
**Thermal insulating products
for building applications —
Determination of freeze-thaw
resistance**

*Produits isolants thermiques destinés aux applications du bâtiment —
Détermination de la résistance aux effets du gel-dégel*

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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 163, *Thermal performance and energy use in the built environment*, Subcommittee SC 1, *Test and measurement methods*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 88, *Thermal insulating materials and products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 16546:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

- Revision of Clause 4, the test is performed by an automatic process only;
- Revision of Clause 7 with a more detailed procedure.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Thermal insulating products for building applications — Determination of freeze-thaw resistance

1 Scope

This document specifies the equipment and test method for determining the effects of successive cycling from dry conditions at $-20\text{ }^{\circ}\text{C}$ to wet conditions at $20\text{ }^{\circ}\text{C}$ on the mechanical properties and moisture content of thermal insulating products.

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 29469, *Thermal insulating products for building applications — Determination of compression behaviour*

ISO 16535, *Thermal insulating products for building applications — Determination of long-term water absorption by immersion*

ISO 16536, *Thermal insulating products for building applications — Determination of long-term water absorption by diffusion*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

freeze-thaw resistance

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

4 Principle

The test is performed continuously using an automatic process of cycling between the specified conditions.

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 has been subjected to 300 successive cycles from dry conditions at $-20\text{ }^{\circ}\text{C}$ to wet conditions at $20\text{ }^{\circ}\text{C}$. Testing shall be performed in conjunction with one of the following long-term water absorption tests:

- a) water absorption by diffusion, according to ISO 16536;

b) water absorption by total immersion, according to ISO 16535.

The chosen long-term water absorption test a) and/or b) will depend on the intended application and is provided in the relevant product standard. Freezing takes place in air, thawing in water.

5 Apparatus

5.1 Cold chamber, capable to maintain 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 keeping the test specimen in position.

The test chamber containing the specimens shall be capable of meeting and maintaining the conditions according to [5.1](#), [5.2](#) and [Figure 2](#).

5.3 Balance, capable of determining the mass of a test specimen to an accuracy of 0,1 g.

5.4 Compression testing machine, including measuring devices, according to ISO 29469.

6 Test specimens

6.1 General

The freeze-thaw test shall be made using the same test specimens, referred to as set A, which have been used for the determination of long-term water absorption by diffusion, according to ISO 16536, or by total immersion, according to ISO 16535.

The compressive test shall be performed on specimens prepared from set A and referred to as set B1 and set B2.

6.2 Dimensions of test specimens

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

The test specimens for set A shall be squares with squarely cut edges having sides of (500 ± 1) mm or (200 ± 1) mm depending on the chosen water absorption test.

6.3 Number of test specimens

The number of test specimens for set A shall be sufficient to allow two sets of test specimens for the compression test (set B1 and B2) to be prepared.

The number and dimensions of test specimens for each set B1 and B2 for the compression test shall be as specified in the relevant product standard or any other relevant technical specification. In the absence of such a specification, the number and dimensions of test specimens shall be as defined in ISO 29469.

6.4 Preparation of test specimens

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

Cutting of the test specimens shall be by a method that does not change the original structure of the product. Any skins, facings and/or coatings shall be retained.

6.5 Conditioning of test specimens

The test specimens shall be stored for at least 6 h at $(23 \pm 5) ^\circ\text{C}$. In case of dispute, they shall be stored at $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity (RH) for the time specified in the relevant product standard for a minimum of 6 h.

In tropical countries, different conditioning and testing conditions may be more relevant. In this case, the conditions shall be $(27 \pm 5) ^\circ\text{C}$ and $(65 \pm 5) \%$ RH and be stated clearly in the test report.

7 Procedure

7.1 General

Carry out the freeze-thaw test in accordance with the procedure indicated in [Figure 1](#). All test specimens shall be taken from the same sample.

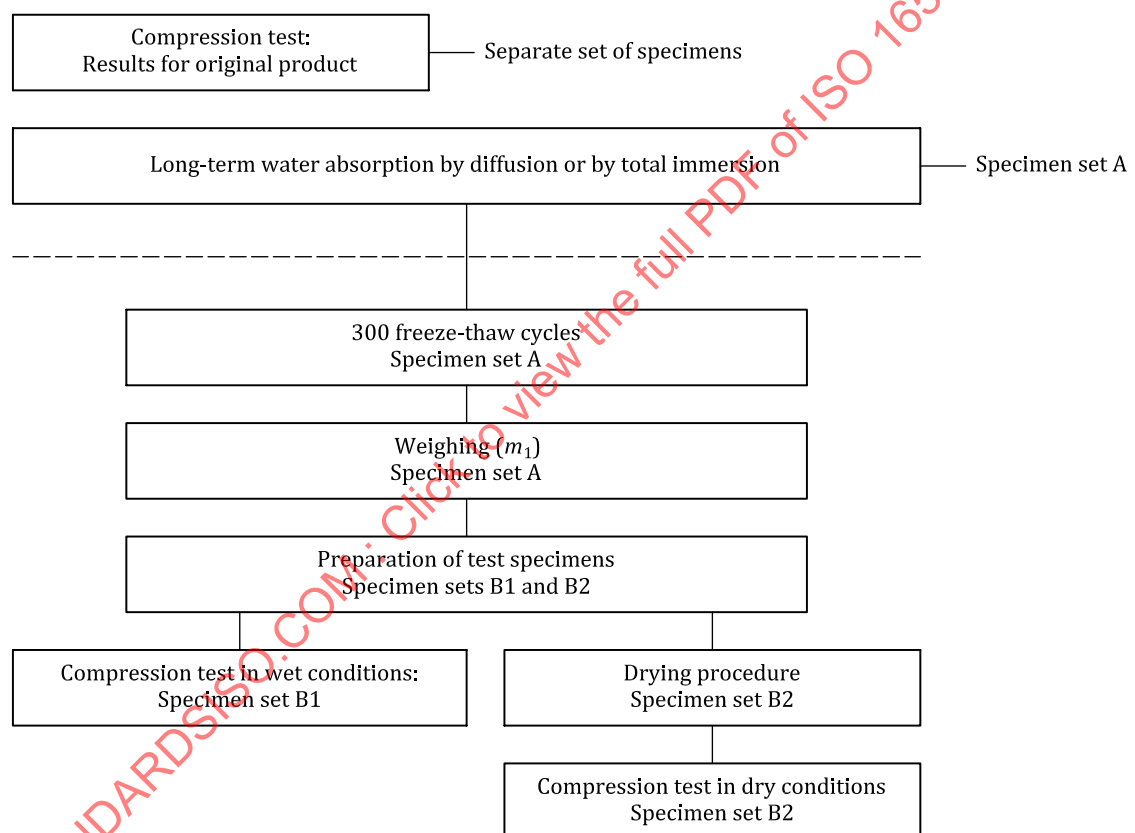


Figure 1 — Flowchart for the test procedure

7.2 Preliminary tests

Determine the compression behaviour of the original product, in accordance with ISO 29469 (σ_m or σ_{10}).

Determine the long-term water absorption of the test specimens using either ISO 16536 or ISO 16535.

Record m_0 , the mass of test specimens at the end of the diffusion test ($m_0 = m_D$) or the total immersion test ($m_0 = m_{28}$).

7.3 Water absorption frost thaw cycling

To avoid loss of water, the specimens shall be handled carefully and moved directly to the next stage of the process without shaking or resting them on another surface. For sanitary reasons and to avoid additional loss of water, they shall not make contact with other laboratory surfaces during the test.

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. Maintain the temperature at $(20 \pm 2) ^\circ\text{C}$ for 1 h. The water depth between the top edge of test specimen and the water surface in the tank shall be (5 ± 2) cm.

An open cage to keep the specimens in an upright and fixed position is recommended.

An immediate continuation between the different stages and cycles shall be ensured.

Stages do not start (see [Figure 2](#)) until the given temperature in the testing box is reached.

Continue the test for 300 cycles (see [Figure 2](#)).

After 300 cycles, mass m_1 shall be determined within about 10 s after dismounting each of the test specimens (set A) to the nearest 0,1 g.

Surface water is removed by patting and turning the specimens on a dry paper towel.

When there are breaks longer than 1 h, e.g. during the night or the weekend, the test specimens shall be left in the cold chamber.

Determine the mass m_1 , of each of the test specimens (set A) to the nearest 0,1 g after completion of all cycles.

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

7.4 Compression behaviour

Prepare the test specimens for set B1 and set B2, according to the relevant product standard.

Determine the compression behaviour of the test specimens from set B1, in accordance with ISO 29469, following the conditions as described hereafter (wet conditions within 24 h of the last freeze-thaw cycle, without further preconditioning).

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. In the absence of such a specification, the test specimens shall be dried until reaching constant mass. For the purpose of this test, the constant mass is considered to have been reached when the change in mass, measured at an interval of (24 ± 1) h is less than 0,5 % of the total mass at a minimum drying temperature of $40 ^\circ\text{C}$.

NOTE Commonly used drying conditions are $60 ^\circ\text{C}$ for about 7 days (specimen $W_v \leq 3$ vol. %) or about 14 days (specimen $W_v > 3$ vol. %).

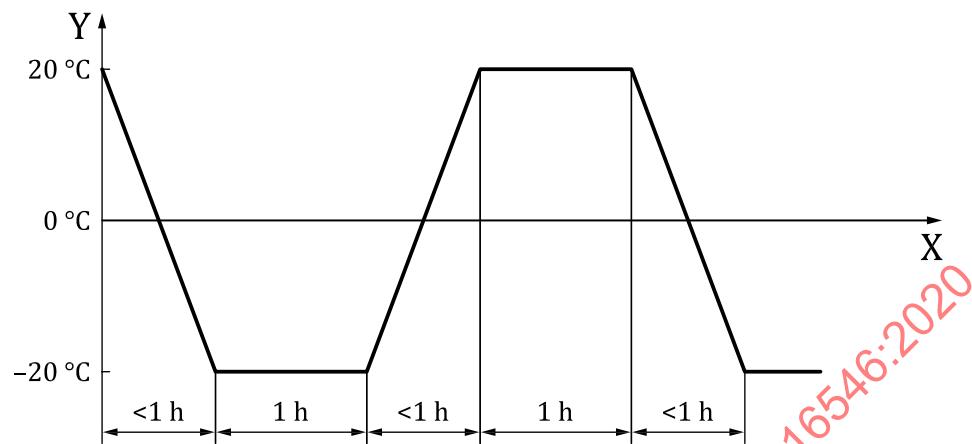
Determine the compression behaviour of the test specimens in accordance with ISO 29469 from set B2 (compression behaviour in dry conditions, $\sigma_{m,dry}$ or $\sigma_{10,dry}$).

7.5 Interruption of the procedure

To avoid freeze-drying when there are breaks longer than 1 h during the freeze thaw cycling process, e.g. during the night or the weekend, the test specimens shall be left in the cold chamber and wrapped in a foil [$\geq 50 \mu\text{m}$ polyethylene (PE) film has proved to be sufficient].

Wrapping instructions: fold the open end of the foil backwards over the specimen and fix along the whole length of the opening with adhesive tape.

In case the freeze-thaw cycling process is not carried out immediately after the test for long-term water absorption, the wet test specimens shall also be wrapped in polyethylene film and stored in a freezer to avoid draining.



Key

X time (h)

Y temperature (°C)

Figure 2 — Test cycle duration

8 Calculation and expression of results

8.1 Water absorption

The test results, W_m or W_v , shall be the mean values of the individual values.

Calculate the water absorption for each test specimen, W_m or W_v , in per cent by mass or in per cent by volume using [Formulae \(1\)](#) 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 g;

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

V is the volume of the test specimen, in cm³;

ρ_w is the density of water, assumed to be 1 g/cm³;

W_m shall be rounded to the nearest 0,1 per cent by mass;

W_v shall be rounded to the nearest 0,1 volume per cent.