# INTERNATIONAL STANDARD

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# Ageing of thermal insulation materials — Determination of the long-term change in thermal resistance of closed-cell plastics (accelerated laboratory test methods)

*Vieillissement des matériaux isolants thermiques — Détermination du changement à long terme de la résistance thermique des plastiques alvéolaires à cellules fermées (méthodes d'essai de laboratoire accélérées)*

Reference number
ISO 11561:1998(E)

**ISO 11561:1999(E)**

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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 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 11561 was prepared by Technical Committee ISO/TC 163, *Thermal insulation*, Subcommittee SC 1, *Test and measurement methods*.

Annexes A and B of this International Standard are for information only.

## Introduction

The purpose of this International Standard is to determine the ageing (long-term decrease in thermal resistance) of closed-cell cellular plastic materials and products which have properties that, due to diffusion of contained gases, change with time. The thermal resistance and its rate of change will vary with product variability, temperature and thickness, and also within the thickness due to cross-sectional variability and the effects of natural or applied surface skins or protective facings.

The long-term thermal resistance is one property required for establishing design thermal performance under service conditions and for determining life-time energy requirements.

This International Standard contains two procedures based on the conditioning of thin slices at room temperature, since conditioning at elevated temperatures can induce changes in a material other than those due to diffusion processes. The first, method A, relates to the core material only. An alternative, method B, is a simplified test to determine a conservative value of a design life-time thermal performance of a product. Two informative annexes provide essential background information on the ageing process and on the factors to be considered when measurements are required on faced products.

The phenomenon and mechanisms of ageing have been known and understood for many years. The use of a blowing agent produces a relatively uniform cell size and initial high thermal resistivity. However, during the subsequent life of the foam, the principle component gases in the air diffuse into the cells and this increases the cell gas pressure, effectively increasing the thermal conductivity of the gas mixtures. In addition, some of the blowing agent is absorbed by or dissolved into the polymer matrix, saturating it, while the remainder diffuses out. This inward diffusion is influenced by appropriate diffusion coefficients. These in turn are influenced by the temperature, effective cell diameter in the direction of movement of air components, and the nature of the polymer matrix.

Since the diffusion of nitrogen and oxygen molecules into the cells is very much faster than the outward diffusion of the generally used larger molecule (the blowing agent), the whole ageing process is a combination of two stages:

a) a primary stage (thermal drift) due to the significant rate of change of cell gas composition (usually complete within 5 years);

b) a secondary stage where air diffusion is complete but there is still very slow outward diffusion of the blowing agent (a period much greater than 10 years and estimated in some cases to be over 100 years).

# Ageing of thermal insulation materials — Determination of the long-term change in thermal resistance of closed-cell plastics (accelerated laboratory test methods)

## 1   Scope

This International Standard specifies two laboratory test methods, based on slicing and scaling techniques, to determine the long-term changes in the thermal resistance of closed-cell (normally 90 %) cellular plastic materials that contain gases which, through diffusion processes, affect the properties of a foam with time.

Using standard methods for the measurement of thermal resistance, method A consists of periodic measurements performed over a short time interval on thin specimens conditioned in a controlled ambient temperature environment. The results of relative change with time are used in conjunction with a mathematical technique to derive the thermal resistance of greater thicknesses of the material as a function of time.

Method B describes a simple test to determine a conservative design life-time value (25 years and longer) for an unfaced, closed-cell, cellular plastic product. This method is limited currently to unfaced homogeneous materials. For this method, multiple specimens of the core and surfaces of materials with variations in the slope of the primary stage thermal resistivity and a time relationship of less than 10 % within a sample are considered to be homogeneous. Generally, products with natural skins or with density deviations normally found with such products may be considered acceptable for test by this technique.

## 2  Normative references

The following normative document contains 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 edition of the normative document 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 7345, *Thermal insulation — Physical quantities and definitions*.

ISO 8301, *Thermal insulation — Determination of steady-state specific thermal resistance and related properties — Heat flow meter apparatus*.

ISO 8302, *Thermal insulation — Determination of steady-state thermal resistance and related properties — Guarded hot plate apparatus*.

ISO 9346, *Thermal insulation — Mass transfer — Physical quantities and definitions*.

## 3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 7345 and ISO 9346, and the following apply.

NOTE    A list of symbols is given in Table 1.

**3.1**
**ageing**
process by which the physical, mechanical and thermal properties of a material, product, or system change with time

NOTE 1    Accelerated tests can introduce effects that will not occur during natural ageing. Care should be taken when developing such test methods and methods of evaluation of test results.

NOTE 2    Examples of ageing processes are the settling of some low-density fibrous materials and gas diffusion in cellular plastics.

NOTE 3    Whatever the process, ageing is always strongly related to the environmental or service conditions to which the material, product or system is exposed and to its shape, size and finish. Accurate prediction of ageing effects should always consider these items.

**3.2**
**aged value**
value of a property of a material, product or system after exposure to known environmental conditions for a specified time

**3.3**
**accelerated aged value**
aged value obtained through laboratory test or through reproducible prediction models for a specified time interval and specified environmental conditions aimed at reproducing frequently encountered service conditions

NOTE    The actual size, working environment and expected life-time should be compared carefully with those implied in the definition of an aged value.

**3.4**
**design life-time**
time interval during which an installed material, product or system should maintain its design performance

NOTE    Life-time depends upon the serviceability of the installation, for example, the design life-time for a thermal insulation material, product or system used in a building might be at least 25 years.

**3.5**
**effective diffusion coefficient**
material property which relates the rate of gas transport to the gas pressure difference across the material having a specific thickness at a specific temperature

**3.6**
**primary stage**
portion of the ageing process where changes in thermal properties are influenced primarily by diffusion of gaseous components such as nitrogen and oxygen into or out of the closed-cell material

**3.7**
**scaling factor**
ratio of the squares of the product and test specimen thicknesses

NOTE    This ratio represents the acceleration rate being applied to the ageing process of a cellular plastic due to thickness differences.

**3.8**
**secondary stage**
portion of the ageing process where changes in thermal properties are influenced primarily by the diffusion of blowing agents usually high molecular weight gases out of the closed-cell material

**3.9**
**thickness of damaged surface layer (TDSL)**
average thickness of surface cells, on one surface, which are ruptured or otherwise damaged during preparation of the test specimen

**3.10**
**transition point**
estimated age of a closed-cell cellular plastic when the ageing process changes from the primary to secondary stage

NOTE     This is also called the transfer point.

**Table 1 — Symbols**

| Symbol | Quantity | Unit |
|--------|----------|------|
| $a$ | thermal diffusivity (thermal diffusion coefficient) | $m^2/s$ |
| $\lambda$ | thermal conductivity | $W/(m \cdot K)$ |
| $\rho$ | density | $kg/m^3$ |
| $c_p$ | specific heat capacity at constant pressure | $J/(kg \cdot K)$ |
| $D$ | effective gas diffusion coefficient | $m^2/s$ |
| $D_0$ | effective gas diffusion coefficient of a reference slab | $m^2/s$ |
| $d$ | thickness of specimen (slices) | m |
| $d_0$ | thickness of a reference slab | m |
| $F$ | numerical coefficient | |
| $F_0$ | Fourier number | |
| $i$ | day $i$ of ageing period | day |
| $n$ | ageing period | days |
| $r$ | thermal resistivity | $m \cdot K/W$ |
| $r_0$ | initial thermal resistivity | $m \cdot K/W$ |
| $r_t$ | thermal resistivity of a test specimen after time $t$ | $m \cdot K/W$ |
| $R$ | thermal resistance | $m^2 \cdot K/W$ |
| $R_{av}$ | average thermal resistance during ageing period | $m^2 \cdot K/W$ |
| $R_0$ | initial thermal resistance | $m^2 \cdot K/W$ |
| $R_t$ | thermal resistance of a test specimen after time $t$ | $m^2 \cdot K/W$ |
| $R_n$ | thermal resistance on last day of ageing period | $m^2 \cdot K/W$ |
| $S$ | scaling factor | |
| $t$ | time | day |
| $t_0$ | initial time | day |
| $T$ | temperature | K |
| $T_1, T_2$ | uniform surface temperatures of a heated or cooled slab | K |
| $T_m$ | mean slab temperature | K |
| TDSL | thickness of damaged surface layer | m |

## 4 Test methods — General

### 4.1 Conditions

A summary of the analytical model that serves as the basis for the slicing and scaling method is given in annex A. The experimental procedure is based on the assumption that the material characteristics of thin specimens are equivalent to those of the material being investigated, i.e. specimens of reduced thickness have the same effective diffusion coefficient and initial cell gas content as those of the full-thickness material and that one-dimensional diffusion is the dominating factor.

Conditioning of full-thickness samples to obtain a service life 25 years or longer value requires too long a period of testing.

### 4.2 Effects of influencing parameters

Annex A also contains brief details of other important factors which affect the ageing process:

— errors due to small thickness used;

— thickness of damaged surface layers;

— ageing prior to preparation of specimen;

— inhomogeneities in the material;

— high and/or inconsistent open-cell content.

## 5 Method A — Test to determine time-dependent change in thermal properties of core materials

### 5.1 Principle

Method A is a general procedure for determining change in thermal resistance at any time due to reduced conditioning time. Random samples of a cellular plastic product having a thickness greater than 25 mm are selected and, from each, separate thin test specimens are prepared having a uniform thickness of approximately 10 mm, but not less than 6 mm, with allowance being made for cell damage at each cut surface. In order to minimize the effects of preparation on resultant thermal performance, it is recommended that the specimen thickness be such that the TDSL is no greater than 5 % of the geometric thickness. Measurements of thermal resistance, of the specimens are made at regular time intervals from an initial starting time, as soon as possible after manufacture, until values of the ratio of the thermal resistance to that at time zero indicate that the specimen has passed from the primary into the secondary stage of the ageing process. A value for the aged thermal resistance of the product is then obtained by use of scaling factors.

### 5.2 Sampling

Select in conformance with appropriate standard sampling procedures. The pieces shall be prepared as soon as possible and practical after production (a recommended minimum of 3 days). This is to ensure that the cell gas content within the sample is stabilized and representative of the initial conditions.

### 5.3 Preparation of specimens

**5.3.1** Condition the block, board, spray-applied or foamed-in-place samples at $(23 \pm 2)$ °C and $(50 \pm 5)$ % relative humidity of air in a climate-controlled room (usually less than 2 days).

**5.3.2** Depending upon the original thickness cut or otherwise, prepare each sample into separate thin slices of uniform thickness. Discard a surface slice if it is protected by any type of diffusion-tight membrane. The overall area should be not less than the equivalent minimum metering area of the test apparatus. To avoid undue warpage of a test specimen for large total area apparatus, it may be necessary to cut slices as two pieces: one to provide the central test section a little larger than the apparatus metering area, plus a second surrounding annular section of the same thickness.

**5.3.3** When measurements are to be carried out on several slices stacked together, the edges of each should be marked to ensure that correct realignment of the stack can be maintained after slicing, conditioning of separate slices, and testing.

NOTE     Several suitable general techniques for slicing and preparing uniformly thick specimens with minimum amounts of surface damage have been developed and shown to provide repeatable and comparable results.

Mount the sample on a lathe, holding it in position by means of air suction. Perform the slicing by multiple cuts on alternate surfaces with a counter-rotating, delicatessen-type, meat-slicing blade.

The sample may be similarly sliced using a band saw with a fine tooth blade. Mount the slice on the vacuum table of a grinding machine and maintain it in position by air suction. Multiple cuts are performed on alternate surfaces to obtain the required thickness.

For thermoplastic materials, a thin hot-wire cutter may be used.

## 5.4  Measurement of dimensions of slices

**5.4.1**  Measure the length and width of each slice to $\pm$ 2 mm.

**5.4.2**  Measure the thickness of each slice to $\pm$ 0,02 mm. For a stack of slices, measure the thickness to $\pm$ 0,2 mm.

**5.4.3**  If a thermal resistivity value is required, calculate an effective thickness by reducing the value obtained in 5.4.2 by the thickness of the damaged surface layer (TDSL), obtained by measurement, or use the amount equivalent to two cell diameters.

NOTE     For many materials one cell diameter has been found to be representative of the TDSL.

**5.4.4**  It is recommended that steps 5.4.1 and 5.4.2 be repeated on one slice after the measurements of thermal resistance have been completed, in order to ascertain that dimensional or other changes have not occurred during the test period.

## 5.5  Measurement of thermal resistance and resistivity of slices

**5.5.1**  Measurements shall be undertaken at one selected temperature either in accordance with ISO 8301 (standard heat flow meter method) or ISO 8302 (guarded hot plate methods) in a climate-controlled room. When a specimen of stacked, separately conditioned, slices is measured, a sufficient number shall be stacked together to ensure that the thickness-independent thermal conductivity is obtained.

NOTE     Experience has indicated that for a density greater than 30 kg/m$^3$, a thickness in excess of 40 mm is adequate.

**5.5.2**  The recommended mean temperatures of the test are either $(10 \pm 2)$ °C or $(23 \pm 2)$ °C. The maximum temperature difference established across the test specimen shall not exceed $(24 \pm 1)$ °C or be less than 5 °C.

NOTE     The mean temperature should not affect the relative behaviour. Two mean test temperatures have been included to allow for the differences between the practices used in Europe and elsewhere in the world.

**5.5.3**  Determine the initial thermal resistance $R_0$ (also, if needed, the derived thermal resistivity $r_0$) of the single slice or stacked slices in accordance with the slected standard method more than one day after slicing.

**5.5.4**  Condition the slices separately at $(23 \pm 2)$ °C and $(50 \pm 5)$ % RH and determine the thermal resistance $R_t$ at regular intervals following each conditioning period. Calculate the relative thermal resistance ratio $R_t/R_0$ and plot the value versus the logarithm of time. (If required the relative thermal resistivity ratio may be used.)

**5.5.5**  Repeat step 5.5.4 until the values of $R_t/R_0$ or $r_t/r_0$ provide a linear relationship in the plateau regime beyond the transition point (see figure A.1).

## 5.6  Calculation of long-term thermal resistance for a product

**5.6.1**  Although different thicknesses of "thin" specimens are being used, a standard or reference value of 10 mm is recommended in order that reliable comparisons of results can be made.

**5.6.2** Determine the average of the thermal resistance values of the specimens and, using the scaling factor concept, convert the curve of experimental results obtained in 5.5 to one for a 10 mm thickness using equation (A.11).

**5.6.3** Use the value obtained in 5.6.2 with a scaling factor to derive the thermal resistance of the full thickness of the number of samples tested at the specific time required.

**5.6.4** As an example, the thermal resistance after 25 years $R_{25}$, and an average value $R_{av,25}$ over the same period for a 50 mm thick product can be derived as follows.

a) Thermal resistance after 25 years for a 50 mm product:

A period of 25 years is 9125 days. Thus for a thickness of 10 mm and according to equation (A.11), this is equivalent to a time:

$$t = 9125\,S = 9125\,(10/50)^2 = 365 \text{ jours} \tag{1}$$

From the curve obtained for the 10 mm slices, determine the thermal resistivity at day 365, $r(10;365)$,

thus

$$r(50;9125) = r(10;365) \tag{2}$$

and

$$R_{25} = 0{,}05\,r(50;9125) \tag{3}$$

b) Average thermal resistance over 25 years for a 50 mm product:

According to equation (A.5), the average thermal resistivity over 25 years (9125 days) will be equal to the thermal resistivity after $9125/\sqrt{10} = 2886$ days.

Thus for a slice thickness of 10 mm, this is equivalent to a time

$$t = 2886\,S = 2886\,(10/50)^2 = 115 \text{ days} \tag{4}$$

From the curve obtained for the 10 mm slices, determine the thermal resistivity at day 115, $r(10;115)$,

thus

$$r_{av}(50;9125) = r(50;2886) = r(10;115) \tag{5}$$

and

$$R_{av,25} = 0{,}05\,r_{av}(50;9125) \tag{6}$$

**5.6.5** Where required, determine appropriate aged thermal resistance values for the product by applying a scaling factor.

# 6 Method B — Simplified test to determine a design life-time thermal resistance of an unfaced product

## 6.1 Principle

Method B is based on principles and criteria described clauses 4 and 5. It involves measuring the thermal resistance of a stack of approximately 10 mm thick slices cut from the original product after they have been conditioned at room temperature individually for $(91 \pm 7)$ days. The method has been shown experimentally to yield a $(25 \pm 2)$ year value for a thickness of 100 mm. For other thicknesses, there may be a small error which is a function of the diffusion coefficient and thermal conductivities of the gases in question.

NOTE    This has been found to have less than a 5 % effect for practical thicknesses.

## 6.2 Procedure

Select replicate samples and test pieces in accordance with 5.2. The period between the production and preparation of test pieces shall not exceed 1 year.

Mark the edges of test pieces to ensure that correct realignment of a stack of the specimen slices can be attained after cutting and subsequent conditioning.

Cut each replicate test piece into a number of thin slices (10 ± 1) mm thick, retaining the surface skins where possible. No additional surface preparation is required.

Measure the thickness of each slice and of each stack of realigned slices before and after conditioning.

Condition the individual slices at (23 ± 2) °C and (50 ± 5) % RH for (91 ± 7) days.

Following the conditioning, reassemble the specimens into replicate complete stacks.

Measure the thermal resistance of each stack in accordance with ISO 8301 or ISO 8302 at a mean temperature of (10 ± 2) °C or (23 ± 2) °C and a maximum temperature difference of (24 ± 1) °C. A sufficient number of slices shall be stacked together to ensure that the thickness-independent thermal conductivity is obtained.

Average the results of the appropriate number of replicates.

## 7 Precision

Due to the long time periods involved in obtaining data on full sized specimens, no definitive statement can be made on the precision and bias of this method.

For method A, limited information available from a study in the USA over 3 to 5 years indicates that the agreement between actual performance and that derived from slicing and scaling tests is much better than 10 %.

For method B, results from tests in Scandinavia over longer periods of time on 100 mm thick specimens indicate that agreement between the results by the two methods is within 5 %.

## 8 Test report

The test report shall contain at least the following information:

a)   all details necessary to identify the product tested;

b)   a reference to this International Standard (ISO 11561) and a statement of compliance, including a list of any deviations;

c)   identification of testing organization;

d)   identification of client or sponsor of the test;

e)   date of manufacture, where known, and date of receipt for evaluation;

f)   date and details of specimen preparation;

g)   use of method A or method B;

h)   description of test specimens and their relationship to the sample supplied;

i)   dimensions, mass and derived bulk density;

j)   effective thicknesses of test specimen where required;

k)  description of test apparatus, including references to appropriate International Standard and any deviations;

l)  mean temperature and temperature difference used in the tests;

m)  date of start of test period on single slices or of conditioning of slices of a stack and dates of subsequent thermal resistance  tests on single slices, or thermal resistance tests on stacked slices;

n)  long-term thermal resistance value(s) for required time period(s) using the averaged results on one or more slices of the replicate specimens with appropriate scaling factor(s);

o)  a design life-time value in accordance with 3.4 from the measurement on the stacked slices of the averaged values of the replicate specimens using the simplified test.

# Annex A
## (informative)

# Analytical model

## A.1   Accelerated ageing

Several "accelerated" ageing tests have been developed over the years in an attempt to determine the long-term thermal performance behaviour, and some have been incorporated into national standards. In general these methods involve measurements at regular time intervals from some initial or "zero" time of the thermal resistance of a uniformly thick (25 mm or greater) specimen(s), conditioned either at some controlled elevated temperature or at ambient room temperature. Depending upon the standard, the elevated temperatures range between 60 °C and 100 °C, and times vary from 90 days to 10 days. Also, the conditions 180 days or longer at room temperature have been used.

The long-term thermal resistance of the specimens is then determined from an extrapolation, based on some mathematical model, of the curve generated by plotting thermal resistance against time. A number of models of varying complexity have been developed, especially over the last 10 years. These have been instrumental in allowing an improved understanding of the ageing process to be obtained and also in reducing, to some extent, the uncertainty in the derived long-term resistance value.

None of the above procedures has proved to be totally satisfactory as a generally accepted procedure for estimating long-term thermal resistance. For some materials, in addition to increasing gas diffusion rates by an order of magnitude, exposure to elevated isothermal temperatures for long periods of times (i.e. conditions not normally experienced by the material in use) can or may cause other changes in properties not associated with the ageing process and thus provide incorrect information. Furthermore, recent intense search for alternative blowing agents to replace CFCs requires that a more rapid test method be available.

Thus the most acceptable method would require that the elevated temperature parameter be eliminated and that conditioning be carried out at or close to room temperature. This involves either longer times or reduced thicknesses. Significant and promising research work has been carried out in various countries on different types of cellular plastics using test specimens having uniform thicknesses of 10 mm or less and measuring changes of thermal performance over much shorter time periods.

The present test methods are based on using this concept, which is commonly described as the slicing and scaling technique.

## A.2   Choice of thin specimen

In most studies of ageing of cellular plastics, the thermal resistivity $r$ (the reciprocal of thermal conductivity) is expressed as a function of the logarithm of time:

$$r = F (\log t) \tag{A.1}$$

Furthermore, the results are presented as a dimensionless thermal resistivity using a value of $r$ at an initial "zero" time (usually a few days) as a reference level:

$$r_t/r_0 = F (\log t) \tag{A.2}$$

This provides a means to understand the general character of the ageing process without paying undue attention to absolute values.

If $R_t$ is the thermal resistance after time $t$ and $R_{av,n}$ is the average thermal resistance over a period of $n$ days, then:

$$R_{av,n} = \frac{\Sigma R_t}{n} \tag{A.3}$$

Assuming a logarithmic dependence of $R$ with time, equations (A.1) and (A.2) give:

$$(R_i - R_n)/(R_0 - R_n) = 1 - \lg i/\lg n \tag{A.4}$$

where $R_0$ and $R_n$ are the thermal resistance on the first and the last day of the ageing period.

Thus the average thermal resistance $R_{av}$ over $n$ days is equal to the thermal resistance $R_t$ after $t$ days, where

$$t = \frac{n}{\sqrt{10}} \tag{A.5}$$

Consider a period of 25 years (9125 days) as being a reasonable life time for an unsliced cellular plastic. Then it will take a period of 9125/3,16 or 2886 days to establish an average thermal resistance value.

## A.3 Analogy of heat and mass transfer

Both the ingress of nitrogen and oxygen into a closed-cell cellular plastic slab and the egress of a blowing agent from the cells can be described in a manner analogous to that used to describe heating or cooling of a slab.

Consider a slab at uniform temperature $T_1$, having its surface lowered to $T_2$. The mean slab temperature $T_m$ is given by:

$$T_m = (T_0 - T_2)/(T_1 - T_2) = 8\pi^2 \Sigma \, 1/(2n+l)\exp\left[-F_0(2n+l)^2\pi^2\right] \quad (n = 0) \tag{A.6}$$

where

$$F_0 = \frac{\alpha t}{d^2} = \lambda / \rho \, c_p \tag{A.7}$$

For large values of $F_0$ (i.e. long times or small thicknesses), the series in equation (A.6) converges rapidly. Thus equation (A.6) can be simplified to:

$$(T_m - T_2)/(T_1 - T_2) = 8\pi^2 \exp\left(-F_0\pi^2\right) \tag{A.8}$$

This implies that for a particular value of time (for a given thickness and thermal diffusivity), the process is strictly exponential. This is called the advanced stage of the diffusion process. Gas transfer can be treated similarly as a diffusion process where gas pressure corresponds to temperature, rate of pressure change to temperature change, and effective gas diffusion coefficient to thermal diffusivity.

Thus gas-transfer phenomena can be analysed similarly with the aid of equations (A.6) and (A.8). Equation (A.8) indicates that the logarithm of relative (dimensionless) pressure depends linearly on $F_0$, for large values of $F_0$, while equation (A.6) states that it is always proportional to $F_0$. To the extent that equation (A.6) describes the gas transfer process providing $F_0$ is constant, the scaling of the transfer process is carried out by determining the rate of diffusion for a thin specimen and relating it to a thick one. Thus to compare the diffusion rate evaluated for a slab with any set of properties ($d$ and $D$) with a reference slab ($d_0$, $D_0$) the measured ageing time must be multiplied by a scaling factor:

$$t = t_0 S \tag{A.9}$$

$$S = (d_0^2 D/d^2 D_0) \tag{A.10}$$

Assuming that $D = D_0$ for the material in question (i.e. it is homogeneous), the scaling factor may be expressed as a ratio involving thickness only, i.e.

$$S = d_0{}^2/d^2 \tag{A.11}$$

EXAMPLE

$$t_{10} = \frac{t_{100}(d_{10})^2}{(d_{100})^2} = 25 \times 365 \frac{(0,01)^2}{(0,1)^2} = 91 \text{ days} \tag{A.12}$$

## A.4   Modelling of the ageing process and use of scaling factors

Various models are available to illustrate the typical changes experienced in the ageing process. The thermal resistivity of the cell-gas changes, both as a function of location in the slab and the condition of ageing (elapsed time, temperature and effective diffusion coefficients of the nitrogen, oxygen and high-molecular-weight blowing agent affecting the cell gas compositions).

Using a given set of parameters for an "ideal" material, a model can be used to calculate the ageing characteristics. Each of the cell gases can be treated separately and the high-molecular-weight blowing agent alone has little effect since its molar concentration does not change in the absence of other gases; even though the diffusion rate for oxygen is six times higher than that for nitrogen, the latter gas plays the dominant role because its concentration is four times that of oxygen. However, changes in thermal performance due to air entering the cells and lowering the relative concentration of high-molecular-weight blowing agent become readily apparent.

A typical calculated characteristic curve of ageing for three thicknesses of an ideal material is illustrated schematically in Figure A.1. The ageing can be represented approximately by separate linear fits to a primary and a secondary stage. The transition point (in practice a zone) plays an important role in the analysis of thermal performance of cellular plastics since it corresponds to the change in the rate of the diffusion process.

The effect of thickness on the characteristics of the ageing curve is readily apparent. As the thickness is reduced, the time period is also reduced until the thermal resistivities become an exponential function of time.

Figures A.2 and A.3 present experimental curves against time and scaled time using two thicknesses of material. It is seen that by using the scaling factor $S$ all layers now follow a common curve. Thus measurements on thin specimens provide data from which the corresponding behaviour of a thick specimen of the same material can be obtained.

## A.5   Effects of influencing parameters

The advantages in the approach of using thin specimens and scaling factors are that this relatively simple technique can be used irrespective of gas content, diffusion coefficient, cell size or radiative properties of the polymer. However, a number of factors have to be considered in the overall application of the method. In particular, the application of scaling factors depends upon two parameters, namely the ratio of the square of thicknesses, and the ratio of the effective diffusion constants. Thus it is essential that measurements be made on a "correct" effective thickness. In addition the material or product studied should have uniform properties through the thickness (i.e. the diffusion constants should not differ widely between surface and core layers).

### A.5.1   Thickness errors

In practice, actual test specimens are generally of the order of 10 mm in the thickness. Currently, methods of preparing pieces of suitable area for the thermal resistance measurements can achieve a uniform thickness in the above regime with a maximum deviation of ±10 % and generally much less.
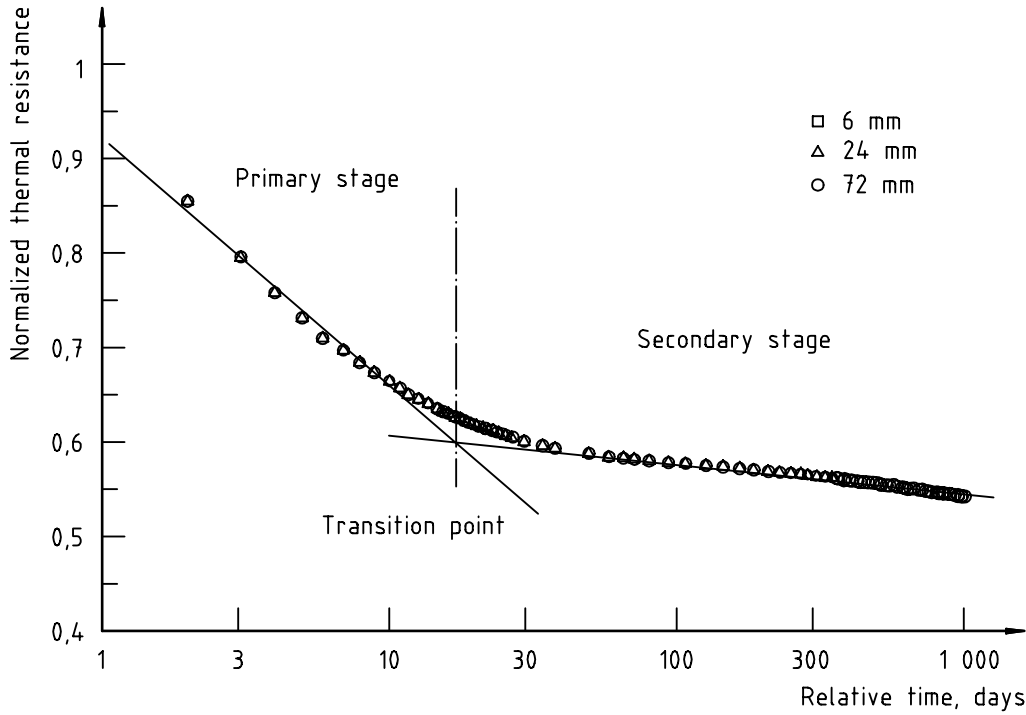
**Figure A.1 — Relative thermal resistance of three thicknesses of a cellular plastic foam after application of a scaling factor**
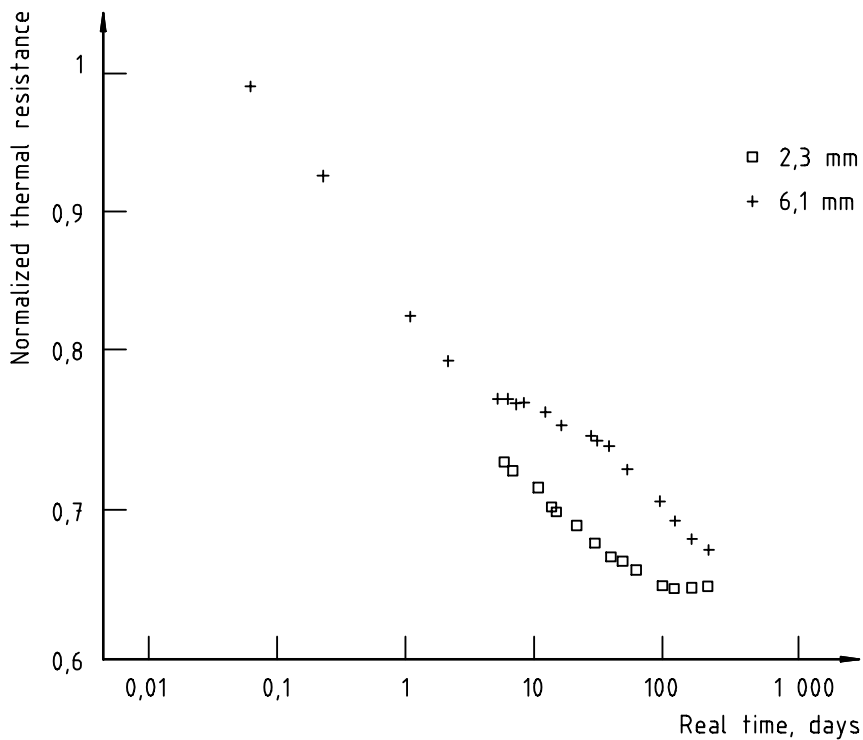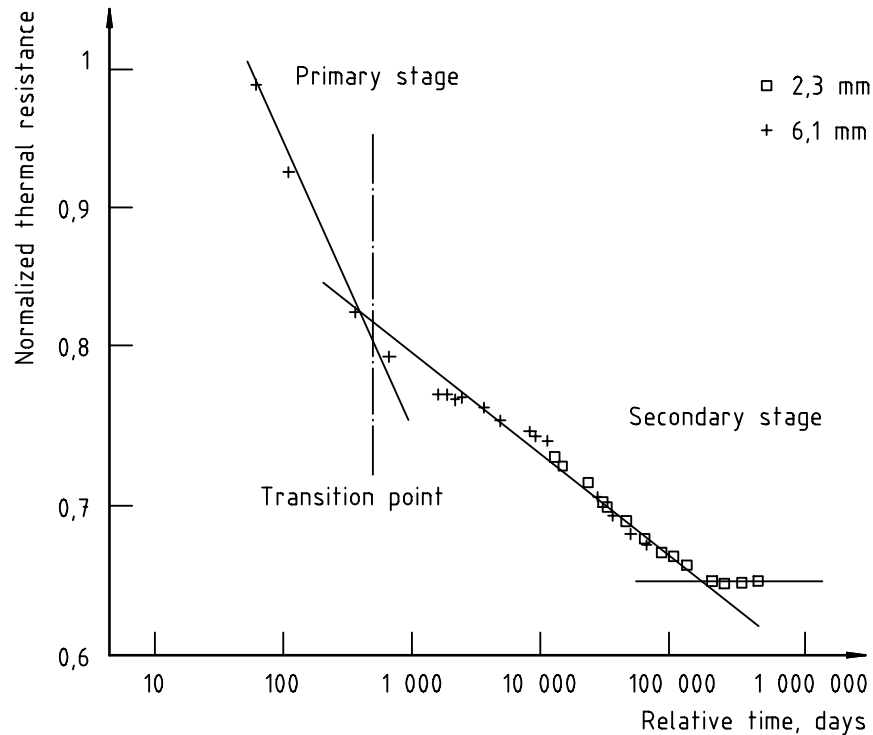


**Figure A.2 — Normalized thermal resistance of two thicknesses of a rigid closed-cell plastic foam as a function of real time**

**Figure A.3 — Normalized thermal resistance of two thicknesses of a rigid closed-cell plastic foam after application of the scaling factor**

## A.5.2 Damaged surface layers

Cutting of test specimens will increase the fraction of open cells in the particular material. This, reduces the "effective" thickness of the specimen and becomes more significant as the overall thickness is reduced. To correct for this reduction in thickness, the concept of TDSL has been introduced.

Measurements of effective specimen volume using either a gas burette or air comparison pycnometer and comparing this to the measured geometric volume provides the most accurate means of obtaining the effective thickness. The former is the most suitable and precise for specimens having a large surface-to-volume ratio, but a standard test method has not yet been developed.

In general, it has been found that this surface layer damage amounts to a maximum of one cell diameter on each cut surface. This is the amount that is recommended to be used until a suitable test method has been developed. The use of ratios of values minimizes the effects of this parameter.

The impact of air in the surface cells of a product can be estimated approximately as follows:

$$\lambda = \frac{d_s - 2x}{d_s/\lambda_s - 2x/\lambda_{air}} \tag{A.13}$$

Here $\lambda_{air}$ corresponds to a cellular plastic with a certain cell diameter $x$ filled with air. For a cell diameter of 0,5 mm, the value of $\lambda$ is 0,038 W/m·K. (This will vary from product to product.)

Thus, for a 100 mm thick product with a 0,5 mm cell diameter

$$\lambda = \frac{0,01 - 0,001}{0,01/0,028 - 0,001/0,038} = 0,0272 \text{ W/m·K} \tag{A.14}$$

The corresponding measured value was found to be 0,028 W/m·K.

### A.5.3   Ageing prior to preparation of test specimen

In studies of ageing phenomena, tests have usually been undertaken on test specimens prepared "immediately" (usually less that 5 days) after manufacture. In practice, however, the time between manufacture and test specimen preparation may be much longer. This parameter can influence the length of the conditioning period.

### A.5.4   Material inhomogeneities

In practice many "homogeneous" cellular plastic materials consist of a uniform core of closed cells of a material of a relatively constant density bounded by higher density surface layers. In some cases, the open cell content may exceed 10 % or there may be open cell non-uniformities within the core. In addition, for cellular plastics products a wide variety of facers or other surface membranes are often applied to reduce ageing effects.

These differing surface layers will affect the diffusion constants differently from those for the core material. Thus, the current method applies to the core only or to material where it can be shown that the diffusion rates for core and skin layers are similar (<10 %) such that an average value can be used. Alternatively corrections to material inhomogenities may be considered.

# Annex B

(informative)

# Example of the determination of long-term thermal resistance of faced products

## B.1 General

The present test method has been developed for unfaced homogeneous materials where an approximate constant diffusion coefficient can be assumed to exist for the total thickness including the higher density layers.

However, in practice, most current cellular plastic thermal insulation products, especially those based upon, polyurethanes (including polyisocyanurates) and phenolics, are composites. They are combined with layers of other types of thermal insulation and/or one or both of the surfaces are covered with coatings, facings, membranes or other surface skins, all serving some functional purpose depending upon the product application. In some cases, these skins are applied as an integral part of the product during the continuous-line foam production process, while in others they are applied separately following manufacture and cutting of the core foam material.

Any of these added surface layers will affect immediately the rate of diffusion of any fluids into or out of the product. Thus the present method is not satisfactory for determining the long-term thermal resistance of these practical products. In general, the ageing of the faced product will be slower than that of the core foam alone.

## B.2 Ageing factors

The ageing characteristics of these faced products is a somewhat complex subject due to the effects of several additional non-reproducible variables on the behaviour of the core. Thus, the core material may have well-defined behaviour but the product based on the core may exhibit highly variable behaviour, even between lots.

### B.2.1 Type and permeability of applied covering

Many different coverings are used and many thicknesses can be involved depending upon the application.

In general, where thermal insulation layers (e.g. perlite or fibrous) are added to form a composite product, the insulation materials have high gas permeability, and any overall effects on the ageing behaviour is relatively small.

For many spray-applied products, various types of polymer or similar coatings are used at different thicknesses (from less than 0,5 mm to well in excess of 1 mm). Providing they are applied correctly, these low permeability coatings impede gas exchange, depending upon both gas permeability and thickness.

The majority of faced products are those having two so-called "impermeable" facings other than thick metal sheets applied to the surfaces. These can vary from single layers of aluminium foil, plastics and fibre-reinforced plastics, papers and, more recently, composite layers of combinations of the above. In general, the thicknesses used are much less than 0,25 mm and often less than 0,1 mm. These types of material have low gas permeability and can be very effective barriers to ageing providing that the facing remains completely whole. However, as the facing thickness is reduced, there is a tendency for pin holes, flaws and similar imperfections to occur. Thus, gas permeability increases due not only to the reduced thickness but also to the imperfections, resulting in an overall loss of effectiveness.

### B.2.2 Adhesion of applied covering

The effectiveness of the facing also depends upon the use of a low-permeability adhesive and complete adhesion of the facing to the total surface of the product. Poor adhesion is manifested by the appearance of bubbles or wrinkles at the surface.

### B.2.3 Interaction between foams and applied coverings

In certain cases, at the time the facing is applied, interactions can occur which cause uncertain effects in the overall permeability of the product. Examples include spray-applied materials, where the chemistry of the coating and polymer foam are incompatible, or where the effect of the temperature of application produces cracks or other imperfections in the surface layers. In continuous-line production material, the speed at which the line moves in relation to the temperature history of the foam during the foaming process can produce shear cracks in the foam layers at the facing/foam interface. Although the facing may appear to adhere completely to the foam, gas exchange can take place along this shear-plane layer.

## B.3 Prediction of long-term thermal resistance of cellular plastic products

Due to the effect of so many variables, a test method (especially a relatively simple one) to determine this complex long-term behaviour is not a viable possibility at the present time. Other means have to be explored. Although, as discussed, it has limitations, a test procedure involving higher temperature exposure can be considered as one basis to characterize a foam and a faced product based on the foam.

NOTE     A considerable amount of long-term testing has been carried out in Europe and North America, where foams are conditioned at elevated constant temperatures especially after an initial "stabilization" period. In Europe, the conditioning is carried out at 70 °C for 63 days, while in North America it is undertaken at 60 °C for 90 days. In each case the chosen elevated temperature is "close" to the maximum that occurs locally in buildings. Results of these tests indicate that this conditioning may produce measured thermal conductivity values which are comparable to those obtained in the asymptotic regime, i.e. thermal resistivity in the secondary stage.

## B.4 Elevated-temperature test procedure

Select random samples of foam in accordance with appropriate sampling procedures.

Place the samples in a controlled elevated-temperature environment in accordance with the appropriate national requirements.

Condition them in this environment for the period of time appropriate to the national requirements.

At the end of this period, in accordance with required testing criteria, cut appropriate test specimens of suitable dimensions for the test apparatus from each sample panel.

Measure the thermal resistance and derived thermal conductivity of each specimen, using the heat flow meter method specified in ISO 8301 or the guarded hot plate method of ISO 8302, at the temperature appropriate to the national requirements.

Average the test results for the number of test specimens involved.

If there is any adjustment required by the relevant materials standard being used, it is then added to the average value of measured thermal conductivity to provide the value on which the long-term thermal resistance of the foam or product is to be derived.

## B.5 Calculation of long-term resistance of faced products

The ageing behavior of products can be modelled as a function of the properties of the core material, as measured above, or preferably by the basic test method modified by allowances for the effect of the permeability of the facings, adhesives and other variables. The latter are either determined directly or estimated from qualitative measurements.

The gas permeability of the particular facings and adhesive can be measured independently by appropriate standard methods.

The presence of holes and similar imperfections can be determined visually. Allowance for their effect can be made on a unit area basis.

The degree of adhesion of the facing and the existence and extent of any shear-plane layers can be determined by use of a dye penetrant technique. In this, a specimen is completely immersed, then evacuated and dried, followed by a visual observation. The existence of poor adhesion and any shear-plane layer is indicated by coloured areas. Allowance for their effect can be made on a unit area or equivalent unit area basis.

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