piece. Carry out the weighings with an accuracy of \pm 0,1 g.

5.5 Expression of results

Calculate the bulk volume V_b of the test piece (in m^3) using the following equation:

$$V_b = S \times t$$

where:

S is the area in square metres;

t is the thickness in metres.

Calculate the bulk density ρ of the test piece in kilograms per cubic metre using the equation:

$$\rho = \frac{m}{V_b}$$

where:

m is the dry mass in kilograms determined in 5.4;

V_b is the bulk volume in cubic metres.

5.6 Test report

Report the data required by clause 11, the mass and dimensions of each test piece, reference to the method for thickness, and the individual values for each test piece and the mean for each item.

6 Determination of resilience

6.1 Definition

Resilience is the ability of ceramic fibre products to spring back after compression to 50 % of thickness. It is the ratio of the thickness of a product after the application and relaxation of a compressive force, to its original thickness.

6.2 Principle

Calculation of the ratio, expressed as a percentage, of the thickness of a product to its initial thickness after application of a compressive stress sufficient to reduce the initial thickness to 50 % for a given period of time.

6.3 Apparatus

6.3.1 Thickness gauge

6.3.2 Compression testing machine, capable of applying the compressive stress at a given rate and provided with means for measuring the test piece deformation.

6.3.3 Ventilated oven, capable of being maintained at $110\text{ °C} \pm 5\text{ °C}$.

6.4 Test pieces

6.4.1 Dimensions

The dimensions of the test piece shall be $100\text{ mm} \times 100\text{ mm} \times$ (nominal thickness). Compression of the test pieces when cutting out shall be avoided.

6.4.2 Drying

Dry the test pieces in accordance with 5.3.

6.5 Procedure

Determine the thickness according to clause 4. Set the compression testing machine to give a constant deformation rate of 2 mm/min.

Place the test piece in the compression tester and compress at the given rate until the test piece thickness has been reduced by 50 %.

NOTE 1 If a record of compressive stress versus thickness is required, the compressive stress at regular percentage reductions of the original thickness should be recorded.

Keep the test piece at 50 % of its initial thickness for 5 min and then remove the majority of the pressure applied by the testing machine but just maintaining a nominal pressure, of either 350 Pa \pm 7 Pa for products with a bulk density < 96 kg/m³ or 725 Pa \pm 15 Pa for products with a bulk density \geq 96 kg/m³. After 5 min, determine the thickness according to clause 4.

NOTE 2 Other values for reduction of the thickness can be chosen by agreement between the parties. The same procedure should be used.

6.6 Expression of the results

Calculate resilience, as a percentage to the nearest 0,5 %, from the following equation:

$$R = \frac{d_f}{d_0} \times 100$$

Calculate permanent deformation, as a percentage to the nearest \pm 0,5 %, from the following equation:

$$PD = 1 - \left(\frac{d_f}{d_0} \right) \times 100$$

where

d_f is the thickness after testing;

d_0 is the initial thickness.

6.7 Test report

Report the data required by clause 11, the dimensions of the test pieces and the thickness method, also any value for reduction of the thickness, if different from 50 %, individual values of permanent deformation/resilience, and the mean values of permanent deformation/resilience.

7 Determination of permanent linear change on heating

7.1 Principle

Determination of the permanent linear change of the dimensions of test pieces held at a prescribed temperature and for a prescribed time interval. The permanent linear change is expressed as the ratio of the difference between the initial dimension and the dimension after testing measured between platinum wire markers inserted into the test piece surface on the initial dimension.

7.2 Apparatus

7.2.1 Furnace, gas or electric (oxidizing atmosphere) in which the temperature distribution shall not exceed $\pm 10\text{ }^{\circ}\text{C}$ and the dimensions of which shall be such as to comply with the test piece placing requirements (see 7.4.3.1) and the temperature measurement requirements (see 7.4.3.2).

7.2.2 Measuring devices, optical such as a cathetometer, accurate to $\pm 0,01\text{ mm}$, or callipers, accurate to $\pm 0,05\text{ mm}$.

7.2.3 Thermocouples, a minimum of two shall be used to measure the temperature and temperature distribution over the space occupied by the test pieces.

7.3 Test pieces

7.3.1 Dimensions

The dimensions of the test pieces shall be $100\text{ mm} \times 100\text{ mm} \times$ (actual thickness), care being taken to record the direction of rolling of the product.

7.3.2 Drying

Dry the test pieces in accordance with 5.3.

7.4 Procedure

7.4.1 Test piece preparation

On the diagonals of the upper $100\text{ mm} \times 100\text{ mm}$ surface of each test piece, and 10 mm to 15 mm away from the edges, insert four platinum wire markers so that they are approximately 75 mm apart.

These markers shall be approximately $0,5\text{ mm}$ in diameter, their length being such as to leave 1 mm or 2 mm protruding above the surface when they are inserted at a depth corresponding to at least $3/4$ of the test piece thickness.

NOTE For boards and pre-formed shapes, the platinum wire markers can be replaced by painted marks.

Test pieces shall be placed on the lower $100\text{ mm} \times 100\text{ mm}$ surface.

7.4.2 Measurements

Measure the distance between the markers, the measurements being made parallel to the edges of the test pieces. Measurements made by an optical device shall be accurate to $\pm 0,05\text{ mm}$ and shall be used as the reference method. Measurements made with callipers shall be accurate to $\pm 0,1\text{ mm}$. The method of measurement shall be stated in the test report.

7.4.3 Heating

7.4.3.1 Placing of test pieces

Place each test piece on a support piece cut from the same material, each support piece being used for one test only. For easier handling, place the support piece on a shaped refractory support 10 mm to 15 mm in thickness.

Place test pieces in the kiln so that:

- a) they are at least 50 mm apart;
- b) they are at least 50 mm away from the heating elements.

7.4.3.2 Temperature measurement and distribution

Measure the temperature using at least two thermocouples. Place the thermocouple junctions at 10 mm to 20 mm over the upper surface of the test pieces. For the hot furnace method, either mount the thermocouples through the wall and roof of the furnace, or on the ceramic fibre support piece which supports the test piece. During the soaking time, the temperatures recorded by the two thermocouples shall not differ by more than 10 °C, and the mean temperature shall not differ by more than 10 °C from the test temperature.

7.4.3.3 Test temperature

The test temperature shall be the service temperature of the product quoted by the manufacturer or that agreed between parties.

7.4.3.4 Heating methods

Test pieces shall be subjected to one of the two heat treatment methods described below, the method being agreed between parties, with the electric furnace slow heat method used as the reference method.

7.4.3.4.1 Hot furnace method

Place the test pieces directly into a furnace pre-heated to test temperature. The soaking time shall start when the temperature has reached test temperature again after introduction of the test pieces. Hold the test temperature to within ± 10 °C for 24 h.

7.4.3.4.2 Slow heat method

Place the test pieces in the furnace and raise the temperature in the furnace at one of the heating rates given in Table 2.

Hold the test temperature to within ± 10 °C for 24 h. At the end of this period, cool the test pieces by at least 200 °C within 30 min.

Table 2 — Heating rates for the slow heat method

Test temperature °C	Temperature range °C	Heating rate °C/min
$\leq 1\ 250$	ambient to 50 < test temperature last 50	5 to 10 1 to 2
1 250 to 1 500	ambient to 1 200 1 200 to 50 < test temperature last 50	5 to 10 2 to 5 1 to 2
$> 1\ 500$	ambient to 1 200 1 200 to 50 < test temperature last 50	< 20 < 10 < 2

7.4.3.5 Measurement of the test pieces after the test

Allow test pieces to cool to room temperature, then measure the distances between markers as specified in 7.4.2.

7.5 Expression of the results

For each test piece, calculate the permanent linear change as the mean of two values measured in two different directions, expressed as a percentage of the initial length measured between the platinum wire markers. As the test result, take the mean of the values recorded for each direction on each of the test pieces.

7.6 Test report

Report the data required by clause 11, as well as the bulk density (determined in accordance with clause 5), means of measuring test temperature, the heating method, the method of measurement, the result in each direction, the mean for each direction and the mean for each test piece.

8 Determination of thermal conductivity

8.1 Definition

The coefficient of thermal conductivity is a value corresponding to the intensity of a thermal flow which runs through a wall as a consequence of a thermal gradient normally directed to the wall. It is designated as λ and expressed in $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

8.2 Principle

Determination of the coefficient of thermal conductivity of a panel of the test material placed in conditions such that:

- a) it is evenly heated on one of its faces;
- b) side thermal flows are as restricted as possible;
- c) thermal energy transmitted by the panel is collected by a calorimeter, fitted with an outer guard.

Using this method, it is essential that the heat flow be perpendicular to the surface of the panel.

8.3 Apparatus

8.3.1 Calorimeters

8.3.1.1 Dimensions

The centre calorimeter shall cover an area of 76 mm \times 76 mm. The guard and the calorimeter shall cover an area of at least 230 mm \times 230 mm. For a typical arrangement of the apparatus, see Figure 2.

8.3.1.2 Water circulating system

The measurement calorimeter and guard shall each be provided with one water inlet and one water outlet. Inlets and outlets shall be located in such a way that any heat transfer between two adjacent calorimeters is avoided. The temperature of incoming water shall be within + 3 °C or – 1 °C of room temperature. The variation of the temperature of incoming water shall not exceed 0,5 °C/h. The pressure of incoming water shall be constant to 1 %.

8.3.1.3 Measurement of the water temperature

The equipment used shall be capable of measuring the difference in temperature between inlet and outlet water to an accuracy of $\pm 0,05$ °C.

8.3.2 Heating

Heating shall be electrical and shall ensure uniform heat distribution over the whole surface of the test piece. The heating control shall be capable of holding the temperature constant to ± 10 °C.

Heating rates shall be in accordance with Table 2.

8.4 Test pieces

8.4.1 Dimensions

The test piece shall consist of one or more layers of the material to be tested and shall be at least 230 mm × 230 mm in length and width, and between 45 mm and 100 mm in thickness. Each layer shall cover the whole surface of the calorimeters and guard.

When the nominal thickness of the product is < 40 mm, use three layers at least; when the nominal thickness is between 40 mm and 50 mm, use two layers.

Where the nominal thickness is > 50 mm, use a single layer.

8.4.2 Drying

Dry the test pieces in accordance with 5.3.

8.5 Procedure

8.5.1 Placing of the test pieces

Prepare four machined studs of insulating refractory for each layer with a diameter of 17 mm ± 0,5 mm and a height of at least 9/10 of the nominal thickness of the product to be tested. Punch four holes in each layer to receive the studs at the four corners of the test panel.

Position the first thermocouple in the centre of the centre calorimeter, then position the first layer and compress it to the top of the studs using a silicon carbide plate. Remove the plate and position the second thermocouple straight above the first one. As before, position a second layer to the thickness of the studs and continue for the required number of layers. Compress the last layer, like the others, with a silicon carbide plate which will remain in contact with the layer and its upper thermocouple during the whole procedure.

If the test piece comprises one or two layers only, thermocouples shall be inserted within these layers so as to have five thermocouples if possible.

8.5.2 Measurement of temperature gradients

Two thermocouples give the temperatures of the hot and cold faces of the test piece.

The other thermocouple each give the temperatures of the hot face and of the cold face of two adjacent layers of material. The measurement of these temperatures and the combinations in thickness of each of the layers when they are in contact give:

- a) ten temperature gradients and ten mean temperatures for a test piece made up of four layers (five thermocouples);
- b) six temperature gradients and six mean temperatures for a test piece made up of three layers (four thermocouples).

8.5.3 Measuring conditions

The hot face of the test piece shall be heated to a temperature consistent with the limit of service of the product or, for high temperature products, to the operational limit of the apparatus used. For this hot face temperature, maintain the temperature of the heating elements until the hot face temperature does not vary by more than 5 °C during a 2 h period and the heat flow measured by means of the calorimeters does not vary by more than 2 % during the same period. Maintain the flow in the centre calorimeter at between 120 ml/min and 200 ml/min, this flow being constant to 1 %.

Adjust the water flow of the guard calorimeter so as to ensure the same outlet water temperature in this calorimeter and the centre calorimeter. After a soaking time of at least 24 h, make three to five readings at 30 min intervals, of the water temperature at the input and output of the calorimeters.

8.6 Expression of the results

Calculate the coefficient of thermal conductivity from the formula:

$$\lambda = \frac{QL}{A(T_2 - T_1)}$$

where:

$$Q = m(t_2 - t_1)c$$

Q is the quantity of heat flowing through the test pieces in Joules per second;

λ is the calculated thermal conductivity in Watts per metre Kelvin;

m is the average mass flow rate of water through the calorimeter in kilograms per second;

t_1 is the inlet water temperature in degrees centigrade;

t_2 is the outlet water temperature in degrees centigrade;

T_1 is the cold face temperature of a test piece or layer in degrees centigrade;

T_2 is the corresponding hot face temperature of a test piece or layer in degrees centigrade;

L is the distance between the thermocouples used to measure T_1 and T_2 in metres;

A is the effective area of the centre calorimeter in square metres;

c is the specific heat capacity of water at mean temperature of the inlet and outlet water of the calorimeter in Joules per kilogram Kelvin.

Applying this formula to each layer of the test piece and to their combinations in thickness, it is possible to obtain six points of the graph:

$$\lambda = f(T_m)$$

where T_m is the mean temperature in Kelvin.

NOTE The values for c given in Table 3 may be used to obtain, by interpolation, a value for the calculation.

Table 3 — Specific heat capacity of water

Temperature °C	c J·kg ⁻¹ ·K ⁻¹
15	4 185,5
20	4 181,6
25	4 179,3

The calculation of the coefficient of thermal conductivity as a function of the actual temperature in each point of the test piece is given in annex A.

8.7 Test report

Report the data required by clause 11, with the bulk density (determined in accordance with clause 5), the heat flow conditions and the mean temperature for each point in the graph and also the maximum and minimum temperatures at this point.

9 Determination of tensile strength

9.1 Definition

The tensile strength is the maximum force that the material can withstand before it fails. It is expressed in pascals (Pa). It is given together with the bulk density determined by a geometrical method.

NOTE The tensile strength is also known as the parting strength.

9.2 Principle

A test piece of prescribed dimensions is cut from the product or the item, and the tensile strength is determined by causing rupture of the test piece at room temperature.

9.3 Apparatus

9.3.1 Tensile testing machine, provided with two pairs of jaws allowing the clamping of the test piece over a 75 mm × 40 mm area. This device shall be capable of straining the test-piece in tension at a constant cross-head speed and shall be Class B as defined by ISO 7500-1.

9.4 Test-pieces

9.4.1 Dimensions

Cut test pieces (230 mm ± 5 mm) × (75 mm ± 2 mm) × (nominal thickness). As far as possible, take test pieces side by side and at random along the longitudinal axis of the product. The longest dimension of the test piece (230 mm) shall be parallel with the manufacturing direction of the product.

NOTE By agreement between parties, and for supplementary tests, test pieces can be taken perpendicular to the manufacturing direction.

9.4.2 Drying

Dry the test pieces in accordance with 5.3. Measure the thickness, the width of the test piece and the tensile strength immediately after drying.

9.5 Procedure

The thickness of the test piece is measured in its strained area according to clause 4, using the dial gauge comparator and the width measured using the steel tape.

Clamp the test piece at both ends so that a surface area of 75 mm × 40 mm is held in the jaws of the testing machine.

The rate of the tensile stress shall be variable so that the piece deformation occurs at a constant speed of 100 mm/min during the whole test. Apply the stress parallel to the manufacturing direction of the product.

Reject results from test pieces where parting occurs at the level of the jaws and carry out repeat determinations until the number of results required by Table 1 is obtained.

For the calculation of the results, use the maximum stress recorded over the course of a test finishing with the parting of the test piece.

9.6 Expression of the results

The tensile strength is given by the formula:

$$R_m = \frac{F}{w \cdot t}$$

where:

R_m is expressed in pascals;

F is the maximum parting force in newtons;

w is the initial width of the active part of the test piece in metres;

t is the initial thickness of the test piece in metres.

The test result is expressed by the mean of those determinations together with the bulk density of the product determined according to clause 5.

9.7 Test report

Report the data in accordance with clause 11, as well as the direction of taking out test pieces, the type of tensile testing machine, the bulk density of the product, and the mean of five determinations carried out on five test pieces.

10 Determination of shot content

10.1 Definition

The shot content is the percentage of non-fibrous particles that would be retained on a 75 µm nominal aperture sieve complying with the requirements given in ISO 565.

NOTE This method is not applicable to products with less than 5 % shot content.

10.2 Principle

Fibrous particles are separated from the shot after firing to burn off organic matter that may be present. Separation is achieved by reduction in the fibre length to release the shot, followed by elutriation.

10.3 Apparatus

10.3.1 Furnace

10.3.2 Balance, weighing to an accuracy of ± 0,1 g;

10.3.3 Compression cylinder, comprising a cylinder of 50 mm ± 5 mm internal diameter into which a hardened steel plunger is fitted with a clearance of 0,13 mm;

10.3.4 Pressing machine, capable of applying a 25 kN load;

10.3.5 Glass elutriator, comprising a dispersion chamber, the diameter of which is less than the diameter of the elutriation column, and a water inlet with constant rate flow; an elutriation column, the diameter of which is between 29 mm and 76 mm, and the minimum volume is 0,75 dm³; a mixer with a glass bowl of capacity one litre.

Examples of glass elutriator are shown in Figures 3a and 3b.

10.4 Treatment of test pieces

10.4.1 General

Take out a sample of at least 20 g and prepare this sample according to one of the following methods:

- a) the stirring method;
- b) the crushing method.

The stirring method is to be used as the reference method.

10.4.2 Stirring method

Fire the sample in an oxidizing atmosphere to its maximum service temperature for 30 min in order to obtain sufficient devitrification. Weigh the sample after firing, to $\pm 0,1$ g.

Fill the mixer with approximately 700 cm³ of water at $20\text{ °C} \pm 10\text{ °C}$ and start the mixer at low speed. Gradually introduce the sample into the mixer, brushing the transfer container to ensure that all the sample has been transferred. Once transfer is complete, increase the mixer speed to a minimum of 250 s⁻¹ and allow to run for 5 min.

10.4.3 Crushing method:

Fire the sample in a furnace at $925\text{ °C} \pm 25\text{ °C}$ for 30 min. Weigh the sample to $\pm 0,1$ g. Introduce the sample into the compression cylinder, crush it twice by means of a press, applying a pressure of at least 10 MPa to the plunger, care being taken to break packs of fibres with a spatula after each pressure application. Transfer the sample to a 250 ml beaker, add 150 cm³ water and stir thoroughly to disperse the sample.

10.5 Procedure

Transfer the whole sample to the elutriation dispersion chamber. Add water until it flows through the column at a rate calculated according to the formula given below and at a temperature of $20\text{ °C} \pm 10\text{ °C}$. Carry on elutriation for 15 min. Drain the elutriator and recover the shot which has settled at the bottom of the column either by sieving (75 μm mesh), by decantation or by filtration.

Dry the shot at $110\text{ °C} \pm 5\text{ °C}$ to constant mass. Constant mass can be considered as achieved when mass variation between two weighings carried out within an hour interval does not exceed 0,1 %. Weigh the shot to $\pm 0,1$ g.

Calculate the flowrate according to the diameter of the elutriation column as follows :

$$q_v = 0,689 D^2$$

where

q_v is the flowrate in cubic centimetres per minute;

D is the diameter of the column in millimetres (Figure 3a) or average diameter of the column in millimetres (Figure 3b).

.....

10.6 Expression of results

Calculate the shot content, in percent, from the following formula:

$$C = \frac{m_{\text{sh}}}{m_{\text{ini}}} \times 100$$

where

C is the shot content expressed as a percentage;

m_{sh} is the mass of the shot in grams;

m_{ini} is the initial mass of the sample after firing, in grams.

10.7 Test report

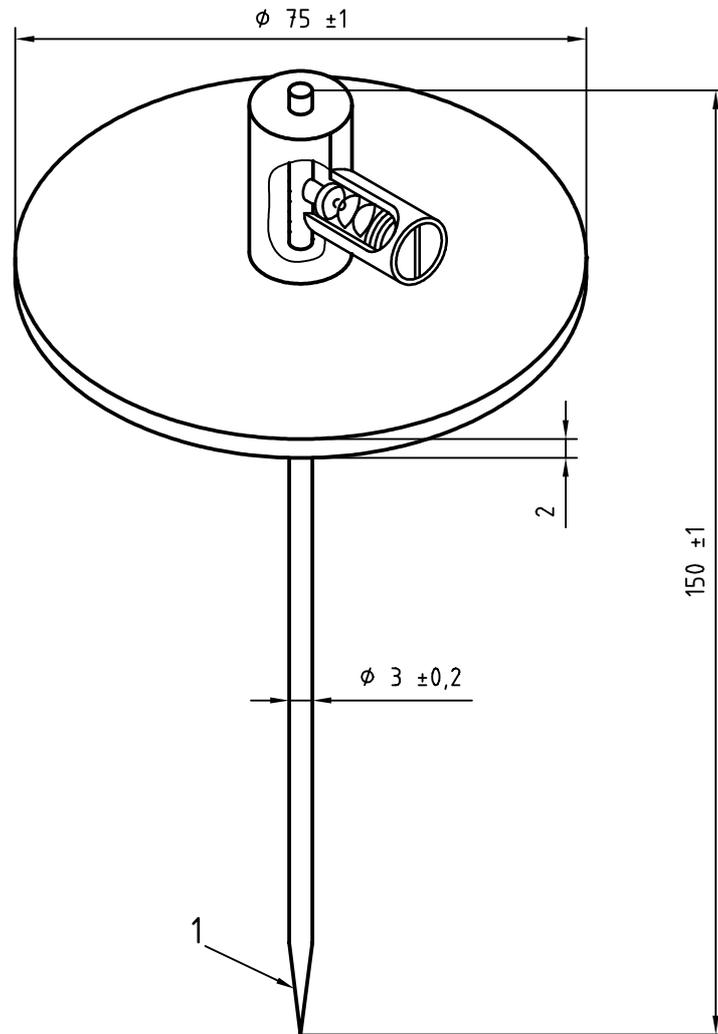
Report the data required by clause 11 and the mean value of the shot content.

11 Test report

The test report shall contain the following general information.

- a) the name of the testing establishment;
- b) the date of the test;
- c) reference to this International Standard, i.e. ISO 10635;
- d) the designation of the product tested, as given in clause 1;
- e) the number of items tested;
- f) the number of test pieces per item;
- g) the specific information given for each method.

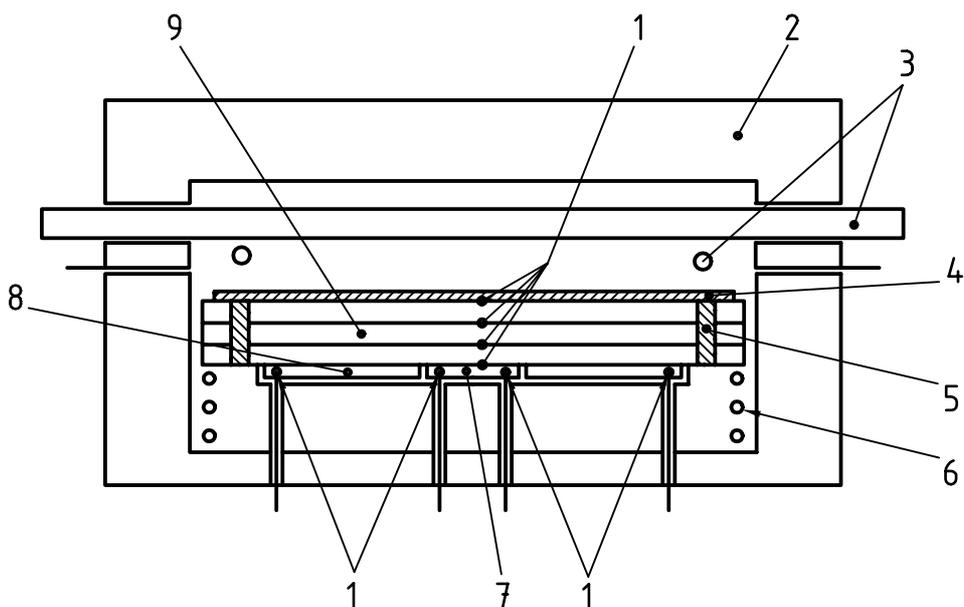
Dimensions in millimetres



Key

- 1 Taper to a sharp point

Figure 1 — Depth gauge for thickness measurements by the needle method

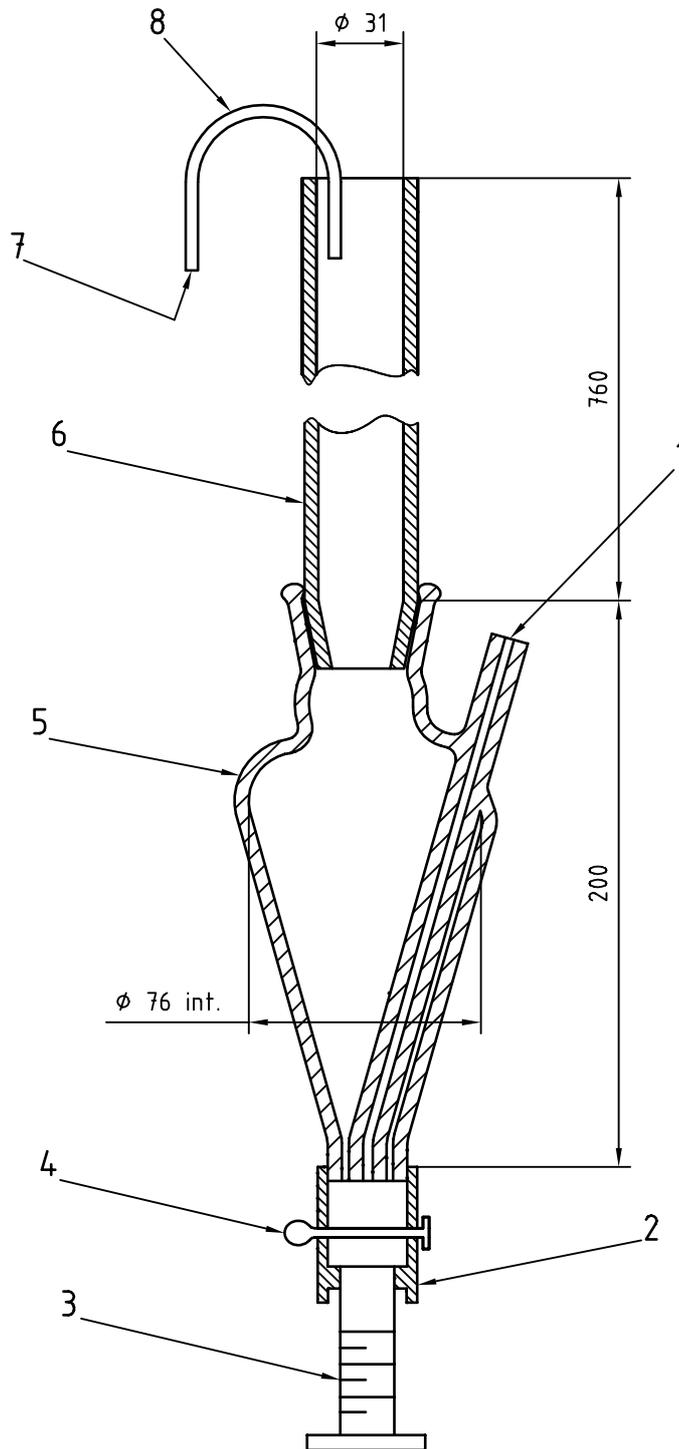


Key

- | | | |
|--------------------|---------------------------|---------------------------|
| 1 Thermocouples | 4 Silicon carbide slab | 7 Centre calorimeter |
| 2 Movable roof | 5 Insulating prop | 8 Inner guard calorimeter |
| 3 Heating elements | 6 Outer guard calorimeter | 9 Multi-layer test pieces |

Figure 2 — Thermal conductivity apparatus (diagrammatic section through heating chamber)

Dimensions in millimetres

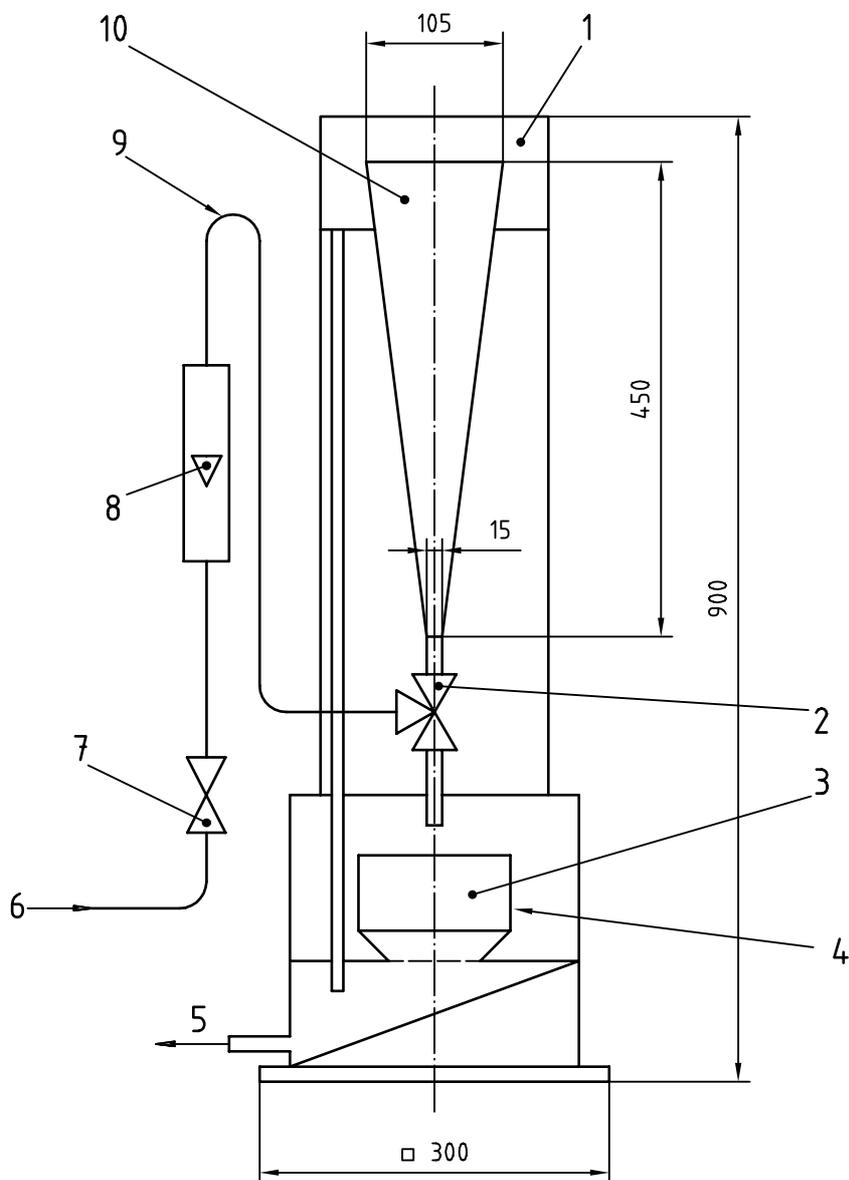


Key

- | | |
|---------------|----------------------|
| 1 Water inlet | 5 Dispersion chamber |
| 2 Rubber tube | 6 Elutriation column |
| 3 Shot trap | 7 Water overflow |
| 4 Clip | 8 Siphon |

Figure 3a — Example of apparatus for measurement of shot content

Dimensions in millimetres



Key

- | | |
|--------------------------|--|
| 1 Water overflow chamber | 6 Water inlet |
| 2 Three-way tap | 7 Regulating valve |
| 3 Shot trap | 8 Flow meter |
| 4 Filter paper | 9 13 mm diameter tube |
| 5 Water outlet | 10 Conical elutriation chamber, volume 1,5 l |

Figure 3b — Example of apparatus for measurement of shot content

Annex A (informative)

Calculation of the coefficient of thermal conductivity as a function of the actual temperature in each point of the test piece

On the assumption that, for a fibre product, the variation law of the thermal conductivity coefficient is most often put in the form

$$\lambda = AT^{(1/2)} + BT^3$$

A and B being numbers depending on the material and determined from the following equation

$$\lambda \frac{T_2}{T_1} = \int_{T_1}^{T_2} \frac{(AT^{1/2} + BT^3)dT}{T_2 - T_1}$$

the mean value for λ between two temperatures T_1 and T_2 therefore is

$$\lambda \frac{T_2}{T_1} = \frac{\frac{2}{3}A}{T_1 - T_2} (T_2^{3/2} - T_1^{3/2}) + \frac{\frac{1}{4}B}{T_1 - T_2} (T_2^4 - T_1^4)$$

considering that each layer of the test piece leads to four equations with two unknowns, whence A and B are determined.

From the expression $\lambda = AT^{1/2} + BT^3$, it is possible to calculate the actual value of λ as a function of the temperature and to draw the thermal conductivity curve $\lambda = f(T)$.

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