Testing hardened concrete —

Part 9: Freeze-thaw resistance — Scaling

ICS 91.100.30



National foreword

This Draft for Development was published by BSI. It is the UK implementation of CEN/TS 12390-9:2006.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated not later than 3 years after its publication by the European organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted by Technical Committee B/517, Concrete, to Subcommittee B/517/1, Concrete production and testing.

A list of organizations represented on B/517/1 can be obtained on request to its secretary.

The BSI committee responsible for the voting decision have concerns over the application of the test in practice. Whilst there are no values of performance given in this Technical Specification, the originators of the tests have values they apply in practice and these are known to others. It is accepted that a concrete satisfying these values will be likely to perform very well in extreme freeze/thaw conditions. However, in the United Kingdom there are many concretes that perform adequately in the prevailing environmental conditions, but if tested against these criteria would fail by a substantial margin. During the preparation of the standard the United Kingdom considered the possibility of using a reference concrete of known, satisfactory performance alongside that under test or, the use of different test conditions or acceptance criteria. However, once weight losses approach the values obtained from concrete with a long satisfactory performance record the results are not reliable. In the opinion of the UK, modifications to the test method are required.

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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Testing hardened concrete - Part 9: Freeze-thaw resistance - Scaling

Prüfung von Festbeton - Teil 9: Frost- und Frost-Tausalz-Widerstand - Abwitterung

This Technical Specification (CEN/TS) was approved by CEN on 25 June 2005 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Foreword

This Technical specification (CEN/TS 12390-9:2006) has been prepared by Technical Committee CEN/TC 51 "Cement and building limes", the secretariat of which is held by IBN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: Austria, Belgium, 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.

Introduction

Concrete structures exposed to the effects of freezing and thawing need to be durable, to have an adequate resistance to this action and, in cases such as road construction, to freezing and thawing in the presence of deicing agents. It is desirable, especially in the case of new constituents or new concrete compositions, to test for such properties. This also applies to concrete mixes, concrete products, precast concrete, concrete members or concrete in situ.

Many different test methods have been developed. No single test method can completely reproduce the conditions in the field in all individual cases. Nevertheless, any method should at least correlate to the practical situation and give consistent results. Such a test method may not be suitable for deciding whether the resistance is adequate in a specific instance but will provide data of the resistance of the concrete to freeze-thaw-attack and freeze-thaw-attack in the presence of de-icing agents.

If the concrete has inadequate resistance then the freeze-thaw attack can lead to two different types of damage, namely to scaling (surface weathering) and to internal structural damage. This part of this standard covers only testing for scaling resistance.

This Technical Specification has one reference method and two alternative methods. For routine testing either the reference method or one of the two alternative methods may be used with the agreement of the parties involved. In case of doubt, and if there is no such agreement, the reference method is used.

The application of limiting values will require the establishment of the correlation between laboratory results and field experience. Due to the nature of the freeze-thaw action, such correlation would have to be established in accordance with local conditions.

1 Scope

This Technical Specification describes the testing of the freeze-thaw scaling resistance of concrete both with water and with sodium chloride solution. It can be used either to compare new constituents or new concrete compositions against a constituent or a concrete composition that is known to give adequate performance in the local environment or to assess the test results against some absolute numerical values based on local experiences.

Extrapolation of test results to assess different concretes i.e. new constituents or new concrete compositions, requires an expert evaluation.

NOTE In some cases the test methods may not be suitable for testing special concretes e.g. high strength concrete or permeable concrete. In these cases the result is to be treated with caution. These tests may not identify aggregates that are subject to occasional 'pop-outs'.

There is no established correlation between the results obtained by the three test methods. All tests will clearly identify poor and good behaviour, but they differ in their assessment of marginal behaviour

There are two types of concrete deterioration when a freeze-thaw attack occurs, scaling and internal structural damage. Test methods on internal structural damage are described in a CEN Technical Report CEN/TR 15177 "Testing the freeze-thaw resistance of concrete - Internal structural damage".

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 12390-2, Testing hardened concrete — Part 2: Making and curing specimens for strength tests.

ISO 5725 (all parts), Accuracy (trueness and precision) of measurement methods and results.

EN 60751, Industrial platinum resistance thermometer sensors (IEC 60751:1983 + A1:1986).

3 Terms and definitions

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

3.1

Freeze-thaw resistance

resistance against alternating freezing and thawing in the presence of water alone

3.2

Freeze-thaw resistance with de-icing salt

resistance against alternating freezing and thawing in the presence of de-icing salt

3.3

Scaling

loss of material at the surface of concrete due to freeze-thaw attack

3.4

Internal structural damage

cracks inside concrete which cannot be seen on the surface, but which lead to an alteration of concrete properties, e. g. reduction of the dynamic modulus of elasticity

4 Making of test specimens

Except where details are specified in Clauses 5, 6 and 7 (e. g. the curing) prepare the test specimens in accordance with EN 12390-2. Concrete that requires vibrating for compaction is compacted on a vibrating table. The prestorage conditions concerning temperature and moisture are documented.

The maximum aggregate size Dmax is restricted to one third of the mould length.

5 Slab test (reference method)

5.1 Principle

Slab specimens, sawn from concrete test specimens (Figure 1), are subjected to freeze-thaw attack in presence of a 3 mm deep layer of de-ionised water or 3% sodium chloride (NaCl) solution. The freeze-thaw resistance is evaluated by the measurement of mass scaled from slab after 56 freeze-thaw cycles.

5.2 Equipment

- **5.2.1** Equipment for making 150 mm concrete cubes according to EN 12390-2.
- **5.2.2** Climate controlled room or chamber with a temperature of (20 ± 2) °C and an evaporation of (45 ± 15) g/(m² h). Normally this is obtained with a wind velocity ≤ 0.1 m/s and a relative humidity of (65 ± 5) %. The evaporation is measured from a bowl with a depth of approximately 40 mm and a cross section area of (225 ± 25) cm². The bowl is filled up to (10 ± 1) mm from the brim.
- **5.2.3** Diamond saw for concrete cutting.
- **5.2.4** Rubber sheet, (3 ± 0.5) mm thick which is resistant to the salt solution used and elastic down to a temperature of -27 °C.
- **5.2.5** Adhesive for gluing the rubber sheet to the concrete specimen. The adhesive is resistant to the environment in question.
- NOTE Contact adhesive has proved to be suitable.
- **5.2.6** Expanded Polystyrene cellular plastic, (20 ± 1) mm thick with a density of (18 ± 2) kg/m³ or alternative thermal insulation with at least a heat conductivity of 0,036 W/(m·K).
- **5.2.7** Polyethylene sheet, 0,1 mm to 0,2 mm thick.
- **5.2.8** Freezing medium, consisting either of 97 % by mass of tap water and 3 % by mass of NaCl (for test with de-icing salt) or of de-ionised water only (for test without de-icing salt).
- **5.2.9** Freezing chamber with temperature and time controlled refrigerating and heating system with a capacity such that the time-temperature curve presented in Figure 4 can be followed for every position where a specimen is placed. The freezer has a good air circulation. The open-mesh shelves in the freezer are level. No deviation from the horizontal plane shall exceed 3 mm per metre in any direction.
- **5.2.10** Thermocouples, or an equivalent temperature measuring device, for measuring the temperature in the freezing medium on the test surface (see Figure 3) with an accuracy within \pm 0,5 K.
- **5.2.11** Vessel for collecting scaled material. The vessel is suitable for use at temperatures up to 120 °C without mass loss and is resistant to attack by sodium chloride.
- **5.2.12** Suitable paper filter for collecting scaled material, optional.
- **5.2.13** Brush, with short (about 20 mm), stiff bristles for brushing off material that has scaled.
- **5.2.14** Spray bottle, containing tap water for washing off scaled material.

- **5.2.15** Drying cabinet, controlled at a temperature of (110 ± 10) °C.
- **5.2.16** Balance, with an accuracy within ± 0.05 g.
- **6.2.17** Vernier callipers, with an accuracy within ± 0.1 mm.

5.3 Preparation of test specimens

The test requires four specimens, one from each of four cubes.

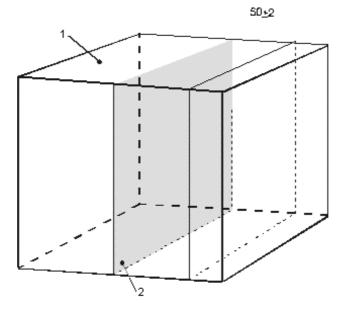
During the first day after casting the cubes are stored in the moulds and protected against drying by use of a polyethylene sheet. The air temperature is (20 ± 2) °C.

After (24 \pm 2) h, the cubes are removed from the moulds and placed in a bath with tap water having a temperature of (20 \pm 2) °C.

When the cubes are 7 days old, they are removed from the water bath and placed in the climate chamber (5.2.2), where they are stored until the freeze-thaw testing starts.

At 21 days a (50 ± 2) mm thick specimen is sawn from each cube perpendicular to the top surface so that the saw cut for the test surface is located in the centre of the cube, see Figure 1. The range in mean thickness of a specimen shall not exceed 2 mm.

Dimensions in millimetres



Key

- 1 Top surface at casting
- 2 Test surface

Figure 1 — Location of test specimen and test surface in sawn cube

Directly after sawing, wash the specimen in tap water and wipe off the excess water with a moist sponge. Measure all dimensions of the specimen to an accuracy of \pm 0,5 mm by using vernier callipers (5.2.17). Without delay, return it to the climate chamber ensuring that the test surface is vertically with a space between the specimens of at least 50 mm.

When the concrete is (25 ± 1) days old, rubber sheet is glued to all surfaces of the specimen except the test surface. Place a string of glue or silicone rubber around the test surface in the join between the concrete and the rubber. The surface area remaining after the application of the glue string shall be not less than 90 % of the original surface area of the specimen. The edge of the rubber sheet reaches (20 ± 1) mm above the test surface. After fixing the rubber sheet the specimen is returned to climate chamber.

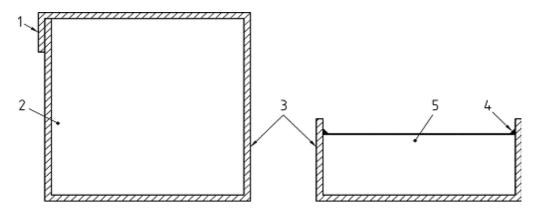
CEN/TS 12390-9:2006

NOTE 1 The adhesive is normally spread on the concrete surfaces as well as on the rubber surfaces. The manner of gluing the rubber sheet illustrated in Figure 2 has been proved suitable.

When the concrete is 28 days old, pour a layer about 3 mm deep of de-ionised water at a temperature of (20 ± 2) °C on the top surface. This resaturation continues for (72 ± 2) h at (20 ± 2) °C during which time the layer is to be maintained at about 3 mm.

NOTE 2 For a specimen with the test area of 150 mm x 150 mm, 67 ml de-ionised water gives an approximately 3 mm thick layer.

Before the test, all surfaces of the specimen except the test surface are thermally insulated with (20 ± 1) mm thick polystyrene cellular plastic (5.2.6) according to the test set-up in Figure 3. Another material or thickness providing equivalent thermal insulation can be used instead.



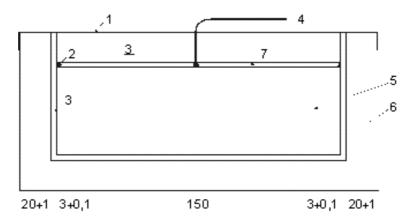
Key

- 1 Overlap
- 2 Test surface
- 3 Rubber sheet
- 4 Glue string
- 5 Specimen

Figure 2 — Sealing the test specimen

Start the test when the specimens are 31 days old. Not earlier than 15 min before the specimens are placed in the freezing chamber (5.2.9), replace the de-ionised water on the test surface with 67 ml of the freezing medium (5.2.8), to obtain an average thickness of 3 mm, at a temperature of (20 ± 2) °C.

Dimensions in millimetres



Key

- 1 Polyethylene sheet
- 2 Glue string
- 3 Rubber sheet

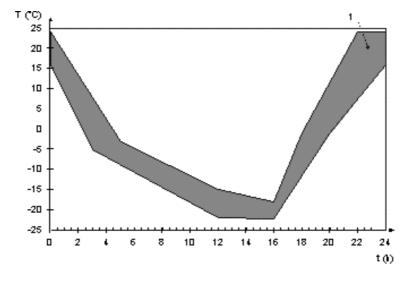
- 4 Temperature measuring device in contact with the test surface
- 5 Specimen
- 6 Thermal insulation
- 7 Freezing medium

Figure 3 — The test set-up used for the freeze-thaw test

The freezing medium is prevented from evaporating by applying a nearly flat, horizontal polyethylene sheet (5.2.7) as shown in Figure 3. The polyethylene sheet remains flat throughout the test so that the distance between the sheet and the surface of the freezing medium is at least 15 mm.

5.4 Test procedure

To begin the test, place the specimens in the freezing chamber at the cycle phase time (0 \pm 30) min according to Figure 4. After the specimens have been placed in the freezing chamber, subject them to repeated freezing and thawing. Monitor the temperature continuously in the freezing medium at the centre of the test surface for at least one specimen in the freezing chamber. During the test, the temperature in the freezing medium shall fall within the shaded area shown in Figure 4. The temperature shall exceed 0 °C during each cycle for at least 7 h but not more than 9 h. The air temperature in the freezer shall never fall below -27 °C.



Key

1 Temperature range at the centre of the test surface

Figure 4 — The time (t) -temperature (T) cycle in the freezing medium at the centre of the test surface

The points specifying the shaded area in Figure 4 are given in Table 1.

Table 1 — Points specifying the shaded area in Figure 4

upper limit		lower limit		
t in h	T in °C	t in h T in °C		
0	+ 24,0	0 + 16,0		
5	- 3,0	3 - 5,0		
12	- 15,0	12 - 22,0		
16	- 18,0	16 - 22,0		
18	- 1,0	20	- 1,0	
22	+ 24,0	24	+ 16,0	

NOTE 1 To obtain the correct temperature cycle for all the specimens it is necessary to have a good air circulation in the freezing chamber.

NOTE 2 It is recommended that the number of specimens in the freezer is always the same. If only few specimens are to be tested, the empty places in the freezer should be filled with blanks, unless it has been shown that the correct temperature cycle is achieved without this precaution.

After (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 cycles, carry out the following procedure for each specimen during the thawed phase of the solution between 20 h to 24 h according to Figure 4:

- a) Collect material which has scaled from the test surface in the vessel (5.2.11). Rinse the surface using the spray bottle (5.2.14) and brush it (5.2.13) to remove the scaled material.
- b) Apply fresh freezing medium to the test surface. 67 ml are required for 150 mm x 150 mm test area.
- c) Return the specimen to the freezer.
- d) Carefully pour out the liquid in the vessel.

Note 3 It is recommended to pour the liquid through a suitable paper filter, especially where small amounts of scaled material are concerned.

The vessel containing the scaled material and the filter if used is dried to constant mass at (110 ± 10) °C and weighed to the nearest 0,1 g. The cumulative mass of the dried scaled material after n freeze-thaw cycle is determined by equation 1. Record the value rounded to the nearest 0,1 g.

$$m_{s,n} = m_{s,before} + (m_{v+s(+f)} - m_{v(+f)})$$
 (1)

where

m_{s, n} is the cumulative mass of dried scaled material after n freeze-thaw cycle rounded to the nearest 0,1 g;

ms, before is the cumulative mass of dried scaled material calculated by the measuring occasion before

 $m_{v+s(+f)}$ is the mass of the vessel containing the dried scaled material and the filter if used rounded to the nearest 0,1 g;

m_{v(+f)} is the mass of the empty vessel and the dry filter if used rounded to the nearest 0,1 g.

5.5 Expression of results

For each measurement and each specimen calculate S_n , the cumulative amount of scaled material per unit area after n cycles, in kilograms per square metre, by the equation:

$$S_{n} = \frac{m_{s,n}}{A} \cdot 10^{3} \tag{2}$$

where

 S_n is the mass of scaled material related to the test surface after the n-th cycle in kg/m²,

m_{s, n} is the cumulative mass of dried scaled material after n freeze-thaw cycle determined by equation 1;

A is the total area of the test surface, calculated from the length measurements before the glue string is applied and rounded to the nearest 100 mm².

The mean value and the individual values for each specimen after 56 cycles are used for evaluating the scaling resistance.

5.6 Test report

The test report shall contain at least the following information:

- a) Reference to this Technical Specification;
- b) origin and marking of the specimens;
- c) concrete identification;
- d) composition of the freezing medium (5.2.8);
- e) amount of cumulative scaled material for each specimen as well as the mean value in kilograms per square metre rounded to the nearest 0.02 kg/m^2 , after (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 freeze-thaw cycles;
- f) visual assessment (cracks, scaling from aggregate particles, leakage of water or salt solution) before the start and after (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 cycles;
- g) any deviations from the reference test procedure (e. g. 5.7);
- h) optional: Composition of the concrete.

5.7 Alternative applications

The reference test method is restricted to specimens with dimensions of approximately 50 mm x 150 mm x 150 mm, where the test starts at an age of 31 days and where a sawn surface is tested. The same test principle can, however, also be used for other conditions. It is normally the method of making and curing specimens that differs from the reference test procedure. Examples of alternative applications are:

- a) Other specimen geometries can be used but the thickness should always be (50 ± 2) mm. For example, the method is suitable for testing slices from cores drilled from structures or for testing precast units.
- b) Top surfaces and surfaces cast against formwork can be tested instead of sawn surfaces.
- c) Other curing conditions can be used and the concrete age may differ from 31 days at the start of the freeze thaw testing.
- d) Other de-icing agents than NaCl can be used.
- e) The number of freeze-thaw cycles may exceed 56. In some cases, e.g. for testing paving blocks, 28 cycles instead of 56 cycles may be used.

When alternative applications are used, the specimens are sawn to a thickness of (50 ± 2) mm 10 days before the start of the freeze-thaw test. During these 10 days the specimens are stored in the climate chamber for 7 days and then resaturated for 3 days as in the reference method, unless other curing conditions are of special interest. A

3 mm thick layer of the freezing medium is poured on to the test surface before the start of the freeze-thaw test. The test then continues according to the reference method.

All deviations from the reference method shall be noted in the test report.

6 Cube test (alternative method)

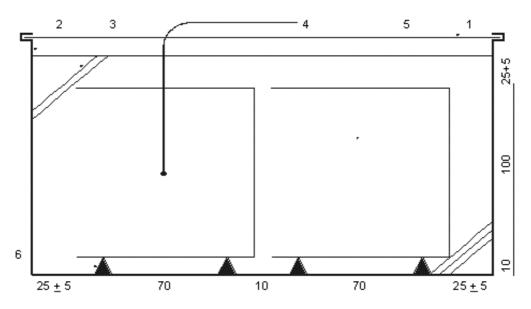
6.1 Principle

Cube specimens, immersed in de-ionised water or 3 % sodium chloride (NaCl) solution, are subjected to freeze-thaw attack. The freeze-thaw resistance is evaluated by the measurement of mass loss of the cubes after 56 freeze-thaw cycles.

6.2 Equipment

- **6.2.1** Equipment for making 100 mm concrete cubes according to EN 12390-2.
- **6.2.2** Climate controlled room or chamber with a temperature of (20 ± 2) °C and an evaporation of (45 ± 15) g/(m² h). Normally this is obtained with a wind velocity ≤ 0.1 m/s and a relative humidity of (65 ± 5) %. The evaporation is measured from a bowl with a depth of approximately 40 mm and a cross section area of (225 ± 25) cm². The bowl is filled up to (10 ± 1) mm from the brim.
- **6.2.3** Containers for the freeze-thaw test: Brass (semi hard brass 63:37) or stainless steel watertight containers with a width of (120 ± 15) mm, a length of (260 ± 15) mm and a height of (150 ± 15) mm (see Figure 5). The sheet metal is about 1 mm thick. The containers are closed with lids which are designed so that they cannot be lifted off when the containers are flooded; containers with sliding lids as shown in Figure 5 and Figure 6 have proved successful. The lid of one container has an opening which can be closed (see Figure 6) for measuring the temperature in the centre of one cube.

Dimensions in millimetres



Key

- Sliding lid
- 2 Container for specimens
- 3 Freezing medium

- 4 Temperature measuring device in the centre of a cube
- 5 Specimen
- 6 Spacers 10 mm high

Figure 5 — Container with specimens

6.2.4 Freezing medium, consisting either of 97 % by mass of tap water and 3 % by mass of NaCl (for test with de-icing salt) or of de-ionised water only (for test without de-icing salt).

- **6.2.5** Spacer (10 \pm 1) mm high placed on the container bottom to support the specimen and to guarantee a defined thickness of the liquid layer between the test surface and the container (see Figure 5).
- **6.2.6** An automatically-controlled freeze-thaw chest with a flooding device. Instead of the automatically-controlled chest a freezer and a water bath or a freeze-thaw chest with a secondary cooling circulation can be used.

The performance of the freeze-thaw chest or the freezer and the water bath is designed so that it is possible to maintain the temperature cycle in Figure 7 for each of the cubes placed in it.

6.2.7 Thermocouples, or an equivalent temperature measuring device, for measuring the temperature in the centre of the cube (see Figure 6) with an accuracy within 0,5 K.

NOTE A continuous recording device is particularly suitable for logging the temperature with which the temperature can be measured and recorded at least every 10 min over a period of 24 h.

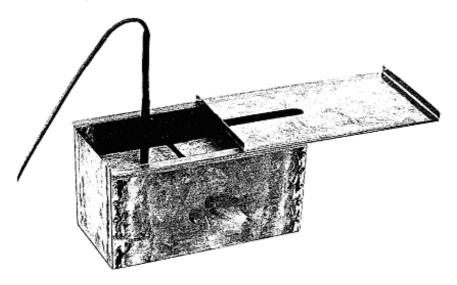


Figure 6 — Container with cubes and temperature sensor

- **6.2.8** Suitable paper filter for collecting scaled material, optional.
- **6.2.9** Brush, with short (about 20 mm), stiff bristles for brushing off material that has scaled.
- **6.2.10** Spray bottle, containing tap water for washing off scaled material.
- **6.2.11** Drying cabinet, controlled at a temperature of (110 ± 10) °C.
- **6.2.12** Balance, with an accuracy within \pm 0,05 g.

6.3 Preparation of test specimen

The test requires four 100 mm cubes (2 containers with 2 cubes each).

Lightly apply a demoulding agent to the internal surfaces of the cube moulds and wipe them with a dry sheet directly before they are filled with concrete so that the test results are not affected by excessive residues of the release agent.

During the first day after casting the cubes are stored in the moulds and protected against drying by use of a polyethylene sheet. The air temperature is (20 ± 2) °C.

After (24 \pm 2) h, the cubes are removed from the moulds and placed in a bath with tap water having a temperature of (20 \pm 2) °C.

When the cubes are 7 days old, they are removed from the water bath and placed in the climate chamber (6.2.2), where they are stored for 20 days.

6.4 Test procedure

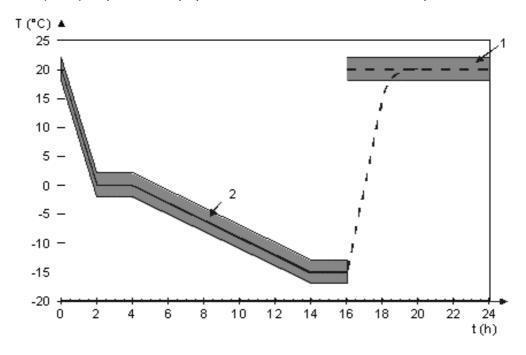
At 27 days, 1 day before the start of freezing test, determine the mass of the 4 cubes to an accuracy of 1 g. Then place the four cubes provided for the freezing test in two containers (6.2.3) so that the faces which were uppermost during casting are perpendicular to the base of the container and that there is about 10 mm distance between the cubes. Pour in freezing medium (6.2.4) until it covers the cubes by (25 ± 5) mm.

After 24 hours, determine the mass of each cube to an accuracy of 1 g and calculate the quantity of freezing medium absorbed in 24 hours from the increase in mass.

At 28 days place the containers with closed lids containing the cubes immersed in the freezing medium, evenly distributed, in the freeze-thaw chest (6.2.6) or the freezer. Start the freeze-thaw cycle. Change the containers around once a week; turn them through 180° and inter-change them on a cyclic basis.

Control the temperature of the freeze-thaw chest so that the temperature at the centre of the cube corresponds to the solid line in Figure 7 and shall not leave the shaded area in the diagram. The air temperature in the cooling chest should not fall below $-25\,^{\circ}$ C. Table 2 contains the mean value, the upper and the lower limit of the temperature curve as function of time.

NOTE The number of containers in the chest or freezer should always be the same. If only a few cubes are to be tested, containers with blanks (cubes) are put in for this purpose. The containers are not stacked on top of one another.



Key

- 1 Temperature of the water bath
- 2 Temperature in the centre of a 100 mm cube

Figure 7 — Temperature behaviour pattern in the centre of a cube

The points specifying the shaded area in Figure 7 are given in Table 2.

Immediately after the 16 h cooling phase if a freeze-thaw chest with air cooling is used, flood the chest with water or put the containers into a water bath at (20 ± 2) °C so that the water stands (20 ± 5) mm below the brims of the containers. The thawing phase lasts a total of 8 h. Keep the water moving at all times, and heat or cool so that the water temperature at all points in the chest or water bath is (20 ± 2) °C during the entire thawing process. If a freeze-thaw chest with secondary cooling is used, the freezing and the thawing of the specimens will be carried out by the cooling liquid.

Check the water temperature during the thawing process when first using the chest or the water bath and after approximately every 56 freeze-thaw cycles. 15 min before the end of the 8 h thawing phase, pump the water out of the chest over a maximum time of 15 min. Where a water bath is used, remove the containers from the bath.

<i>t</i> in h	<i>T</i> in °C				
	Upper limit	Nominal value	lower limit		
0	+ 22,0	+ 20,0	+ 18,0		
2	+ 2,0	0,0	- 2,0		
4	+ 2,0	0,0	- 2,0		
14	- 13,0	- 15,0	- 17,0		
16	- 13,0	- 15,0	- 17,0		

Table 2 — Points specifying the shaded area in Figure 7

If, in exceptional cases, it is necessary to interrupt the test or during the weekend, if non-automatic test equipment is used, the containers with the cubes remain in frozen state at (-15 ± 2) °C.

After (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 freeze-thaw cycles, carry out the following procedure during the thawed state of the specimens:

- a) Check the cubes visually to determine whether cracks or other substantial changes have occurred and whether the loss is on the surfaces or has occurred at the edges.
- b) Brush the cubes with the brush (6.2.9) using a light pressure in such a way that the pieces which have already been loosened are detached and can be collected. Pour the liquid carefully out of the container (and through a suitable filter).
- c) Before the start of the next cooling phase, fill the container with the cubes with fresh freezing medium (6.2.5) at (20 ± 2) °C. Return the container to the chest.
- d) Dry all the pieces which have been detached by the freezing action (from the container, from the filter and the above-mentioned brushings) to a constant mass at (110 ± 10) °C. The cumulative mass of dried scaled material is determined to an accuracy of 0,1 g by equation (3):

$$m_{s,n} = m_{s,before} + m_{c+f+b}$$
 (3)

where

m_{s, n} is the cumulative mass of dried scaled material after n freeze-thaw cycle rounded to the nearest 0,1 g;

m_{s, before} is the cumulative mass of dried scaled material calculated by the measuring occasion before;

 m_{c+f+b} is the mass of dried scaled material in the container, filter and obtained by brushing rounded to the nearest 0,1 g.

6.5 Expression of the results

Calculate the liquid absorption L for each cube before the start of the freeze-thaw cycles as a percentage by mass to the nearest 0,1 % by the equation:

$$L = \frac{m_{28d} - m_{27d}}{m_{27d}} \cdot 100 \tag{4}$$

where

 m_{27d} is the mass of the air dry cube at 27 days, in grams;

 m_{28d} is the mass of the saturated cube at 28 days, in grams

and determine the mean value of the four cubes to the nearest 0,1 %.

For each measurement calculate the loss *P* of two cubes in each container as a percentage by mass to the nearest 0,1 % by the equation:

$$P = \frac{m_{s,n}}{m_o} \cdot 100 \% \tag{5}$$

where

m_o is the mass of two air dry cubes (for one container) at 27 days, in grams;

m_{s, n} is the cumulative mass of the dried scaled material calculated by equation (3)

and determine the mean value for the two containers to the nearest 0,1 %.

The mean value and the individual values for loss in mass after 56 cycles are used for evaluating the scaling resistance.

6.6 Test report

The test report shall contain at least the following information:

- Reference to this Technical Specification;
- b) origin and marking of the specimens;
- c) concrete identification;
- d) composition of the freezing medium (6.2.5);
- e) loss of mass of the cubes for each container as well as the mean value in percentage by mass to the nearest 0,1 %, after (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 freeze-thaw cycles;
- f) visual assessment (notes on cracks, substantial changes in the cube and type of loss loss of material at the surfaces or edges) after (7 ± 1) , (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 freeze-thaw cycles. Unevenly distributed deterioration of the cubes should be mentioned;
- g) any deviations from this alternative test procedure (e. g. 6.7);
- h) optional: Liquid absorption by the cubes (mean) during the 24 hour water or NaCl-solution storage before the start of the freezing test in percentage by mass to the nearest 0,1 %;
- optional: Composition of the concrete.

6.7 Alternative applications

The alternative test method is restricted to 100 mm cubes, where the test starts at an age of 28 days. The same test principle can, however, also be used for other conditions. It is normally the method of making and curing specimens that differs from the standard test procedure. Examples of alternative applications are:

a) Other specimen geometries can be used but the specimens shall always be 80 mm to 100 mm in thickness and width. For example, the method is suitable for testing slices from cores drilled from structures, paving blocks or precast units.

- b) Other curing conditions can be used and the concrete age may differ from 28 days at the start of the freeze thaw testing.
- c) Other de-icing agents than NaCl can be used.
- d) The number of freeze-thaw cycles may exceed 56.

When alternative applications are used, the specimens are stored in the climate chamber for 20 days and then resaturated for one day in the freezing medium as in this alternative method, unless other curing conditions are of special interest. The test then continues according to this alternative method.

All deviations from this alternative method shall be noted in the test report.

7 CF/CDF-test (alternative method)

7.1 Principle

CF/CDF specimens, obtained by splitting a 150 mm cube mould with a centralised PTFE plate, are subjected to freeze-thaw attack in presence of de-ionised water (CF-test) or 3 % sodium chloride (NaCl) solution (CDF-test). The freeze-thaw scaling resistance is evaluated by the measurement of mass scaled from specimens after 28 freeze-thaw cycles (CDF test using 3 % sodium chloride (NaCl) solution) or after 56 freeze-thaw cycles (CF test using de-ionised water).

7.2 Equipment

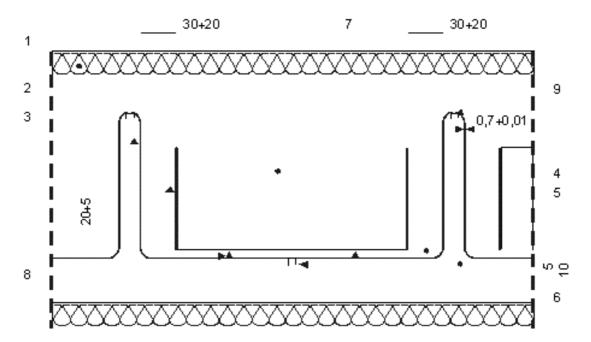
- **7.2.1** Equipment for making 150 mm concrete cubes according to EN 12390-2.
- **7.2.2** PTFE plate (Polytetrafluorethylene) or other materials with an equivalent hydrophobic surface serving as mould for the test surface. The geometry of the plate is adapted to the 150 mm cube mould and the thickness is less than 5 mm.
- **7.2.3** Climate controlled room or chamber with a temperature of (20 ± 2) °C and an evaporation of (45 ± 15) g/(m² h). Normally this is obtained with a wind velocity ≤ 0.1 m/s and a relative humidity of (65 ± 5) %. The evaporation is measured from a bowl with a depth of approximately 40 mm and a cross section area of (225 ± 25) cm². The bowl is filled up to (10 ± 1) mm from the brim.
- **7.2.4** Lateral sealing consists of solvent-free epoxy resin or aluminium foil with butyl rubber, durable to temperatures of -20 °C and resistant against the attack of the de-icing solution.
- **7.2.5** Freezing medium, consisting either of 97 % by mass of tap water and 3 % by mass of NaCl (for test with de-icing salt) or of de-ionised water only (for test without de-icing salt).
- **7.2.6** Unit for adjusting liquid level, i. e. by suction device. The suction device may consist of a capillary tube with a spacer of (10 ± 1) mm that is connected with e. g. a water jet pump to suck up the excessive liquid in the test containers.
- **7.2.7** Test containers (see Figure 8). The specimens are stored in stainless steel containers during the freeze-thaw cycles. The stainless sheet metal is (0.7 ± 0.01) mm thick. The size of the test container is selected in such a way that the thickness of the air layer between the vertical side of the specimen and the test container is restricted to (30 ± 20) mm.

The same test containers can be used for capillary suction. Other containers can be used if they assure an equivalent arrangement for capillary suction. During the capillary suction the test container is closed with a cover. The cover has an incline to prevent any possible condensation water from dripping onto the specimens.

- **7.2.8** Spacer (5 ± 0.1) mm high placed on the container bottom to support the specimen and to guarantee a defined thickness of the liquid layer between the test surface and the container (see Figure 8).
- **7.2.9** Temperature controlled chest (see Figure 8). For temperature control during the freeze-thaw cycle, a chest with a liquid cooling bath is used. The temperature of the cooling bath is controlled by an appropriate device. The

heating and cooling capacity and the control unit is capable of maintaining the temperature regime at the reference point within \pm 0,5 K with full loading by the test containers with the test specimens. The temperature deviation of the cooling liquid is limited at \pm 0,5 K at the minimum temperature and at \pm 1 K at other temperatures. A constant time shift between the test containers is acceptable.

Dimensions in millimetres



Key

- 1 Lid of the chest
- 2 Test container
- 3 Lateral sealing
- 4 Freezing medium
- 5 Cooling liquid

- 6 Reference point under the centre of the median container in the chest
- 7 Specimen
- 8 Spacer 5 mm high
- 9 Supports for adjusting the containers

Figure 8 — Principle arrangement of the CF/CDF-test during the freeze-thaw cycles

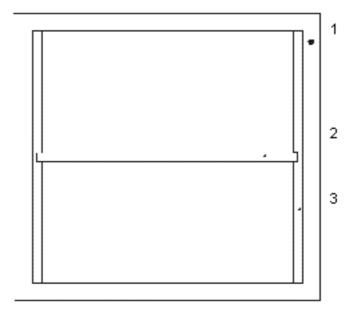
The chest is equipped with supports for the test containers above the cooling bath to ensure an immersion depth of the bottom of the test containers of approximately (20 ± 5) mm.

- **7.2.10** A temperature gauge with an accuracy of \pm 0,05 K at 0 °C is recommended for the measurement. The reference temperature is measured in the cooling bath liquid below the bottom of the test container. The temperature gauge is in the form of a rectangular container with dimensions (40 x 5 x 5 \pm 0,2) mm. A surface (5 x 40) mm of the temperature gauge is secured so that the long side of the probe lies in the direction flow. The time constant (t –90 %) of the probe (without securing device), determined according to EN 60751 in a flow water bath, should be (6,3 \pm 0,8) s. The minimum temperature of –20 °C is used for calibration.
- **7.2.11** Ultrasonic bath. The size of the ultrasonic bath is sufficiently large. The test container does not have a mechanical contact to the ultrasonic bath. The minimum distance between the test container and the lower surface of the bath amounts to 15 mm. The bath should provide the following power data: ERS power in the range of 180 W to 250 W; HF peak power under double half-wave operation in the range of 360 W to 500 W; frequency in the range of 35 kHz to 41 kHz.
- **7.2.12** Suitable paper filter for collecting scaled material, optional.
- **7.2.13** Drying cabinet, controlled at a temperature of (110 ± 10) °C.
- **7.2.14** Balance, with an accuracy within ± 0.05 g.
- **7.2.15** Vernier callipers, with an accuracy within ± 0.1 mm.

7.3 Preparation of test specimens

The test requires five specimens. The test surface of each specimen is approx. 140 mm x 150 mm.

The PTFE plate (7.2.2) is centred in the mould in order to divide the mould into halves. The vertical PTFE plate serves as a mould surface. The centred plate can be fixed by two other plates which are placed vertically (see Figure 9). The concrete surface at the PTFE plate is the test surface. The PTFE plate is not treated with any demoulding agent.



Key

- 1 Form work 150 mm x 150 mm x 150 mm
- 2 Centred PTFE disk
- 3 Lateral PTFE disk

Figure 9 — Arrangement of PTFE plates

During the first day after casting the cubes are stored in the moulds and protected against drying by use of a polyethylene sheet. The air temperature is (20 ± 2) °C.

After (24 ± 2) h, the cubes are removed from the moulds and placed in a bath with tap water having a temperature of (20 ± 2) °C.

When the cubes are 7 days old they are removed from the water bath and placed in the climate chamber (7.2.3), where they are stored for surface drying for 21 days until the freeze-thaw testing starts.

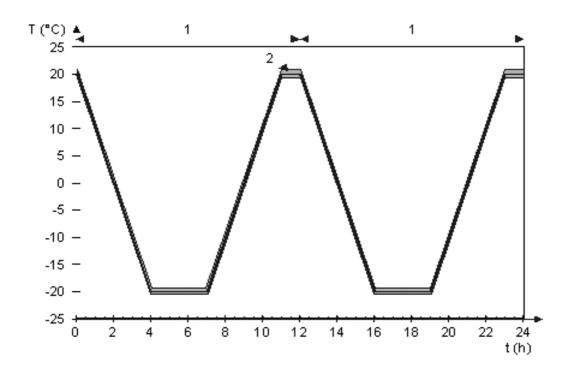
Between 21st and 26th day after casting the specimens the lateral surfaces are either covered with aluminium foil glued with butyl rubber or sealed with a solvent-free epoxy resin. Immediately after this treatment the specimens are returned to the climate chamber.

7.4 Test procedure

The freeze-thaw test starts after 28 days with the resaturation of the specimens.

Following dry storage, the specimens are placed in the test containers on the (5 ± 0.1) mm high spacers with the test surface downwards (see Figure 8). Subsequently, the freezing medium is poured into the container to a height of (10 ± 1) mm without wetting the specimen's top.

NOTE This can be achieved by filling to approx. 13 mm and removing the surplus solution by means of suction device (7.2.6).



Key

- 1 Freeze-thaw cycle
- 2 Temperature measured at the reference point

Figure 10 — Temperature curve

During the capillary suction the test container is closed. The capillary suction period is seven days at a temperature of (20 ± 2) °C. Check and adjust the liquid level above at regular intervals, depending on the suction capacity of the material during capillary suction. The weight gain of the specimens is measured.

Before starting the freeze-thaw cycles, remove loosely adhering particles and dirt from the test surfaces of the specimens by treatment in the ultrasonic bath (7.2.11) described in 7.4 a. The material removed is discarded.

A 12 hour freeze-thaw cycle is applied (see Figure 10). The temperature cycle in the chest (7.2.10) is monitored continuously at the reference point.

The points specifying the temperature curve in Figure 10 are given in Table 3.

Table 3 — Points specifying the shaded area in Figure 10

<i>t</i> in h	₹ in °C			
2 111 11	upper limit	nominal value	lower limit	
0	+ 21,0	+ 20,0	+ 19,0	
4	- 19,5	- 20,0	- 20,5	
7	- 19,5 - 20,0 -		- 20,5	
11	+ 21,0	+ 20,0	+ 19,0	
12	+ 21,0 + 20,0		+ 19,0	

After (4 ± 1) , (6 ± 1) , (14 ± 1) and 28 freeze-thaw cycles (CDF-test) or (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 freeze-thaw cycles (CF-test), carry out the following procedure for each specimen while the temperature is above 15 °C.

a) To remove loosely adhering scaled material from the test surface, the test container is dipped into the contact liquid of an ultrasonic bath (7.2.11) and subjected to ultrasonic cleaning for three minutes.

b) The solution comprising the scaled material is filtered. The suitable paper filter is subsequently dried at (110 ± 10) °C for 24 hours and cooled for (60 ± 5) min at a temperature of (20 ± 2) °C and a relative humidity of (65 ± 5) %. The mass of the filter containing the dried scaled material m_{s+f} is weighed to 0,1 g. The mass of the empty filter m_f is determined before with the same accuracy. The mass of the dried scaled material $m_{s, n}$ is determined by equation (6):

$$\mathbf{m}_{s,n} = \mathbf{m}_{s,before} + (\mathbf{m}_{s+f} - \mathbf{m}_{f}) \tag{6}$$

where

m_{s. n} is the cumulative mass of dried scaled material after n freeze-thaw cycle rounded to the nearest 0,1 g;

m_{s, before} is the cumulative mass of dried scaled material calculated by the measuring occasion before;

m_{s+f} is the mass of the dried filter with the scaled material rounded to the nearest 0,1 g;

m_f is the mass of the dry filter rounded to the nearest 0,1 g.

- c) Before the start of the next freeze-thaw cycle pour fresh freezing medium (7.2.5) into the container to a height of (10 ± 1) mm without wetting the specimen's top.
- d) Return the container to the chest.

7.5 Expression of test results

For each measurement and each specimen calculate S_n , the cumulative amount of scaled material per unit area after n cycles, in kilograms per square metre, by equation (7):

$$S_n = \frac{m_{s,n}}{A} \cdot 10^3 \tag{7}$$

where

S_n is the mass of scaled material related to the test surface after the n-th cycle in kg/m²,

m_{s.n} is the cumulative mass of dried scaled material after n freeze-thaw cycle determined by equation (6),

A is the test surface in mm², calculated on the basis of the linear dimensions as the average of at least two measurements determined to the nearest 0,5 mm.

The mean value and the standard deviation of the scaled material are evaluated. The mean value and the individual values for each specimen after 28 cycles (CDF-test) or 56 cycles (CF-test) are used for evaluating the scaling resistance.

7.6 Test report

The test report shall contain at least the following information:

- a) Reference to this Technical Specification;
- b) origin, size and marking of the specimens;
- c) concrete identification;
- d) the composition of the freezing medium (7.2.5);
- e) amount of cumulative scaled material for each specimen as well as the mean value and the standard deviation in kilograms per square metre rounded to the nearest 0,001 kg/m², after (4 ± 1) , (6 ± 1) , (14 ± 1) and 28 cycles (CDF-test) or (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 cycles (CF-test);

- f) visual assessment (cracks, scaling from aggregate particles) before the start and after (4 ± 1) , (6 ± 1) , (14 ± 1) and 28 cycles (CDF-test) or (14 ± 1) , (28 ± 1) , (42 ± 1) and 56 cycles (CF-test);
- g) any deviations from this alternative test procedure (e. g. 7.7);
- h) optional: Mass of solution sucked up during the capillary suction period for each specimen as well as the mean value and the standard deviation;
- i) optional: The composition of the concrete.

7.7 Alternative applications

The alternative test method is restricted to at least five specimens whereby each specimen has a dimension of approx. 70 mm x 140 mm x 150 mm, where the test starts at an age of 28 days. The same test principle can, however, also be used for other conditions. It is normally the method of making and curing specimens that differs from the standard test procedure. Examples of alternative applications are:

- a) Other specimen geometries may be used provided the thickness shall be (70 \pm 5 mm) and the total test surface area shall be \geq 0,08 m². For example, the method is suitable for testing slices from cores drilled from structures, paving blocks or for testing precast units.
- b) It is permissible to insert two PTFE plates (7.2.2) at two opposed vertical sides. In this case, the specimens are cut through the centre between the two test surfaces after storage under water. For larger aggregate size the PTFE plate can be placed only at one side of the mould.
- c) If the strength development of the specimens is low the curing time in the mould can be increased. The storage time in tap water is then decreased by the same amount.
- d) Other curing conditions can be used and the concrete age may differ from 28 days at the start of the freeze thaw testing.
- e) Other de-icing agents than NaCl can be used.
- f) The number of freeze-thaw cycles may exceed 28 (CDF-test) or 56 (CF-test).

When alternative applications are used, the specimens are stored for surface drying in the climate chamber (7.2.3) for 21 days and then resaturated for 7 days in the freezing medium as in this alternative method, unless other curing conditions are of special interest. The test then continues according to this alternative method.

All deviations from this alternative method shall be noted in the test report.

8 Precision data

The precision estimates from each of the tests, given as coefficients of variation in Table 5;

- a) are derived from data for repeatability and reproducibility determined from inter-laboratory trials carried out in accordance with ISO 5725 and published in: Breit, W.; Siebel, E.: Standard methods for testing the resistance of concrete to freezing and thawing Round robin test. Milestone Report Work Package 3, European Research Project MAT1-CT94-0055, Forschungsinstitut der Zementindustrie, Report No. B1489/3, Düsseldorf, Juni 1998;
- b) are based on the functional relationships between scaling (general mean m) and coefficients of variation (V_r) given in Table 4 for each of the three test methods described in this document;
- c) give an indication of the differences between test results for the reference method at one specific scaling level;
- d) apply only to scaling caused by the action of 3 % sodium chloride solution as the freezing medium".

Table 4 — Functional relation between scaling and repeatability and reproducibility of coefficient of variation (scaling with 3 %-sodium chloride solution)

Test		Repeatability coefficient of variation			Reproducibility coefficient of variation			
method	I linite for m		$v_r = a \cdot m^b$			$v_{R} = a \cdot m^{b}$		
		а	b	R²	а	b	R²	
Slab	kg/m²	17,00	- 0,24	0,99	31,00	- 0,23	0,95	
CDF	kg/m²	15,00	- 0,12	0,91	31,00	- 0,18	0,94	
Cube	% by mass	27,00	- 0,35	0,89	54,00	- 0,26	0,50	

Table 5 — Precision data for appropriate scaling level

Test method	Society lovel	Coefficient of variation [%]		
rest method	Scaling level	repeatability $v_{\rm r}$	reproducibility v_{R}	
Slab	1,0 kg/m²	17	31	
CDF	1,5 kg/m²	14	29	
cube	3 % by mass	18	38	

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