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**Thermal-insulating materials —  
Determination of freeze-thaw resistance**

*Matériaux d'isolation thermique — Détermination de la résistance au gel-dégel*

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## Foreword

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The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20394 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 10, *Cellular plastics*.

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# Thermal-insulating materials — Determination of freeze-thaw resistance

## 1 Scope

This International Standard specifies the equipment and procedures for determining the effect, on the mechanical properties and moisture content of a product, of the successive cycling of the product from dry conditions at  $-20\text{ }^{\circ}\text{C}$  to wet conditions (water) at  $+20\text{ }^{\circ}\text{C}$ . It is applicable to thermal-insulation products.

The method is intended to simulate freeze-thaw effects on thermal-insulation products which are frequently exposed to water and low-temperature conditions, e.g. inverted roofs and unprotected ground insulation.

This test method is not recommended for all thermal-insulation products. It will normally be clear from the product standard if this International Standard is applicable to a particular product.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2896, *Rigid cellular plastics — Determination of water absorption*

ISO 20392, *Rigid cellular plastics — Determination of long-term compressive creep*

ISO 20393, *Rigid cellular plastics — Determination of long-term water absorption by diffusion*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **freeze-thaw resistance**

ability of a product to withstand repeated wetting followed by freezing conditions, this ability being quantified in terms of the water absorption and the change in compression behaviour

## 4 Principle

The freeze-thaw resistance is determined as the change in the amount of water absorbed and the change in compression strength or stress of a test specimen which is subjected to 300 successive cycles from dry conditions at  $-20\text{ }^{\circ}\text{C}$  to wet conditions (water) at  $+20\text{ }^{\circ}\text{C}$ . The freezing takes place in air, the thawing in water. The freeze-thaw exposure is carried out in conjunction with one of the following long-term water absorption tests:

- a) water absorption by diffusion as specified in ISO 20393;
- b) water absorption by total immersion as specified in ISO 2896.

The water absorption test to be used will be indicated in the relevant product standard.

## 5 Apparatus

**5.1 Cold chamber**, at a constant temperature of  $(-20 \pm 2) ^\circ\text{C}$ .

**5.2 Water tank**, with a constant water temperature of  $(20 \pm 2) ^\circ\text{C}$ , equipped with a device for holding the test specimen in position.

NOTE Normally, no accelerated thermal-exchange facilities, e.g. fan assistance in the cold chamber or turbulent water circulation in the water tank, are provided.

**5.3 Balance**, which permits reading to 0,1 g.

**5.4 Compression-testing machine**, including measuring devices, as specified in ISO 20392.

## 6 Test specimens

### 6.1 General

The test specimens used for this method (set A) shall be the same test specimens which have been used for the determination of long-term water absorption by diffusion in accordance with ISO 20393 or by total immersion in accordance with ISO 2896.

### 6.2 Dimensions

The thickness of the test specimens shall be the original product thickness.

The test specimens for set A shall be square, with squarely cut edges, having sides of length  $(500 \pm 1)$  mm or  $(200 \pm 1)$  mm, depending on the water absorption method chosen.

### 6.3 Number

The number of test specimens in set A will depend on the sizes of the two sets of specimens for the compression test (sets B1 and B2) which are prepared from set A.

The number of test specimens in each of sets B1 and B2 for the compression test, and their dimensions, shall be as specified in the relevant product standard or another international technical specification. In the absence of such a specification, the number of specimens and their dimensions shall be as defined in ISO 20392.

### 6.4 Preparation

If possible, the test specimens shall be cut so that they do not include original product edges.

The preparation of the specimens shall be by methods that do not change the original structure of the product. Any skins, facings and/or coatings shall be retained.

### 6.5 Conditioning

The test specimens shall be conditioned for at least 6 h at  $(23 \pm 5) ^\circ\text{C}$ .

In cases of dispute, they shall be conditioned at  $(23 \pm 2) ^\circ\text{C}$  and  $(50 \pm 5) \%$  relative humidity for the time stated in the relevant product standard, but in any case for a minimum of 6 h.

## 7 Procedure

Carry out the freeze-thaw exposure by the procedure described below and illustrated in Figure 1. All test specimens in both set A and the separate set for compression testing of the original product shall be taken from the same sample.

Determine the compression behaviour ( $\sigma_m$  or  $\sigma_{10}$ ) of the original product in accordance with ISO 20392.

Determine the long-term water absorption of the test specimens using either ISO 2896 or ISO 20393, recording the mass of each test specimen at the end of the water absorption test as  $m_0$ .

Place the test specimens in the cold chamber and maintain the temperature at  $(-20 \pm 2)^\circ\text{C}$  for 1 h. Remove the test specimens from the cold chamber and immerse them in water maintained at a temperature of  $(20 \pm 2)^\circ\text{C}$  for 1 h.

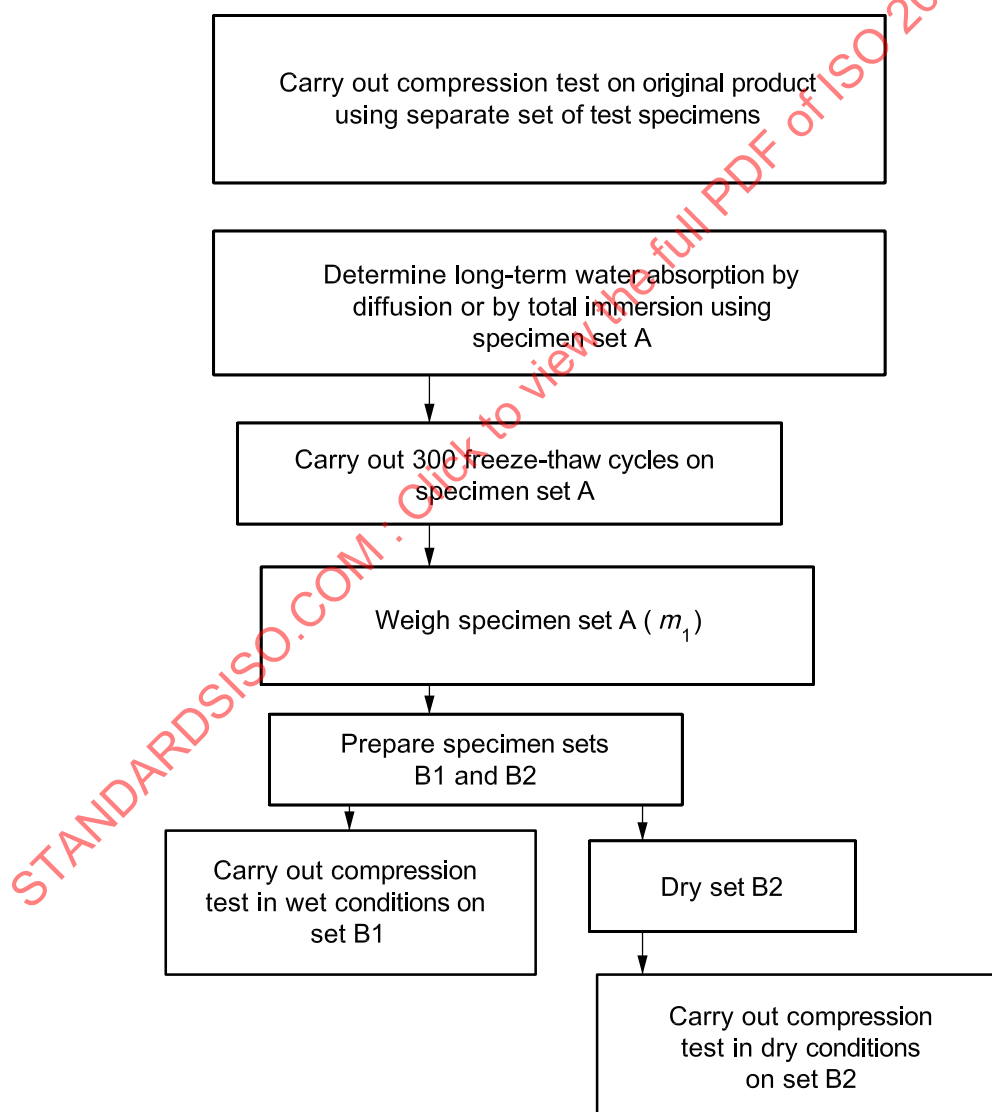
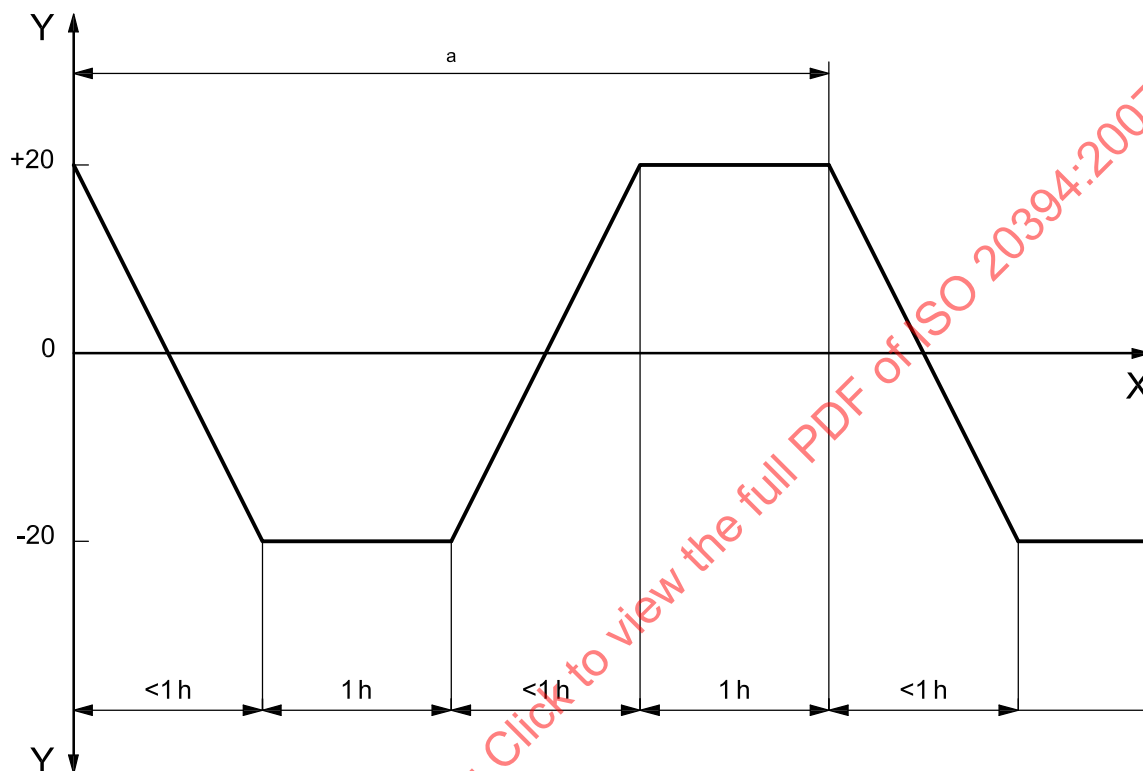


Figure 1 — Flow chart for the test procedure

Continue the freeze-thaw exposure for a total of 300 cycles (see Figure 2).

If there are interruptions of longer than 1 h in the specified freeze-thaw cycle, e.g. during the night or the weekend, the test specimens shall be left in the cold chamber during the interruption.

On completion of the 300 cycles, determine the mass,  $m_1$ , of each of the test specimens (set A) to the nearest 0,1 g.



**Key**

X time

Y temperature, °C

a 1 cycle.

**Figure 2 — Test cycle duration**

Examine the test specimens visually for defects, e.g. cracks, blisters.

Prepare the test specimens for set B1 and set B2.

Within 24 h of the last freeze-thaw cycle, determine, in accordance with ISO 20392, the compression behaviour of the test specimens in set B1 (compression behaviour in wet conditions,  $\sigma_{m,wet}$  or  $\sigma_{10,wet}$ ).

Dry the test specimens from set B2 in a ventilated drying chamber for the time and temperature specified in the relevant product standard or any other technical specification agreed between the interested parties. In the absence of such a specification, dry the test specimens to constant mass (considered to have been reached when the change in mass between two consecutive weighings at 24 h intervals is less than 0,5 % of the total mass at a drying temperature of at least 40 °C).

**NOTE** Commonly used drying conditions are 105 °C for 24 h, 70 °C for 4 days and 40 °C for 7 days.



Determine, in accordance with ISO 20392, the compression behaviour of the test specimens in set B2 (compression behaviour in dry conditions,  $\sigma_{m,dry}$  or  $\sigma_{10,dry}$ ).

Ideally, the freeze-thaw exposure is carried out immediately after the determination of the long-term water absorption. In the event that this is not possible, wrap the wet test specimens in polyethylene film and store them at ambient laboratory conditions.

## 8 Calculation and expression of results

### 8.1 Water absorption

Calculate the water absorption for each test specimen,  $W_m$  or  $W_V$ , in percent by mass or in percent by volume, respectively, using Equation (1) or (2):

$$W_m = \frac{m_1 - m_0}{m_0} \times 100 \quad (1)$$

$$W_V = \frac{m_1 - m_0}{V \times \rho_w} \times 100 \quad (2)$$

where

$m_1$  is the mass of the test specimen after 300 freeze-thaw cycles, in grams;

$m_0$  is the mass of the test specimen at the end of the water absorption by diffusion or by total immersion, in grams;

$V$  is the volume of the test specimen, in cubic centimetres;

$\rho_w$  is the density of water, assumed to be 1 g/cm<sup>3</sup>.

Express the result of the test as the mean value of the individual values calculated above, rounding the mean value of  $W_m$  to the nearest 0,1 % by mass and the mean value of  $W_V$  to the nearest 0,1 % by volume.

### 8.2 Changes in the compression behaviour

For each test specimen, calculate the change in compression behaviour determined in wet conditions and in dry conditions,  $\Delta\sigma_{wet}$  and  $\Delta\sigma_{dry}$ , in percent, using Equation (3) or (4) and Equation (5) or (6):

$$\Delta\sigma_{wet} = \frac{\sigma_{m,wet}}{\sigma_m} \times 100 \quad (3) \quad \text{or} \quad \Delta\sigma_{wet} = \frac{\sigma_{10,wet}}{\sigma_{10}} \times 10 \quad (4)$$

$$\Delta\sigma_{dry} = \frac{\sigma_{m,dry}}{\sigma_m} \times 100 \quad (5) \quad \text{or} \quad \Delta\sigma_{dry} = \frac{\sigma_{10,dry}}{\sigma_{10}} \times 100 \quad (6)$$

where

$\sigma_{m,wet}$  is the compressive strength of the test specimen in wet conditions, in kilopascals;

$\sigma_{10,wet}$  is the compressive stress of the test specimen at 10 % relative deformation in wet conditions, in kilopascals;

$\sigma_{m,dry}$  is the compressive strength of the test specimen in dry conditions, in kilopascals;

$\sigma_{10,dry}$  is the compressive stress of the test specimen at 10 % relative deformation in dry conditions, in kilopascals;