BRITISH STANDARD

BS EN 12371:2010

Natural stone test methods — Determination of frost resistance

ICS 73.020; 91.100.15

BSI
British Standards

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National foreword

This British Standard is the UK implementation of EN 12371:2010. It supersedes BS EN 12371:2001 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/545, Natural stone.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Natural stone test methods - Determination of frost resistance

Méthodes d'essai pour pierres naturelles - Détermination de la résistance au gel

Prüfverfahren für Naturstein - Bestimmung des Frostwiderstandes

This European Standard was approved by CEN on 20 February 2010.

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Foreword

This document (EN 12371:2010) has been prepared by Technical Committee CEN/TC 246 “Natural stones”, the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

This document supersedes EN 12371:2001.

This European Standard is one of the series of standards for tests on natural stone.

Test methods for natural stone consist of the following European Standards:

EN 1925 Natural stone test methods – Determination of water absorption coefficient by capillarity

EN 1926 Natural stone test methods – Determination of uniaxial compressive strength

EN 1936 Natural stone test methods – Determination of real density and apparent density, and of total and open porosity.

EN 12370 Natural stone test methods – Determination of resistance to salt crystallisation

EN 12372 Natural stone test methods – Determination of flexural strength under concentrated load

EN 12407 Natural stone test methods – Petrographic examination

EN 13161 Natural stone test methods – Determination of flexural strength under constant moment

EN 13364 Natural stone test methods – Determination of the breaking load at dowel hole

EN 13373 Natural stone test methods – Determination of geometric characteristics on units

EN 13755 Natural stone test methods – Determination of water absorption at atmospheric pressure

EN 14066 Natural stone test methods – Determination of resistance to ageing by thermal shock

EN 14146 Natural stone test methods – Determination of the dynamic modulus of elasticity (by measuring the fundamental resonance frequency)

EN 14147 Natural stone test methods – Determination of resistance to ageing by salt mist

EN 14157 Natural stone test methods – Determination of the abrasion resistance

EN 14158 Natural stone test methods – Determination of rupture energy

EN 14205 Natural stone test methods – Determination of Knoop hardness

EN 14231 Natural stone test methods – Determination of the slip resistance by means of the pendulum tester

EN 14579 Natural stone test methods – Determination of sound speed propagation

EN 14580 Natural stone test methods – Determination of static elastic modulus

EN 14581 Natural stone test methods – Determination of linear thermal expansion coefficient

It is intended that other ENs should call up this European Standard as the basis of evaluation of conformity.

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NOTE It is not intended that all natural stones products should be subjected regularly to all the listed tests. Specifications in other standards should call up only relevant test methods.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This European Standard specifies a method to assess the effect of freeze/thaw cycles on natural stones (see EN 12670 for terminology, and EN 12440 for denomination). The standard contains provision for both a shorter technological test (Test A) to assess the effect of freeze/thaw cycles on the relevant performance characteristics and an identification test (Test B).

NOTE Some marbles, as defined in EN 12440, undergo changes in physical properties as a result of the test conditions rather than the freeze/thaw cycles. In these cases, additional tests (for example EN 14066) should be applied.

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.

EN 1926, *Natural stone test methods — Determination of uniaxial compressive strength*

EN 12372, *Natural stone test methods — Determination of flexural strength under concentrated load*

EN 13161, *Natural stone test methods — Determination of flexural strength under constant moment*

EN 13364, *Natural stone test methods — Determination of the breaking load at dowel hole*

EN 14066, *Natural stone test methods — Determination of resistance to ageing by thermal shock*

EN 14146, *Natural stone test methods — Determination of the dynamic modulus of elasticity (by measuring the fundamental resonance frequency*

3 Principle

The frost resistance of natural stone units is determined by a test comprising cycles of freezing in air and thawing in water.

4 Symbols

M_{d0} mass of the dry specimen before immersion in water and before starting the cycles, in grams

M_{s0} mass of the saturated specimen after immersion in water and before starting the cycles, in grams

M_{h0} apparent mass of the specimen in water before starting the cycles, in grams

M_{dn} mass of the dry specimen at Nc cycles, in grams

M_{sn} mass of the saturated specimen at Nc cycles, in grams

M_{hn} apparent mass of the specimen in water at *n* cycles, in grams

V_{b0} apparent volume of the specimen before freezing, in millilitres

V_{bn} apparent volume of the specimen at Nc cycles, in millilitres

ΔV_b change in apparent volume of the specimen, as a percentage

E_0 dynamic elastic modulus of the dry specimen before freezing, in Megapascals

E_n dynamic elastic modulus of the dry specimen at *n* cycles, in Megapascals

ΔE change in dynamic elastic modulus, as a percentage

Nc number of cycles completed before failure for identification test

5 Apparatus

5.1 A freezing tank of sufficient capacity to hold the required number of specimens, possibly with an automatic control system to programme the freezing and thawing cycles within the chamber with a tolerance of ± 2,0 °C.

NOTE The tests can be executed manually if no suitable automated system is available.

5.2 A temperature recording system capable of measuring temperature to ± 0,1 °C.

5.3 A device for measuring dynamic elastic modulus in accordance with EN 14146.

5.4 A weighing instrument with an accuracy of at least 0,01 % of the mass to be weighed.

5.5 A ventilated oven capable of maintaining a temperature of (70 ± 5) °C.

6 Preparation of the specimens

6.1 Sampling

The sampling is not the responsibility of the test laboratory except where specially requested.

For technological tests the number of specimens shall be in accordance with the appropriate standard (see also Table 1). Two sets of specimens are required, one to be tested after being subjected to the freeze/thaw cycles, the other to be tested without freeze/thaw cycling. Each set shall be randomly selected from the body of stone to be tested. In addition, one extra specimen is used to monitor the temperature of the specimens.

For an identification test, seven specimens, which are considered representative of the body of stone being tested, shall be selected. Use one of the seven specimen to monitor the core temperature.

Table 1 — Summary of the application of the freeze/thaw test

	Technological test (Test A)		Identification test (Test B)
	Flexural strength	Compressive strength	
Specimen size in mm	50 × 50 × 300 (see Note 1 and 3)	(see Note 2)	50 × 50 × 300 (see Note 3)
Minimum number of specimens	21	21	7

NOTE 1 Specimen size according to EN 12372 or EN 13161.

NOTE 2 Specimen size according to EN 1926.

NOTE 3 For specific stones with tendency to split (shale, phyllite, …), the thickness can be reduced to the thickness of use with a minimum of 13 mm. If the thickness is less than the minimum thickness required in EN12372 or EN 13161, the span for testing the flexural strength should be equal to (180 ± 1) mm

6.2 Size of specimens

6.2.1 Technological test (Test A)

Where the test is carried out to determine the effect of freeze/thaw cycles on performance characteristics the specimens shall be in accordance with the appropriate standard. The appropriate standards are EN 12372 or EN 13161 for flexural strength, EN 13364 for breaking load at a dowel hole, EN 14066 for resistance to thermal shock, and EN 1926 for compressive strength.

6.2.2 Identification test (Test B)

The specimens are in the form of rectangular prisms with dimensions of 50 mm × 50 mm × 300 mm (except for Note 3 in Table 1). The long axis shall be parallel to the anisotropy planes.

6.2.3 Putting reference marks on the specimens

Initial defects and all irregularities have to be marked with indelible marks on the specimens.

To ensure that the various dynamic elastic modulus measurements performed before and after the freeze/thaw cycles are done at the same points on the specimens, make indelible marks, in the form of points, on the relevant faces of the specimens.

Trace an indelible line at the point of the axis of the two supports on which the specimens will be placed during the determination of the dynamic elastic modulus (in flexural mode).

6.3 Monitoring core temperature

One specimen shall be fitted with a device (for example a thermocouple) to measure the temperature of the specimen during freezing and thawing cycles. The device shall be placed in a hole that is drilled parallel to the long axis of the specimen. The diameter of this hole has to be appropriate to the diameter of the device. The centre of the hole shall be equidistant from the edges of the specimen to a minimum depth of 50 mm from the top. If cubes are being used, the hole shall be (25 ± 5) mm deep. The device shall be placed in the hole which is then sealed to avoid water penetration.

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If several sets of specimen are tested at the same time in the freezing tank, it is allowed to use only one specimen for the monitoring of the core temperature providing that its porosity is equal to or lower than the one of the tested specimen.

6.4 Drying the specimens

The specimens are dried at a temperature of (70 ± 5) °C to constant mass. This is assumed to have been attained when the difference between two weighings at an interval of (24 ± 2) h is not greater than 0,1 % of the first of these two masses. The measurement of the dry specimens is the initial value M_{d0}. If the specimens are to be used for an identification test, the measurements of the modulus of elasticity shall be according to EN 14146. The measurement taken at this point is regarded as the initial value, E_0.

6.5 Immersion of the specimens

Place the specimens upright in a container at least 15 mm from adjacent specimens. Then add tap water at (20 ± 10) °C up to half the height of the specimens (time t_0). At time t_0 + (60 ± 5) min add tap water until the level of the water reaches three-quarters of the height of the specimens. At time t_0+ (120 ± 5) min add tap water until the specimens are completely immersed under (25 ± 5) mm of water. The specimens then need to be left completely immersed for (48 ± 2) h.

6.6 Determination of the apparent volume

If the specimens are to be used for an identification test, make measurements of the apparent mass in water and the mass in air (the specimen is surface dried after removal from the water) before starting the cycles (M_{h0},and M_{s0} respectively) and at Nc cycles (M_{hn} and M_{sn} respectively).

6.7 Number of cycles

6.7.1 Technological test (Test A)

For technological tests the number of cycles are given in the appropriate product standard.

6.7.2 Identification test (Test B)

For an identification test the client specifies the maximum number of cycles. If no value is specified, continue the test until the specimens are classed as failed up to a maximum of 168 cycles. The criteria for failure are given in 7.3.2.5 (see also 7.3.2.1 to 7.3.2.4). In addition, further guidance for specific uses may be found in relevant national annexes or specifications.

7 Test procedure

7.1 Arrangement of the specimens in the freezing tank

The specimens are placed in the tank with either the long axis vertical or horizontal and in such a way that they do not come into contact with each other or with the sides of the tank. They are positioned at least 10 mm apart, and at least 20 mm away from the tank sides. The reference specimen containing the temperature measuring device is placed in the middle of the specimens to be tested.

After 14, 56, 84 and 140 cycles (or less if convenient), turn the specimens through 180° around the horizontal axis.

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7.2 Description of the freezing and thawing cycles

Each cycle consists of a 6 h freezing period in air, followed by a 6 h thawing period during which the specimens are immersed in water. The cycles shall be repeated until the specimens fail or up to the given maximum number of cycles.

During the cycle, the changes in the temperature at the centre of the monitored specimen shall remain within the zone shown in Figure A.1. The range of permitted temperatures is the result of different rates of cooling, freezing and thawing which are related to the apparent density, porosity and the water absorption of the stone under test.

Each cycle is as follows in Table 2.

Table 2 — Stages of the cycles

	Temperature at the centre of the monitored specimen	**Time**
Start of cycle T_0	≥+5 °C ≤ +20 °C	T_0
Stage 1	≤ 0 °C ≥ -8 °C	T_0 + 2,0 h
Stage 2	≤ -8 °C ≥ -12 °C	T_0 + 6,0 h
Stage 3 By T_0 +6,5 h	Total immersion	T_0 + 6,5 h
Stage 4	≥ +5 °C ≤ +20 °C	By T_0 + 9,0 h
Stage 5	≥ +5 °C ≤ +20 °C	T_0 + 12,0 h

If the temperature of the stone does not remain within imposed limits of the zone (see Figure A.1), the chamber shall be adjusted so that it does and any deviations noted in the test report. If the test is being carried out manually, then Stage 5 can be extended to T_0 + 24,0 h.

For the identification test, different numbers of cycles and conditions may be used if particular requirements are specified by the client (see Annex A). Any changes in the conditions shall be noted in the test report.

NOTE It is important that the specimens are thawed in water as this ensures that they are water impregnated at the start of the next freezing period.

If the test is to be interrupted, other than for testing, at any time then the specimens are to be immersed in water at (20 ± 5) ºC. If the interruption exceeds 96 h, the specimens are to be taken out of water and wrapped to avoid evaporation.

7.3 Control measurements to determine the freeze/thaw resistance

7.3.1 Technological test (Test A)

7.3.1.1 Visual inspection

A visual inspection may be carried out in accordance with 7.3.2.2.

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7.3.1.2 Other methods (for example change in flexural strength)

After the completion of the required number of cycles, the specimens shall be tested in accordance with the appropriate standard.

7.3.2 Identification test (Test B)

7.3.2.1 General

Two criteria are used to assess the action of freezing and thawing cycles on the specimens:

a) Before testing and after 14, 56, 84, 140 and 168 cycles (or after reaching the maximum number of cycles Nc):

   — visual inspection;

   — measurement of the dynamic elastic modulus (Young´s modulus).

b) Before testing and after having reached the maximum number of cycles (Nc):

   — measurement of the apparent volume.

All results shall be recorded in an appropriate form.

7.3.2.2 Visual inspection

After the freeze-thaw cycles, the specimens are examined on all faces and sides and their behaviour is scored using the following scale:

0 specimen intact.

1 very minor damage (minor rounding of corners and edges) which does not compromise the integrity of the specimen.

2 one or several minor cracks (≤ 0,1 mm width) or detachment of small fragments (≤ 30 mm^2 per fragment).

3 one or several cracks, holes or detachment of fragments larger than those defined for the ‘2’ rating, or alteration of material in veins, or the specimen shows important signs of crumble or dissolution.

4 specimen with major cracks or broken in two or more or disintegrated.

The number of cycles at which the score of the visual examination attains 3 shall be noted (see also 7.3.2.5).

7.3.2.3 Measurement of apparent volume

Repeat the same measurements at the end of cycles (Nc) to determine M_{hn} and M_{sn} respectively.

Dry the specimens (see 6.4) to determine M_{dn}.

The initial apparent volume is expressed by:

$$V_{bo} = (M_{s0} - M_{h0}) \quad (1)$$

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At Nc cycles, the apparent volume is expressed by:

$$V_{bn} = (M_{sn} - M_{hn}) \quad (2)$$

NOTE For the purposes of this test, the density of water is assumed to be 1 000 kg/m^3.

The percentage change in apparent volume (ΔV_b) at *n* cycles is calculated as follows:

$$\Delta V_b = \frac{((M_{s0} - M_{h0}) - (M_{sn} - M_{hn})) \times 100}{(M_{s0} - M_{h0})} \quad (3)$$

7.3.2.4 Measurement of the dynamic elastic modulus

By measuring the change in the dynamic elastic modulus during the freezing-thawing cycles it is possible to detect some deterioration, such as microcracking. The dynamic elastic modulus (Young´s modulus) shall be determined in accordance with EN 14146.

It is necessary to dry the specimens to constant mass (see 6.4) and then perform the measurement. The specimens are then re-immersed in water in accordance with 6.5 before the freeze/thaw cycle is resumed.

The percentage decrease in dynamic elastic modulus (Young´s modulus) is calculated as follows:

$$\Delta E = \frac{(E_0 - E_n) \times 100}{E_0} \quad (4)$$

where

E_0 is the initial measurement in a dry condition (see 6.4) performed before cycling starts;

E_n is the measurement in a dry condition (see 6.4) after *n* cycles.

The number of cycles at which the decrease of dynamic elastic modulus reaches 30 % shall be noted.

7.3.2.5 Deterioration

The test continues until two or more of the specimens are classed as failed using either of the following criteria:

— score of the visual inspection attains 3;

— decrease of dynamic elastic modulus reaches 30 %.

8 Expression of the results

8.1 Technological test (Test A)

The change in performance is determined from the individual results using methods given in the relevant product standard.

8.2 Identification test (Test B)

The number of cycles completed (Nc), the reasons for failure and the results of all measurements prior to failure are recorded.

9 Test report

The test report shall contain the following information:

a) unique identification number of the report;

b) number, title and date of issue of this European Standard;

c) name and address of the test laboratory and the address where the test was carried out if different from the test laboratory;

d) name and address of the client;

e) it is the responsibility of the client to supply the following information:

- petrographic name of the stone;
- commercial name of the stone;
- country and region of extraction;
- name of the supplier;
- direction of any existing plane of anisotropy (if relevant to the test) to be clearly indicated on the sample or on each specimen by means of two parallel lines;
- name of the person or organization which carried out the sampling;
- surface finish of the specimens (if relevant to the test);

f) date of delivery of the sample or of the specimens;

g) date when the specimens were prepared (if relevant) and the date of testing;

h) number of specimens in the sample;

i) dimensions of the specimens;

j) for the technological test:

- percentage change in performances as a result of subjecting the specimens to freeze/thaw cycles and the number of cycles performed;

or for the indication test:

- maximum number of cycles given by the client, and the number of cycles before deterioration occurred (Nc);

k) statement on measurement uncertainty (where appropriate);

l) all deviations from the standard and their justification;

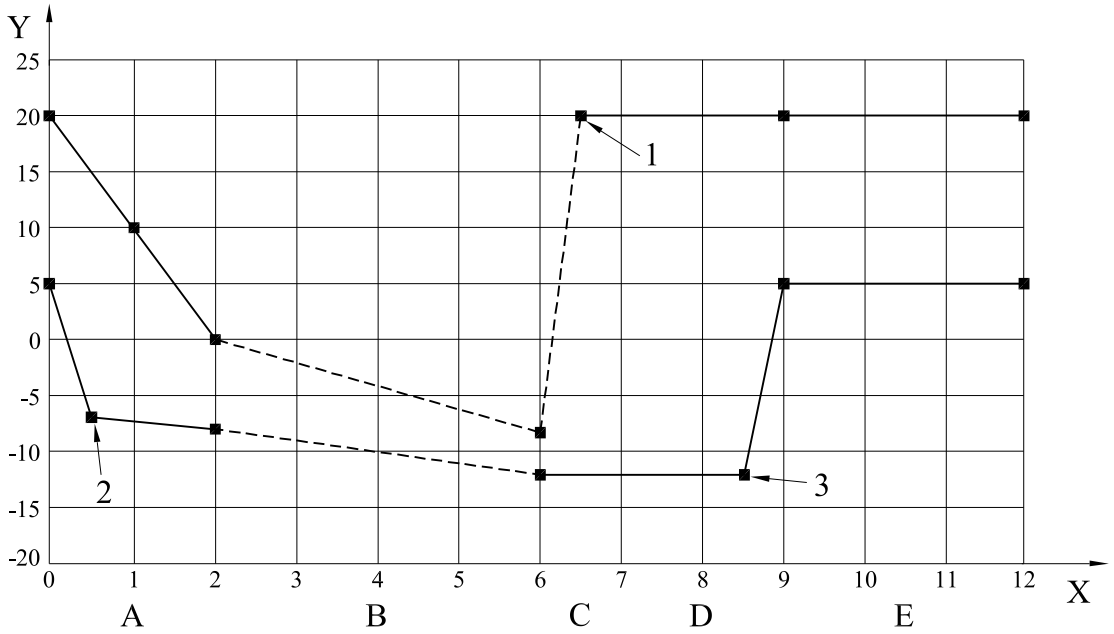
m) remarks.

The test report shall contain the signature(s) and role(s) of the person(s) responsible for the testing and the date of issue of the report. It shall also state that the report shall not be partially reproduced without the written consent of the test laboratory.

Annex A
(informative)

Test cycles

For some specific uses it may be appropriate to use different test cycles, for example freezing in water, freezing to a lower temperature, or testing specimens embedded in non-porous siliceous granules. In these cases, other methods may be followed provided these variations are clearly stated in the test report.



Key

1 T = 6,5 h; Temp. = +20 °C
2 T = 0,5 h; Temp. = -7 °C
3 T = 8,5 h; Temp. = -12 °C
A Stage 1
B Stage 2
C Stage 3
D Stage 4
E Stage 5

_____ : imposed limit of the zone
- - - - - - - - - - : indicative limit of the zone

Figure A.1 — Zone of permitted temperatures at the centre of the monitored specimen during a freezing and thawing cycle

Bibliography

EN 12440, *Natural stone — Denomination criteria*

EN 12670, *Natural stone — Terminology*

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