# INTERNATIONAL STANDARD

ISO 10545-12

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# Ceramic tiles —

# Part 12:

Determination of frost resistance

Carreaux et dalles céramiques — Partie 12: Détermination de la résistance au gel



#### ISO 10545-12:1995(E)

### **Foreword**

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International Standard ISO 10545-12 was prepared by Technical Committee ISO/TC 189, Ceramic tile.

ISO 10545 consists of the following parts, under the general title Ceramic tiles:

- Part 1: Sampling and basis for acceptance
- Part 2: Determination of dimensions and surface quality
- Part 3: Determination of water absorption, apparent porosity, apparent relative density and bulk density
- Part 4: Determination of modulus of rupture and breaking strength
- Part 5: Determination of impact resistance by measurement of coefficient of restitution
- Part 6: Determination of resistance to deep abrasion for unglazed tiles
- Part 7: Determination of resistance to surface abrasion for glazed tiles
- Part 8: Determination of linear thermal expansion

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- Part 9: Determination of resistance to thermal shock
- Part 10: Determination of moisture expansion
- Part 11: Determination of crazing resistance for glazed tiles
- Part 12: Determination of frost resistance
- Part 13: Determination of chemical resistance
- Part 14: Determination of resistance to stains
- Part 15: Determination of lead and cadmium given off by glazed tiles
- Part 16: Determination of small colour differences
- Part 17: Determination of coefficient of friction

# Ceramic tiles —

# Part 12:

Determination of frost resistance

## 1 Scope

This part of ISO 10545 specifies a method for determining the frost resistance of all ceramic tiles intended for use in freezing conditions in the presence of water.

## 2 Principle

After impregnation with water, submission of the tiles to a cycle between + 5 °C and -5 °C, all the sides of the tiles being exposed to freezing during a minimum of 100 freeze-thaw cycles.

#### 3 Apparatus and materials

**3.1 Drying oven**, capable of being operated at  $(110 \pm 5)$  °C.

Microwave, infrared or other drying systems may be used provided that it has been determined that the same results are obtained.

- **3.2 Balance**, accurate to 0,01 % of the mass of a test specimen.
- 3.3 Apparatus for impregnation with water after evacuation, by means of a vacuum pump capable of lowering the air pressure by  $(60\pm4)$  kPa in a tank containing the tiles.
- **3.4 Freezer**, capable of freezing at least 10 tiles having a minimum total surface area of 0,25 m<sup>2</sup>, supported in such a manner that the tiles shall be separated.

#### 3.5 Chamois leather.

- **3.6 Water**, maintained at a temperature of  $(20 \pm 5)$  °C.
- 3.7 Thermocouple, or another suitable temperature measuring device.

## 4 Test specimens

#### 4.1 Sample

A minimum area of 0,25 m<sup>2</sup> and not less than 10 whole tiles shall be used. The tiles shall be free from defects. Relevant defects are cracks, crazing, holes, nipped edges and nipped corners.

If tiles with defects have to be tested, the defects shall be marked with a permanent stain prior to the test and these defects shall be examined after testing.

#### 4.2 Preparation of test specimens

The tiles shall be dried in the oven (3.1) adjusted to (110  $\pm$  5) °C, until constant mass is reached, i.e. until the difference between two successive weighings at intervals of 24 h is less than 0,01 %. The dry mass of each tile  $(m_1)$  shall be recorded.

#### 5 Impregnation with water

**5.1** After cooling to ambient temperature, place the tiles vertically in the dry vacuum tank (3.3) with no contact between them and no contact with the vacuum tank.

Connect the vacuum tank to the vacuum pump and evacuate to a pressure ( $60\pm2.6$ ) kPa below atmospheric pressure. Bring the water (3.6) into the tank containing the tiles whilst maintaining this pressure and cover the tiles with at least 50 mm head of water. Maintain the evacuation at the same pressure for a further 15 min and then restore atmospheric pressure

Prepare the chamois leather (3.5) by wetting and wringing out by hand. Place it on a flat surface and lightly dry each side of each tile in turn. Dab any relief surfaces with the chamois leather.

Record the wet mass of each tile  $(m_2)$ .

**5.2** The initial water absorption,  $E_1$ , expressed as a percentage by mass, is calculated using the equation

$$E_1 = \frac{m_2 - m_1}{m_1} \times 100$$

where

 $m_1$  is the mass of each dry tile;

 $m_2$  is the mass of each wet tile.

## 6 Procedure

Select one of the thickest tiles that is under test. It shall be one that is considered to have representative properties of that test sample. Drill a 3 mm diameter hole from the centre of one edge to a maximum distance of 40 mm from the edge of the tile. Insert a thermocouple (3.7) and seal the hole with a piece of thermally insulating material (for example expanded polystyrene). If it is not possible to drill a hole in this way, place a thermocouple on the centre of one face of a tile and stick a second tile over this. Place all the tiles under test vertically in the freezer (3.4), supported in such a way that there is a space between each one and an air flow over all the surfaces. Place the tile with the thermocouple in the middle of the test samples. The temperature of the thermocouple defines the temperature of all the tiles under test. Only in the case of repeated tests with similar test specimens can this be omitted, and then occasional checks shall be made with a thermocouple in a tile. All temperature measurements shall be accurate to ± 0,5 °C.

Lower the tile temperature to -5 °C at a rate not exceeding 20 °C/h. Maintain the tile temperature below -5 °C for 15 min. Immerse the tile in or spray it with water (3.6) until a tile temperature above +5 °C is reached. Maintain the tile temperature above +5 °C for 15 min. Repeat the cycle a minimum of 100 times. Interruption of the cycles shall be permitted only if the tiles are kept submerged in water above +5 °C.

Weigh the tiles after the test  $(m_3)$  and then dry them to constant mass  $(m_4)$ . The final water absorption,  $E_2$ , expressed as a percentage by mass, is calculated using the equation

$$E_2 = \frac{m_3 - m_4}{m_4} \times 100$$

where

m<sub>3</sub> is the mass of each wet tile after the test;

 $m_4$  is the mass of each dry tile after the test.

After 100 cycles, examine the glazed or proper surfaces and the edges of the tiles with the naked eye or with spectacles if usually worn, at a distance of 25 cm to 30 cm with an illumination of approximately 300 lx. They may be examined at intermediate stages to expedite the reporting, if there is reason to believe that they might sustain damage relatively early in the test. Record all damage that is observed on the glazed or proper surfaces and the edges of the tiles.

#### 7 Test report

The test report shall include the following information:

- a) reference to this part of ISO 10545;
- an identification of the tiles, including any surface relief, if necessary;
- c) the number of test specimens in the sample;
- d) the initial water absorption,  $E_1$ :
- e) the final water absorption, E<sub>2</sub>;
- a description of defects before the test and all damage on the glazed or proper surfaces and the edges of the tiles after the freeze-thaw test;
- g) the number of damaged tiles after 100 cycles.

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