INTERNATIONAL STANDARD ISO 2582

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Cork and cork products — Determination of thermal conductivity — Hot plate method

Liège et produits en liège — Détermination de la conductivité thermique — Méthode de la plaque chaude

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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 2582 was developed by Technical Committee ISO/TC 87, *Cork*, and was circulated to the member bodies in April 1977.

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

Czechoslovakia	Mexico	Turkey
France	Romania	United Kingdom
Hungary	South Africa, Rep. of	Yugoslavia
Italy	Spain	

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

Germany

Cork and cork products — Determination of thermal conductivity — Hot plate method

0 INTRODUCTION

The determination of the thermal conductivity in stationary regime of low conductivity materials may be performed by :

— **comparative measurement** : for routine checks such as are required for quality control; or for the determination of the thermal conductivity of moist materials, etc.

NOTE — Such measurements require nothing more than simple but reliable apparatus calibrated in relation to other apparatus serving as reference.

— **absolute measurement** : for the measurement and checking of materials that must be subject to specific tests under the provisions of public or private contracts; for the provision of technical data for expert appraisal; for the calibration of checking apparatus; and the provision of reference values for technical, scientific or industrial publications.

The hot plate method meets this second purpose and is dealt with in this International Standard.

The continuous progress of measurement techniques makes it difficult to standardize a given type of apparatus since this might hinder the future improvement of measuring procedures. Therefore, this International Standard concentrates mostly on laying down and standardizing the procedural conditions required to make the measurements. It gives, as an example, the general description of an apparatus meeting those requirements and indicates the procedure to be followed when using such an apparatus.

1 SCOPE

This International Standard specifies a hot plate method for the determination of the thermal conductivity of cork and cork products.

2 FIELD OF APPLICATION

The field of application of this International Standard is defined as follows :

2.1 Temperature range

Between − 180 and + 200 °C.

2.2 Thermal conductivity

Thermal conductivity of the material not exceeding 3,5 $W \cdot m^{-1} \cdot K^{-1}$ (3 $kcal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$).

2.3 Nature of materials

a) Homogeneous materials and compact granular mixtures of appreciably continuous granulometry and of size not exceeding, for the largest dimension of the grain, 1/4 of the thickness of the sample (unless special specifications apply to the material to be tested).

NOTE — A material is considered to be homogeneous when the value of the thermal conductivity is not affected by a change in thickness or in area, for the size range within which it is normally available and used.

b) Structures containing cavities statistically distributed and with a mean diameter of less than 3 mm.

NOTE — Insufficiently homogeneous materials may be tested according to this method. However, they should never be reported as having been subjected to a thermal conductivity test and the relevant test report shall refer instead to the determination of the thermal resistance of material for a given thickness and composition.

2.4 Humidity of materials

The method is valid only if the transfer of moisture through the material during the measuring procedure remains negligible (see 6.2).

3 DEFINITIONS

3.1 heat flow rate : The quotient of the quantity of heat flowing through a surface, by the time.

3.2 density of heat flow rate through a surface : The quotient of the heat flow rate through a surface, by the area of that surface.

3.3 thermal conductivity : The quotient of the density of heat flow rate through a surface, by the temperature gradient.

For homogeneous materials at given temperature and moisture content and under steady-state conditions, the thermal conductivity is equal to the quotient of the quantity of heat passing through unit area in unit time, by the temperature gradient in the direction perpendicular to the surface.

3.4 thermal resistance : The quotient of the temperature difference which maintains a heat flow under steady-state conditions, by the heat flow rate.

4 PRINCIPLE

A heat source called a "hot plate" supplies heat energy. A constant heat flow through two test pieces of the material, located one on each side of the guarded hot plate, is maintained, producing parallel isothermal planes. After having crossed the test pieces, the energy is dissipated by two cooling plates located one on each side of the test pieces.

The energy supplied under steady-state conditions to the hot plate is measured and the temperature gradient in the test pieces is determined.

A graph is plotted showing how the coefficient of thermal conductivity varies with the mean temperature of the test pieces, or the coefficient of thermal conductivity at a given mean temperature is determined. The above-mentioned graph shall be plotted within the temperature range indicated by the manufacturer as being the range within which the material should be used. Should there be changes in the material deriving from variations in temperature, wait until equilibrium is restored before making measurements.

The temperature gradient in the test pieces is to be in the region of 1 to 3 $K \cdot cm^{-1}$, the temperature difference between cold and hot surfaces being as low as possible and in any case always less than 15 K.

Results of measurements may be considerably affected by the moisture content of the samples.

5 APPARATUS

As stated in the Introduction, with regard to the apparatus emphasis is placed on defining the conditions required rather than on constructional details.

5.1 General description

Two identical test pieces are placed on each side of a hot plate. Two cooling plates are then placed one on each side of the assembly thus formed (see figure 1).

Normally, this assembly is to be placed horizontally in a container holding the insulating material intended for edge insulation. This container should be air-tight when the test is conducted at lower than room temperature or when specifications call for a given relative humidity.

The apparatus shall be built to provide for testing test pieces 0,50 m square; these dimensions may be reduced to 0,30 m for samples of low thickness (see clause 6).

5.2 Hot plate (heating plate)

The hot plate (see figure 2) consists of a central part holding the heating elements and locked between two metal plates which stabilize the temperature. The hot plate is divided into three sections : a central measuring square, a first guard section and a second section called edge overheating section.

5.2.1 *Central part*

The central part has a maximum thickness of 3 mm and is made up of heating wires set in a heat-resisting and highly electrically insulating material such as asbestos, mica, refractory cement, etc., in such a way as to result in a compact thermally conducting element and to avoid the presence of air inclusions.

The measuring section and first guard section have one wire only, wound in square spirals at a regular spacing of not more than 10 mm. Potential taps are located on the spiral which marks the limit between the measuring section and the guard section so that the power which is dissipated in the measuring section can be evaluated. The first heating element ends 30 to 40 mm away from the edge of the plate, where a second guard section starts, made up of independent square spirals intended for edge overheating.

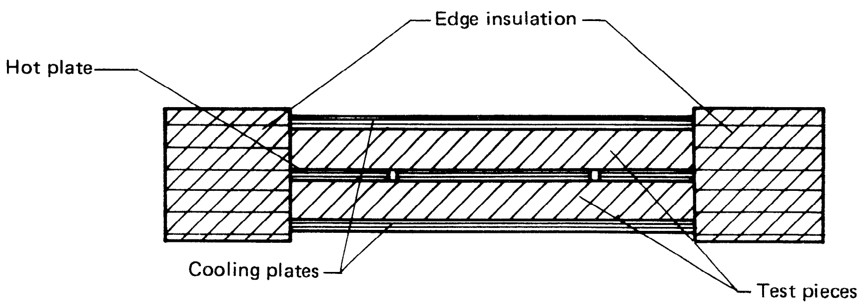

FIGURE 1

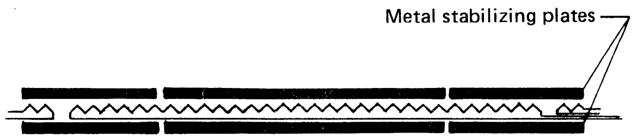

FIGURE 2

The resistance of the electrical insulation between heating elements and stabilizing plates shall be higher than 1 MΩ at a temperature of 200 °C. The electrical insulation between heating elements shall also exceed 1 MΩ. After cooling to room temperature, moisture regain shall not result in lowering the insulation to less than 1 MΩ.

5.2.2 *Stabilizing plates*

The stabilizing plates shall have a maximum thickness of 5 mm and shall be made of metal of high thermal conductivity. They shall have a very smooth finish but shall not be polished. Departure from flatness shall not exceed 0,1 mm.

The measuring section, which is an independent square, shall be separated from the remainder of the plate by a milled slot resulting in a gap 1 to 3 mm wide.

The dimensions of the measuring section are taken from the centre of the above-mentioned gap and shall be 0,25 m or 0,15 m on each side (depending on whether the test pieces are 0,50 m or 0,30 m square, respectively).

5.2.3 *Power supply to hot plate*

The power supply to the guarded hot plate, which shall be stabilized within less than 0,5 %, may be either a.c. (distortion lower than 5 %) or d.c.

For a.c., the power shall be measured by means of a wattmeter class 0,5 or 0,2 allowing for an evaluation of electrical power to within 1 %.

For d.c., the power shall be measured by the potentiometric method to a maximum uncertainty of accuracy of 1 % of the power.

5.2.4 *Power supply to overheating section*

The power supplied to the overheating section shall also be stabilized. This overheating is usually automatically controlled and shall provide for correction of isotherms so that the absolute value of the temperature difference of the differential thermocouples located between the central square and first guard section is less than 0,02 K [see 5.4 b)].

5.3 Cooling plates

The cooling plates shall each contain a fluid circuit in the form of a nearly square double spiral which allows fluid to circulate in both directions for a better stabilization of temperature.

The temperature on the surface of the plates shall be stabilized at better than 0,1 °C and the fluid supply to the plates shall be controlled by thermostats adjusted to ensure this stabilization. The adjustment for each plate shall be identical.

5.4 Thermocouples

The temperature is measured by means of thermocouples, calibrated to within 1 μV, and a potentiometric bridge allowing for readings with an error of less than 10^{-3} in relative value (or with an error of less than 2 μV for low values of potential difference).

These thermocouples shall be made of wire of diameter not larger than 0,2 mm. The cold junctions shall be immersed to the same depth in melting ice held in a Dewar flask. The temperature in the Dewar flask is to be checked by a precision thermometer.

The thermocouples intended to measure the temperature of the surfaces shall be electrically insulated from the heating and cooling plates by insulation having a resistance of at least 1 GΩ.

a) Five thermocouples shall be set on each side of the test pieces, as shown in the diagram of figure 3 (thermocouples, a, b, c, d and 1).

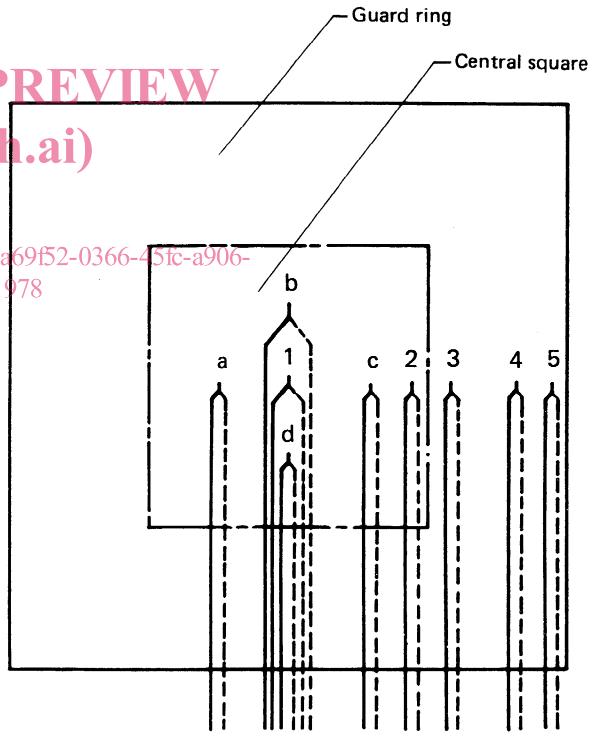

FIGURE 3

The central thermocouple (1) is intended primarily to measure the surface temperature; the four series-installed differential thermocouples (a, b, c and d) are intended to check the homogeneity of the temperature on their plane and also to improve the accuracy of the determination of the temperature difference between hot and cold surfaces.

Optionally, four more thermocouples (2, 3, 4 and 5) may be installed to check the distribution of temperatures from the centre to the sides of the test pieces.

b) There shall be at least twenty differential thermocouples, series-installed (see figure 4), to measure the temperature differences between the central square and the first guard ring.

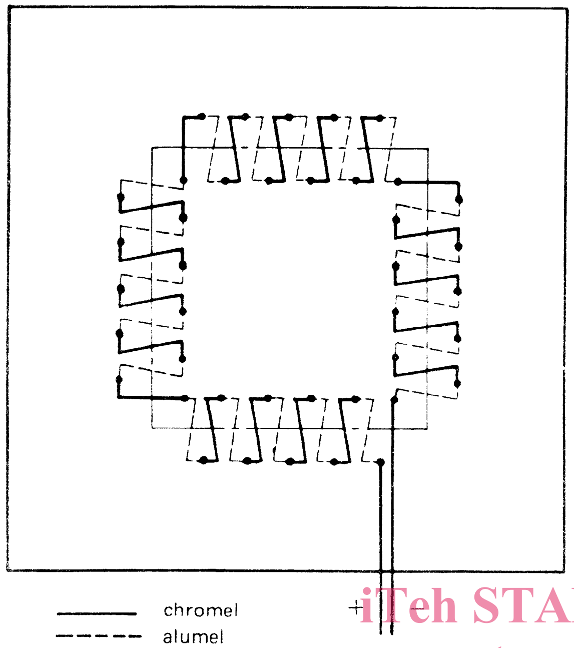

FIGURE 4

Each junction shall be at a distance of 15 mm from the boundary between the central square and the guard ring. The wires forming the isothermal guard of each junction shall be at least 30 mm long and set parallel to the side of the square which they check.

5.5 Edge insulation

5.5.1 Edge insulation may be ensured by means of a heat-insulating material at least 0,25 m thick, composed of a granular material less than 2 mm in grain size and with the lowest possible thermal conductivity, preferably lower than that of the material being tested and in any case not exceeding 0,05 $W \cdot m^{-1} \cdot K^{-1}$ (0,04 $kcal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$).

5.5.2 Measurements which are to be made when cooling to lower than room temperature shall be carried out in an air-tight container to avoid the transfer of moisture to the material during the procedure.

An additional cooling device for the insulating material is mandatory when the cooling plates have to be at lower than room temperature. This is to avoid any reverse edge flow and thus to rectify the isotherms in the test pieces by correcting the outward edge flow.

5.5.3 The quantity of material needed for edge insulation may be reduced by adjusting temperature and humidity. In such cases the adjusted room temperature should be selected so as to allow for the easy correction of the outward edge flow through the overheating of the guard ring, and the degree of humidity should be set at a level which results in the absence of moisture transfer between the test pieces and the surrounding atmosphere.

6 TEST PIECES

The samples from which the test pieces are to be prepared shall be taken in accordance with the specifications applying to the material to be tested.

6.1 Cutting the test pieces and determining their apparent density

6.1.1 When dealing with solid, one-piece materials, the test pieces must be identical, must come from the same source and must have plane and parallel surfaces. The tolerances allowed on flatness and parallelism shall be laid down in the specification applying to the material being tested.

Test pieces with 0,50 m sides shall if possible have a thickness e under 7 cm and in any case not exceeding 9 cm. Minimum thickness shall be 1 cm in every case. In the case of material which is not manufactured with a thickness of at least 1 cm, the test report shall mention only the determination of the thermal resistance of the material in question.

Test pieces with 0,30 m sides shall if possible have a thickness under 3,5 cm, and in any case not exceeding 4,5 cm. Minimum thickness remains unaltered (1 cm).

The difference between thickness of each of the two test pieces and their average thickness must be lower than 1 % of this average thickness.

After cutting the test pieces, determine their mass and dimensions and calculate their apparent density.

6.1.2 In the case of powder and granulated products the apparent density shall be determined in accordance with the standardized method applying to the product in question.

6.1.3 For fibrous materials or materials liable to deformation, in slabs or sheets, determine the mass of the test pieces and calculate the apparent density on the basis of the nominal thickness of the product.

The apparent densities of the two pieces shall not differ by more than 6 % for solid materials or by more than 4 % for materials liable to deformation.

6.2 Conditioning of test pieces

Test pieces shall be conditioned according to the specification applying to the material being tested.

Measure

- the mass of the test pieces as submitted to the test;
- the mass of the test pieces after testing, so as to determine any possible deviation in mass due to loss or regain of moisture or to any other factor; the test is valid only if no alteration in the mass of test pieces has occurred;

— the mass of the test pieces in the dry condition.

Calculate

— the apparent density of the test pieces in the dry condition and as submitted to the test;

— their moisture content by mass and by volume.

NOTE — In general, the dry condition is considered conventionally to be the state of equilibrium of the material in a ventilated oven set at 70 °C, air intake coming from an atmosphere at 20 °C and 65 % relative humidity. Special drying conditions may be required under the specifications applying to certain materials.

7 PROCEDURE

7.1 Setting-up procedure

Place the test pieces in the testing device. Test pieces of materials liable to deformation (insulating sheet materials, fibrous materials, etc.) have to be positioned allowing for a spacing between heating and cooling plate equal to the nominal thickness of the material being tested. This spacing may be obtained by any means provided that it prevents the formation of interfering thermal short-circuits between hot and cold plates and introduces a minimum of contact surface. For example, small non-deformable blocks may be used to form an outer rim to the device.

After the normal check of the electrical insulation of the several measuring elements, start the supply of electric power to the hot plate so as to reach the required temperature gradient, taking a hypothetical value for the thermal conductivity. Start the cooling fluid circulation to obtain the selected average temperature. Adjust edge overheating if this is controlled by hand; if automatically controlled, the overheating will progress with the development of the steady-state condition of the test piece.

7.2 Determination

7.2.1 The first measurements are generally made 12 to 24 h after starting the test. Further measurements are made at least at 4 h intervals. (The thermal capacity of the heating plate in the presence of highly insulating materials results in a considerable time constant.)

An example of a table for recording measurements is given in the annex.

7.2.2 The measurements and calculation are based on a given mean test temperature.

The table for recording measurements is to be filled in as follows :

Insert in columns 2 and 3 the power and the density of heat flow, and in columns 4 to 7 the mean temperatures of the hot surfaces (θ_{mh1}, θ_{mh2}) and of the cold surfaces (θ_{mc1}, θ_{mc2}) of the two test pieces.

Insert in columns 8 and 9 the mean temperature of each test piece, equal to the average of the mean temperatures of the hot and cold surfaces of the test piece during the test :

$$\theta_{m1} = \frac{\theta_{mh1} + \theta_{mc1}}{2} \qquad \theta_{m2} = \frac{\theta_{mh2} + \theta_{mc2}}{2}$$

Insert in column 10 the mean test temperature, equal to the average of the mean temperatures of the two test pieces :

$$\theta_m = \frac{\theta_{m1} + \theta_{m2}}{2}$$

Insert in columns 11 and 12 the temperature difference between the surfaces of each test piece, i.e. the difference between the mean temperatures of the hot and cold surfaces of the test piece :

$$\Delta T_1 = \theta_{mh1} - \theta_{mc1} \qquad \Delta T_2 = \theta_{mh2} - \theta_{mc2}$$

Insert in column 13 the mean difference in test temperatures, equal to the average of temperature differences in the two test pieces :

$$\Delta T_m = \frac{\Delta T_1 + \Delta T_2}{2}$$

Insert in column 14 ϵT, which is the temperature difference of the differential thermocouples located between the central square and the first guard section.

7.2.3 The steady-state condition is considered as having been attained once the following requirements are met :

a) the difference of the average temperature ϵT between the central square and the guard ring (at least twenty thermocouples) shall not exceed

$$\epsilon T \leqslant \frac{2 \times 10^{-4} \times \Delta T_m}{e}$$

where e is the mean thickness, in metres, of the test pieces.

b) the temperature difference between hot surfaces of the test pieces shall not exceed 1 % of the mean difference in test temperatures :

$$\theta_{mh1} - \theta_{mh2} \leqslant \frac{\Delta T_m}{100}$$

c) the temperature differences between the surfaces of each test piece do not deviate by more than 2 % of the mean difference in test temperatures;

$$\Delta T_1 - \Delta T_2 \leqslant \frac{2 \Delta T_m}{100}$$

d) at least three measurements made at intervals of at least 4 h shall show

— no power variation in excess of 1 % to either side of the average value;

— no variation of the mean difference ΔT_m between the surfaces of the test pieces in excess of 1 % to either side of the average value;

— no variation of the temperature imposed on the cold plates in excess of 0,1 K.

8 EXPRESSION OF RESULTS

8.1 The thermal conductivity, λ_m, expressed in watts per metre kelvin, at the mean test temperature is given by the formula

$$\lambda_m = \frac{\Phi \times e}{S \times \Delta T_m}$$

where

Φ is the heat flow rate in watts, equal to half of the electric power dissipated in the heating plate;

e is the average thickness, in metres, of two test pieces;

S is the surface area, in square metres, of the central square up to the gap separating it from the first guard section, dimensions being measured from the centre of the gap;

ΔT_m is the mean difference in test temperatures as defined in 7.2.2.

NOTES

1 For each value of the average flow or of the average temperature, the thermal conducitivity λ_m may be determined in relation to several values, positive or negative, of the difference ϵT between the central square and the guard section.

It is permissible to interpolate λ_m for $\epsilon T = 0$ provided that the interpolation graph be enclosed with the test report.

2 The conversion factor to convert λ_m into kilocalories per hour metre kelvin is 0,86 :

λ_m (kcal·h^{-1}·m^{-1}·K^{-1}) = 0,86 λ_m (W·m^{-1}·K^{-1})

8.2 The variation of the thermal conductivity or of the thermal resistance in relation to the mean test temperature shall be shown by a graph.

9 TEST REPORT

The test report shall include the following information :

a) the characteristics identifying the material;

b) the dimensions of the test pieces;

c) the conditioning, in case this is required by the specification applying to the material tested, and the behaviour of the test pieces during the conditioning process or the measuring process, as well as the humidity conditions having prevailed during the test;

d) the value of the average thermal conductivity calculated for each mean test temperature, as well as the graph showing the variation in thermal conductivity with that of the mean test temperature.

NOTE — When the material is not sufficiently homogeneous to permit a report on its thermal conductivity to be issued but nevertheless has the characteristics needed to be tested by this method, the report shall mention all the characteristics referred to above and furthermore the value of the average thermal resistance calculated for each mean test temperature and, if warranted, a graph plotted to show the variation of the thermal resistance with that of the mean test temperature.

e) reference to this International Standard;

f) any procedural details that may be optional or not specifically foreseen in this International Standard and also any possible occurrence that might have affected the results.

ANNEX

EXAMPLE OF TABLE FOR RECORDING MEASUREMENTS

(Not part of the standard)

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Date, hour	Power P on hot plate	Flow density $\frac{P}{2S}$	θ_{mh1}	θ_{mh2}	θ_{mc1}	θ_{mc2}	θ_{m1}	θ_{m2}	θ_m	ΔT_1	ΔT_2	ΔT_m	ϵT	λ_m	
	W	$W \cdot m^{-2}$	°C	°C	°C	°C	°C	°C	°C	K	K	K	K	$W \cdot m^{-1} \cdot K^{-1}$	$kcal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$