

Products and systems
for the protection and
repair of concrete
structures — Test
methods —
Determination of loss of
mass of hydrophobic
impregnated concrete
after freeze-thaw salt
stress

The European Standard EN 13581:2002 has the status of a
British Standard

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National foreword

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Products and systems for the protection and repair of concrete structures - Test method - Determination of loss of mass of hydrophobic impregnated concrete after freeze-thaw salt stress

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Produkte und Systeme für den Schutz und die Instandsetzung von Betontragwerken - Prüfverfahren - Bestimmung des Masseverlustes von hydrophobiertem Beton nach der Beanspruchung durch Frost-Tausalz-Wechsel

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Foreword

This document (EN 13581:2002) has been prepared by Technical Committee CEN/TC 104 "Concrete and related products", the secretariat of which is held by DIN.

It has been elaborated by Subcommittee SC 8 "Products and systems for the protection and repair of concrete structures", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by February 2003, and conflicting national standards shall be withdrawn at the latest by February 2003.

The annex A is normative and the annex B is informative.

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

1 Scope

This European Standard is one of a series dealing with products and systems for the protection and repair of concrete structures. It specifies a method for determining the loss of mass after freeze-thaw salt stress in sodium chloride solution. It can be used to test the resistance of hydrophobic impregnated concrete as well as the untreated concrete. There are two types of concrete deterioration when a freeze-thaw attack occurs: surface scaling and internal damage.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1766, *Products and systems for the protection and repair of concrete structures - Test methods - Reference concrete for testing*.

3 Symbols

Symbol	Explanation	Unit
C	Difference between the reached number of cycles of untreated and treated test cubes	-
C	Consumption of impregnant during treatment	g/m ²
C_{abs}	Quantity of fluid absorbed of one test cube during immersion in 3 % NaCl-solution	%
$C_{abs, m}$	Quantity of fluid absorbed of four test cubes during immersion in 3 % NaCl-solution; Mean value of treated an untreated test cubes	%
C_m	Mean consumption of impregnant during treatment	g/m ²
C_n	Consumption of impregnant for each face of the cube during treatment	g/m ²
C_u	Reached number of cycles of untreated test cubes	-
C_t	Reached number of cycles of treated test cubes	-
n	Indicating the number of cycles carried out	-
ΔW_n	Change of mass after the n cycle	%
W_n	Mass of a test cube after n cycles	g
W_0	Mass of a test cube before immersion for 24 h in 3 % NaCl solution	g
W_{t1}	Mass of test cube immediately prior to treatment	g
W_{t2}	Mass of test cube immediately after treatment	g
W_e	Mass of a test cube after immersion for 24 h in 3 % NaCl solution	g

4 Principle

The principle of the test method described in this standard is to compare the loss of mass and visible defects of treated and untreated test cubes from the same batch of concrete. The difference between the reached cycles of treated and untreated test cubes characterizes the effectiveness of a hydrophobic impregnant.

5 Apparatus

- 5.1 Eight moulds for concrete cubes (100 mm x 100 mm x 100 mm).
- 5.2 Laboratory or chamber maintained at constant temperature (21 ± 2) °C and relative humidity of (60 ± 10) %.
- 5.3 Four brass or stainless steel watertight containers with a width of (120 ± 10) mm, a length of (260 ± 10) mm and a height of (150 ± 10) mm. The sheet metal shall be approximately 1 mm thick. The containers are closed with lids which shall be designed so that they cannot be lifted off when the chamber is flooded. The lid of one container shall have an opening for placing a thermocouple to measure the temperature in the centre of one test cube.
- 5.4 Thermocouples, or an equivalent temperature measuring device with which the temperature shall be measured to an accuracy of 0,5 °C. A continuous recording device shall be used measuring and recording the temperature at least every 10 min over a period of 24 h.
- 5.5 An automatically-controlled freezing chamber with a flooding device. The performance of the freezing chamber shall be designed so that it is possible to maintain the temperature cycle referred to Figure 1 for each of the test cubes placed in it.
- 5.6 Brush, with short (about 20 mm), soft bristles for brushing off scaled material.
- 5.7 Spray bottle, containing tap water for washing off scaled material.
- 5.8 Drying cabinet, for a temperature of at least 110 °C.
- 5.9 Balance, with an accuracy within $\pm 0,1$ g.
- 5.10 Desiccator cabinet containing silica gel
- 5.11 Fume cupboard
- 5.12 Sodium-chloride solution; 3 percent by weight.
- 5.13 One 5 l beaker with suitable spacers to support test cubes.
- 5.14 One 150 mm diameter petri dish with 2 spacers glued to bottom of dish to support the test cubes during treatment.

6 Preparation of test samples

Eight 100 mm concrete test cubes shall be cast from a single batch of type C (0,70) concrete and cured for 28 days according to EN 1766. No mould oil or release agent shall be used. After removal from the curing tank, the test cubes shall be cleaned with tap water using a soft brush (5.6) to remove any loose material. The surface of the test cubes shall not be grit blasted.

The test cubes shall then be conditioned for at least 60 days at a temperature of (21 ± 2) °C and a relative humidity of (60 ± 10) %.

7 Treatment

Four test cubes (No. 1 - 4) shall be treated in a fume cupboard (5.11) with the fan on after the conditioning of the specimens.

Each cube shall be treated by dipping each face in the impregnant. Immediately prior to treatment of each face, the cube shall be weighed (W_{t1}). 60 ml of the material shall be measured into a petri dish 150 mm in diameter (5.14).

One side of the cube, supported on the 2 mm plastic spacers, shall be dipped in the material for (120 ± 5) s, and then removed. Excess liquid on the cube shall be allowed to drain back into the dish (5.14) and the cube immediately reweighed (W_{t2}). The excess material in the dish is then to be discarded. This procedure shall be repeated for first side of the other two cubes.

The consumption (C_n) of the material for each face of the cube shall be calculated as follows :

$$C_n = \frac{W_{t2} - W_{t1}}{0,01} \text{ in grams by square meters} \quad (1)$$

The above procedure shall be repeated on the remaining faces of each of the cubes and the average consumption for each cube shall be calculated.

The mean consumption (C_m) for the three test cubes shall be calculated.

If the consumption is below the manufacturer's recommended value the time of dipping can be extended.

If it is not practical to treat test cubes by this method, the treatment should be carried out in accordance with the manufacturer's instructions.

The cubes shall be stored in a climate (21 ± 2) °C and (60 ± 10) % R. H. for 14 days after the treatment suitably supported to allow air circulation around each of the 6 faces.

8 Test procedure

One day before the start of the freezing test, the mass of the 8 test cubes is determined with an accuracy of 0,1 g (W_0).

The 8 test cubes provided for the freezing test shall then be placed in four containers (5.3) so that the faces which were uppermost during manufacture are perpendicular to the base of the container and that there shall be about 10 mm distance between the test cubes and the steel containers.

The containers shall be filled with 3 % sodium-chloride-solution (5.12) until the solution covers the test cubes by more than 20 mm and less than 30 mm. 24 h after the start of this storage the mass of each test cube shall be determined again to an accuracy of 0,1 g (W_e) and the quantity of fluid absorbed in 24 h (C_{abs}) is calculated from the increase in mass following the equation 2 :

$$C_{abs} = \frac{W_e - W_0}{W_e} \cdot 100 \text{ in \%} \quad (2)$$

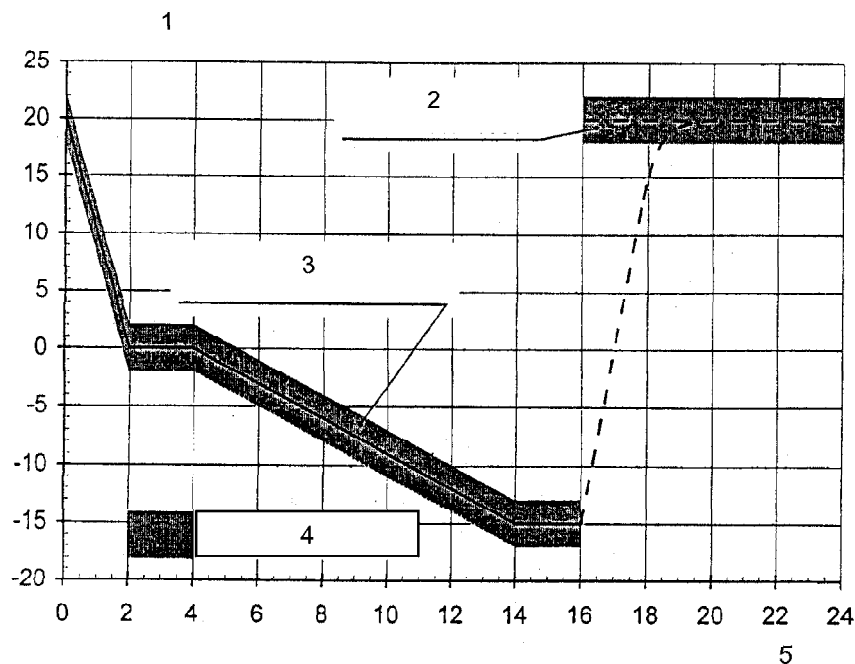
The means values $C_{abs, m}$ are to be calculated from the single values C_{abs} of treated and untreated test cubes.

The temperature of the freezing chamber (5.5) shall be controlled so that the temperature at the centre of the test cube corresponds to the solid line in Figure 1 and shall not leave the shaded area in the diagram. The air temperature shall not fall below - 25 °C.

Immediately after the determination of W_e the containers containing the test cubes immersed in 3 % sodium-chloride-solution shall be placed with closed lids in the freezing chamber ensuring that they are evenly distributed. The freeze-thaw cycle then begins. The containers shall be changed around once a week; they are turned through 180° and interchanged on a cyclic basis. The number of containers in the freezing chamber shall always be the same. If only a few test cubes are to be tested, containers with "blank test cubes" shall be put in for this purpose. The containers shall not be placed on top of each other.

Immediately after the 16 h cooling/freezing phase, the freezing chamber is flooded with water or the containers are put into a water bath of $(21 \pm 2)^\circ\text{C}$ so that the water level shall be (20 ± 5) mm below the brims of the containers. The thawing phase shall last a total of 8 h. 15 min before the end of the 8 h thawing phase the water is pumped out of the freezing chamber for a maximum time of 15 min. The water shall always be in circulation and be kept at a constant temperature at $(21 \pm 2)^\circ\text{C}$ during the entire thawing process. The test shall last 50 cycles. One cycle lasts 24 h (see Figure 1).

If, in exceptional cases, it is necessary to interrupt the test, the temperature of the containers with the test cubes shall be maintained at $(-15 \pm 2)^\circ\text{C}$.



Key

- 1 Temperature in $^\circ\text{C}$
- 2 Temperature of the water bath
- 3 Temperature in the center of a 100 mm cube
- 4 Temperature range
- 5 Time in h

Figure 1 — Temperature behaviour pattern in the centre of a test tube

The breakpoints specifying the shaded area in Figure 1 are given in Table 1 :

Table 1 — Limit values of the temperature curve shown in Figure 1 (shaded area)

Time in h	Temperature in °C	
	upper limit	lower limit
0	23	19
2	2	- 2
4	2	- 2
16	- 13	- 17

After every 5 cycles, the following procedure is carried out after the thawed state of the specimens :

- the test cubes shall be checked visually to determine whether cracks or other substantial changes have occurred and whether the weathering loss is on the surfaces or has occurred at the edges ;
- the test cubes shall then be brushed off using a light pressure in such a way that the pieces which have already been loosened are detached and then slightly dried with a paper towel ;
- the mass of the test cubes W_n ($n = 5, 10, 15, 20, 25, 30, 35, 40, 45, 50$ indicating the number of cycles) is determined to an accuracy of 0,1 g.

The change of mass (ΔW_n) shall be calculated using the equation 3 :

$$W_n = \frac{W_n - W_0}{W_0} \cdot 100 \text{ in \%} \quad (3)$$

The mean values $\Delta W_{n,m}$ are to be calculated from the single values ΔW_n of treated and untreated test cubes. It is allowed to neglect one single value with strong deviations ;

- before the start of the next cycle the container with the test cubes is filled with renewed 3 % sodium-chloride-solution at (21 ± 2) °C. The container is returned to the freezing chamber.

A flow chart showing the test procedure is given in Annex B (informative).

9 Expression of results

The mean values of the change of mass ΔW_n in % of the treated and untreated test cubes ($W_0, W_e, W_{15} \dots W_m$) are shown in a graphic above the number of cycles (see Annex A (normative)). The intersection between the curve and the 0 % line indicates the reached number of cycles. The difference between the reached number of cycles (C) of untreated (C_u) and treated (C_t) test cubes shall be calculated.

10 Test Report

The test report shall contain the following information :

- a) a reference to this European standard ;
- b) name and address of the test laboratory ;
- c) identification number and date of the test report ;
- d) name and address of the manufacturer or supplier of the product(s) ;
- e) name and identification marks or batch number of the product(s) ;
- f) date of supply of the product ;
- g) date of preparation of the test specimens and any deviation from the prescribed method of preparation ;
- h) numbering of cubes ;
- i) details of the concrete mix design ;
- j) conditions of storage of prepared specimens prior to test ;
- k) consumption C and mean consumption C_m of impregnant during treatment ;
- l) the mean liquid absorption by the test cubes ($C_{abs,m}$) in % to the nearest 0,1 % by weight during the 24 h sodium-chloride-solution-storage before the start of the freezing test ;
- m) the mean mass change of the test cubes (ΔW_n) in % to the nearest 0,1 % by weight, after every 5 cycles up to 50 cycles ;
- n) visual assessment (notes on cracks, substantial changes in the test cube and type of weathering loss, loss of material at the surfaces or edges) after every 5 cycles up to 50 cycles. Uneven distributed deterioration of the test cubes shall be mentioned ;
- o) graphic according to clause 9 ;
- p) calculated difference in number of cycles between treated and untreated test cubes ;
- q) details of the test equipment used ;
- r) any deviation of the test method specified ;
- s) date of the test.

Annex A
(normative)

Graphic change of mass above cycles

1

2

Key

- 1 Loss of mass (% by weight)
- 2 Number of cycles

Figure A.1 — Determination of the reached number of cycles (Example)

Annex B (informative)

B.1 Types and quality of specimens

Usually, specimens of type C (0,70) in accordance with EN 1766 shall be used as standard substrate. If there is a special interest of the parties concerned, specimens of type C (0,45) in accordance with EN 1766 can be used.

When using specimens of type C (0,45), the test report must document the reasons.

B.2 Material for moulds

For the fabrication of the specimens, no mould oil shall be used. Moulds shall be manufactured from non-absorbent rigid material, not attacked by cement paste or polymers. Appropriate materials could be PE-sheets or Polytetrafluorethylene.

B.3 Flow chart of test procedure

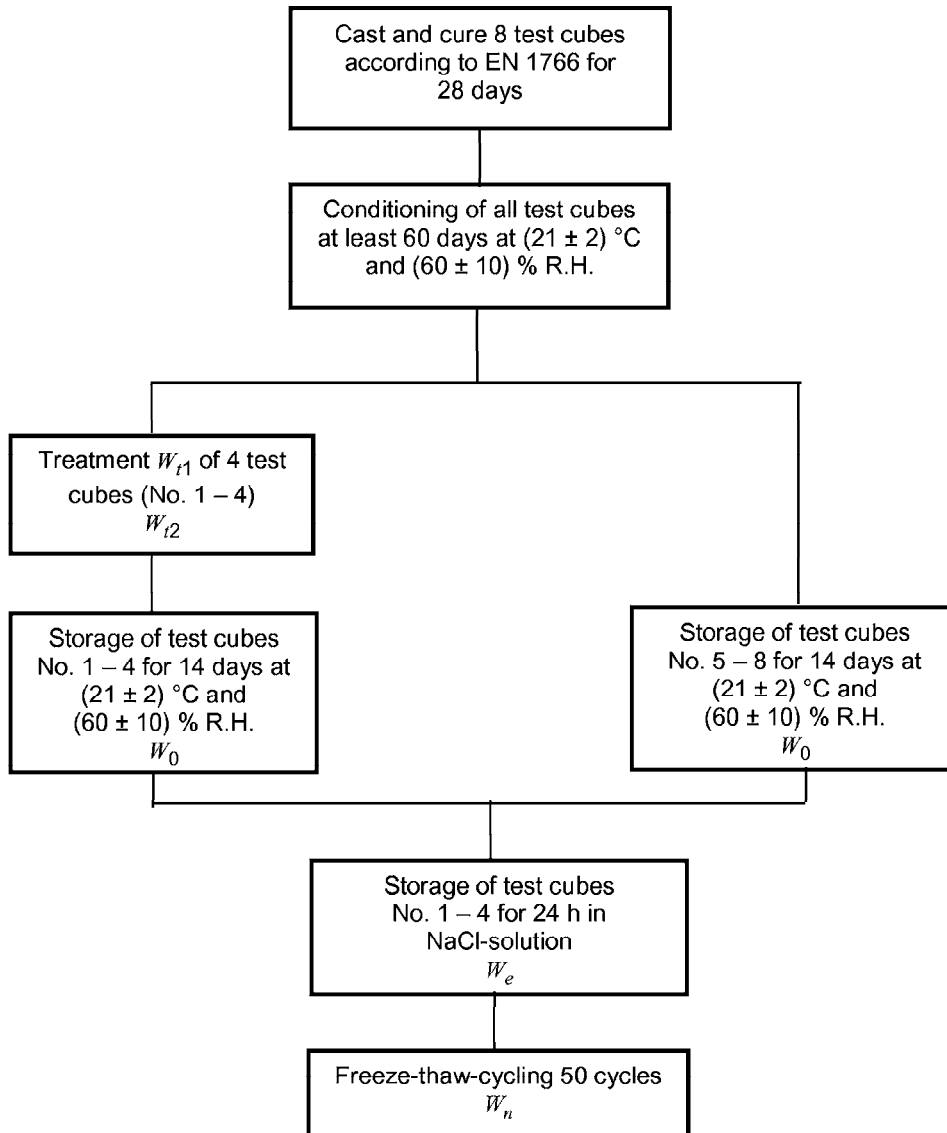


Figure B.1 — Preparation and test procedure

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