Freeze-thaw testing
CSLA Project - Task 1. Literature Review

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Summary
In this report multiple freezing and thawing and scaling test methods as well as indirect test methods are described. Comparison between different test methods is difficult. The selection of a suitable test method depends on the objective of research or whether for quality control.

There is no "right" freezing and thawing resistance test method. All of them have been made for their own purpose and use in any other purpose has to be done in great care. It must be kept in mind that the criteria for durability are also greatly dependent on environmental loading and test methods used.

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Preface

This report is an integral part of the research performed with the CSLA project – Concrete Service Life Assessment: Modelling frost attack degradation in the presence of chlorides. The main objective of CSLA project is to investigate the influence of freeze-thaw deterioration of concrete on the transport characteristics of chloride ion in concrete.

The report presents a literature review on testing methods on freezing and thawing performance of concrete.

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Abstract

In this report multiple freezing and thawing and scaling test methods as well as indirect test methods are described. It can be noticed that many tests are actually measuring different properties of concrete. Tests have also different parameters even when they are meant for measuring the same property. These make comparison between different test methods very difficult. The selection of a suitable test method depends on the target of research or quality control.

There is no "right" freezing and thawing resistance test method. All of them have been made for their own purpose and use in any other purpose has to be done in great care. It must be kept in mind that the criteria for durability is also greatly dependent on environmental loading and test methods used.

As Lindmark (1998) stated, “a good test method should classify different materials in the same way that exposure in the field would. The test method must not allow concretes of inferior quality to be used, but at the same time must not be too restrictive.”
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1. Introduction

Freeze thaw testing methods can be divided in direct and indirect methods. In direct methods samples are subjected to alternating freezing and thawing. Salt solution may used in testing if salt frost durability is of interest. The idea is to simulate real freezing and thawing environmental load by accelerated testing. Because all of these tests have to be accelerated the real freezing and thawing environmental load and possible deterioration will not happen. This risk is present in all of these direct test methods.

In indirect methods the idea is to measure property that is affecting frost resistance or deterioration mechanism. Risk in these indirect methods is that only one property is measured at a time and there may be some other properties which may have even higher effect on freeze thaw behaviour. If there is a valid theoretical background behind the indirect methods they are normally faster and many times have acceptable accuracy.

One additional factor that has to be taken into account is the ageing of concrete. Ageing and interaction with environment change all properties of concrete. Also freezing and thawing deterioration properties are changing. At early age this change is fast as hydration is proceeding. Environmental interactions which are not necessarily deteriorative (sometimes on the contrary) also change the chemistry, porosity and mechanical properties of concrete. These all have an effect on freezing and thawing behaviour of concrete. In testing methods traditionally concrete age is quite a low and environmental interaction is omitted.

When choosing suitable testing methods it also has to be taken into account what purpose the test methods have been developed for and for which purpose they would be used. Many of the test methods have been developed for quality control purposes. In quality control tests the acceptability of concrete and uniform quality of it from batch to batch is checked. In research where for instance deterioration mechanisms are of interest these test methods might not be suitable.

Because of these many factors choosing a suitable test method has to be based on valid theoretical background and good understanding of the test methods. This report tries to increase the understanding on most commonly used test methods.
2. Freeze-thaw test methodologies

Traditional freeze-thaw test methods for assessing the frost resistance of cement-based materials are various and well described in the literature. Volume change, mass change or change in dynamic modulus of elasticity is often used as a descriptor of durability. These characteristics are of significant practical value but they do not provide any clear direct evidence for a particular mechanism of freeze-thaw action. No single laboratory test method can fully reproduce the conditions in the field in all individual cases. A good enough testing method should at least correlate to the practical situation and give consistent results.

The application of limiting values will require the establishment of the correlation between laboratory results and field experience. This correlation must be established in accordance with local conditions. [CEN/TS 12390-9, CEN/TR 15177, Rønning 2001, Rønning 2010]

2.1 Freeze-thaw resistance – internal structural damage

2.1.1 Freezing and thawing

In the European standard [CEN/TR 15177 (2006)] there are three different methods for the estimation of the freeze-thaw resistance of concrete with regard to internal structural damage. These methods produce relatively consistent results. No single test method is established as a reference test method. The methods include:

- Slab-Test in CEN/TR 15177 (2006) which is based on the earlier Swedish standard SS 13 72 44 (so called “Borås-method”),
- CIF-method in CEN/TR 15177 and
- Beam test in CEN/TR 15177.

Table 1 presents an overview of the test methods according to CEN/TR 15177.

In North America the most commonly used testing standard for freeze-thaw resistance is


This standard includes two methods:

- Rapid Freezing and Thawing in Water (Procedure A) and
- Rapid Freezing in Air and Thawing in Water (Procedure B).

In the ASTM C666 method, the freezing rate is very high. There has been criticism on the ASTM C666 method because it does not represent well natural freeze-thaw exposure. Anyway this method is widely known and frequently used. In Europe, the RILEM TC 117-FDC work group has decided that the ASTM C666 method will not be accepted as a CEN-testing method.

In Finland it was still in 2012 possible to use a national beam method for freeze-thaw resistance assessment:

- SFS 5447 (1988) (Freeze-thaw durability; beams 500×100×100 mm³).

This method essentially includes freezing in air (−20 °C) and thawing in water (<40 °C, end of thawing cycle temperature +20 °C). SFS 5447 is a very loosely defined method. For instance in standard there is no instructions for acceptance criterion or even acceptance methods.
### Table 1. Overview of the test methods (freeze-thaw, internal structural damage) according to CEN/TR 15177. [CEN/TR 15177]. (Figures from [Boos & Giergiczny 2010])

<table>
<thead>
<tr>
<th>Test parameters</th>
<th>Slab test</th>
<th>CIF-test</th>
<th>Beam test</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Storage</strong> (T = ca. 20 °C) after de-moulding (at 24 h), before freeze-thaw</td>
<td>Water: for 6 d</td>
<td>Water: for 6 d</td>
<td>Plastic film: for 6 d</td>
</tr>
<tr>
<td>  (at 24 h), before freeze-thaw</td>
<td>Climate: for 21 d</td>
<td>Climate: for 21 d</td>
<td>Water: for 21 d</td>
</tr>
<tr>
<td>  De-ionised water (3 mm) on the top surface: for 3 d</td>
<td>Freezing medium (5 mm) underneath: for 7 d</td>
<td>Freezing medium (5 mm) underneath: for 7 d</td>
<td></td>
</tr>
<tr>
<td><strong>Specimen (typical)</strong></td>
<td>With a sawn surface: 150x150x50 mm³</td>
<td>Splitting a 150 mm cube mould with a polytetrafluorethylene (PTFE) plate with no de-moulding agent (= test surface, downwards): 150x150x70 mm³</td>
<td>A cast beam: 400x100x100 mm³</td>
</tr>
<tr>
<td><strong>Number of specimens</strong></td>
<td>4 (from different cubes)</td>
<td>≥25 (total test area ≥0.08 m²)</td>
<td>≥5</td>
</tr>
<tr>
<td><strong>Age at freeze-thaw</strong></td>
<td>31 d</td>
<td>28 d</td>
<td>28 d</td>
</tr>
<tr>
<td><strong>Test direction/method</strong></td>
<td>One-way</td>
<td>One-way</td>
<td>All directions</td>
</tr>
<tr>
<td>  Test liquid (3 mm) on the top surface</td>
<td>Test liquid (5 mm) underneath</td>
<td>- Freezing in air, thawing submerged</td>
<td></td>
</tr>
<tr>
<td><strong>Freezing medium (test liquid)</strong></td>
<td>De-ionised water or 3% NaCl solution</td>
<td>De-ionized water or 3% NaCl solution</td>
<td>De-ionized water</td>
</tr>
<tr>
<td><strong>Duration of a cycle</strong></td>
<td>24 h</td>
<td>12 h</td>
<td>12 h</td>
</tr>
<tr>
<td><strong>Freeze-thaw cycle:</strong> T_mil/T_max</td>
<td>In the freezing medium (test liquid): (-20±2 °C)/(+20±4) °C</td>
<td>In the cooling bath liquid: (-20±0.5 °C)/(+20±1) °C</td>
<td>At the centre of the beams: (-20±2) °C/(+13±8) °C</td>
</tr>
<tr>
<td>  Chest temperature (with good air circulation) not below 27 °C</td>
<td>  - At the centre of the beams: (-20±2) °C/(+13±8) °C</td>
<td>  - Water bath: (+13±8) °C</td>
<td>  - Chest temperature not below -25 °C</td>
</tr>
<tr>
<td><strong>Cooling speed below +0 °C</strong></td>
<td>Ca. 1.7 °C/h</td>
<td>10 °C/h</td>
<td>Ca. 3.3 °C/h</td>
</tr>
<tr>
<td>  From +0 °C to -20 °C in 12 hours</td>
<td>From +0 °C to -20 °C in 2 hours</td>
<td>From +0 °C to -20 °C in 6 hours</td>
<td></td>
</tr>
<tr>
<td>  Test liquid</td>
<td>Cooling bath liquid</td>
<td>  Centre of the beams</td>
<td></td>
</tr>
<tr>
<td><strong>Duration/Number of freeze-thaw cycles</strong></td>
<td>56 d/56 cycles</td>
<td>28 d/56 cycles</td>
<td>28 d/56 cycles</td>
</tr>
<tr>
<td><strong>Additional information</strong></td>
<td>Measurements during the thawed phase when temperature is above 15 °C</td>
<td>Measurements during the thawed phase when temperature is above 15 °C</td>
<td>Every 7 d the beams are turned 180°</td>
</tr>
<tr>
<td>  - Back to the freezing chamber at the cycle phase (0±30) min.</td>
<td>  - Also to be placed in different positions (vertically) in the chest</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Measurement of resistance</strong></td>
<td>Dilation (reference procedure), RDM by UPTT or RDM by FF after 7, 14, 28, 42 and 56 freeze-thaw cycles</td>
<td>Dilation, RDM by UPTT (reference procedure) or RDM by FF after 7, 14, 28, 42 and 56 freeze-thaw cycles</td>
<td>RDM by UPTT or RDM by FF after 7, 14, 28, 42 and 56 freeze-thaw cycles</td>
</tr>
<tr>
<td><strong>Alternative applications (examples)</strong></td>
<td>Other curing conditions or concrete age at the start of the freeze-thaw cycling</td>
<td>Other curing conditions or concrete age at the start of the freeze-thaw cycling</td>
<td>Other curing conditions or concrete age at the start of the freeze-thaw cycling</td>
</tr>
<tr>
<td>  - Other specimen geometries (but h (thickness) must be 50 ±2 mm)</td>
<td>  - Other specimen geometries (but h (thickness) must be 70 ±5 mm, and total test surface area must be ≥0.08 m²)</td>
<td>  - Other specimen geometries (but d = (100±20) mm and h/d ≥ 2.0)</td>
<td></td>
</tr>
<tr>
<td>  - Top surfaces or surfaces cast against formwork (instead of sawn surfaces)</td>
<td>  - Two PTFE plates in the mould at two opposed vertical sides, forming the test surfaces</td>
<td>  - e.g., slices from cores</td>
<td>  - &gt;56 cycles</td>
</tr>
<tr>
<td>  - &gt;56 cycles</td>
<td>  - Other de-icing agent than 3% NaCl solution</td>
<td>  - Other de-icing agent than 3% NaCl solution</td>
<td>  - &gt;56 cycles</td>
</tr>
</tbody>
</table>

In [By 50 2012] this method (SFS5447) is an optional quality control method in addition to CEN/TR 15177 Slab test and air pore analysis (Spacing factor – see Chapter 3.1.2). Based on the demanded design service life and exposure class, the amount of freeze-thaw cycles is 100 or 300. The acceptance criterion is based on either RDM as measured by UPTT (≥75%) or relative flexural or splitting tensile strength (≥67%). [By 50, 2012].

In [By 50 2012] this method (SFS5447) is an optional quality control method in addition to CEN/TR 15177 Slab test and air pore analysis (Spacing factor – see Chapter 3.1.2). Based on the demanded design service life and exposure class, the amount of freeze-thaw cycles is 100 or 300. The acceptance criterion is based on either RDM as measured by UPTT (≥75%) or relative flexural or splitting tensile strength (≥67%). [By 50, 2012].
Table 1. (Continue, internal damage).

<table>
<thead>
<tr>
<th>Test report (main results)</th>
<th>Freezing medium</th>
<th>Mean relative values for freeze-thaw resistance (structural damage) after the specified number of freeze-thaw cycles, and the same values individually for each specimen</th>
<th>Freezing medium</th>
<th>Mean relative values for freeze-thaw resistance (structural damage) after the specified number of freeze-thaw cycles, and the same values individually for each specimen</th>
<th>Mean relative values for freeze-thaw resistance (RDM, structural damage) after specified number of freeze-thaw cycles, and the same values individually for each specimen</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<td>--------------------------------------------------------------------------------------------------------------------------</td>
<td></td>
<td>--------------------------------------------------------------------------------------------------------------------------</td>
<td>--------------------------------------------------------------------------------------------------------------------------</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Water uptake [%] (mean values and for each specimen)</td>
<td></td>
<td>Visual assessment (cracks, scaling from aggregate particles) before the start and after the specified number of cycles</td>
<td>Composition of the concrete</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Visual assessment (cracks, scaling from aggregate particles, leakage of freezing medium) before the start and after the specified number of cycles</td>
<td></td>
<td>Visual assessment (cracks, scaling from aggregate particles) before the start and after the specified number of cycles</td>
<td>Composition of the concrete</td>
</tr>
<tr>
<td>Additional results</td>
<td></td>
<td>Water uptake [%] (mean values and for each specimen)</td>
<td></td>
<td>Composition of the concrete</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Visual assessment (cracks, scaling from aggregate particles) before the start and after the specified number of cycles</td>
<td></td>
<td>Composition of the concrete</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Composition of the concrete</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1) Water = In water; Plastic film = Wrapped in plastic film; Climate = at (ca. RH 65% and T = 20 °C).
2) RDM = Relative Dynamic Modulus of elasticity; UPTT = Ultrasonic Pulse Transit Time; FF = Fundamental transverse Frequency.

2.1.2 Dilation

An out-of-date Finnish freeze-thaw method is
- SFS 5448 (Freezing dilation).

This method is no longer in use in Finland. [SFS 5448 1988].

There is also an out-of-date ASTM dilation method
- ASTM C671-94.

This method was withdrawn in year 2003 with no replacement. It covered the determination of the test period of frost immunity of concrete specimens as measured by the length of time of water immersion required to produce critical dilation when subjected to a prescribed slow-freezing procedure. [ASTM C671-94]

The testing methods [SFS 5448] and [ASTM C671-94] are based on the fact that there will be a permanent dilation when concrete freezes with a high enough water content. Microcracks will be created, and this can be detected based on macroscopic specimen volume/length increase. A reason why SFS 5448 was withdrawn was the high variation in testing results. One reason for this was that it was not possible to measure reliably the dilation in low freezing temperatures by the measurement devices available. A further reason was that SFS 5448 includes pre-drying at 105 °C before water saturation. Severe drying is known to affect the concrete pore structure. Instead, the principle in ASTM C671 is that pre-treatment is as close to natural circumstances as possible. For SFS 5448 it was also found to be difficult to get an even water saturation in high strength concrete specimens after drying. In ASTM C671 a practical problem was that for concretes with very good frost resistance it was difficult to achieve a high enough degree of water saturation within a reasonable testing period. Anyway, freezing dilation measurements are generally considered to be a good method to detect internal degradation caused by freeze-thaw [Kang et al. 2012].

2.1.3 Critical Degree of Saturation

Fagerlund (1972) has developed a special testing method called:
- ‘Critical Degree of Saturation Method’ (SCR-method) (Figure 1).
This method combines freeze-thaw and capillary water absorption. The original testing method was suggested to be as a general method for the determination of internal frost resistance. [Fagerlund 1972] It basically gives the possibility to also predict a sort of potential service life, and to make quantitative comparisons between quite different types of concretes. The test procedure as applied to concrete was described in a RILEM Tentative Recommendation published in 1977 [RILEM 1977]. At the same time, the potential of this method for testing concrete was investigated in an international co-operative test [Fagerlund 1977]. The deviation in the test results between different laboratories was remarkably small.

![Graph](image1.png)

**Figure 1. Examples of critical degree of saturation test methods, for OPC-concrete with w/c = 0.45. a) Determination of the critical degree of saturation from measurement of dynamic modulus of elasticity (E). b) Results of a capillary absorption test with 30 mm thick slices. [Fagerlund 2004]**

Later on Fagerlund (1999) has presented that in reality, under certain circumstances, some more water might enter the concrete. Examples of such circumstances are:

- Repeated freeze-thaw under very moist conditions. Then, water might be forced in by other, more forceful mechanisms than these acting during isothermal water uptake.
- Condensation of moisture under dense surface layers due to temperature gradients.

These water uptake mechanisms are not considered in the test as it was initially designed. [Fagerlund 1999]

Also one obstacle for a wider use of this method was considered to be the fact that this method is laborious and time-consuming. Fagerlund (1999) suggested also modifications to the testing method, e.g. the use thin slice specimens (thickness 10 mm) instead of larger and thicker ones for the critical degree of saturation determination (S_{CR}-determination). Lately Luping (2010) has made some preliminary trials with this more practical method [Fagerlund 1999, Luping 2010]. These results showed that S_{CR}-determination was feasible in a short time with one single freeze-thaw cycle. A new procedure was suggested by Luping (2010) for the measurement of critical degree of saturation, natural water uptake and also water uptake during freeze-thaw caused by the pumping effect. All of these parameters are then used as input data in practical service life models with regard to frost attack. Also the effect of minimum temperature is included in this
testing procedure (Figure 2). The total period needed for the testing is 4 weeks, i.e. shorter than the slab test (8 weeks; 56 cycles). [Luping 2010]

Figure 2. Example of results for a non-air entrained concrete (w/c = 0.46; air ca. 3%, cement 475 kg/m³) with somewhat different degrees of initial saturation after different number of freeze-thaw cycles, and with different minimum freezing temperatures using modified critical degree of saturation test method. [Luping 2010]

Vesikari (1998a, 1998b, 1999a, 1999b) has adapted Fagerlund’s SCR-model to predict service life of concrete structures by a computer simulation technique. [Vesikari 1998a, Vesikari 1998b, Vesikari 1999a, Vesikari 1999b]

2.2 Freeze-thaw resistance – scaling

There are several test methods in use, many of them also European, to evaluate concrete scaling. They each have their own particularities:
- concrete curing and preservation before the test,
- test specimens (shape, dimensions, production/casting and further preparation),
- freeze-thaw cycle (duration, upper and lower limits for temperature, evolution of temperature, etc.),
- the number of freeze-thaw cycles,
- the surface that is in contact with the freezing medium/salt solution (only upper or lower face, fully immersed),
- the type of thawing salt (NaCl or CaCl₂),
- the temperature that is to be checked for regulating the freeze-thaw cycles (freezing medium, the air in the climate chamber, the concrete specimen),
- the way to collect the scaled material (by rinsing, ultrasonic cleaning),
- how often and how many times in all the amount of scaled off material is determined,
- the way in which test specimens are cooled during the freezing periods (with air or liquid).

In the European testing specification [CEN/TS 12390-9:2006(E)] there are three testing methods for concrete freeze-thaw scaling:
- Slab-Test in CEN/TS 12390-9 which is based on the earlier Swedish standard SS 13 72 44 (so called “Borås-method”),
- CIF-method in CEN/TS 12390-9 and
There is no established correlation between the results obtained by these three test methods. All tests will identify poor and good behaviour, but they differ in their assessment of marginal behaviour. The slab test is the reference method.

Table 2. Overview of the test methods (scaling) according to CEN/TS 12390-9. [CEN/TS 12390-9]. Figures from [Boos & Giergiczny 2010]

<table>
<thead>
<tr>
<th>Test parameters</th>
<th>Slab test (reference method)</th>
<th>CF/CDF-test</th>
<th>Cube test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Storage 1) (T = ca. 20 °C after de-moulding (at 24 h), before freeze-thaw</td>
<td>- Water: for 6 d</td>
<td>- Water: for 6 d</td>
<td>- Water: for 6 d</td>
</tr>
<tr>
<td></td>
<td>- Climate: for 21 d</td>
<td>- Climate: for 21 d</td>
<td>- Climate: for 20 d</td>
</tr>
<tr>
<td></td>
<td>- De-ionised water (3 mm) on the top surface: for 3 d</td>
<td>- Freezing medium (5 mm) underneath: for 7 d</td>
<td>- Immersed in the freezing medium: for 1 d</td>
</tr>
<tr>
<td>Specimen (typical) = l×d×h</td>
<td>With a sawn surface: 150×150×50 mm³</td>
<td>Splitting a 150 mm cube mould with a polytetrafluorethylene (PTFE) plate with no de-moulding agent (= test surface, downwards): 150×150×70 mm³</td>
<td>Cubes: 100×100×100 mm³ (casted with light use of de-moulding agent, wiping with a dry sheet directly before casting)</td>
</tr>
<tr>
<td>Number of specimens</td>
<td>4 (from different 150 mm cubes)</td>
<td>≥5 (total test area ≥0.08 m²)</td>
<td>4 (2 containers with 2 cubes in each)</td>
</tr>
<tr>
<td>Age at freeze-thaw</td>
<td>31 d</td>
<td>28 d</td>
<td>28 d</td>
</tr>
<tr>
<td>Test direction/method</td>
<td>- One-way</td>
<td>- One-way</td>
<td>- All directions</td>
</tr>
<tr>
<td></td>
<td>- Test liquid (3 mm) on the top surface</td>
<td>- Test liquid (5 mm) underneath</td>
<td>- 10 mm spacers on the bottom of the containers, 10 mm distances between the cubes, cubes covered by test liquid (25±5 mm)</td>
</tr>
<tr>
<td>Freezing medium (test liquid)</td>
<td>3% NaCl solution or de-ionized water</td>
<td>3% NaCl solution (CDF-test) or de-ionized water (CF-test)</td>
<td>3% NaCl solution or de-ionized water</td>
</tr>
<tr>
<td>Freeze-thaw cycle: $T_{\text{min}}/T_{\text{max}}$</td>
<td>- In the freezing medium (test liquid): (-20±2) °C/(+20±4) °C</td>
<td>- In the cooling bath liquid: (-20±0.5) °C/(+20±1) °C</td>
<td>- In the centre of the cubes: (-20±2) °C/(-15±2) °C</td>
</tr>
<tr>
<td></td>
<td>- Chest temperature (with good air circulation) not below -27 °C</td>
<td></td>
<td>- Chest temperature not below -25 °C</td>
</tr>
<tr>
<td></td>
<td>- In the cooling bath liquid: (-20±0.5) °C/(+20±1) °C</td>
<td></td>
<td>- Flooding the chest with water, or the containers are moved to a water bath ((+20±2) °C</td>
</tr>
<tr>
<td>Cooling speed below +0 °C</td>
<td>- Ca. 1.7 °C/h</td>
<td>- 10 °C/h</td>
<td>- Ca. 1.2 °C/h</td>
</tr>
<tr>
<td></td>
<td>- From +0 °C to -20 °C in 12 hours</td>
<td>- From +0 °C to -20 °C in 2 hours</td>
<td>- From +0 °C to -15 °C in 10...14 hours</td>
</tr>
<tr>
<td></td>
<td>- Test liquid</td>
<td>- Cooling bath liquid</td>
<td>- Centre of the cubes</td>
</tr>
<tr>
<td>Duration/Number of Freeze-thaw cycles</td>
<td>56 d/56 cycles</td>
<td>28 d/56 cycles</td>
<td>56 d/56 cycles</td>
</tr>
<tr>
<td>Additional information</td>
<td>- Measurements during the thawed phase (between 20–24 h)</td>
<td>- Measurements during the thawed phase when temperature is above 15 °C</td>
<td>- Change the containers around every 7 days: turn them through 180° and inter-change them on a cyclic basis</td>
</tr>
<tr>
<td></td>
<td>- Back to the freezing chamber at the cycle phase (0±30 min)</td>
<td></td>
<td>- Measurements during the thawed state of the specimens</td>
</tr>
<tr>
<td>Measurement of resistance</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 2 (continued, scaling.)

<table>
<thead>
<tr>
<th>Test parameters (examples)</th>
<th>Slab test</th>
<th>CF/CDF-test</th>
<th>Cube test</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Other curing conditions or concrete age at the start of the freeze-thaw cycling</td>
<td>- Other curing conditions or concrete age at the start of the freeze-thaw cycling</td>
<td>- Other curing conditions or concrete age at the start of the freeze-thaw cycling, Other specimen geometries (but h (thickness) must be 70±5 mm, and total test surface area must be ≥0.08 m²) Two PTFE plates in the mould at two opposed vertical sides, forming the test surfaces &gt;28 cycles for CDF-test or &gt;56 cycles for CF-test Other de-icing agent than 3% NaCl solution</td>
<td></td>
</tr>
<tr>
<td>- Other specimen geometries (but h (thickness) must be 50 ± 2 mm)</td>
<td>- Other specimen geometries (but h (thickness) must be 70±5 mm, and total test surface area must be ≥0.08 m²)</td>
<td>- Other curing conditions or concrete age at the start of the freeze-thaw cycles, Other specimen geometries (but 80–100 mm in thickness and width) &gt;56 cycles Other de-icing agent than 3% NaCl solution</td>
<td></td>
</tr>
<tr>
<td>- Top surfaces or surfaces cast against formwork (instead of sawn surface)</td>
<td>- Two PTFE plates in the mould at two opposed vertical sides, forming the test surfaces</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- &gt;56 cycles or &lt;56 cycles (e.g. 28 cycles for paving blocks)</td>
<td>- &gt;28 cycles for CDF-test or &gt;56 cycles for CF-test</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Other de-icing agent than 3% NaCl solution</td>
<td>- Other de-icing agent than 3% NaCl solution</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Test report (main results)</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Freezing medium</td>
</tr>
<tr>
<td>- Mean values for cumulative scaled of material after the specified number of freeze-thaw cycles, and the same values individually for each specimen (kg/m²)</td>
</tr>
<tr>
<td>- Visual assessment (cracks, substantially changes in the cubes, and type of loss – surfaces/edges)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Additional results</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Visual assessment (cracks, scaling from aggregate particles, leakage of freezing medium) before the start and after the specified number of cycles</td>
</tr>
<tr>
<td>- Composition of the concrete</td>
</tr>
<tr>
<td>- Liquid (freezing medium) absorption during the 24 h liquid storage before the freeze-thaw (w.-%)</td>
</tr>
<tr>
<td>- Composition of the concrete</td>
</tr>
</tbody>
</table>

Water = In water; Climate = at (ca. RH 65% and T = 20 °C).

There are also freeze-thaw methods for surface scaling with salt where a saturated salt solution is used as the freezing medium, and specimens are immersed in this solution for freezing. In the SFS 5449 (1988) method, specimens (100 mm cubes) are moved directly from the thawing bath (water, +20 °C) to freeze in a saturated salt solution at -15 °C. Scaling is determined as a volume change of the specimens. This method includes significant temperature shocks, and it is not considered to sufficiently simulate natural freeze-thaw exposure with de-icing salt. [SFS 5449 (1988), Werse 1976]

The Swedish Standard SS 13 72 44 (´Bohås method´) [Svensk standard SS 13 72 44, 1992] is nearly the same as the ‘Slab test’ in [CEN/TS 12390-9], and can also be considered as an improved version of the ASTM C672 method. The ASTM C672 method is intended for use in evaluating the surface resistance qualitatively by visual examination. [Svensk standard SS 13 72 44, ASTM C672/C672M-12]

The French XP P18-420 standard presents the particularity of including specification on storage before scaling test. During the last 7 days of curing, the samples are cut parallel to the bottom side of the mould, to a height of (70 ±2) mm. These specimens are stored in a climatic chamber (20 ±2) °C and (65 ±5)% RH for (14 ±1) days. Following dry storage, the tested surface is pre-humidified with tap water for (72 ±4) hours. After this 31-day conservation period, a 3% NaCl salt solution replaces the pre-humidification water and covers the exposed surface for 56 freeze-thaw cycles. A freeze-thaw cycle lasts 24 hours with a period of 4 hours at -20 °C and another period of 5 hours at +20 °C. [Bouteille et al. 2010]
In Table 3 there is basic information on three additional freeze-thaw methods for the surface scaling with salt [ÖNORM B 3306, ASTM C672/C672M-12, ISO/DIS 4846].

<table>
<thead>
<tr>
<th>Test parameters</th>
<th>ÖNORM B 3306</th>
<th>ASTM C672/C672M-12</th>
<th>ASTM C672/C672M-12</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimens</td>
<td>Slabs: 50 mm x 300 cm²</td>
<td>Slabs: &gt;75 mm x 460 cm²</td>
<td>Cube or prism</td>
</tr>
<tr>
<td>Curing</td>
<td>- at +20 °C</td>
<td>- at +20 °C</td>
<td>- at +20 °C</td>
</tr>
<tr>
<td></td>
<td>- 14 d in water</td>
<td>- 14 d in a moist room</td>
<td>- 14 d in a moist room</td>
</tr>
<tr>
<td></td>
<td>- 14 d in laboratory</td>
<td>- 14 d in laboratory</td>
<td>- 14 d in laboratory</td>
</tr>
<tr>
<td>Treatment before freeze-thaw</td>
<td>8 hours before the freeze-thaw salt solution on the surface</td>
<td>No pre-treatment</td>
<td>7 days before the freeze-thaw salt solution on the surface</td>
</tr>
<tr>
<td>De-icing agent</td>
<td>- 3% NaCl solution</td>
<td>- 4% NaCl solution</td>
<td>- Ca. 3% NaCl solution</td>
</tr>
<tr>
<td></td>
<td>- New solution after every 5 cycles</td>
<td>- New solution after every 5 cycles</td>
<td>(30g NaCl/100 cm³ H₂O)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>- New solution after every 5 cycles</td>
</tr>
<tr>
<td>Freeze-thaw cycle duration</td>
<td>24 h =16 h + 8 h</td>
<td>24 h = 16…18 h + 6…8 h</td>
<td>24 h = 16…18 h + 6…8 h</td>
</tr>
<tr>
<td>Minimum and maximum temperatures</td>
<td>-18…-20 °C</td>
<td>-18 ±2 °C</td>
<td>-18 ±2 °C</td>
</tr>
<tr>
<td></td>
<td>- +15…+22 °C</td>
<td>- +23 ±2 °C</td>
<td>- At laboratory (+20 °C)</td>
</tr>
<tr>
<td>Number of cycles</td>
<td>25</td>
<td>50</td>
<td>25/50</td>
</tr>
<tr>
<td>Measurement of resistance</td>
<td>- Visual evaluation</td>
<td>- Visual evaluation</td>
<td>- Visual evaluation</td>
</tr>
<tr>
<td></td>
<td>- Depth of deterioration</td>
<td>- Depth of deterioration</td>
<td>- Photographs</td>
</tr>
<tr>
<td></td>
<td>- Visible aggregate particles</td>
<td>- Evaluation scale: 0–5 (no deterioration – heavy deterioration &amp; big aggregate particles exposed)</td>
<td>- Evaluation based on surface weight loss, depth of deterioration and rate of deterioration</td>
</tr>
<tr>
<td></td>
<td>- Durable when no changes in the surface, or depth of deterioration is less than 1 mm</td>
<td>- Often also measurement of the amount of scaled off material (after 50 cycles scaling of ca. 0.8 kg/m² corresponds evaluation to class 3–4 [Thomas 1997])</td>
<td></td>
</tr>
</tbody>
</table>

2.3 New and future methods for freeze-thaw

A good test method should classify different materials in the same way that exposure in the field would do. The test method must not allow concretes of inferior quality to be used, but at the same time must not be too restrictive. [Lindmark 1998] Lately there have been many studies dealing with the correlation of freeze-thaw test methods with field exposure. The basic reason for the severity of most laboratory tests is most probably access to water at all times, which is not always the situation in the field. [Rønning 2001, Milachowski et al. 2012, Pigeon et al. 2003, etc.]

According to Boyd & Hooton (2007), relating field exposure to standard testing methods can be difficult. The first reason is naturally the variable nature of environmental conditions. In testing, the moisture content of the specimens and also the maturity of the concrete at the initiation of testing are important to consider. Casting an exterior concrete in June, several months before the freezing season begins, will most probably produce concrete more resistant to scaling than a similar concrete cast in October. According to Boyd & Hooton (2007), standard test methods should be able to take such variability into account if they are to be used to evaluate concretes for specific projects. [Boyd & Hooton 2007]

According to Rønning (2001, 2010), moisture load during freeze-thaw laboratory testing and under field conditions should be known better to be able to fairly compare different concretes and binding materials in different environments. For instance, the influence of early age submerged curing and long term moisture
pumping in freeze-thaw testing should be evaluated in comparison with real field freeze-thaw circumstances. [Rønning 2001, Rønning 2010]

In the CEN TC 51/WG 12/TG 4 working group, the test method for freeze-thaw scaling [CEN/TS 12390-9] is a current subject undergoing review. The aim is to find the correct way for the assessment of freeze-thaw resistance especially for ‘milder’ exposure. One question is also if [CEN/TS 12390-9] is suitable for binding materials with slowly developing properties. [Rønning 2010]

Milachowski et al. (2012) studied e.g. how intermediate drying periods, moisture content and minimum temperature (-0 °C/-20 °C) affected scaling in freeze-thaw testing with de-icing salt. They found that scaling resistance was clearly dependent on the moisture content. Intermediate dry periods increased the number of freeze-thaw cycles that were affectless to damage. Initial scaling rates subsequent to dry periods were also reduced. It was detected that water in capillary pores increased during freeze-thaw and decreased during drying. Instead the content of physically bound water predominantly in gel pores increased also in the case of freeze-thaw with intermediate drying periods. This was expected to be because of an increased degree of saturation due to higher ion concentration in gel pore water. Continuous freeze-thaw load led to a coarsening of the pore structure. Instead, after freeze-thaw load with intermediate drying, a change in the pore structure to finer as well as to coarser pores was observed. The increase of the fraction of pores in the range of gel pores (<0.003 µm) was expected to be due to a blockage of surface capillary pores by crystallization of salt. Increasing the minimum temperature from -20 to -10 °C clearly reduced scaling. It was also found that the chloride content under freeze-thaw exposure with 3% NaCl-solution in laboratory significantly exceeded typical contents due to capillary suction and diffusion. After all, it was concluded that further research is needed to quantify the exposure parameters affecting scaling and to implement them in a practical design model.

Lindmark (1998) has presented, based on his frost-salt scaling theory, suggestions on how to improve freeze-thaw scaling testing methods (especially ‘Slab test’). These suggestions are presented shortly below [Lindmark 1998]:

- Clarify the true climatic conditions which may be expected in different types of use.
- Allow for the changes in micro structure which can be expected to develop during the lifetime of the concrete structure - special attention to those types of concrete which are known to develop a coarser porous micro structure over time.
- Temperature cycle (‘slab test’) applies a low minimum temperature for a short time. This might, according to the described mechanism, represent too mild an exposure – a low minimum temperature means a large portion of the pore system will be clogged with ice. This will reduce permeability and thus to some extent hinder micro ice body growth.
- Furthermore, in cold regions, the temperature of a concrete structure may remain at sub-freezing levels for much longer periods of time than the 16 hours in ‘slab test’. This means that, in reality, there will be much more time for micro ice bodies to grow and to exert pressure.
- In the ‘slab test’, salt solution is applied to the specimen surface throughout the entire test. It is doubtful whether this is a realistic simulation of conditions along a highway. Since the continuous presence of the de-icer solution may lower the freezing point of the pore solution, micro ice body growth may be reduced. In reality, however, salt is applied mainly during periods of sub-freezing temperatures and its concentration may vary. According to the
hypothesis by Lindmark (1998), the period of above-freezing temperatures acts mainly to reduce moisture uptake and micro ice body growth.

- Based on the above considerations, it seems reasonable to test concrete intended for highways with a frost cycle which does not allow the pore solution to melt completely and which reaches a minimum temperature which is chosen on the basis of available recorded climatic data for the actual construction site.
- The concrete might be frozen with tap water on its surface and the salt added when freezing has begun in the pore solution.
- For example, the frost cycle may oscillate between -3 °C and -13 °C, using 12 hours for freezing and 12 hours for melting – this ways the possibility that the de-icer will act to inhibit the severity of the salt frost attack is significantly reduced.
- The number of frost cycles should be chosen from recorded climatic data.
- It is also important that cooling acts over the surface to which the de-icer solution is applied - otherwise the favoured depth of ice body growth will be displaced, possibly to too great a depth.

For instance in [Boyd & Hooton 2007], comparison of the scaling results from the three different laboratories emphasized one of the well-known problems with freeze-thaw scaling resistance tests – lack of good repeatability. It was very difficult to obtain consistent results from different laboratories.

In Europe it was found (1993–1997), based on e.g. some round robin test results performed before introducing [CEN/TS 12390-99] and [CEN/TR 15177], that if not strictly specified and controlled some parameters in freeze-thaw testing affect the test results easily. For instance it was found that the pre-treatment and storage of the specimens must be clarified. It was also discovered that the methods for assessing damage of the internal structure did not lead to consistent results. Also maintenance of the specified temperature was considered important. After this information European cooperation project was conducted. Based on the results from this project, including first inter-laboratory trials, some modifications were made to the European testing methods. The precision estimates in [CEN/TS 12390-99] are based on the round robin results (25 laboratories, 11 countries) performed after these modifications (Figure 3). For investigation scaling due to freeze-thaw attack with de-icing salt the round robin test showed that the precision data for the coefficient of variation for repeatability was in the range from 14% to 18% and for reproducibility from 29% to 38%. The test methods (Slab-, CDF- and Cube-test) were regarded as being nearly equally precise (Table 4). [Siebel & Breit 1999, Breit & Siebel 1998]

![Figure 3](image-url)
Table 4. Precision data for appropriate scaling level. [CEN/TS 12390-99]

<table>
<thead>
<tr>
<th>Test method</th>
<th>Scaling level</th>
<th>Coefficient of variation (%)</th>
<th>repeatability $V_r$</th>
<th>reproducibility $V_{Rr}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slab</td>
<td>1.0 kg/m²</td>
<td>17</td>
<td>31</td>
<td></td>
</tr>
<tr>
<td>CDF</td>
<td>1.5 kg/m²</td>
<td>14</td>
<td>29</td>
<td></td>
</tr>
<tr>
<td>Cube</td>
<td>3% by mass</td>
<td>18</td>
<td>38</td>
<td></td>
</tr>
</tbody>
</table>

In [Breit & Siebel 1998] calculation of precision data for scaling without de-icing salt was impossible due to very low scaling rates. Testing the internal deterioration the round robin test showed that the two test methods CIF- and Beam-test were suitable leading to a yes or no statement concerning the assessment of freeze-thaw attack. It was concluded that to achieve a quantitative statement further research work is needed.

It was also concluded that especially the questions concerning the comparability of the results achieved by different test methods, and the applicability of results achieved under laboratory conditions to practical conditions, have to be clarified by further investigations. [Siebel & Breit 1999, Breit & Siebel 1998]

The current ASTM C672 method has been reported to be more severe than field experience. A concern has been the severity of osmotic effects in samples not pre-saturated with salt solution. A one week pre-saturation period may balance the ions between the saline solution on the top surface and the top layers’ voids, decrease the osmotic pressure and result in less salt scaling. Based on this, there is a new proposed replacement method for ASTM C672 called ‘BNQ standard’ (developed by Bureau de Normalisation du Quebec). [Ahani & Nokken 2012]

In [Klieger 1957] a testing method for scaling where there was a water layer on the concrete surface during the freezing phase and before thawing at ca. 23 °C flake calcium chloride was used as the de-icer. After the thaw period (ca. 4 hours), the salt solution was washed off and replaced by fresh water for the freezing portion of the cycle (ca. 20 hours). After a certain number of cycles there was a numerical rating (0–5) of the scaling (“no scaling” – “severe scaling”). With this method scaling was severe for the non-air-entrained concretes after 5–83 cycles. For the air-entrained concretes, scaling was dependent on the other testing parameters (mainly curing time and temperature and binding material). Both only very slight scaling after 250 cycles and also severe scaling after less than 60 cycles were included. It must be noted that this testing series also included cases with only very short curing times.

Recently electrical methods have also been used to study the freezing-thawing process. In [Perron & Beaudoin 2002] it is suggested that these methods have promise as a basis for development of new rapid methods of durability testing and new test standards.

Dielectric spectroscopy (or electrochemical impedance spectroscopy) measures the dielectric properties of a medium as a function of frequency. This method was found to be sensitive to nucleation and growth of ice crystals. Perron & Beaudoin (2002) developed a method for simultaneous measurement of length change and impedance characteristics of cement paste on cooling and warming. A feature of the impedance spectra, the frequency dispersion (or depression) angle was found to contain information on pore structure effects as they relate to mass transfer of unfrozen water, pore blocking, nucleation of ice crystals and pores filling with ice. [Perron & Baudeoin 2002]
In an on-going Belgium project ‘Freeze-Thaw Resistance Criteria of concretes’ (2010–2014), a comparison of testing methods is being performed. A final aim of this project is the determination of freezing resistance criteria of concrete. This comparative study will be achieved with the help of three test methods: the Slab test (reference test method of the CEN/TS 12390-9), the ISO-DIS 4846 (most currently used in Belgium for measurement of scaling in the presence of the combined action of freeze-thaw cycles and de-icing salts), and the CEN/TR 15177 method for the measurement of mechanical performance loss after the exposition to certain number of freeze-thaw cycles (cooling in air and thawing in water). The concretes with and without air entertaining agents are studied using all three test methods. Also evaluation is done on the influence of carbonation (curing of concretes before the test) on the resistance to freeze-thaw scaling. [http://www.cric.be/default/en-us/research/activitesrecherche/researchfrostcyclus.aspx]
3. **Indirect testing methods – related to freeze-thaw**

3.1 **Microscopy - optical and electron**

Both optical and scanning electron microscopy (SEM) are frequently used to study and quantify concrete microstructure and changes in it caused by some deterioration mechanics. Here some examples are presented on the use of microscopy especially in the studies important for freeze-thaw durability and chloride penetration. These studies include e.g. studies on air entrainment quality, frost cracking and ice formation and propagation in cement paste.

3.1.1 **Optical thin and plane sections**

Optical thin section study is today a common and widely used technique to study cement based materials microstructure. These studies are made by using direct light, polarized light or fluorescent light microscopy. [NT BUILD 381-1991, ASTM C856-11, ASTM C457-90, VTT-TEST-R003-00]. Thin section consists of a thin slice of concrete impregnated with fluorescent epoxy glued to an object glass and protected by a cover glass. Fluorescent epoxy penetration is achieved through application of a vacuum to assist epoxy impregnation. The thickness required for analysis of cement-based material is 25–30 µm. The final size of a thin section is e.g. 35 mm x 55 mm x 25 µm, but can also be different.

Materials with crystalline structure cannot be studied using ordinary white light principally because the light vibrates in all directions. The polarized light microscopy can overcome these problems and specific properties of a material can be detected in specific orientations.

Because of the porous nature of concrete and cement paste, it is possible to fill the voids and pore spaces with resin (epoxy) that has been mixed with a fluorescent dye. As fluorescent epoxy is located in original voids and cracks in the concrete, including the large capillary pores of cement paste, this method highlights strongly the presence of all kinds of pores and cracks in cement paste.

The estimation of concrete water-cement ratio (w/c) is also possible by a thin section study. In this it is very important that the petrographer is experienced, and the quality of the thin section is good. The standard deviation of w/c can be as low as 0.02–0.03 (Round Robin test) [Jakobsen & Brown 2006]. Fluorescent epoxy fills the capillary pores during vacuum impregnation. The amount of epoxy in pores is proportional to the capillary porosity, and the original water-cement ratio. The brightness or the cement paste filled with fluorescent epoxy gives information on the capillary porosity, and thus also on the w/c ratio of the paste. Dense cement paste appears darker while a porous cement paste with high w/c appears lighter. By using comparison thin sections with w/c from 0.35 to 0.70, and by comparing the shade of the paste to these known laboratory mixes the w/c of the concrete can be determined.

Also homogeneity of the cement paste, i.e. local variation in w/c can be detected. Lack of homogeneity can be related to the original mixing efficiency. Microcracks can be easily detected in thin sections. Also unhydrated cement grains as well as different kinds of agglomerates (e.g. silica) can be detected. Some reaction products can also be seen, e.g. needle like ettringite. Normally in the preparation phase of a thin section the specimen is dried at 30–50 °C before impregnation with epoxy. This drying phase may cause microcracking in materials sensitive to
drying cracking. Secondary microcracks caused by cutting, grinding or drying must be distinguished from the primary shrinkage microcracks.

3.1.2 Air pore analysis (e.g. spacing factor) – VTT-TEST-R-00-11 (NT BUILD 381), EN 480-11 and ASTM C457

In optical concrete air pore analysis normally the main determined parameter is Powers´ spacing factor (L). It is an attempt to calculate the fraction of paste within some distance of an air void (paste-void proximity). Also other parameters can be determined, and are also used for the calculation of Powers´ spacing factor, or for the evaluation of concretes air pore structure, e.g. specific surface area of the air pores, amount (vol.-%) of air pores in different size classes i.e. air pore size distribution or volume of air pores below some size, e.g. <0.300 mm pores or <0.800 mm pores [Vesikari 1985].

There are somewhat different methods and standards for concrete air pore analysis and determination of Powers´ spacing factor. The most known methods are:

- ASTM C457 (especially in U.S.A and Canada)
- EN 480-11 (in Europe)
- NT BUILD 381 (in Nordic countries) and
- VTT-TEST-R-00-11 (in Finland).

Preparation of the samples for the analysis may be different (thin sections and different kinds of plane sections, Figure 4), and there are also some small differences in the analysis itself. This means that there may also be some differences in the results. If sample preparation is excluded, the size rating of the pores in the analysis will have some effect on the results. As there is no precise criterion to distinguish between entrained and entrapped air there are different criterions adapted for that. For instance in a study by Kang et al. (2012) entrained air was defined as voids less than 500 µm in chord size. [Kang et al. 20129]. Kang et al. used linear transverse method in which the length of a line intersecting the pores i.e. chord length is measured.

In Figure 5 there is a comparison where the spacing factor is calculated based on the pores 0.020–0.800 µm (chord size), and on the other hand based on all the pores >0.020 mm, i.e. including also all compaction pores. In this case the analysis was in both cases according to VTT-TEST-method and rating of the pore sizes was according to the intersection length in the pores in linear traverse method. [VTT-TEST-R-00-10, ASTM C457]
3.1.3 Quality of air pore structure – homogeneity, clustering, coalescence and agglomeration

Air void system with sufficiently high air content and small spacing factor can work properly only when the voids are spread out through the whole paste. Air-void clustering is a phenomenon in which entrained air voids cluster typically especially around coarse aggregate particles. When air voids coalescence bigger air voids are created instead of individual smaller pores. According to Kang (2010) homogeneity of air pore structure, and for instance air pore clustering and agglomerates do have an effect on freeze-thaw scaling (Figure 6). [Kang 2010]

In a study by Kang (2010) it was noticed that the clustering seemed to coincide with ITZ around aggregates. [Kang 2010]
A visual petrographic rating system was developed by Kozikowski et al. (2005) [Kozikowski 2005] to establish a standardized method for determining the severity of clustering (no, minor, moderate, and severe air void clustering). A correlation was found between this rating and compressive strength loss (Figure 7). Problematic air void clustering was found to coincide with late additions of water to the concrete mixture. The type of air-entraining admixture used in the concrete was also found to have a major influence on clustering. In this research the main thing was the effect of air void clustering on compressive strength. [Kozikowski 2005]

In [Jim Grove 2006] the clustering or coalescing of air voids was expected to be caused by the air entraining admixture itself, mixing issues, or the absorptive or dry aggregate. In the last case, water moves toward absorptive aggregate and carries the admixture with it. If air entraining admixture is not soluble enough it may accumulate at the aggregate surface as water is absorbed causing a disproportionate amount of admixture and thereby create bubbles near the surface of the aggregate as the mixing continues. The movement of water...
towards the aggregate may also carry the air bubbles towards the aggregate surface, resulting in coalescing. Over-vibration of the concrete during construction can also cause unhomogeneous air pore structure. Entrapped big air pores are a sign of inadequate consolidation. [Jim Grove 2006]

3.1.4 Scanning Electron Microscopy (SEM) + X-Ray facility

The chloride content of hardened concrete can be determined using a scanning electron microscopy (SEM) fitted with an energy dispersive X-Ray facility. However extensive sample preparation is needed. Flat surface is needed, and grinding must be done with chloride free lubricants with low chloride solubility. Also this technique has limited application as primary analysis technique as there has been found to be only a non-linear relationship between this X-Ray method and more conventional methods.

3.1.5 Frost cracking intensity/properties by optical microscopy

According to [Applied Petrography Group 2010] cracks are classified using the following terms:

- **Macrocrack**: These are cracks that are readily visible to the naked eye without the aid of a stereo binocular microscope and are typically >0.1 mm wide.
- **Macroscopic cracks**: These cracks are visible in the hand specimen or with the aid of a stereo binocular microscope and are typically >0.01 mm wide.
- **Fine crack**: These are cracks that are only readily visible with a stereo binocular microscope or in thin section. Cracks of this type are typically between 0.01 and 0.10 mm wide.
- **Microcracking**: These cracks cannot be detected with a stereo binocular microscope. They are typically <0.01 mm wide and are most easily seen in petrographic thin sections containing fluorescent dye and by using fluorescent illumination.

In the work by Jacobsen et al. (1996) crack density caused by rapid freeze-thaw exposure was measured on polished sections impregnated with a red dye (Hornain et al.1986). This impregnation procedure involves no drying that may increase microcracking. Two polished sections were made of each level of deterioration. Crack densities were measured by counting the number of cracks traversing parallel lines approximately 5 mm apart. 1500 mm traverse length was counted for each level of deterioration. [Jacobsen et al. 1996]

Sample preparation technique for crack detection using dye impregnation is also presented in [Hornain et al. 1996]. The procedure includes the impregnation of a concrete section with a colored dye (‘Irgacete red powder’) diluted in ethanol. During further preparation concrete is constantly kept humid to avoid cracking caused by drying. Other methods usually require drying of the sample prior to impregnation. Cracks >1 µm can be detected with this technique. Also more porous zones in cement paste, such as ITZ, can be detected.

Fluorescent dye can also be used to facilitate the detection of cracks. The water saturated concrete sample is immersed in a bath filled with the fluorescent dye solution, and the pore water is slowly replaced by the coloured solvent by a counter-diffusion process. This depends on the size of the specimen but takes typically ca. 4 days. Excess fluorescent dye is removed by slightly polishing the surface. The technique has been found to be well suited for the study of cracks in concrete. Its main drawback is that it takes many days to prepare a sample. [Hornain et al. 1996]
Normal thin section preparation technique or a vacuum-impregnated reground plane section technique can also be used. The latter technique includes drying in an oven at a temperature of 35 °C for 24 h before vacuum impregnation (1 bar) with epoxy resin containing fluorescent dye. [NT BUILD 381, Glinicki & Litorowicz 2006]

The quantification of the crack network has been determined by using the oriented secants method or stereological total projection method. Recent developments in microscopy and image processing techniques have facilitated efforts towards investigation of the crack system in concrete. When image-analysis systems are used for crack detection and analysis application of appropriate shape criteria to distinguish cracks from porous zones and spherical air voids are normally used. Crack system parameters have been correlated with e.g. chloride diffusion properties. [Glinicki & Litorowicz 2006]

For the description of the crack pattern the following parameters have been used [Glinicki & Litorowicz 2006]:

- Dendritic length of cracks, L [mm] – total length of all the dendrites of each crack on the image.
- Average width of cracks, W [mm] – total crack area per total dendritic length.
- Area of cracks, A [mm²] – total area of each crack on the image.
- Density of cracks, LA [mm/mm²] – total dendritic length of cracks per image area.
- Areal fraction, AA [mm²/mm²] – ratio between the area of the counted cracks to the entire area of the active image.
- Degree of crack orientation, – determined as a classical stereological method of the oriented secants on a plane.
- Distribution of crack width.

3.1.6 Directional cooling stage – studies on ice penetration

Carlos (2008) has examined ice penetration into air-entrained voids by using a directional cooling stage (DCS) mounted onto an optical microscope. The DCS consisted of two copper plates, thin electrical heat pads, motor, control box, and temperature bath reaching temperatures as low as -30 °C. Unidirectional solidification was performed using a thin sample placed between a microslide and a glass cover (Figure 8). Images were taken at various time intervals during the experiment using a digital camera.

By this method the kinetics of ice formation was studied. High cooling rates were used (40 °C per hour and higher). Cross plane polarization was used to indicate if grains and grain boundaries were present in the ice structure. Polarized light was also used for some samples to obtain clearer images of the area between the ice formation and the solution, of any grain boundaries that may have occurred, and of any brine pockets in the ice. [Carlos Jr 2008] Directional solidification tests are commonly used in biological and thermal and fluid science in order to study liquid-solid interface morphologies during phase transitions.
Figure 8. Illustration of Directional Cooling Stage (DCS). v is the velocity of the glass microslide and sample. TH and TL are the high and low temperatures of the copper plates. [Carlos Jr 2008]

Hardened cement paste samples have been studied and imaged using the directional cooling stage (DCS) by Corr (2001) and Wang et al. (1996). The cement paste samples were sawn, glued with epoxy on a microslide and ground to an 80 µm thickness. [Carlos Jr 2008, Corr 2001, Wang et al. 1996]

3.1.7 Ice formation and morphology in entrained air voids by LTESEM

Corr et al. (2004) have used low temperature scanning electron microscopy (LTSEM) to study ice formation and morphology in entrained air voids. LTSEM is closely related to the standard SEM, except that the specimen is kept at a cryogenic temperature (-190 °C) during imaging. An image of an entrained air void in a cement paste specimen cured at RH 100% for 7 days before freezing at -7 °C for 24 h prior to imaging is presented in Figure 9a. Figure 9b shows the same air void after sublimation of the ice. [Corr et al. 2004]

Figure 9. Discrete ice crystals a) before and b) after sublimation. Images are taken by using low temperature scanning electron microscopy (LTSEM). [Corr et al. 2004]
3.2 Permeation and porosity methods

Methods that give information on concrete porosity and transport properties are known to be very essential in all durability research. Precise characterisation of the physical structure of the concrete and cement paste is extremely difficult, if not impossible. This is why average values of permeability, porosity, diffusivity and sorptivity have to be used to model the transport mechanisms through the complex pore structure. For concrete durability especially near surface permeability and porosity properties are essential. [Long et al. 2001]

3.2.1 Protective pore ratio

An old Finnish standard [SFS 4475, 1988] has been used as an official quality control method for freeze-thaw resistance both with and without salt exposure. It determines so called protective pore ratio (p_r). Protective pore ratio is the volume proportion of all concrete pores that will maintain empty in water storage according to the standard (i.e. all air pores, including compaction pores). Principally this method gives the volume of air pores in relation to capillary and gel pores. Total pore volume was determined by using 15 ±1.5 MPa water pressure for 24 h. In [By 32 1992] the demand for p_r was 0.17–0.25. This method was not allowed in every case because in some cases it was not applicable (low workability mixes). High strength concrete does not get fully saturated in the water storage of the standard. This means that too high proportion of all the pores will be defined as air pores (too high p_r).

3.2.2 Water penetration under pressure

Water penetration under pressure is a method to get information on concrete durability properties. In general the water tightness of concrete depends on capillary porosity, its connectivity and the pore structure (tortuosity and disconnection). These parameters are directly related to the w/b ratio and the progress of cement hydration. Capillary disconnection is very difficult for w/c-ratio higher than 0.60, and impossible for w/c of 0.70. With e.g. some additional fine materials, the decrease and increase of water penetration depth is also related to the filler effect, heterogeneous nucleation and the dilution effect (a consequence of replacing a part of cement by the same quantity of limestone). [Ramezanianpour 2010, Persson 2001]

In EN 12390-8 (2009) method water is applied under pressure to the surface of hardened concrete. The specimen is then split and the depth of penetration of the waterfront is measured. For instance in [Ramezanianpour 2010] this method was used to evaluate durability properties of concretes containing limestone cements. For the mixes with no limestone the penetration depth at 28 d was from 11 mm to 17 mm, when w/c-ratio was from 0.37 to 0.55. At 180 d this penetration depth was from 3.5 mm to 10 mm. In Finland EN 12390-8 method is used as a quality control method if concrete must be watertight [by 50 (2012)]. Water penetration (at ≥28 d) must be less than 100 mm after being exposed to (500 ±50) kPa water pressure for (72 ±2) hours.

3.2.3 Absorption (capillary absorption)

ASTM C1585-11 method can be used to determine the rate of absorption (sorptivity) of water by hydraulic cement concrete by measuring the increase in the mass of a specimen resulting from absorption of water as a function of time when only one surface of the specimen is exposed to water. The specimen is conditioned at a standard relative humidity to induce a consistent moisture
condition in the capillary pore system. The exposed surface of the specimen is immersed in water and water ingress of unsaturated concrete is dominated by capillary suction during initial contact with water.

In [Şahmaran & Li 2009] ASTM-sorptivity test was conducted to determine how load-induced micro-cracks of Engineered Cementitious Composites (ECC) affect the capillary suction (absorption) of concrete. The presence of micro-cracking in ECC significantly altered the transport properties (Figure 10).

\[ \text{Sorptivity as the function of the number of microcracks. [Şahmaran & Li 2009]} \]

In Finland the so called Capillary water uptake test has been used widely in research. [VTT-TEST 385-86] In this method the specimens are normally 25 mm thick (area normally 100 × 100 mm²). In this testing there is first a drying period at 50 °C, and after that capillary water absorption through one specimen surface. Normally water absorption time is 14 d. The principles in the Capillary water uptake test are presented in Figure 11. [Vesikari & Kuosa 1999]

\[ \text{Figure 11. The principles in Capillary water uptake test for concrete. In “1” capillary pores are filled with water, and in “2” air pores are filled with water. In the “nick point” (t̿ₚ) all capillary and gel pores are expected to be filled with water. After that air pores are filled slowly. [Vesikari & Kuosa 1999]} \]

3.2.4 Mercury intrusion porosimetry (MIP)

Mercury intrusion porosimetry (MIP) has been a widely used technique for characterization of the pore size distribution of cement-based materials. The volume of mercury that intrudes is used to determine the pore volume, the pore size being calculated from the intrusion pressure. However, the technique has
several limitations, among which are the ink bottle effect and a cylindrical pore geometry assumption that lead to inaccurate pore size distribution curves. MIP only indicates the accessibility of the overall porosity as a function of pore size. This is especially pronounced for the pores or microcracks in the interfacial zone. Only if these pores or cracks provide a continuous passageway, they are recorded correctly by MIP in terms of volume and size. [Kelly et al. 1998, Eksröm 2003]

The method of drying can also damage or change the microstructure of the pores and the high pressure used in MIP can further damage and alter the microstructure of the pores. Besides the simplifying pore-geometry assumptions necessary for analysing the results are not representative of the structure. [Eksröm 2003, Kelly et al. 1998]

3.2.5 Wood’s metal intrusion porosimetry (WMIP)

To avoid limitations in MIP, as the ink bottle effect, Willis et al. (1998) used Wood’s metal as the intruding liquid instead of mercury. The used in their experiment had a chemical composition of 50% bismuth, 26.7% lead, 13.3% tin, and 10% cadmium by weight. This material has a freezing point of 65.5 °C, a specific gravity of 9.7 g/cm³, and is insoluble in water. Willis et al. (1998) used scanning electron microscopy and imaging techniques to the sample after intrusion. The molten Wood’s metal solidifies within the pore structure of the sample, which allows it to be sectioned and observed in the scanning electron microscopy. Wood’s metal intrusion porosimetry (WMIP) provided the possibility for quantitative characterization of the spatial geometry of pores in cement-based materials. It was found that that the size distributions obtained from MIP were shifted more towards smaller pores than those obtained from WMIP and image analysis. [Willis et al. 1998]

3.2.6 Thermoporosimetry and cryoporometry

Thermoporosimetry and cryoporometry provide a method to pore size distribution that is fully or partly saturated with a liquid. The method is based on thermodynamic conditions of the liquid-solid transformation (melting-solidification) of capillary condensate inside a porous body. A small crystal in a liquid melts at a lower temperature than the bulk liquid. Thus, if a liquid is imbibed into a porous material, and then frozen, the melting temperature will provide information on the pore-size distribution, i.e. the freezing point depression and the melting point depression can be used to determine the pore radius (melting point depression is mostly used). The difference in transition temperature between confined and bulk solvent can be detected calorimetrically by differential scanning calorimetry (DSC). The melting point depression is inversely proportional to the pore size. This method is applicable to pores with radius 2–200 nm. [Aligizaki 2006]

3.2.7 Low heat calorimetry

In low heat calorimetry (LHC) the measurement of length-change is combined with calorimetry. This gives more information than the measurement of dilation only. LHC is based on ideas of Verbeck and Klieger (1958). When measuring the length-change curve in a calorimeter there is no place for a rig. Instead an equipment must be hanged inside the calorimeter so that the sensor only measures the movement of the specimen and not movements of the calorimeter itself. The calorimeter and a sensor detect the ice-formation at the same time. For instance it has been detected when the ice-formation ends and the specimen contracts. [Fridh 1999]
3.3 Other methods

3.3.1 Ultrasonic pulse transit time measurements (UPTT) and resonant frequencies of vibrations (FF)

Especially in the freezing and thawing test of concrete, elastic-wave methods are applied to estimate the frost damage non-destructively. In 'slab test' according to [CEN/TR 15177 (2006)] length change measurement by extensometer is the reference measurement procedure but there are also two alternative elastic-wave methods for that. One alternative method is based on the ultrasonic test, which measures the transmission time of the longitudinal wave (P wave, Ultrasonic pulse transit time, UPTT), while the other alternative method measures the resonant frequencies of vibrations (Fundamental transverse frequency, FF). [CEN/TR 15177 (2006), Ohtsu 2011]

Ohtsu (2011) has found that the relative modulus obtained from P-wave velocity (UPV, transmission velocity of the longitudinal wave) is comparable to that from the tangential modulus of elasticity in the compression test, and it was reasonably recommended to estimate the frost damage non-destructively. In the study by Ohtsu (2011) the difference between the frost damages estimated by the two alternative methods, i.e. UPTT and FF, was practically little, because the concrete samples were not heavily deteriorated. [Ohtsu 2011]

UPTT technique is one of the most popular non-destructive techniques used in the assessment of concrete properties. In UPTT-technique the propagation velocity of a pulse of vibrational energy which has passed through a concrete medium is measured. Knowing the direct path length between the transducers, and the time of travel, the pulse velocity through the concrete can be obtained. The transducers may be located directly opposite each other (direct transmission), diagonally to each other; that is, the transducers are across corners (diagonal transmission) or attached to the same surface and separated by a known distance (indirect transmission). Pulse velocity is proportional to the square root of the elastic modulus and inversely proportional to the square root of the mass density of the concrete. All of the concrete between the transmitter and the receiver affects the measured property. Pulse velocity is affected by e.g. concrete strength and cracks. UPTT-method is widely used to detect internal freeze-thaw deterioration in concrete specimens. It is also used for concrete strength estimation, and to detect concrete uniformity, and changes in concrete properties with time. There are several standards for UPTT-measurements. These standards discuss somewhat differently on the factors that affect pulse velocity in concrete, such as reinforcement, temperature, humidity, size and shape of the specimen, etc. There are also commercially available instruments for UPTT-measurements, e.g. the V-meter (USA) and the Portable Ultrasonic Non-destructive Digital Indicating Tester PUNDIT (UK). [Komloš et al. 1996, Long et al. 2001]

Selleck et al. (1998) has found that ultrasonic pulse velocity (UPV) was not very sensitive to changes caused by distributed microcracking. Distributed cracking was induced by freeze-thaw cycling and salt-scaling. Measurements were correlated with damage observed using optical microscopy. As such, salt scaling had little or no effect on the UPV, while the freeze-thaw cycles consistently reduced the UPV. However, changes in the UPV were small compared to the degree of physical damage that was noted visually and quantified by the dynamic modulus of elasticity. For example, a 25–50% change of dynamic modulus corresponded to only 3–9% change of UPV. There are also studies showing that UPV-method is not satisfactory for the estimation of concrete compressive
strength. This is because UPV is affected by parameters which have little effect upon the mechanical properties of interest. Also existing standardized ultrasonic methods are based on the assumption that the bulk pulse velocity is independent of ultrasonic frequency used. However, test results show that this is not the case. [Selleck et al. 1998, Mirmiran & Wei, Popovics & Rose 2004]

Test method [ASTM C215-08] relies on changes in resonant frequency and has proven to be a good gauge of material degradation due to freeze thaw damage. It is specifically referenced within [ASTM C666/C666M-03(2008)] for freeze thaw evaluation. Values for the dynamic modulus of elasticity may result from widely different resonant frequencies of specimens of different sizes and shapes of the same concrete. Therefore, it is not advisable to compare results from specimens of different sizes or shapes. [ASTM C215-08]

3.3.2 Dielectric and ultrasonic measurements by Fabbri et al. (2009)

Fabbri et al. (2009) have studied the evolution of the ice content of porous media submitted to sub-zero temperatures by dielectric and ultrasonic measurements. These quantitative methods were successfully applied to samples of sintered glass beads, Boom clay and hardened cement paste. The ice content curves were analyzed with the help of thermoporometry concepts in order to characterize the pore size distribution. The two apparatus presented predicted with a good accuracy the amount of ice formed within a consolidated porous medium. It was concluded that they represent a cheaper alternatives to the low temperature calorimetry. [Fabbri et al. 2009]

3.3.3 Combination of Calorimetric, expansion and acoustic methods by Kaufmann (2004)

In a series of experiments, the freezing process, its initiation, continuation and the resulting damage, was studied by Kaufmann (2004). He used a combination of calorimetric, expansion and acoustic methods to monitor heat release, mechanical deformation and damage during a series of frost cycles. It was possible to get an insight into supercooling, salt segregation, ice front penetration and thawing characteristics. It was possible to separate ice formation into two main phases, i.e. an instantaneous nucleation part and a progressive ice penetration (percolation) part. Based on the results, a qualitative sequential damage model was proposed. [Kaufmann 2004]

The heat flow induced by the phase transition was detected by the calorimetric method. With low degree of saturation only small pores were saturated, and the lower was the observed initial freezing point. A well-marked freezing peak is observed near the homogenous nucleation temperature (-40 °C) perhaps originating from strong supercooling in isolated pores (Figure 12a). Figure 12b presents the thawing phase for specimen saturated with a sodium chloride solution. For very high salt content, an additional peak at about -20 °C, perhaps because of the result of salt segregation, not far from the eutectic temperature, is observed.
Due to the very different longitudinal wave velocities in water and in ice, the pulse velocity in a porous specimen is significantly increased when its pore water freezes. This effect was used by Kaufmann (2004) to monitor the beginning of ice formation in concrete blocks. A test arrangement with a liquid layer on top of the surface was chosen. As the pulse velocity within the concrete is much higher than the one of ice, the superficial frost medium (water, salt solution) did not influence the result. By this method, for instance the detection of the effects of salt solution vs. pure water on concrete surface, ice front penetration and supercooling were possible (Figure 13).

Kaufmann (2004) used also expansion experiments to study the ice formation and the damage evolution. To include liquid transport he had a liquid layer on the specimen surface (thin plates with 10 mm thickness). An increasing length change with increasing number of frost cycles was detected. To demonstrate that the observed length changes coincide with deterioration, the acoustic emissions of concrete plates were continuously monitored during the frost cycles. [Kaufmann 2004]

**Figure 13. a) Test arrangement to monitor the beginning of ice formation in a concrete block; b) Temperature and pulse velocity at freezing while frost medium was 3% sodium chloride solution. [Kaufmann 2004]**

### 3.3.4 Neutron diffraction technique

In neutron diffraction technique the advantage of using neutrons lies in both their sensitivity to hydrogen and the penetrating nature of the radiation. Neutron diffraction technique is similar to X-ray diffraction but due to their different
scattering properties, neutrons and X-rays provide complementary information. A neutron diffraction pattern provides information of the structure of the material.

The Schulson et al. (2000) used neutron diffraction technique to study hexagonal ice in hardened cement. They studied nearly saturated cement pastes (w/c = 0.36) and used cylindrical specimens (d 5 mm, h 50 mm). Heavy water (D$_2$O) was used instead of light water to reduce incoherent scattering. They showed for the first time that the pore water transformed mainly to hexagonal ice (I$_h$ crystal structure), upon slow cooling below the (bulk) equilibrium freezing point of heavy water (+3.65 °C) (Figure 14). The ice formed nearly continuously, implying a range of freezing sites; and at any temperature below the bulk freezing point there was more ice upon heating than cooling, indicating freeze-thaw hysteresis. The results were used to calculate the pore size distribution (was mainly 3–30 nm). [Schulson et al. 2000]

![Figure 14. Neutron diffraction patterns from nearly saturated hardened cement at 298, 248, and 227K (25, -25 and -46 °C). I$_h$ denotes hexagonal ice; I$_c$ denotes cubic ice. [Schulson et al. 2000]](image)

Swainson & Schulson (2001) used also neutron diffraction technique to study ice and water within a hardened cement paste during freeze-thaw. They used also a saturated rod of hardened Portland cement paste (w/c = 0.40), and examined the behaviour of it during two slow freeze-thaw cycles (+24 – -46 °C). They concluded that neutron diffraction technique gives a direct and independent quantification of the amount of ice and liquid water in the pore system of hardened cement paste as a function of temperature. In [Chatterji 2002] there is a discussion on the study by Swainson & Schulson (2001). In this discussion the negligible changes to the pore size distribution and geometry that occurred during two cycles of freeze-thaw cycles is wondered. The main reason for these negligible changes was expected to be too much drying of the specimens before any freeze-thaw.

### 3.3.5 Electrical resistivity

Cai & Liu (1998) used concrete resistivity measurements to evaluate freezing water amount during cooling. Concrete is conductive due to ions contained in concrete pore solution, such as Ca$^{2+}$, OH$^-$, Na$^+$, K$^+$ etc. Since ice can be regarded as nonconductor, when some amount of concrete pore solution freezes under negative temperatures, the electrical conductivity of concrete decreases because of the decreasing amount of conductive pore solution. Therefore, by studying the decreasing rate of the electrical conductivity of concrete, the amount
of frozen pore solution at different temperatures within a freeze-thaw cycle, namely, ice formation process, can be evaluated.

The test set up is presented in Figure 15. In the test concrete types with w/c 0.60, 0.40 and 0.30 (W6, W4 and W3 respectively) with and without air entrainment (A) was measured. The results show that most of the freezing happens above -10 °C and also that melting is happening in much higher temperatures (Figure 16).

Figure 15. Test setup for concrete resistivity measurement. [Cai & Liu, 1998]

The method is quite simple and it looks to give good macroscopic indication of freezing phenomena inside concrete sample.
Figure 16. Electrical conductivity of concrete during cooling and heating cycle. w/c ratios 0.60, 0.40 and 0.30 (W6, W4 and W3 respectively) with and without air entrainment (A). [Cai & Liu, 1998]
4. Conclusions

In this report multiple freezing and thawing and scaling test methods as well as indirect test methods are described. It can be noticed that many tests are actually measuring different properties of concrete. Tests have also different parameters even when they are meant for measuring the same property. These make comparison between different test methods very difficult. The selection of a suitable test method depends on the target of research or quality control.

There is no "right" freezing and thawing resistance test method. All of them have been made for their own purpose and use in any other purpose has to be done in great care. It must be kept in mind that the criteria for durability is also greatly dependent on environmental loading and test methods used.

Most of the standardized test methods have been prepared for quality control purposes. For research purposes different tests are widely used. Best understanding on concrete behaviour is obtained by using multiple different methods parallel (both direct and indirect).
5. References


5.1 Standards

ASTM C457-71. Standard Recommended Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete. 15 s.


ASTM C671-77. Standard Test Method for Critical Dilatation of Concrete Specimens Subjected to freezing. 6 s.


ASTM C856-11 Standard Practice for Petrographic Examination of Hardened Concrete. 17 p.


CEN/TR 15177. Measurement of mechanical performance loss after the exposition to certain number of freeze-thaw cycles.


NT Build 492. Nordtest method. Concrete, mortar and cement-based repair materials: Chloride migration coefficient from non-steady-state migration experiments. 8 p.


XP P18-420 1995. AFNOR. Béton – Essai d'écaillage des surfaces de béton durci exposées au gel en présence d'une solution saline (Concrete – Scaling test for hardened concrete surfaces exposed to frost in the presence of a salt solution) In French.