

BAW Code of Practice

Frost Resistance Tests for Concrete (MFB)

Issue 2012



BAW Codes of Practice and Guidelines Publisher

Bundesanstalt für Wasserbau (BAW) Kussmaulstrasse 17 76187 Karlsruhe, Germany

P. O. Box 21 02 53 76152 Karlsruhe, Germany

Tel.: +49 721 9726-0 Fax: +49 721 9726-4540

info@baw.de www.baw.de

No part of this bulletin may be translated, reproduced or duplicated in any from or by any means without the prior permission of the publisher: © BAW 2012

Table	of Contents	Page
Modific	cations	1
Previou	us Editions	1
Prelimi	nary remarks	1
1	Introduction	1
2	Literature and Normative references	2
3	Terms and definitions	3
4	Equipment	3
5	Test specimens	7
5.1	General requirements	7
5.2	Preparation of the standard test specimens	7
5.2.1	Specimens for testing concrete	7
5.2.2	Test specimens for testing sprayed mortar and sprayed concrete	9
5.3	Non-standard test specimens	10
6	Test procedure	10
6.1	General	10
6.2	Dry storage	11
6.3	Presaturation	11
6.3.1	Preparation and sealing of the test specimens	11
6.3.2	Presaturation with test liquid by capillary suction	12
6.4	Freeze-thaw testing	12
7	Measurements	12
7.1	Sequence of measurements with respect to scaling, moisture uptake and ultrasonic transi	t time 12
7.2	Determination of surface scaling	13
7.2.1	Procedure	13
7.2.2	Evaluation of scaling	14
7.3	Measurement of the moisture uptake	14
7.3.1	Procedure	14
7.3.2	Evaluation of the moisture uptake	14
7.4	Measurement of the ultrasonic transit time (internal damage)	15
7.4.1	Test arrangement	15
7.4.2	Calibration	15
7.4.3	Procedure	16
7.4.4	Evaluation of the internal damage	16

8	Evaluation of the freeze-thaw resistance after the CIF test	17
8.1	Relevant acceptance criterion - internal damage	17
8.2	Additional acceptance criterion - scaling	18
9	Evaluation of the freeze-thaw and de-icing agent resistance after the CDF test	18
9.1	Relevant acceptance criterion - scaling	18
9.2	Additional acceptance criterion - internal damage	18
10	Report	19
11	Requirements for laboratories and reference samples	20
12	Precision data	20
12.1	General	20
12.2	Precision of the CIF test for concrete mixes	20
12.2.1	Measurement of the internal damage - ultrasonic transit time	20
12.2.2	Moisture uptake	21
12.2.3	Scaling	21
12.3	Precision of the CDF test for concrete mixes	21
12.3.1	Scaling	21

List of Tables

Table 6.1:	Storage periods prior to the start of freezing	11
Table 8.1:	Acceptance criteria for internal damage in the CIF test	17
Table 8.2:	Acceptance criteria for scaling in the CIF test	18
Table 9.1:	Acceptance criteria for scaling in the CDF test	18
Table 9.2:	Acceptance criteria for internal damage in the CDF test	18
Table 12.1:	Precision data for the measurement of the internal damage $\ \ $ - ultrasonic transit time in the CIF test	20
Table 12.2:	Precision data for the measurement of the moisture uptake	21
Table 12.3:	Precision data for the measurement of scaling in the CIF test	21
Table 12.4:	Precision data for the measurement of scaling in the CDF test	22
Table 12.5:	Parameters v_0 and d for scaling in the CDF test according to /1/	22

List of Figures

Figure 1:	Capillary suction	4
Figure 2:	Test container with test specimen in a bath with a heating and cooling liquid	4
Figure 3:	Temperature-controlled chest	5
Figure 4:	Control temperature cycle. The measurements referred to in Section 7 may be performed a temperatures over 15 °C (shaded area in graph).	at 5
Figure 5:	Suction device	6
Figure 6:	Ultrasonic bath	6
Figure 7:	Sawing the test specimen and reference sample where the PTFE plates are placed at the sides of the mould	9
Figure 8:	Sequence for the measurement of the ultrasonic transit time	13
Figure 9:	Test arrangement for measuring the ultrasonic transit time	15

Modifications

Referring to the Code of Practice "Frost Resistance Tests for Concrete" (MFB), Issue 2004, the references to standards were updatet.

Previous Editions

Code of Practice "Frost Resistance Tests for Concrete" (MFB), Issue 2004.

Preliminary remarks

The BAW's Code of Practice "Frost Resistance Tests for Concrete" describes methods of testing the freeze-thaw resistance and freeze-thaw and de-icing agent resistance of concrete, sprayed mortar and sprayed concrete. Standardized methods are not yet available for either of these tests.

The CIF test /1/ is to be used for testing the freeze-thaw resistance and the CDF test /2/ for testing the freeze-thaw and de-icing agent resistance. Both test methods have been published as RILEM Recommendations. The following test description is essentially an adaptation of the RILEM Recommendation for the CIF test /1/ which has been modified to take account of waterways engineering requirements. The modifications apply to the preparation, geometry and conditioning of the test specimens, the age at test-ing and the acceptance criteria. Advances in the way the CIF is conducted and the modifications referred to above also apply to the CDF test. In addition to the RILEM Recommendation /1/ and the European prestandard CEN/TS 12390-9, measurement of the ultrasonic transit time has been included in the CDF test in order to enable the degree of internal damage to be determined.

Apart from the different test liquids used, the following test description applies to both the freeze-thaw resistance test and the freeze-thaw and de-icing agent resistance test.

The acceptance criteria laid down for the assessment of the freeze-thaw resistance and the freeze-thaw and de-icing agent resistance are based on tests on the types of concrete and sprayed concrete commonly used in waterways engineering¹ and apply to the assessment of test specimens prepared specifically for suitability and quality testing. The acceptance criteria do not apply to assessments of existing structures undergoing structural inspection.

1 Introduction

The CIF test is used to investigate the resistance to freeze-thaw attack in the presence of water only. The abbreviation "CIF" stands for "Capillary suction, Internal damage and Freeze-thaw test". During the test, the degree of water saturation is increased, initially by isothermal capillary suction and then by defined freeze-thaw cycles (frost suction) corresponding to the uniaxial loading occurring in practice. The CIF test enables the combined measurement of the moisture uptake and the internal damage caused by a number of freeze-thaw cycles with uniaxial heat and moisture flux in the presence of water. The internal damage

¹ Concretes and sprayed concretes commonly used in waterways engineering are generally those complying with the requirements of the Supplementary Technical Contract Conditions - Waterways Engineering (ZTV-W), Sections 215 and 219. It may be necessary to specify separate acceptance criteria for concretes with a high cement paste content and for concretes without air-entraining admixtures and with an atypically low air content which are not usually used in waterways engineering.

caused by moisture uptake and the associated degree of moisture saturation is the key parameter for assessing the freeze-thaw resistance. Surface scaling is also measured.

The CDF test is used to determine the resistance to freeze-thaw attack in the presence of de-icing agents. The abbreviation "CDF" stands for "Capillary suction of De-icing chemicals and Freeze-thaw test". The CDF test enables the combined measurement of the moisture uptake and the internal damage caused by a number of freeze-thaw cycles with uniaxial heat and moisture flux in the presence of a de-fined test solution. A defined solution of de-icing salt (3 % sodium chloride solution) is generally used. Surface scaling is the dominant feature of the freeze-thaw and de-icing agent resistance test and is the decisive factor in the assessment. Internal damage is also measured.

2 Literature and Normative references

- /1/ CIF Test Testmethode zur Bestimmung des Frost-Widerstands von Beton (CIF). Final Recommendation of RILEM TC 176-IDC ,Internal Damage of Concrete due to frost action: Test methods of frost resistance of concrete. Materials and Structures, Vol. 37 - No 274 (12.2004) p. 742-75
- /2/ CDF Test Testmethode zur Bestimmung des Frost-Tausalz-Widerstands von Beton Pr
 üfung mit einer Natriumchloridlösung (CDF). RILEM Recommendation TC117-FDC: Freeze-thaw and de-icing resistance of concrete. Materials and Structures Vol. 29 (1996) 523-528.

CEN/TS 12390-9	Prüfung von Festbeton - Teil 9: Frost- und Frost-Tausalz-Widerstand - Abwitte- rung; Deutsche Fassung CEN/TS 12390-9
DIN 1045-2	Tragwerke aus Beton, Stahlbeton und Spannbeton – Teil 2: Beton – Festlegung, Eigenschaften, Herstellung und Konformität – Anwendungsregeln zu DIN EN 206-1
DIN EN 206-1	Beton – Teil 1: Festlegung, Eigenschaften, Herstellung und Konformität
DIN EN 12350	Prüfung von Frischbeton
DIN EN 12390-1	Prüfung von Festbeton, Teil 1: Form, Maße und andere Anforderungen für Probekörper und Formen
DIN EN 12390-2	Prüfung von Festbeton, Teil 2: Herstellung und Lagerung von Probekörpern für Festigkeitsprüfungen
DIN EN 12504-4	Prüfung von Beton in Bauwerken – Bestimmung der Ultraschallgeschwindigkeit
DIN EN 14488-1	Prüfung von Spritzbeton – Teil 1: Probenahme von Frisch- und Festbeton
DIN ISO 5725	Genauigkeit (Richtigkeit und Präzision) von Messverfahren und Messergebnissen
VDI/VDE 3522	Zeitverhalten von Berührungsthermometern, (Time performance of contact ther- mometers), Juni 1987.

3 Terms and definitions

- a) *Freeze-thaw resistance* is the resistance to alternating freezing and thawing in the presence of demineralized water as the test liquid.
- b) *Freeze-thaw and de-icing agent resistance* is the resistance to alternating freezing and thawing in the presence of a de-icing agent solution as the test liquid.
- c) The *test liquid* is the liquid taken up by the test specimens during the test procedure (Section 4 c).
- d) *Scaling* is the loss of material from the surface of concrete due to freeze-thaw or freeze-thaw de-icing agent attack.
- e) Internal damage is the deterioration of the internal structure of concrete (even without any visible external damage) which leads to changes in the properties of the concrete (such as a reduction in the dynamic modulus of elasticity, the bending tensile strength and the resistance of the boundary zone of the concrete to the penetration of harmful substances).
- f) The *reference point* is the physical measuring point at which the temperature cycle is controlled.
- g) The reference temperature is the temperature measured at the reference point.
- h) The *test surface* is the surface of the test specimen over which the temperature change and the test liquid act on the specimen during the test procedure.
- i) The *ultrasonic transit axis* is the hypothetical shortest distance between the centres of the coupling surfaces of the transmitter and receiver transducers.
- j) The *ultrasonic transit path* is the path over which the ultrasonic transit time is measured; it is the shortest path on the ultrasonic transit axis between the ultrasound transmitter and receiver.
- k) The *ultrasonic transit time* is the time required by an ultrasonic pulse wave to cover the ultrasonic transit path between the transmitter and receiver.
- I) The test liquid is used as the *coupling medium*. It enables a reproducible signal transfer to take place between the transducers and the test specimen.

4 Equipment

- a) Climate chamber: With a temperature of (20 ± 2) °C and a relative humidity of 65 ± 5 %. In the climate chamber the rate of evaporation from a free water surface shall be 45 ± 15 g/(m² h). This is usually achieved with a wind velocity of ≤ 0.1 m/s. The evaporation shall be measured by means of a bowl with a depth of approx. 40 mm and a cross-sectional area of 225 ± 25 cm² which shall be filled up to 10 ± 1 mm from the brim.
- b) Lateral sealing: Aluminium foil with butyl rubber (reference method) or epoxy resin (alternative method). The sealing must be durable at temperatures of -20 °C. It must not be brittle at the minimum temperature nor become detached from the test specimen. A suitable primer shall be used.

and 3 % by mass NACI).

c) Test liquid: Freeze-thaw test (CIF test): Freeze-thaw and de-icing agent resistance (CDF test): demineralized water standard de-icing agent solution (97 % by mass demineralized water

d) Test containers (Figure 1 and Figure 2): The test containers are made of stainless steel. The size of the test containers shall be such that the thickness of the air layer between the vertical sides of the test specimen and the test container is limited to 30 ± 20 mm.^{2,3}. A 5 ± 0.1 mm spacer and a lid are also required.

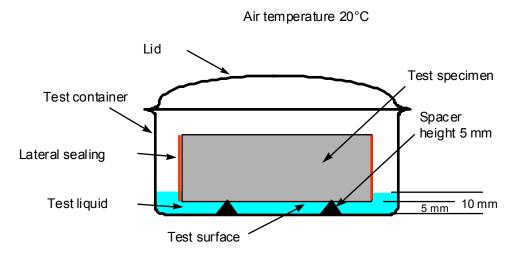


Figure 1: Capillary suction

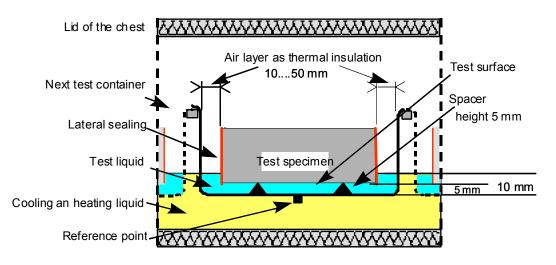


Figure 2: Test container with test specimen in a bath with a heating and cooling liquid

e) *Temperature-controlled chest* (Figure 3): A chest with a bath with a heating and cooling liquid is used. The temperature of the cooling and heating bath is controlled by an appropriate device. The heating and cooling capacity and the control unit must be capable of maintaining the temperature regime at the reference point according to the temperature cycle (Figure 4).

² The air layer between the vertical sides of the test specimen and the test container acts as thermal insulation.

 $^{^{3}}$ The stainless steel containers are available in a variety of modular sizes so that the same boundary conditions can be met for each size of specimen.

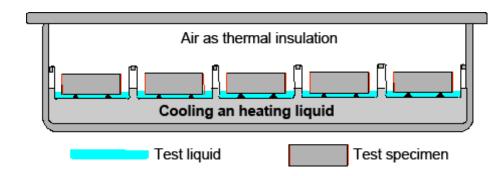
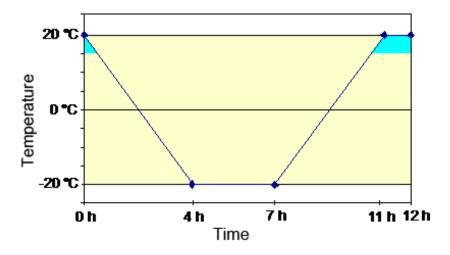


Figure 3: Temperature-controlled chest

The chest must be equipped with supports above the cooling and heating bath to ensure an immersion depth of the bottom of the test containers of 15 ± 3 mm. If the cooling and heating bath is not filled completely with test specimens any gaps shall be occupied by empty test containers, for example.⁴

A test container placed at a representative point of the bath (usually the centre) is used for monitoring and controlling the reference temperature which is measured in the cooling and heating liquid in the bath at the bottom of a test container. The reference point is positioned in good thermal contact in the centre of the base of the container.

A temperature gauge with an accuracy of ± 0.05 K at 0 °C is used for the measurement. It must be in the form of a rectangular container with the dimensions 50 x 6 x 6 mm ± 0.2 mm. It is fixed so that the long side (50 x 6 mm) lies in the direction of the flow. The time constant (t-90 %) of the probe (without securing device), determined according to VDI/VDE 3522 in a flowing water bath, must be 6.3 s ± 0.8 s. The minimum temperature of -20 °C is used for calibration purposes.



The equipment must ensure freeze-thaw cycles according to the temperature cycle shown in Figure 4.

Figure 4: Control temperature cycle. The measurements referred to in Section 7 may be performed at temperatures over 15 °C (shaded area in graph).

A freeze-thaw cycle of 12 hours is applied. Starting at +20 °C, the temperature is lowered at a constant cooling rate of 10 K/h over 4 hours. It is then maintained at a constant -20 °C for 3 hours before being raised to +20 °C at a heating rate of 10 K/h over 4 hours. The temperature is maintained at a constant +20 °C for 1 hour. The temperature cycle is monitored at the reference point. The temperature measured at the reference point may not deviate from the minimum temperature by more than \pm 0.5 K or more than \pm 1 K from all other temperatures. A constant time shift between the test containers is acceptable. The tolerance on the temperature may be exceeded for a maximum of 10 minutes immediately after the first ice formation.

f) Unit for adjusting the liquid level: E. g. a suction device (Figure 5). The suction device may consist of a capillary tube with a spacer of 10 ± 1 mm that is connected to a water jet pump to suck up the excess water in the test containers.

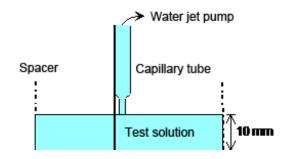
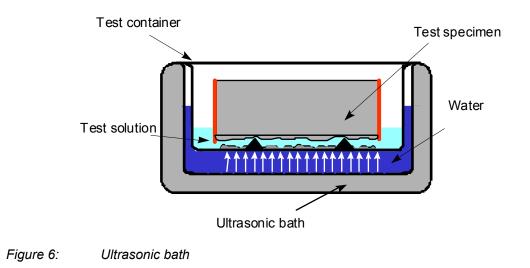


Figure 5: Suction device

g) Ultrasonic bath (Figure 6): The dimensions of the ultrasonic bath shall be sufficiently large so that there is no mechanical contact between the test containers and the ultrasonic unit in the coupling medium. In addition, a minimum distance of at least 15 mm between the test container and the bottom of the bath must be ensured. The ultrasonic bath must comply with the following power requirements: ERS power 250 W; HF peak power 450 W under double half-wave operation; frequency 35 kHz.



⁴ There is no need to cover the test containers if the test is conducted in a cryogenic bath as the lid of the chest provides sufficient protection against evaporation while the walls of the test container act as a cold trap.

- h) Equipment for measuring the ultrasonic transit time: The ultrasonic transit time can be measured by conventional commercially available ultrasonic measuring equipment. The equipment must be suitable for determining the transit times of longitudinal waves in the case of direct transmission in accordance with DIN EN 12504-4. The use of a device indicating the signal pattern (arrival amplitude) is recommended to check the plausibility of the measurements. The transducers must operate in a frequency range between 50 and 150 kHz and should have a diameter of 30 ± 10 mm.
- Test container for ultrasonic transit time measurement: A container made of an electrically nonconductive material (e. g. polymethyl methacrylate) is used for the ultrasonic transit time measurement. The transducers must be mounted such that the transit axis is parallel to and at a distance of 35 mm from the test surface (e. g. in recesses on two opposite sides of the container, see Figure 9). The dimensions of the test container must ensure calibration in accordance with Section 7.4.2.
- j) Calibration test specimen: A calibration test specimen with the dimensions 150 x 120 x 70 mm (± 0.1 mm) is used to calibrate the ultrasonic transit time measurement assembly. The calibration test specimen shall have a defined ultrasonic transit time and be provided with gauge marks.
- k) Specimen tray: A tray (preferably made of 1 mm V2A steel) with handles to facilitate handling of the test specimens during the measurement of the moisture uptake and the internal damage. The dimensions of the tray must be larger than those of the test surface to ensure that all scaled material can be collected. The height of the folded-up edges of the tray must be 10 mm ± 2 mm.
- I) Drying cabinet: A drying cabinet maintained at a temperature of 110 ± 5 °C must be used.
- m) Paper filters: Paper filters are used for collecting the scaled material.
- n) Balance: With an accuracy of ± 0.01 g for measuring the weight of the scaled material.
- o) Balance: With an accuracy of ± 0.1 g for measuring the weight of the test specimens.
- p) Vernier calipers: With an accuracy of ± 0.1 mm.
- q) *PTFE plates*: PTFE plates (e. g. Teflon) with the dimensions 150 x 150 x 2 mm are added to standard moulds with the dimensions 150 x 150 x 150 mm.

5 Test specimens

5.1 General requirements

At least five test specimens are used for each test series and the total test surface must be at least 0.08 m^2 . Five test specimens enable a statistical evaluation to be made and any outliers to be identified. The height of any test specimen must be 70 mm (± 2 mm).

5.2 Preparation of the standard test specimens

5.2.1 Specimens for testing concrete

5.2.1.1 Dimensions

The dimensions of the standard test specimens are $150 \times 150 \times 70 \text{ mm}$ (± 2 mm) (length x width x height). The width of the test specimens may be reduced to 110 mm (minimum dimension).

5.2.1.2 Preparation of test specimens for suitability and quality testing

Concrete production

The test specimens for the suitability and quality testing of concrete mixes are made in 150 mm cube moulds as specified in DIN EN 12390-1. The requirements of DIN 1045-2, subclause 9.8, must be satisfied when mixing the concrete. The test specimens must be cast and compacted on a vibrating table in accordance with DIN EN 12390-2. The time taken to compact the concrete shall be long enough to achieve complete compaction and depends on the consistency of the concrete. The freeze-thaw resistance of concrete depends to a large extent on the pore structure, in particular on the capillary-inactive air content and the density. As a result, concretes that have undergone different degrees of compaction may also differ with regard to their freeze-thaw resistance. Complete compaction of the concrete is therefore particularly important for the assessment of the freeze-thaw resistance in suitability and quality tests. Compaction must be carried out until no more air can be seen to be driven out of the fresh concrete. Segregation or bleeding of the concrete is not permitted.

If admixtures are used, the quantities used shall be the same as those that are likely to be used in practice. The density, air content and consistency of fresh concrete shall be tested and documented as specified in DIN EN 12350.

Two vertical PTFE plates shall be placed firmly in the cube mould, with one each at two opposite sides of the mould. The PTFE plates must not be coated with a release agent. The test surface is the concrete surface cast against the PTFE plate. The maximum size of the aggregate in the concrete must not exceed one third of the shorter ultrasonic transit path (length and width in accordance with 5.2.1.1).

Curing

The test specimens are left in the moulds for 24 ± 2 hours, during which the free upper surfaces are protected against drying out, and subsequently demoulded. The curing time in the moulds may be extended to 48 ± 2 hours if the strength development of the concrete is slow.

After removal from the moulds, the test specimens are stored in tap water with a temperature of (20 ± 2) °C. The length of storage in water after removal from the moulds depends on the time at which presaturation of the test specimens (capillary suction) begins (see Section 6). Except when otherwise agreed, presaturation begins when the compressive strength class is verified. The following periods therefore apply:

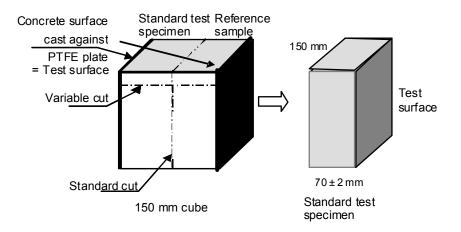
- Concretes conforming to DIN EN 206-1/DIN 1045-2 must be cured in water for 6 days⁵ (until the 7th day); presaturation begins on the 28th day.
- Concretes whose compressive strength class may be verified after 56 days may be cured in water for 13 days⁵ (until the 14th day) if presaturation does not begin until the 56th day.

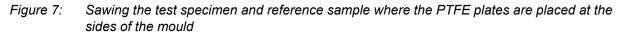
Cutting the test specimens

Immediately after storage under water, the test specimens are sawn to the standard height. Cutting produces a standard test specimen and a reference sample (Figure 7). If a reduction in the specimen width

⁵ One day less if the specimens are cured in the mould for 48 hours.

has been agreed, the test specimens may be cut along the rough upper side to a minimum width of 110 mm (variable cut). Cutting is followed by dry storage.





5.2.2 Test specimens for testing sprayed mortar and sprayed concrete

5.2.2.1 Dimensions

The samples may be cylindrical or rectangular in shape. Standard cylindrical test specimens must be 150 mm in diameter and 70 mm (± 2 mm) high and standard rectangular test specimens shall have 150 mm sides and a height of 70 mm (± 2 mm).

5.2.2.2 Preparation of test specimens for suitability and quality testing

Production and curing of the test slabs

Five separate test slabs in accordance with DIN EN 14488-1 are required for the suitability and quality testing of sprayed mortar and sprayed concrete. The surfaces of the slabs must be left in the as-sprayed condition. After preparation, the test slabs are left in the moulds in a closed room with an air temperature between 15 and 22 °C for 24 ± 2 hours during which the free upper surfaces are covered with damp cloths to protect them against moisture loss. The slabs are then demoulded. The curing time in the moulds may be extended to 48 ± 2 hours if the strength development of the slabs does not permit demoulding after 24 hours.

After removal from the moulds, the slabs are stored in tap water with a temperature of (20 ± 2) °C. Unless otherwise agreed, the slabs must be stored in water for 6 days after demoulding⁶ (until the 7th day).

Cutting the test specimens

Immediately after the slabs have been cured in water one specimen and one reference sample must be cut from each slab by sawing or drilling perpendicular to the as-sprayed surface by the wet-cutting method. The test specimens are sawn parallel to the as-sprayed surface, removing only as much material as is necessary to produce a smooth, closed surface. This surface is the test surface. The specimens are

⁶ One day less if the specimens are cured in the mould for 48 hours.

then reduced to a height of 70 ± 2 mm by sawing them parallel to the test surface. Cutting is followed by dry storage.

5.3 Non-standard test specimens

If non-standard concrete test specimens (e. g. specimens cut from concrete members) are tested, this must be stated in the test report.

The minimum diameter of cores drilled from finished sprayed mortar or sprayed concrete members (e. g. for monitoring the quality of workmanship) depends on the maximum size of the aggregate. The minimum diameter of the specimens is 100 mm for maximum aggregate sizes up to 16 mm and 150 mm for larger maximum aggregate sizes.

The test surface of each specimen must be large enough to completely fit a circle with a diameter of 90 mm. The length-to-height ratio must not exceed 3. The height must be 70 mm (\pm 2 mm).

6 Test procedure

6.1 General

The test procedure consists of three stages: dry storage, presaturation by capillary suction and the freeze-thaw cycles. The test begins after the curing period which takes place directly after the test specimens have been obtained.

The start of presaturation by capillary suction determines the point at which freezing is started. For concretes according to DIN EN 206-1/DIN 1045-2 presaturation begins when the compressive strength class is verified at 28 days. Where the compressive strength class is verified at 56 days presaturation can also begin at 56 days.⁷

The storage periods for test specimens produced in accordance with Sections 5.2.1 or 5.2.2 are specified in the following table:

⁷ Slow-hardening concretes achieve the same performance as rapid-hardening concretes at a greater age. This also applies to the freeze-thaw resistance and the freeze-thaw and de-icing agent resistance. Accordingly, the duration of storage in a damp atmosphere/water or in the climate chamber may be extended to 14 days or 6 weeks respectively for concretes whose compressive strength class is verified at 56 days in accordance with ZTV-W LB 215 or ZTV-W LB 219. For cements with a high proportion of granulated blast furnace slag (its use in concrete with a high freeze-thaw resistance being limited in accordance with DIN EN 206-1/DIN 1045-2) the positive effect of an extended curing period may be less pronounced as an increase in carbonation with a detrimental effect on the weathering characteristics cannot be ruled out if the concrete is stored in the climate chamber for a longer period of time.

Storage after production	Curing ⁸ (In mould and in water) in accordance with Section 5.2	Dry storage in the climate chamber in accor- dance with Section 0	Presaturation by capillary suction in accordance with 6.3
Start of presaturation by capillary suction	Storage period		
28th day	7 d	21 ± 1 d	7 d
56th day	14 d	42 ± 1 d	7 d

Table 6.1: Storage periods prior to the start of freezing

6.2 Dry storage

The test specimens are stored in a climate chamber as specified in Section 4 a) at (20 °C/65 % RH) for surface drying. The storage period according to Table 6.1 is 21 ± 1 days if the concrete is tested at an age of 28 days and 42 ± 1 days if it is tested at an age of 56 days. The test specimens are to be placed on their sides at least 50 mm apart so that the test surfaces are free of obstruction. The change in weight has to be recorded.

During storage in the climate chamber compliance with the permitted rate of evaporation specified in 4 a) must be checked at regular intervals.

6.3 Presaturation

6.3.1 Preparation and sealing of the test specimens

The lateral surfaces of the test specimens must be sealed. The specimens, particularly their lateral surfaces, must be clean and dry. Prior to and after sealing, the specimens are to be weighed with an accuracy of ± 0.1 g in order to determine the reference mass of the unsealed test specimens required to calculate the moisture uptake.

Prior to sealing, the lateral surfaces must be treated with an appropriate primer. One of the two methods described below is to be used to seal the lateral surfaces:

- a) Sealing by aluminium foil with butyl rubber (reference method): The aluminium foil with butyl rubber must be applied to the lateral surfaces with an overlap of 20 mm between three days prior to and immediately before the start of presaturation. The butyl tape must be applied in such a way that a durable connection is ensured.
- b) Sealing with epoxy resin (alternative method): A solvent-free epoxy resin is applied to the lateral surfaces between two to four days prior to the start of presaturation so that an adequate degree of hardening is ensured.

⁸ A period of storage in a damp atmosphere/water longer than that specified in Table 6.1 (e. g. up to the start of the test) may have a detrimental effect on the freeze-thaw and the freeze-thaw and de-icing agent resistance owing to the higher degree of water saturation of the pores at the start of freezing.

6.3.2 Presaturation with test liquid by capillary suction

After dry storage, the test specimens are placed in the test containers on the 5 mm high spacers with the test surface at the bottom. The test liquid is then poured into the containers up to a height of 10 ± 1 mm without wetting the tops of the specimens. The following test liquids are used:

- for the freeze-thaw test (CIF):
 demineralized water
- for the freeze-thaw and de-icing agent resistance test (CDF): 3 % NaCl solution

During capillary suction the test container must be closed with a lid and no condensation must drip from the lid onto the specimen.

Capillary suction take places for a period of 7 days at a temperature of (20 ± 2) °C. The level of liquid must be checked and adjusted at regular intervals during capillary suction, depending on the suction capacity of the material. The increase in weight of the test specimens is measured at regular intervals every two to three days.

6.4 Freeze-thaw testing

Freeze-thaw testing is a cyclic attack in which the specimens are subjected to a temperature cycle in accordance with Section 4 (Figure 4) in a temperature-controlled chest. Generally speaking, 28 freeze-thaw cycles are required for both the freeze-thaw resistance test and the freeze-thaw and de-icing agent resistance test.

Prior to the start of freeze-thaw cycling any loose particles and dirt adhering to the test surface of the specimens shall be removed by treatment in an ultrasonic bath as described in Section 7.2. Discard the material that is removed.

If the cyclic attack is interrupted during the freeze-thaw test (the test equipment breaks down, for example), the test specimens must be left in the test solution and protected against drying out. Long interruptions in particular can influence the test result and must be recorded in the test report and taken into account in the evaluation.

7 Measurements

7.1 Sequence of measurements with respect to scaling, moisture uptake and ultrasonic transit time

Measurements are carried out at the beginning of the freeze-thaw test (0 freeze-thaw cycles) and after every four to six freeze-thaw cycles. Depending on the agreed criterion, either 24 freeze-thaw cycles (quality test) or 28 freeze-thaw cycles (suitability test) are performed.

The measurements must be carried out at temperatures above 15 °C (shaded area in Figure 4).

The measurements must be carried out in the following order:

- 1. Surface scaling
- 2. Moisture uptake
- 3. Ultrasonic transit time (internal damage)

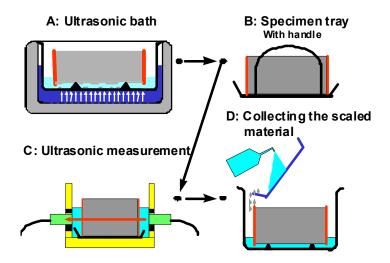


Figure 8: Sequence for the measurement of the ultrasonic transit time

After determining the surface scaling place the test specimen on the specimen tray specified in Section 4 k) to collect any additional scaled material during the subsequent measurements. The scaled material collected on the tray is returned to the test container and taken into account during the next measurement of surface scaling. If the measurement sequence is interrupted, the specimen must be returned to the test container with the test liquid to prevent it drying out.

7.2 Determination of surface scaling

7.2.1 Procedure

The test container is immersed in the contact liquid of an ultrasonic bath and subjected to ultrasonic cleaning for three minutes to remove any loosely adhering scaled material from the test surface whenever measurements are taken (Figure 6).

Filter the test liquid containing the scaled material. The paper filter is then dried at (110 ± 5) °C for 24 hours and left to cool at (20 ± 2) °C and (60 ± 10) % RH for at least one hour. The mass of the filter containing the dried scaled material μ_b is weighed with an accuracy of ± 0.01 g.

Prior to use, the empty paper filter must be dried as described above and its mass μ_f determined with the same degree of accuracy.

The mass of the scaled material μ_s is then: $\mu_s = \mu_b - \mu_f$.

7.2.2 Evaluation of scaling

Calculate the total quantity of scaled material m_n in relation to the test surface after the nth freeze-thaw cycle at each measurement and for each test specimen:

$$m_n = \frac{\sum \mu_s}{A}$$
(1)

 m_n is the total quantity of scaled material in relation to the test surface at each measurement, in g/m^2

- μ_s is the mass of the scaled material at each measurement, accurate to ± 0.01 g. The total is the sum of all measurements up to the *n*th cycle.
- A is the size of the test surface in m², calculated on the basis of the linear dimensions. These are the mean values of at least two measurements rounded to the nearest 0.1 mm.

Determine the mean value and the standard deviation. The result must be checked for outliers.

7.3 Measurement of the moisture uptake

7.3.1 Procedure

After removal of the scaled material the test specimens are placed vertically on an absorbent surface (laboratory towel) to allow water to run off the test surfaces. Carefully dry the lateral surfaces and the upper surface of the specimens with the laboratory towel. To avoid losing any of the scaled material the balance is zeroed with the specimen tray in place (Section 7.1) and the specimen is then weighed on the tray with an accuracy of \pm 0.1 g.

7.3.2 Evaluation of the moisture uptake

The moisture uptake of each test specimen Δw_n after the *n*th cycle is calculated as follows:

$$\Delta w_n = \frac{W_n - W_1 + \Sigma \mu s}{W_0} * 100 \tag{2}$$

- Δw_n is the moisture uptake of each test specimen at each measurement, in % by mass.
- μ_s is the mass of the scaled material, in g, at each measurement, measured to an accuracy of 0.01 g. The total is the sum of all measurements up to the *n*th cycle.
- w₀ is the reference mass of each test specimen without the mass of the sealing material after dry storage, in g.
- w₁ is the mass of each test specimen including the mass of the sealing material prior to the beginning of presaturation, in g.
- w_n is the mass of each test specimen at each measurement, in g.

Determine the mean value and the standard deviation of the increase in mass. The results must be checked for outliers.

7.4 Measurement of the ultrasonic transit time (internal damage)

7.4.1 Test arrangement

A container in accordance with Section 4 i) is used for the ultrasonic transit time measurement. The test liquid used serves as the coupling medium. The temperature of the coupling medium and the test specimen must be (20 ± 5) °C.

The ultrasonic transit time is measured with ultrasonic measuring equipment in accordance with Section 4 h). Place the transducers such that the axis of the ultrasonic transit path is parallel to the test surface and the distance between the transit path and the test surface is 35 mm. The container is filled with the test liquid up to 10 mm below the transducers but not above the top of the specimen. The upper surface of the test specimen must remain dry!

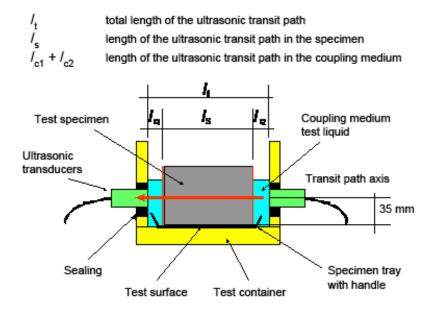


Figure 9: Test arrangement for measuring the ultrasonic transit time

7.4.2 Calibration

The test arrangement must be checked as follows before the start of each measurement cycle:

Reference measurements with a calibration specimen

The ultrasonic measurement equipment is checked by connecting the transducers directly to the calibration specimen at the measurement points by means of a suitable coupling medium (e. g. grease). The ultrasonic transit time measured in this way and the time stated on the calibration specimen must be identical. In the times differ, the ultrasonic transit time must be calibrated (to set it to the time stated on the calibration specimen).

Checking the transit time in the coupling medium

The calibration specimen is placed in the container as shown in Figure 9. The transducers are then moved so that the transit path in the water on each side is 5 mm (\pm 1 mm) (defined transducer spacing I_t).

The transit time in the water is the difference between the transit time for this arrangement and that measured for the calibration specimen.

Alternatively, a defined transducer spacing I_t can be specified by moving the transducers such that the actual measured transit time corresponds to the transit time of the calibration specimen + 10 µs (± 0.1 µs).

7.4.3 Procedure

The specimen is placed on the specimen tray and positioned in the test container for the measurement of the ultrasonic transit time as shown in Figure 9. The ultrasonic transit axes marked on the specimen during the first measurement must be used for all subsequent investigations. The transit time is measured for each specimen on two transit axes that are at right angles to each other.

In the case of rectangular specimens, the coupling points must be midway between the edges of the specimens. Before capillary suction begins, the length of the specimen which is to be crossed by ultrasonic waves is measured to an accuracy of ± 0.1 mm without taking the sealing material on either side of the specimen into account. The shortest ultrasonic transit time is measured to an accuracy of ± 0.1 µs after preliminary storage and after each predefined number of freeze-thaw cycles. The specimen must be moved slightly during the measurement and the shortest transit time determined. Care should be taken to ensure that no air bubbles adhere to the transducers or the sides of the specimens and that the lateral sealing is in full contact with the specimen. During the measurement sequence, the time during which the test surface is in contact with the air must be kept as brief as possible and any wetting of the top of the specimen must be avoided.

7.4.4 Evaluation of the internal damage

The ultrasonic transit time in the coupling medium t_c is calculated from the transit length in the coupling medium I_c and the velocity of the ultrasonic signal in the coupling medium v_c . The transit length in the coupling medium I_c is determined from the difference between the transducer spacing and the specimen dimensions I_s for each transit axis to an accuracy of ± 0.1 mm (Figure 9).

$$t_{c} = \frac{l_{c}}{v_{c}}$$
(3)

 t_c is the transit time in the coupling medium in μ s.

 I_c is the transit length in the coupling medium, calculated from $I_{c1} + I_{c2}$, in mm.

 v_c is the velocity of the ultrasonic signal in the coupling medium. It can be taken as 1490 m/s at (20 ± 5) °C.

The change in the transit velocity τ_n after n freeze-thaw cycles is calculated separately for each specimen and each transit axis using the following equation:⁹

$$\tau_{\rm n} = \frac{t_{\rm cs} - t_{\rm c}}{t_{\rm n} - t_{\rm c}} \tag{4}$$

⁹ The change in length may be neglected.

 τ_n is the relative transit velocity

t_{cs} is the total transit time after capillary suction (cs) in µs, prior to the first freeze-thaw cycle

 t_n is the total transit time after n freeze-thaw cycles in μ s.

It is useful to describe the inner damage by means of the relative dynamic modulus of elasticity Run from the ultrasonic transit time instead of by the transit velocity. In this test method, the relative dynamic modulus of elasticity after n freeze-thaw cycles is calculated by means of the following equation:¹⁰

$$R_{u,n} = \tau_n^2$$
(5)

The mean value calculated from the values of both transit axes expresses the relative dynamic modulus of elasticity of the test specimen. The relative dynamic modulus of elasticity may also be expressed as a percentage.

The mean value and the standard deviation of the relative dynamic modulus of elasticity of a test series must be determined. The result must be checked for outliers.

8 Evaluation of the freeze-thaw resistance after the CIF test

8.1 Relevant acceptance criterion - internal damage

The concrete is considered to be damaged if the relative dynamic modulus of elasticity $R_{u,n}$ is less than 0.75 or 75 %¹¹. The relevant criterion for evaluating the inner damage is the number of freeze-thaw cycles before the damage criterion is reached.

The number of freeze-thaw cycles can be determined by linear interpolation between two adjacent measuring points where the difference in the number of freeze-thaw cycles at each measuring point is less than 6.

The acceptance criterion shall be a number of cycles up to which the damage criterion must always be reached. Unless otherwise agreed in writing, the following acceptance criteria shall apply:

	Suitability test	Quality test and structural inspection
Mean value of test series	≥ 28 freeze-thaw cycles	≥ 24 freeze-thaw cycles

Table 8.1: Acceptance criteria for internal damage in the CIF test

¹⁰ Density, size and Poisson's ratio are neglected in this equation. This is not a serious limitation as the aim of the test is to identify damage and the ultrasonic transit time is the relevant parameter. The dynamic modulus of elasticity is just a parameter that is well-known to engineers.

¹¹ A relative dynamic modulus of elasticity of 75% ensures sufficient differentiation with regard to the modulus of elasticity of undamaged concrete (100 %) in accordance with the precision data. See also the precision data in 12.2.1

8.2 Additional acceptance criterion - scaling

Unless otherwise agreed in writing, the following acceptance criteria shall apply:

Table 8.2: Acceptance criteria for scaling in the CIF test

	Suitability test, quality test and structural inspection
Mean value of test series	≤ 1000 g/m² after 28 freeze-thaw cycles
95 % quantile of the test series	≤ 1750 g/m² after 28 freeze-thaw cycles

9 Evaluation of the freeze-thaw and de-icing agent resistance after the CDF test

9.1 Relevant acceptance criterion - scaling

Unless otherwise agreed in writing, the following acceptance criteria shall apply:

Table 9.1: Acceptance criteria for scaling in the CDF test

	Suitability test, quality test and structural inspection
Mean value of test series	≤ 1500 g/m² after 28 freeze-thaw cycles
95 % quantile of the test series	≤ 1800 g/m² after 28 freeze-thaw cycles

9.2 Additional acceptance criterion - internal damage

The concrete is considered to be damaged if the relative dynamic modulus of elasticity $R_{u,n}$ is less than 0.75 or 75%. The acceptance criterion shall be a number of cycles up to which the damage criterion must always be reached. Unless otherwise agreed in writing, the following acceptance criteria shall apply:

Table 9.2: Acceptance criteria for internal damage in the CDF test

	Suitability test	Quality test and structural inspection
Mean value of test series	≥ 28 freeze-thaw cycles	≥ 24 freeze-thaw cycles

10 Report

The test report must contain at least the following information:

- 1. A reference to this test specification.
- 2. Designation, origin, dimensions and weight of the test specimens on receipt of the samples/after preparation and after drying.
- 3. Sponsor and body responsible for preparation of the test specimens.
- 4. Reference to the type of test, e. g. suitability or quality test.
- 5. Information on the concrete composition and constituents including product designations.
- 6. Parameters of fresh concrete: bulk density, compaction time and compacting factor, air content.
- 7. Duration of storage in water and dry storage.
- 8. Composition of the test liquid.
- 9. Number of freeze-thaw cycles performed.
- 10. Change in the relative dynamic modulus of elasticity from the ultrasonic transit time for each test specimen and the mean value and standard deviation in %, rounded to the nearest 1 %, as a function of the number of freeze-thaw cycles, the intermediate measurements and the final measurement.
- 11. Mass of the scaled material for each test specimen, the mean value and the standard deviation in g/m^2 , rounded to the nearest 1 g/m^2 , as a function of the number of freeze-thaw cycles carried out, the intermediate measurements and the final measurement.
- 12. Mass of the solution absorbed during capillary suction and during the freeze-thaw resistance test (frost suction) for each test specimen, the mean value and the standard deviation in percentage by mass, rounded to the nearest 0.01 % by mass, as a function of the number of freeze-thaw cycles carried out, the intermediate measurements and the final measurement.
- 13. Visual assessment (cracks, scaling of aggregate particles) before the start and at least at the end of the test. The test report shall include a photograph of the test surface before and after the test at least for one representative test specimen by way of illustration.
- 14. Any deviation from the test method described above.
- 15. Assessment of the freeze-thaw resistance and the freeze-thaw and de-icing agent resistance in accordance with the acceptance criteria.

11 Requirements for laboratories and reference samples

The test laboratory performing the suitability and quality tests must have sufficient experience with the test method and must keep at least the following documents:

- 1. Test protocol and test report
- 2. Graphs showing the temperature development during the test period

In addition, the reference samples must either be stored as agreed or sent to the sponsor.

12 Precision data

12.1 General

There are two ways of stating the precision: repeatability and reproducibility. The precision of the CIF and CDF tests was determined for concretes in accordance with Section 5.2.1 on the basis of ISO 5725.

12.2 Precision of the CIF test for concrete mixes

12.2.1 Measurement of the internal damage - ultrasonic transit time

The precision data for the relative dynamic modulus of elasticity (RDM) is shown in Table 12.1. The data applies to laboratory concrete tested in accordance with Section 7.4, where s_r and s_R are the standard deviations of the repeatability and reproducibility respectively and can be calculated from the functional correlation with the relative dynamic modulus of elasticity $R_{u,n}$ using the equations in the fifth line.

Relative dynamic modulus of elasticity	Repeatability s _r	Reproducibility s _R
(RDM)	Standard	deviation
For RDM = 100 %	0.7 %	0.9 %
For RDM = 75 %	5.9 %	7.6 %
Equation*	s _r = 0.2046 R _{u,n} + 0.2122	s _R = 0.2656 R _{u,n} + 0.2750
* Proven field of application: $R_{u,n}$ = 0.70 to 1.0 with R^2 = 0.85 for s_r and R^2 = 0.73 for s_R . These figures apply to laboratory concrete produced as specified in Section 5.2.1		

 Table 12.1: Precision data for the measurement of the internal damage
 - ultrasonic transit time in the CIF test

NOTE: The precision data and the equations given in Table 12.1 are based on the results of the RILEM Round Robin Test of TC IDC, conducted with 9 institutes and three different concrete series.

12.2.2 Moisture uptake

The precision data for the moisture uptake is shown in Table 12.2. The data applies to laboratory concrete tested in accordance with Section 7.3, where s_r and s_R are the standard deviations of the repeatability and reproducibility respectively and can be calculated from the functional correlation with the moisture uptake Δw_n during the frost resistance test using the equations in the sixth line.

Table 12 2 [.]	Precision data for the	measurement of the moisture uptake
		measurement of the moisture uplace

Moisture uptake	Repeatability s _r	Reproducibility s _R
	Standard deviation	
Up to 0.5 % by mass	0.014 % by mass	0.029 % by mass
0.5 to 1.5 % by mass	0.027 % by mass	0.058 % by mass
> 1.5 % by mass	0.054 % by mass	0.115 % by mass
Equation*	$s_r = 0.0265 \Delta w_n + 0.0005$	$s_R = 0.0569 \Delta w_n + 0.0008$
* Proven field of application: $\Delta W_n = 0$ to 2.5 with R ² = 0.30 for s _r and R ² = 0.30 for s _R . These figures apply to laboratory concrete produced as specified in Section 5.2.1.		

NOTE: The precision data and the equations given in Table 12.2 are based on the results of the RILEM Round Robin Test of TC IDC, conducted with 7 institutes and three different concrete series.

12.2.3 Scaling

The precision data for scaling is shown in Table 12.3. The data applies to laboratory concrete tested in accordance with Section 7.2. The precision data for surface scaling obtained in round robin tests solely with freeze-thaw attack is currently only available for the range of 0 to 500 g/m².

Table 12.3: Precision data for the measurement of scaling in the CIF test

Scaling	Repeatability s _r	Reproducibility s _R
	Standard deviation	
0 to 500 g/m ²	120 g/m²	160 g/m²
	(v = 24 %)	(v = 32 %)

NOTE: The precision data and the equations are based on the results of the RILEM Round Robin Test of TC IDC, conducted with 9 institutes and three different concrete series.

12.3 Precision of the CDF test for concrete mixes

12.3.1Scaling

The precision data for scaling is shown in Table 12.4. The data applies to laboratory concrete tested in accordance with Section 7.2. The precision data can be calculated with Equation (6) and the parameters given in Table 12.5 for the acceptance criterion $m_0 = 1,500 \text{ g/m}^2$.

Table 12.4:	Precision data	for the measurement of	of scaling in the CDF test
-------------	----------------	------------------------	----------------------------

Scaling	Repeatability s _r	Reproducibility s _R
Ocaning	Standard deviation	
1,500 g/m²	156 g/m² (v = 10.4 %)	262 g/m² (v = 17.5 %)

The coefficient of variation v depends on the mean value of scaling m for the acceptance criterion m₀ = 1,500 g/m².

$$v = v_0 \cdot \left(\frac{m}{m_0}\right)^d$$

(6)

v coefficient of variation

m mean value of scaling

m₀ acceptance criterion (resistance level)

The parameters v_0 and d for the repeatability and reproducibility respectively are shown in Table 12.5 as an exponential relationship of the mean value of scaling m.

Table 12.5:	Parameters	v_0 and d for scaling in the	CDF test according to /1/
-------------	------------	--------------------------------	---------------------------

	Repeatability	Reproducibility
d	-0.33	-0.29
v ₀	10.4% (v _r)	17.5% (v _R)