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TC 117-FDC: FREEZE-THAW AND DEICING RESISTANCE OF CONCRETE

CDF Test – Test method for the freeze-thaw resistance of concrete - tests with sodium chloride solution (CDF)

Recommendation

Prepared by M. J. Setzer, G. Fagerlund and D. J. Janssen

FOREWORD

During RILEM TC 117-FDC meetings, the drafts of the three procedures recommended by TC 117 and published in Materials and Structures *(Vol. 28, No. 177) were discussed extensively. As already stated in the draft edition, TC 117-FDC will not issue a final recommendation concerning any procedure for which the precision following ISO 5725 is not given. Before the meeting in Sapporo (July 31-August 1, 1995), precision data were published only for the CDF test; this situation has not changed since then. Therefore, RILEM TC 117-FDC unanimously passed the following text proposed by Prof. Janssen:*

"TC 117 directs RILEM to recommend the CDF test for the determination of the scaling resistance of concrete. However, the committee cannot decide whether the draft procedures for the CF, Slab or Cube tests should be recommended until the precision data for these tests have been made available and the committee has had an opportunity to examine these data."

A RILEM recommendation should be restricted to the description and evaluation of the test procedure itself. The regulations should have a substantial scientific basis. Since an acceptance criterion can be modified by reasons which are valid only under certain conditions and economic considerations, a RILEM recommendation could contain at most guideline values.

Due to these considerations, the drafts for the CDF test had to be modified (without CF test). However, the necessary modifications were not so important that they required approval by the complete committee. Therefore, TC 117 established an editorial group responsible for these modifications, which consisted of Prof. Fagerlund, Prof. Janssen and Prof. Setzer, the Chairman of the Committee. This work of the editorial group has now been completed.

As Chairman of RILEM Technical Committee TC 117-FDC, it is an honour for me to thank all the members, and especially the editorial committee, for their very serious and engaged work, which led to a recommendation having sufficient precision for the freeze-thaw and deicing salt resistance of concrete.

M. J. SETZER – July 1996

1. INTRODUCTION

Adequate resistance against freeze-thaw attack with deicing chemicals should be tested.[1] This testing requires both a test procedure with demonstrated precision, *i.e.* repeatability and reproducibility[2], as well as an acceptable correlation of test results to the performance of concrete in practice.

As in other fields such as strength testing, a test procedure for freeze-thaw and deicing salt resistance cannot cover all the variations possible under exposure conditions in practice. This would increase the expenditure. At the same time the scatter would increase and the results would become non-interpretable. The `CDF´ test has been developed to attain a high degree of precision with minimum expenditure on equipment and labour. CDF means Capillary suction of Deicing solution and Freeze-thaw test.[3]

(1) Freeze-thaw and deicing salt resistance has been achieved to date by rules, based on long term practical experience, that prescribe the water/cement ratio, the types of constituent and the amount of artificially entrained air voids. However, new constituents or untried compositions are required for certain practical demands. There are concrete applications where the practical design rules cannot be adopted, such as in dry concrete production of blocks, kerbs and flags. In these cases a reliable test procedure of the freeze-thaw and of the freeze-thaw and deicing salt resistance is necessary.

(2) Any test procedure should be made as precise and reliable as possible by a sufficiently exact definition of the relevant parameters. Its results should be related to practical experience, such as the above-mentioned rules of mix design. The precision should be assessed following ISO 5725. By this methodology, the freeze-thaw or the freeze-thaw and deicing salt resistance, even under unfavourable practical conditions, should be predictable with sufficient reliability.

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2. SCOPE

This procedure allows us to measure the amount of scaling per unit surface area due to a number of well-defined freezing and thawing cycles in the presence of deicing salt - as a rule sodium chloride solution (CDF), and leads to an estimate of the freeze-thaw and deicing salt resistance of the concrete tested.

It can be used to test an exposed surface of concrete structures or of precast concrete products, and to test the constituents of concrete as well as the concrete mix.

3. STANDARDS

prEN-ISO 2736/2-1993: Testing hardened concrete-test specimens. Part 2: Making and curing of test specimens.

ISO 5725/1 to 6-1990: Accuracy (trueness and precision) of measurement methods and results.

4. DEFINITIONS

1. The *freeze-thaw and deicing agent resistance* is the resistance against alternating freezing and thawing in the presence of deicing agent solution as test solution as test liquid.

2. The *test solution* is the solution of 3 mass % sodium chloride and 97 mass % demineralized water.

3. *Scaling* is the loss of material at the surface of concrete due to freeze-thaw or freeze-thaw and deicing attack.

4. The *reference point* is the physical measuring point at which the temperature cycle is controlled.

5. The *reference temperature* is the temperature measured at the reference point.

6. The *test surface* is the surface of the test specimens on which the freeze-thaw and deicing agent resistance is established in this test specification.

5. EQUIPMENT

1. *Climate chamber* with a temperature of 20 ± 2°C and a relative humidity of 65 ± 5 %.

2. Evaporation is measured using an *evaporation bowl* with a depth of approximately 40 mm and a cross-sectional area of 225 ± 25 cm^2.

3. *Lateral sealing* is by epoxy resin or aluminium foil with butyl rubber. Both must be durable at temperatures of - 20°C and resistant against the attack of the deicing solution. They cannot be brittle at the minimum temperature reached.

4. *Test liquid*, consisting of deicing agent solution, *e.g.* 97% by weight of demineralized or distilled water and 3% by weight of NaCl.

5. *Test containers* (Figs. 1 and 2). The specimens are stored in stainless steel containers during the freeze-thaw cycles. The size of the test container should be 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 20 ± 10 mm.[4,5] On the container bottom a 10 ± 0.1 mm high spacer is arranged to support the specimen and to guarantee a defined thickness of the liquid layer between the test surface and the container.

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

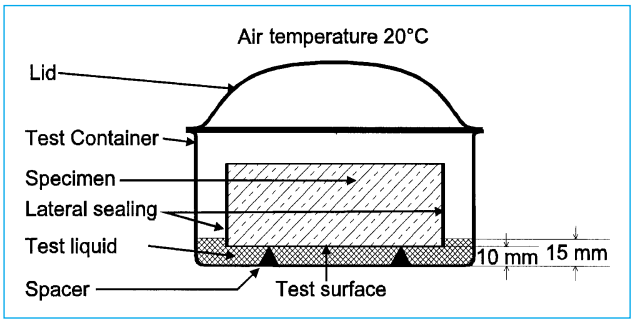

Fig. 1 – Capillary suction.

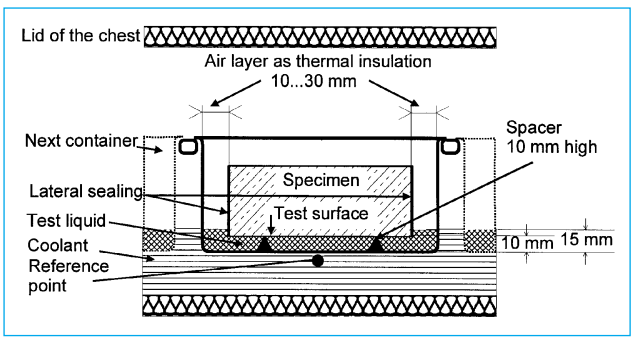

Fig. 2 – Test container with specimen in the liquid cooling bath.

6. *Temperature controlled chest* (Fig. 3). For temperature control during the freeze-thaw cycle and to guarantee uniaxial heat flux, 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 must be capable of maintaining the temperature regime at the reference point within ± 0.5 K with full loading by the test containers with the test

(3) Several different procedures have been developed. However, there is still a lack of precision, i.e. *reproducibility and repeatability. The CDF test has been developed by analysing and optimising the existing procedures and by adopting basic research work on adequate restriction of test parameters.*

(4) The air layer between the vertical side of the specimen and the test container acts as thermal insulation.

(5) The stainless steel containers are matched in several modular sizes so that the same boundary conditions can be met for each specimen form.

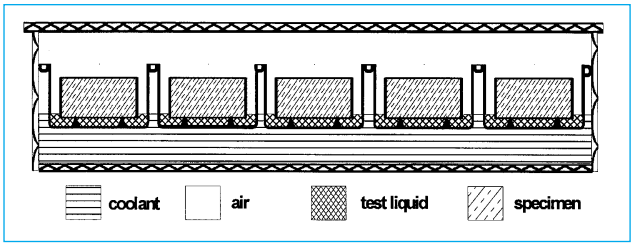

Fig. 3 – Temperature controlled chest.

specimens. The temperature in the bath must be uniform, within a limit of ± 0.5 K at least at the minimum temperature and of ± 1 K at other temperatures. A constant time shift between the test containers is acceptable.

The chest must be 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 mm. During the freeze-thaw cycles, the upper space of the chest containing the specimens must be separated from the cooling bath either by the test containers or by other lids.[6]

The reference temperature is measured in the cooling bath liquid below the bottom of the test container. The gauge is fixed to the container in such a way that a precise distance of 1 mm between the bottom and the measuring point is ensured. A standardised platinum resistance temperature gauge with an electrical resistance of 100 W at 0°C (PT100) is recommended for measurement. For calibration, the minimum temperature of - 20°C should be used. For monitoring and controlling the reference temperature, a test container in the centre of the bath is used.[7]

7. *Ultrasonic bath* (Fig. 4). The size of the ultrasonic bath must be sufficiently large. The test containers must fit in the ultrasonic bath without mechanical contact. Additionally, a minimum distance between the test container and the bottom of the bath of 15 mm must be ensured. The bath should provide the following power data: ERS power 250 W; HF peak power 450 W under double half-wave operation; frequency 35 kHz.

8. *Paper filter* for collecting scaled material.

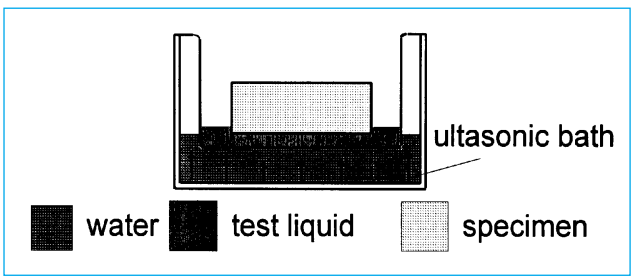

Fig. 4 – Ultrasonic bath.

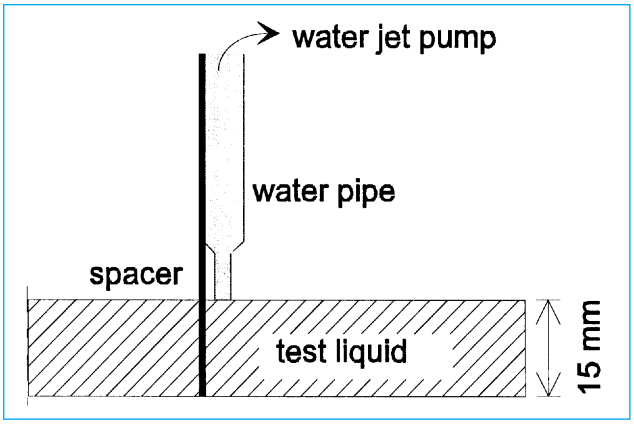

Fig. 5 – Suction device to remove the liquid exceeding the level of 15 mm in the test containers.

9. *Unit for adjusting liquid level*, *i.e.* by a suction device (Fig. 5). The suction device may consist of a capillary tube with a spacer of 15 ±1 mm, that is, connected with a water jet pump to suck up the excessive liquid in the test containers.

10. *Drying cabinet* for a temperature of (110 ± 5)°C.

11. *Balance* with an accuracy within ± 0.01 g.

12. *Vernier callipers*, with an accuracy within ± 0.1 mm.

6. TEST SPECIMENS

6.1 Required total test surface area and number of specimens

For one series, a number of ≥ 5 specimens[8] with a total test surface area of ≥ 0.08 m^2 is recommended.

6.2 Making test specimens for testing concrete mixes or concrete constituents in a mix (reference specimens)

For testing concrete mixes or concrete constituents in a mix, the test specimens are cast and compacted on a vibrating table in 150 mm cube moulds, according to prEN-ISO 2736/2. Centred in the mould is a vertical teflon disk which separates the mould into two halves. The teflon plate must not be treated with any demoulding agent. The concrete surface at the teflon plate is the test surface. For a larger aggregate size, the teflon disk can be arranged at one side.

After 24 ± 2 hours of curing, the specimens are removed from the mould and stored for 6 days (until the age of 7 days) in tap water at 20 ± 2 °C. (If strength

(6) When conducting the test in a cryogenic bath, no cover of the test containers is required, since the chest lid will provide a sufficient evaporation barrier, while the container wall will serve as a cold trap. The geometry of liquid coolant bath ensures a uniaxial thermal attack. No lateral thermal isolation is required, because the thermal conductivity of the ambient air is sufficiently low and because the liquid coolant is capable of adding and removing larger amounts of heat.

(7) Basically, the freeze-thaw test can also be performed in an air-cooled chest. However, a uniaxial heat flux must be ensured as well as a temperature cycle with sufficient precision to establish the same scaling at the test surface. The temperature regime cannot be controlled as precisely as in the liquid cooling bath and must be adjusted.

(8) A minimum number of 5 specimens is recommended for statistical evaluation and for finding possible stragglers.

development of the specimens is low, the curing in the mould can be increased. The storage in tap water is then decreased by the same amount.) This treatment precedes the dry storage.

6.3 Test specimens for testing the surface of concrete structures

The test surface should correspond to the surface of the real structure exposed to weathering. The test surface should be plane and can be of any kind - cast, screeded, sawed or of different texture.[9]

The test surface should be free from any demoulding agent. If a demoulding agent cannot be avoided, this can affect the scaling during the first freeze-thaw cycles and must be taken into account in the assessment.

The height of the test specimens should be comparable to the height of the specimens described in section 6.2. Specimen heights between 50 mm and 150 mm are acceptable.

6.4 Test specimens for testing precast concrete elements

Small precast concrete elements, such as concrete blocks and flags, can be tested directly, independent from the outer shape. If the dimension exceeds 200 mm, the element should be cut. The test surface is the weathered surface of the element and should be plane.

The test surface should be free from any demoulding agent. If a demoulding agent cannot be avoided, this can affect the scaling during the first freeze-thaw cycles and must be taken into account in the assessment.

The height of the test specimens should be comparable to the height of the specimens described in section 6.2. Specimen heights between 50 mm and 150 mm are acceptable.

7. TEST PROCEDURE

The test procedure consists of three steps: the dry storage, the presaturation by capillary suction and the freeze-thaw cycles. The test procedure starts immediately after the curing period. For test specimens made following Section 6.2, this is at the age of 7 days.

7.1 Dry storage

The concrete specimens are stored in the climate chamber (20°C, 65% RH) for surface drying for 21 days. Monitoring of the weight is recommended.

In the climate chamber the evaporation from a free water surface shall be 45 ± 15 g/m^{2}h. The evaporation is measured by weight loss of a water-filled evaporation bowl with a depth of approximately 40 mm and a cross-sectional area of 225 ± 25 cm^2. The bowl shall be filled up to 10 ± 1 mm from the brim. Monitoring of the weight is recommended.[10]

7.2 Preparation of specimens

Between 7 and 2 days before presaturation, the specimens are sealed on their lateral surfaces either with aluminium foil with butyl rubber or with a solvent-free epoxy resin.[11] The specimens must be clean and dry. Before sealing the lateral surfaces, it is recommended to treat them with an adequate primer.

7.2.1 Sealing with aluminium foil with butyl rubber[11]

A piece of aluminium foil with butyl rubber is glued tightly on the lateral surfaces with an overlap of 20 mm.

7.2.2 Sealing with epoxy resin

A solvent-free epoxy resin is laid on the lateral surfaces, whereas the bottom of the specimens and the test surface must be kept free.

7.3 Presaturation of test liquid by capillary suction

Following dry storage, the specimens are placed in the test containers on the 10 mm high spacers with the test surface underneath. Subsequently, the test liquid is filled into the container to a height of 15 ± 1 mm without wetting the specimen's top. (This can be achieved by filling to approximately 17 mm and removing the surplus solution by a capillary tube combined with a spacer of 15 mm and connected to a water jet pump.)

During the capillary suction, the test container must be closed with a cover that should have an incline to prevent any possible condensation water from dripping onto the specimen's top surface.

The capillary suction period is 7 days at a temperature of 20 ± 2°C. During capillary suction, the liquid level should be checked and adjusted as described above, at regular intervals depending on the suction capacity of the material.

The weight gain of the specimens should be measured.

(9) A test specimen not covered by the curing procedure as described in § 6.2 should be treated equivalently.

(10) After the drying period, all freezable water, at least near the test surface, should have evaporated by reaching a water content equivalent to an equilibrium below 70% RH.

(11) When conducting frost/deicing salt tests, this prevents falsified results due to possible lateral scaling.

7.4 Cleaning of test surface before starting the freeze-thaw cycles

Before starting the freeze-thaw cycles, loosely adhering particles and dirt should be removed from the test surfaces of the specimens by treatment in the ultrasonic bath, as described in section 7.6. The material removed is discarded.

7.5 Freeze-thaw testing

7.5.1 Temperature cycle

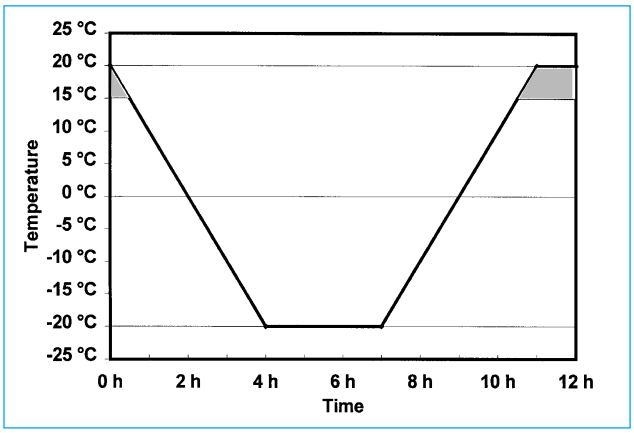

Fig. 6 – Control temperature cycle.

A 12 h freeze-thaw cycle is applied (Fig. 6). Starting at + 20 °C, the temperature is lowered in 4 h with a constant cooling rate of 10 K/h. It is kept constant for 3 h at - 20 °C and increased in 4 h with a constant heating rate of 10 K/h. It is kept constant for 1 h at + 20 °C.[12] The temperature cycle is monitored at the reference point. The deviation of temperature measured at the reference point should not be more than ± 0.5 K at least at the minimum temperature and more than ± 1 K at other temperatures. A constant time shift between the test containers is acceptable.

7.6 Determination of surface scaling

The surface scaling can be measured while the temperature is above 15 °C (shaded area in Fig. 6).

To remove loosely adhering scaled material from the test surface, the test container is dipped into the contact liquid of an ultrasonic bath and subjected to ultrasonic cleaning for 3 minutes.

The solution comprising the scaled material is filtered. The paper filter is subsequently dried at 110 ± 5 °C for 24 h and cooled for 1 h (± 5 min) at 20 ± 2 °C and 60 ± 5 RH. The mass of the filter containing the dried scaled material μb is weighed to 0.01 g precision. The mass of the empty filter μ_f is determined before with the same accuracy.

The mass of the scaled material μs is then: $\mu s = \mu_b - \mu_f$.

The amount of scaling should be determined after 14 and always after 28 freeze-thaw cycles. Additional measurements, *e.g.* after 4 or 6 cycles, are recommended.

8. EXPRESSION OF TEST RESULTS

The following is to be calculated for each measuring occasion and each specimen: the total amount of scaled material related to the test surface after the *n*th cycle m_n:

$$m_n = \frac{\Sigma \mu_s}{A} * 10^6 \text{ g/m}^2 \qquad (1)$$

μ_s is the mass of scaled material of the measurement after n cycles (g) with an accuracy of 0.01 g. The sum is taken over all measurements until the *n*th cycle.

A is the area of the test surface. It is calculated on the basis of the linear dimensions. They are taken 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 should be evaluated. The result should be checked for stragglers.

The mean value and the individual values for each specimen after 28 cycles are used for evaluating the scaling resistance.

9. ASSESSMENT OF THE CDF TEST

The freeze-thaw and deicing salt resistance of the CDF test is assessed after 28 freeze-thaw cycles.

The precision for freeze-thaw and deicing salt resistance (3 % sodium chloride solution) can be given in accordance to ISO 5725.[13]

(12) The cycle corresponds principally to a recommendation agreed upon at the meeting of RILEM TC 117, Freeze-thaw and Deicing Resistance of Concrete, in May 1990. The duration of minimum temperature is maintained, as well as the maximum and minimum temperatures and the constancy of cooling and heating rates. However, the rate is increased and the duration at the maximum temperature decreased to reach a 12 h cycle. It is proved that the difference in scaling is small (Setzer, M.J. and Hartmann, V., 'Verbesserung der Frost-Tausalz-Widerstands-Prüfung - Improved frost/deicing salt resistance testing', Betonwerk und Fertigteiltechnik, Vol. **57**, *Heft 9 (1991) 73-82).*

(13) Further details about correlation with behaviour in practice, the CDF resistance limit, and the precision data are provided in: Setzer, M. J. and Auberg, R., 'Freeze Thaw and Deicing Salt Resistance of Concrete Testing by CDF Method; CDF Resistance Limit and Evaluation of Precision', Mater. Struct. **28** *(1995) 16-31.*
The practical behaviour is dealt with in: Hartmann, V., 'Optimierung und Kalibrierung der Frost-Tausalz-Prüfung von Beton - CDF Test', PhD Thesis, University of Essen, 1993.
The statistical analysis is described in more detail in: Auberg, R., PhD Thesis, University of Essen 1996.
The basic ideas were described first in: Setzer, M.J., 'Prüfung des Frost-Tausalz-Widerstands von Betonwaren'. Essen, Universität Gesamthochschule-Essen, Forschungsberichte aus dem Fachbereich Bauwesen Heft 49 (1990).

9.1 Precision of the CDF test for plastic concrete mixes

Three types of precision are distinguished: repeatability, reproducibility and between laboratory scattering. The precision of the CDF test procedure with a 3% sodium chloride salt solution was evaluated according to ISO 5725 for plastic concrete mixes as described in section 6.2. The coefficient of variation v depends on the mean scaling m related to the resistance level m_0 = 1500 g/m^2:

$$v = v_0 \cdot \left(\frac{m}{m_0} \right)^d \qquad (2)$$

The parameters in equation 2 for reproducibility, repeatability and between laboratory variation as an exponential relationship of mean scaling m are as tabulated below:

	Repeatability	Between laboratory	Reproducibility
d	-0.33	-0.26	-0.29
v_0	10.4%	14.0%	17.5%

10. REPORT

The test report shall contain at least the following information:

a) A reference to this description.

b) Size, origin and marking of the specimens.

c) In the case of the testing of concrete mixes or constituents, the composition of the concrete.

d) The composition of the test liquid.

e) The amount of scaled material for each specimen, as well as the mean value and the standard deviation in g/m2 rounded to the nearest 1 g/m^2, at least after 14 and 28 cycles.

f) The mass of solution sucked up during the capillary suction period for each specimen, as well as the mean value and the standard deviation.

g) Visual assessment (cracks, scaling from aggregate particles) before the start and at least after 14 and 28 cycles.

h) Any deviations from the standard test procedure.