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TEST METHODS OF CONCRETE RESISTANCE TO CHLORIDE INGRESS

METODY BADANIA ODPORNOŚCI BETONU NA WNIKANIE CHLORKÓW

Abstract

The experimental methods for determination of concrete resistance to chlorides – both the chloride permeability test methods (NT Build 492 NordTest – Non-Steady State Migration Test, AASHTO T 277-ASTM C 1202 Test) and the methods for testing concrete resistance to surface scaling due to cyclic freezing and thawing in the presence of NaCl solution (deicing salts, saline sea water, etc.) (e.g. Slab Test according to CEN/TS 12390-9, based on the Borås method according to Swedish Standard SS 13 72 44) are presented in the paper. The Rapid Chloride Test – the method used "in situ" to determine the chloride ion content in concrete is also described.

Keywords: *concrete durability*, *exposure classes (XD, XS, XF)*, *chloride diffusion*, *scaling resistance*

Streszczenie

W artykule przedstawiono doświadczalne metody określenia odporności betonu na działanie chlorków – metody badania przepuszczalności chlorków (NT Build 492 NortTest – Test migracji chlorków przy nieustalonym przepływie, AASHTO T 277-ASTM C 1202 Test) oraz metody badania odporności betonu na powierzchniowe łuszczenie spowodowane cyklicznym zamrażaniem i odmrażaniem w obecności roztworu NaCl (soli odladzających, słonej wody morskiej itd.) (np. Slab Test według normy CEN/TS 12390-9, wzorowany na metodzie Borås według szwedzkiej normy SS 13 72 44). Opisano również Rapid Chloride Test – metodę „in situ" badania zawartości jonów chlorkowych w betonie.

Słowa kluczowe: *trwałość betonu*, *klasy ekspozycji (XD, XS, XF)*, *dyfuzja chlorków*, *odporność na powierzchniowe łuszczenie*

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1. Introduction

Concrete exposed to various environmental conditions can be penetrated by many aggressive ions. Chloride ions affect the durability of concrete subjected to the action of sea water, chloride-bearing air in marine areas and de-icing salts. The penetration of chloride ions can cause corrosion of concrete and corrosion of rebars embedded in concrete.

Corrosion of concrete is caused by the expansive products obtained in the reaction of chloride ions with the components of concrete (mainly calcium hydroxide $Ca(OH)_2$), e.g. expansive alkaline calcium chloride $Ca(OH)_2 \cdot H_2O \cdot CaCl_2$, which increases its volume during crystallization and causes destruction of concrete, is formed during the following reactions:

$$Ca(OH)_2 + 2NaCl \rightarrow CaCl_2 + 2NaOH \tag{1}$$

$$Ca(OH)_2 + CaCl_2 + H_2O \rightarrow Ca(OH)_2 \cdot H_2O \cdot CaCl_2 \tag{2}$$

Chlorides also affect corrosion of steel reinforcement. Chloride ions, CO_2, etc. destroy the protective ferric oxide film which is stable in alkaline (high pH) environment around the steel rebar. This depassivation induces corrosion of steel (lack of the passive layer on the surface of reinforcement leads to the reaction of chloride ions Cl^- with iron, which results in ferric chloride $FeCl_2$; then $FeCl_2$ reacts with water to form rust $Fe(OH)_2$ and the resulting hydrochloric acid HCl causes formation of pits on the surface of reinforcing bars [17, 19]:

$$Fe + 2Cl^- \rightarrow FeCl_2 + 2e^- \tag{3}$$

$$FeCl_2 + 2H_2O \rightarrow Fe(OH)_2 + HCl \tag{4}$$

Corrosion of steel reinforcement causes its expansion in volume due to corrosion products and subsequent cracking and spalling of concrete from the reinforcement.

Moreover, chloride ions (from de-icing salts) together with frost attack can cause another form of concrete deterioration – concrete scaling [7, 17, 19].

The threat of corrosion from chlorides is addressed in the European standard EN 206-1 [35] under two sets of exposure classes: chlorides from sea water (XS) and chlorides other than from sea water (XD). Exposure class XS1 covers concrete that is exposed to airborne salts from sea water but not in direct contact with sea water. The examples of such structures are those situated along the coast beyond the spray zone. Exposure class XS2 includes concrete that is permanently submerged in sea water, i.e. marine and coastal structures. This concrete may be subjected to considerable chloride penetration but significant corrosion may not occur due to the low level of oxygen supply. Exposure class XS3 means that concrete is in the tidal, splash and spray zones. The examples of structures exposed to these conditions include marine and coastal structures. Exposure class XD1 includes the case of concrete of moderate humidity and in contact with airborne chlorides from sources other than sea water, e.g. structures in proximity to highways. Exposure class XD2 covers concrete that is wet, rarely dry, in contact with water containing chlorides other than from sea water. The examples of such structures are swimming pools or structures exposed to industrial waters containing chlorides. Exposure class XD3 means that concrete is exposed to cyclically wet and dry environment and chlorides are not from sea water. The examples of structures are pavements, car park slabs, parts of bridges, highways subjected to de-icing salts or spray from water containing de-icing salts.

The action of chlorides (de-icing agents) together with the freeze-thaw attack is classified in EN 206-1 [35] as: XF2 exposure class when concrete is moderate water saturated, XF4 exposure class in the case of high water saturated concrete. The examples of such structures are pavements, road and marine structures [4, 7, 13, 19, 29].

The problem of concrete resistance to chloride ingress occurs in many various structures. The chloride resistance is one of the most important properties of concrete in the design, construction and maintenance of structures. The paper presents the test methods used for determining the chloride permeability of concrete, chloride content in concrete and concrete scaling caused by frost and de-icing salts. It is also an attempt at a comparative analysis of the afore-mentioned test methods.

2. Chloride permeability of concrete – Standard test methods

The most common methods used to determine the resistance of concrete to the penetration of chloride ions (the chloride diffusion coefficients) are two methods: the **NordTest** – the **Rapid Chloride Migration Test** (RMT), described in the Scandinavian standard **NT Build 492** [39], which is very popular in Europe and the **Rapid Chloride Permeability Test** (RCPT), presented in two American standards **ASTM C 1202** [33] and **AASHTO T 277** [31], which is widespread in the USA and Canada.

In both methods, the penetration of chloride ions is exerted by an external electrical field. The transport processes that take place during the tests are **diffusion** and **electromigration**. Diffusion, which is the transfer of ions from the region of higher ion concentration to the region of lower ion concentration, is the principal chloride ion transport mechanism from the external environment into concrete [13]. Electromigration is caused by the applied voltage. The electrical field is used to accelerate the transfer of chloride ions [8, 18].

In both tests, cylinders with the dimensions of **100 mm (diameter) × 50 mm (thickness)** are used as concrete specimens. The major **differences** between the two methods are the concentration of **NaCl** solution (**3%** in the Rapid Chloride Migration Test /RMT/ versus **10%** in the Rapid Chloride Permeability Test /RCPT/), the voltage used across the electrodes (**30 V** in RMT versus **60 V** in RCPT) and the test duration (from 6 to 96 hours, usually **24 hours** in RMT versus **6 hours** in RCPT).

The RMT and RCPT tests are **non-steady state** tests (in reality, chloride penetration into concrete is a non-steady-state process [8, 18, 23]). The rapid non-steady-state tests are popular, in contrast to the steady-state tests.

Tests for measuring steady-state flux of chlorides are described in the literature [18, 19, 21, 23]. Steady-state tests are very time consuming (it takes several months or even several years to obtain the steady-state chloride flow /diffusion/ across the specimen). The common procedure is the diffusion cell test method (Fig. 1) [18, 21].

In this method a thin specimen forms a barrier between an ion source solution and another solution free of the ions. This allows ions to diffuse in a concentration gradient. When steady-state conditions are achieved, the diffusion coefficient is calculated (using

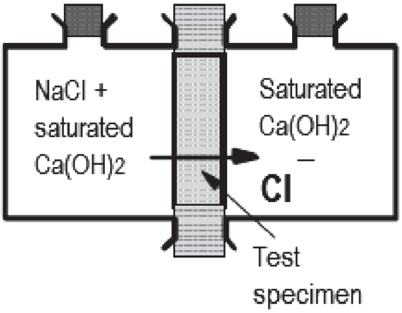


Fig. 1. Test of the chloride resistance of concrete – The steady-state test method. A typical ion diffusion cell [18]

the Fick's First Law of diffusion). Certain migration cell methods in which an electric field is applied to a concrete specimen have been developed in recent years. However, it is difficult to evaluate the diffusion coefficient since all the ions involved in the test affect the flux of chloride ions.

2.1. NT Build 492 (NordTest) method

NT Build 492 (NordTest) – Non-Steady State Migration Test [39], also known as the Rapid Chloride Migration Test (RMT), proposed initially by **Tang and Nilsson** (in 1991) [26, 28] is a very simple and reliable test. The test can be use for **different types of concrete**. To determine the chloride migration coefficient under non-steady state, the depth of chloride penetration in concrete is measured [11, 13, 15, 18, 27]. The concrete specimen (**$Ca(OH)_2$ saturated**) is subjected to the external electrical voltage and chloride ions are forced to move into concrete (Fig. 2). Due to the potential difference between the electrodes, chloride ions migrate from the **10% NaCl solution**, through the concrete specimen, towards the **0.3 M NaOH solution** (Fig. 3), for a defined period of time. Afterwards, the specimen is split and sprayed with $AgNO_3$ – an indicator for chlorides – and thus the chloride penetration depth is measured (Fig. 4). The chloride migration coefficient is calculated on the basis of the value obtained by the measurement.

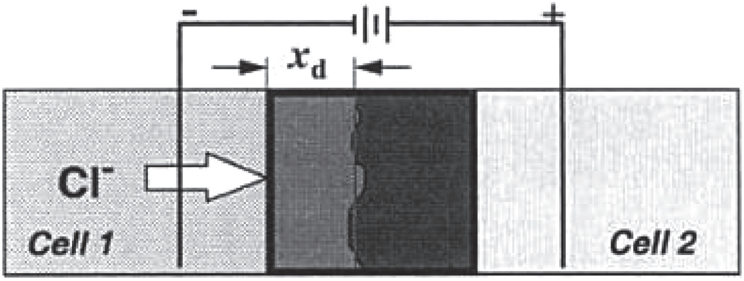


Fig. 2. Test of the chloride resistance of concrete – Schematic diagram of the NT Build 492 (NordTest) method [27]

The method is applicable to the specimens cast in a laboratory or drilled from field structures. The method requires cylindrical specimens with a diameter of 100 mm and thickness of 50 mm, sliced from cast cylinders or drilled cores with a minimum length of 100 mm (e.g. if a Ø100 × 100 mm cast cylinder is used, a 50 mm thick slice should be cut from the central portion of the cylinder; the preparation of the test specimen in the case of longer cast cylinders and drilled cores is described in the standard). Three specimens should be used in the test. After sawing, washing, drying and the vacuum treatment (10–50 mbar /1–5 kPa/ for three hours), the specimens are **saturated with $Ca(OH)_2$** for 19 hours (one hour with the maintained steady vacuum level and subsequently 18 hours after turning off the pump). The saturated concrete sample is set in the test apparatus as shown in Fig. 3.

The catholyte and anolyte reservoirs are filled with the solutions. The catholyte solution is **10% NaCl** solution (100 g NaCl in 900 g distilled or de-ionised water, about 2 N) and the anolyte solution is **0.3 N NaOH** solution (approximately 12 g NaOH in 1 litre water). Thus, the solution on one side of the sample contains chloride ions, while the other solution is chloride free. The electrical potential (**30 V** DC) is applied and the chloride ions are driven into concrete. The initial current is measured. Based on the **initial current**, the **test voltage** and the **test duration** should be selected (as shown in Tab. 1). For example, if the initial current is between 120 and 180 mA, the test voltage is reduced to 15 V DC and the test duration is 24 hours [23, 39].

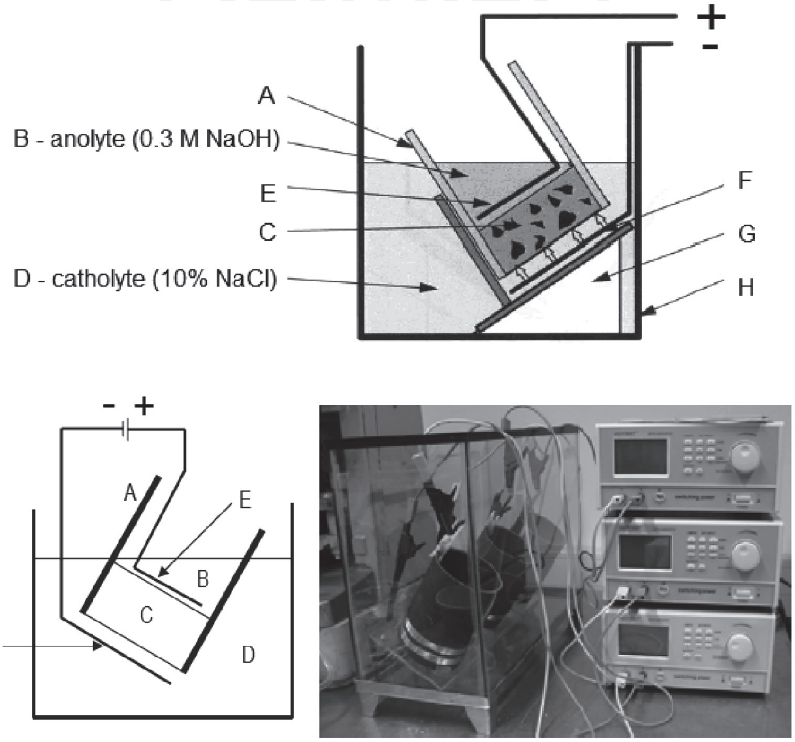


Fig. 3. Test of the chloride resistance of concrete – the NT Build 492 (NordTest) method. Test set-up: A – silicone rubber sleeve, B – anolyte (0.3 M NaOH), C – concrete sample, D – catholyte (10% NaCl), E – anode, F – cathode, G – plastic support, H – plastic box [2, 22, 23]

Table 1

NT Build 492 (NordTest) method – Test voltage and duration [39]

Initial current I_{30V} (with 30 V) [mA]	**Applied voltage U (after adjustment) [V]**	**Possible new initial current I_0 [mA]**	**Test duration t [hour]**
$I_0 < 5$	60	$I_0 < 10$	96
$5 \leq I_0 < 10$		$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$		$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	
$120 \leq I_0 < 180$	15	$60 \leq I_0 < 90$	
$180 \leq I_0 < 360$	10	$60 \leq I_0 < 120$	
$I_0 \geq 360$		$I_0 \geq 120$	6

After a specified period of time the sample is removed and then it is split (in 10-mm thick slices) (see Fig. 4) and sprayed with 0.1 N **$AgNO_3$ (silver nitrate) solution**. When $AgNO_3$ solution is sprayed on concrete containing chloride ions, a chemical reaction occurs. $AgNO_3$ converts to AgCl (silver chloride), a whitish substance ($Ag^+ + Cl^- \rightarrow$ **AgCl /white/**). In the non-chloride area the silver binds with hydroxides present in concrete, creating a brownish colour ($Ag^+ + OH^- \rightarrow AgOH \rightarrow Ag_2O$ /brown/). The chloride penetration depth is measured on the basis of the **white AgCl precipitation** (clearly visible after about 15 minutes) (Fig. 4) [12, 13].

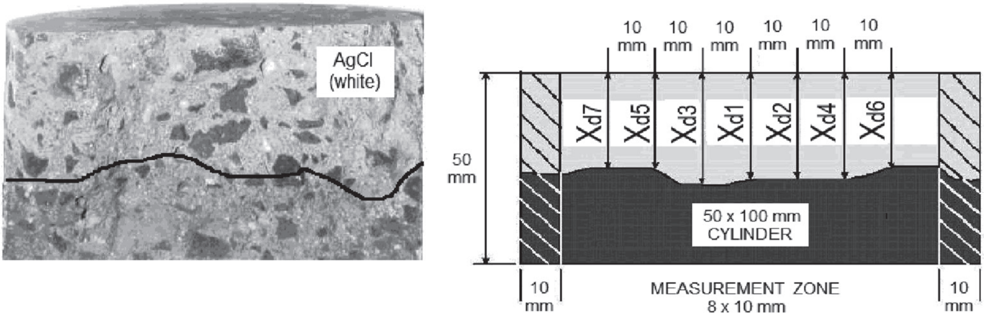


Fig. 4. Test of the chloride resistance of concrete – the NT Build 492 (NordTest) method. Measurement of chloride penetration depths [2, 23, 39]

A quick and easy method of spraying $AgNO_3$ (silver nitrate) on a cross-section of split concrete to identify the **depth of chloride penetration** into a specimen is called the **colourimetric method** (technique) (in which $AgNO_3$ is used as a colourimetic indicator) [3, 12, 22]. The penetration depths should be measured (to an accuracy of 0.1 mm) in the concrete sample starting from the centre towards both edges at intervals of 10 mm (Fig. 4) (using a slide caliper or a suitable ruler). No measurements should be made in the zone within about 10 mm from the edge to obviate the edge effect due to a non-homogeneous degree of saturation or possible leakage [12]. An average of the seven measurements is used for determining the **non-steady-state chloride migration coefficient** (also known as the chloride diffusion coefficient) $\boldsymbol{D_{nssm}}$. The coefficient is calculated on the basis of the voltage magnitude, temperature of anolyte measured at the beginning and the end of test and the depth of penetration of chloride ions, using Eq. (6), obtained from Eq. (5) [2, 28, 39].

$$D_{nssm} = \frac{RT}{zFE}\,\frac{x_d - \alpha\sqrt{x_d}}{t} \tag{5}$$

$$E = \frac{U-2}{L}, \quad \alpha = 2\sqrt{\frac{RT}{zFE}}\, erf^{-1}\left[1 - \left(\frac{2\,C_d}{C_0}\right)\right]$$

where:

D_{nssm} – non-steady-state chloride migration coefficient [m^2/s],
R – gas constant, $R = 8.314$ [J/(°K mol)],
T – average value of the initial and final temperatures in the anolyte solution [K],
z – absolute value of ion valence, for chloride ions $z = 1$,
F – Faraday constant, $F = 9.648 \times 10^4$ [J/(V mol)],
E – electric field [V/m],
U – absolute value of the applied voltage (potential difference across the specimen) [V],
L – thickness of the specimen [m],
x_d – average value of the penetration depths [m],
α – laboratory constant [$m^{1/2}$],
t – test duration [s],
erf^{-1} – inverse of error function,
C_d – chloride concentration at which the colour changes, $C_d = 0.07$ N for Portland cement concrete [mol/dm^3],
C_0 – chloride concentration in the catholyte solution, $C_0 = 2$ N [mol/dm^3].

Since $erf^{-1}\left[1 - \left(\frac{2 \times 0.07}{2}\right)\right] = 1.28$, the equation is simplified to

$$D_{nssm} = \frac{0.0239\,(273+T)\,L}{(U-2)\,t}\left(x_d - 0.0238\sqrt{\frac{(273+T)\,L\,x_d}{U-2}}\right) \tag{6}$$

where:

D_{nssm} – non-steady-state chloride migration coefficient [× 10^{-12} m^2/s],
T – average value of the initial and final temperatures in the anolyte solution [°C],
U – absolute value of the applied voltage [V],
L – thickness of the specimen [mm],
x_d – average value of the penetration depths [mm],
t – test duration [h].

The mathematical formulae (Eq. (5), Eq. (6)) were provided by Tang and Nilsson (in 1991) [28]. Equal (5) has been obtained as the analytical solution of the following equation (describing the flux of chloride ions due to diffusion and migration) (the Nernst–Plank equation) [2, 8, 19, 22, 28]:

$$J = J_D + J_M = -D\left(\frac{\partial C}{\partial x} - \frac{zFE}{RT}C\right) \tag{7}$$

where:

J – total flux of chloride ions [g/ m^2 s],
J_D – flux of chloride ions due to diffusion (the Fick's Law) [g/m^2 s],
J_M – flux of chloride ions due to electromigration (electrically forced migration) [g/m^2 s],
C – concentration of chloride ions [g/m^2],
x – distance from the surface exposed to the source solution of ions [m],
$\partial C / \partial x$ – concentration gradient (at position "x").
T – solution temperature [°K],
D, z, F, E, R – as shown in Eq. (5).

The chloride migration coefficient under non-steady-state (D_{nssm}) is used for evaluating the concrete resistance to chloride penetration, according to the criteria presented in Tab. 2 [15, 26]. The depth of chloride penetration itself may also be a useful parameter.

Table 2

Estimation of the concrete resistance to chloride ion penetration [15, 26]

Chloride migration coefficient D [m^2/s]	**Resistance to chloride penetration**
$< 2 \times 10^{-12}$	very good
$2 - 8 \times 10^{-12}$	good
$8 - 16 \times 10^{-12}$	acceptable
$> 16 \times 10^{-12}$	unacceptable

2.2. ASTM C 1202 and AASHTO T 277 test method

ASTM (American Society of Testing and Materials) C 1202 [29] and AASHTO (American Association of States Highway and Transportation Officials) T 277 [31] test, also known as the Rapid Chloride Permeability Test (RCPT) or the **Coulomb Test**, first developed by **Whiting** (in 1981) [30] is an easy to conduct and time-efficient test. To determine the permeability of concrete to chloride ions, the total charge passed through concrete during a 6-hour period under a 60 V potential difference is measured [8, 11, 18, 20]. Three specimens with the diameter of 100 mm and thickness of 50 mm (obtained by sawing the central part of 100 × 200 mm concrete cylinders) should be used. Before conditioning, the side of the concrete specimen is coated with epoxy. Then the specimen is placed in a vacuum chamber for 3 hours, vacuum saturated with water (de-aerated) for 1 hour and soaked for 18 hours (after turning off the pump). The procedure of sample preparation is the same as that in the NT Build 492 test, but the medium used for saturation is different (water in Coulomb Test versus $Ca(OH)_2$ in the NT Build 492 test). The **water saturated** concrete sample is placed in the test apparatus between two cells as shown in Fig. 5. One of the cells is filled with **3% NaCl** solution and the other cell is filled with **0.3 N NaOH** solution. The system is connected (the NaCl electrode is the cathode, the NaOH electrode is the anode) and a constant voltage of **60 V** DC is applied across the two sides of the sample for **6 hours**. The amount of charge passing through the specimen is measured by recording the current as a function of time. After 6 hours the total **charge** (in Coulombs) is determined by calculating the area under the plot of current versus time [1, 18, 20, 30, 31, 33]. The analysis of **chloride ion concentration in the anode cell** is also conducted by an ion chromatograph at specified intervals.

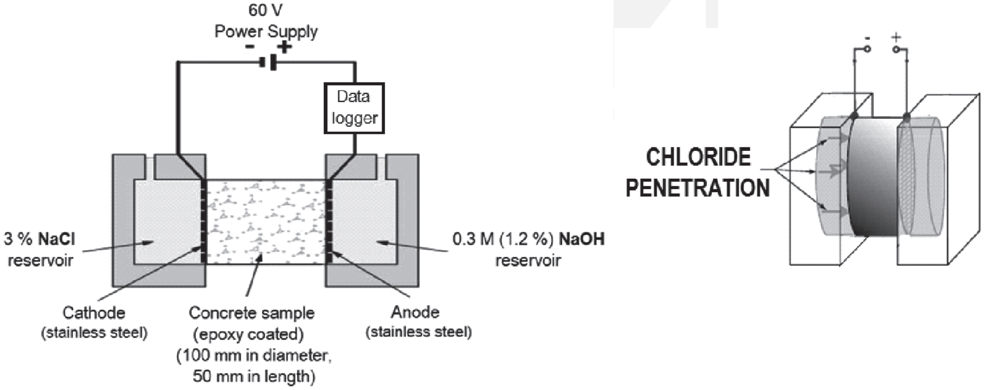


Fig. 5. Test of the chloride resistance of concrete – the ASTM C 1202 / AASHTO T 277 method. Test set-up [2, 22, 23, 30]

ASTM C 1202 [33] and AASHTO T 277 [31] specify the rating of chloride permeability of concrete based on the charge passed, as listed in Tab. 3 [20, 30, 31, 33]. For example, for bridge concrete, which should be of low chloride permeability (w/c ratio is less than 0.4), the required measurement result is between 1,000 and 2,000 Coulombs [9].

Table 3

Chloride permeability of concrete based on charge passed [18, 20, 30, 31, 33]

Charge [Coulombs]	Chloride permeability	Type of concrete
> 4,000	high	high water-to-cement ratio (> 0.5) conventional Portland cement concrete
2,000–4,000	moderate	moderate water-to-cement ratio (0.4–0.5) conventional Portland cement concrete
1,000–2,000	low	low water-to-cement ratio (< 0.4) conventional Portland cement concrete
100–1,000	very low	latex-modified concrete, internally sealed concrete
< 100	negligible	polymer-impregnated concrete, polymer concrete

The Rapid Chloride Permeability Test (ASTM C 1202 and AASHTO T 277 test) has been criticized by many scientists because the charge passed affects **all the ions**, not only **chloride ions** [1, 18, 20]. For concretes with fly ash, ground blast furnace slag, silica fume or other chemical additives the measured charge strongly depends on the composition of concrete (the chemistry of pore solution), i.e. the concentration of various ions (Cl^-, OH^-, SO_4^{2-}, Na^+, Ca^{2+}, etc.) in the pore solution. Due to the presence of the chemical additives the reduction in alkalinity of pore solution (before 90 days) can occur and concrete electrical conductivity (the amount of Coulombs) decreases [20]. The procedure is also under criticism because the high voltage applied (60 V) leads to an increase in temperature, which affects the amount of charge measured in the test [1]. Although the test of Tang and Nilsson (NT Build 492 test) obviates these problems, the ASTM C 1202 / AASHTO T 277 test is still widely used because it is so quick to administer [1, 20].

3. Chloride permeability of concrete – “In situ” test methods

“In situ” tests for the rapid assessment of concrete resistance to chloride ion penetration are very useful to control the durability of existing and newly constructed concrete bridges, pavements, marine structures, etc. [9]. “In situ” diagnosis of concrete is based on the procedures presented in the standards and it is carried out with the special testing set-up (e.g. the **Proove’it** system [35] (Fig. 6)).

The Proove’it system is typically used to measure the total charge passed according to ASTM C 1202 / AASHTO T 277 (the **Rapid Chloride Permeability Test**) (see Section 2.2). This rapid test takes 6 hours. But the system is also applied to conduct longer tests to determine the depth of chloride penetration in accordance with standard NT Build 492 (the **Rapid Chloride Migration Test**) (see Section 2.1).

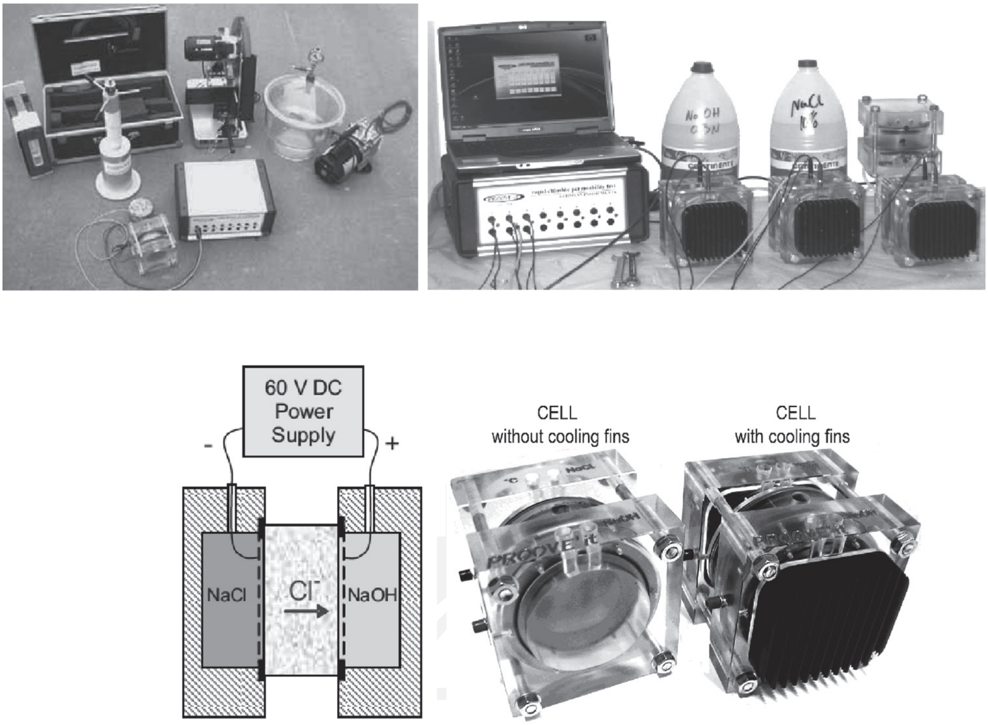
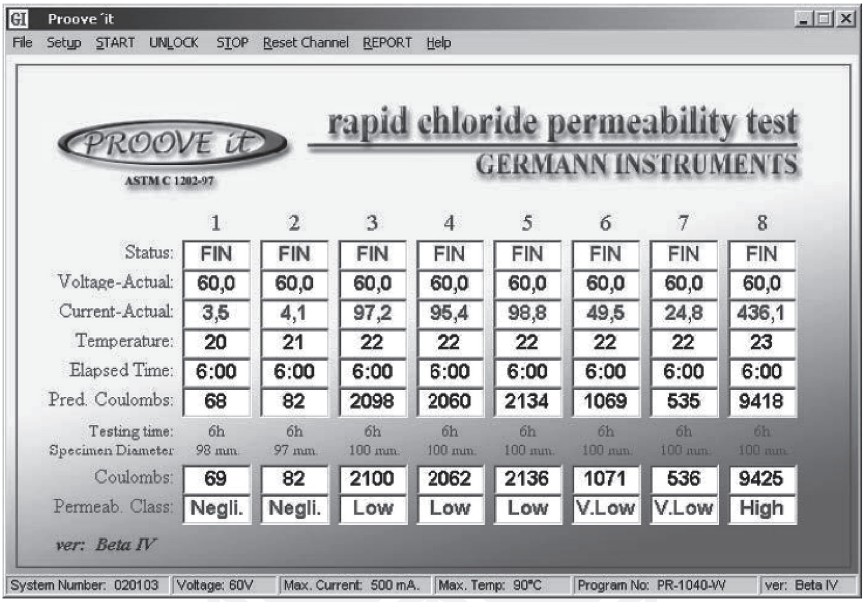


Fig. 6. Test of the chloride resistance of concrete – Proove’it system (with two types of Proove’it cells – with and without cooling fins) [21, 42]

The testing kit includes the following items (Fig. 6):

- moulds for cylindrical specimens (100 mm in diameter and 50 mm in length),
- electrodes (e.g. copper meshes),
- water cooled diamond saw,
- vacuum desiccator,
- vacuum pump,
- sealer (e.g. acrylic) + brush,
- cells (e.g. made of plexiglass) (each cell consists of two reservoirs) (two types – with or without cooling fins /ribs/; cooling fins are needed if the temperature is required to be kept constant, e.g. in the test of chloride ions migration, according to the NT Build 492),
- computer controlled microprocessor power supply,
- temperature probe,
- 3% NaCl solution (ASTM C 1202 test), 10% NaCl solution (NT Build 492 test),
- 0.3 N NaOH solution.

The example of the recorded results after completion of 8 simultaneous tests according to ASTM C 1202 / AASHTO T 277 (the Rapid Chloride Permeability Test) is presented in Fig. 7.



	1	2	3	4	5	6	7	8
Status:	FIN	FIN	FIN	FIN	FIN	FIN	FIN	FIN
Voltage-Actual:	60,0	60,0	60,0	60,0	60,0	60,0	60,0	60,0
Current-Actual:	3,5	4,1	97,2	95,4	98,8	49,5	24,8	436,1
Temperature:	20	21	22	22	22	22	22	23
Elapsed Time:	6:00	6:00	6:00	6:00	6:00	6:00	6:00	6:00
Pred. Coulombs:	68	82	2098	2060	2134	1069	535	9418
Testing time:	6h	6h	6h	6h	6h	6h	6h	6h
Specimen Diameter	98 mm.	97 mm.	100 mm.	100 mm.	100 mm.	100 mm.	100 mm.	100 mm.
Coulombs:	69	82	2100	2062	2136	1071	536	9425
Permeab. Class:	Negli.	Negli.	Low	Low	Low	V.Low	V.Low	High

System Number: 020103 | Voltage: 60V | Max. Current: 500 mA. | Max. Temp: 90°C | Program No: PR-1040-W | ver: Beta IV

Fig. 7. Test of the chloride resistance of concrete – "Proove'it" system. Screen-shot after completion of 8 simultaneous tests according to the ASTM C 1202 [35]

The "Status" line (for the eight cells) is either OFF, ON or FIN (FIN indicates that the test has been completed). The "Voltage-Actual" line points out to the test voltage. The "Current-Actual" line shows the instantaneous current during testing. The "Temperature" line indicates the instantaneous temperature in the reservoir solutions during testing. The "Elapsed Time" line points out to the time since the start-up of each cell. The "Pred. Couloms" line shows the predicted Coulombs at 6 hours, which are estimated after every 5 minutes of testing. The "Testing time" indicates the selected testing time. The "Specimen Diameter" points out to the actual diameter of the specimen. The "Coulombs" line indicates the measured Coulombs at any time during testing. The last line shows the "Chloride ion permeability" classification according to ASTM C 1202.

4. Chloride content in concrete – "In situ" test method

An "in situ" diagnosis of the chloride ion content in concrete can be carried out with a special testing set-up, e.g. the Rapid Chloride Test (RCT) kit (Fig. 8) [41].

The **Rapid Chloride Test** (RCT) is fast and easy to perform. Both hardened and fresh concrete can be tested. A powder sample of hardened concrete, obtained from the structure by drilling and grinding, or a sample taken from fresh concrete is mixed with the extraction liquid in a plastic vial and shaken for 5 minutes. The liquid extracts the chloride ions. Then, a calibrated electrode is submerged into the solution (as shown in Fig. 8) to determine the amount of chloride ions, which is expressed as a percentage of concrete mass (the reading in mV is converted by means of the calibration chart to chloride content) [41].

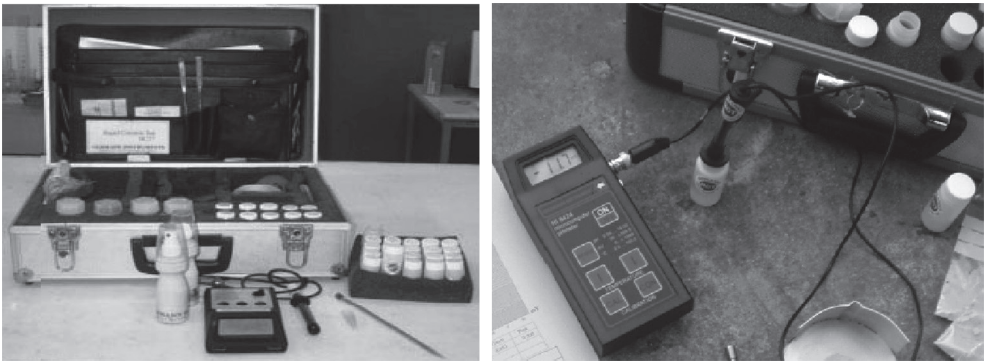


Fig. 8. Test of the chloride content in concrete – Rapid Chloride Test (RCT) system [41]

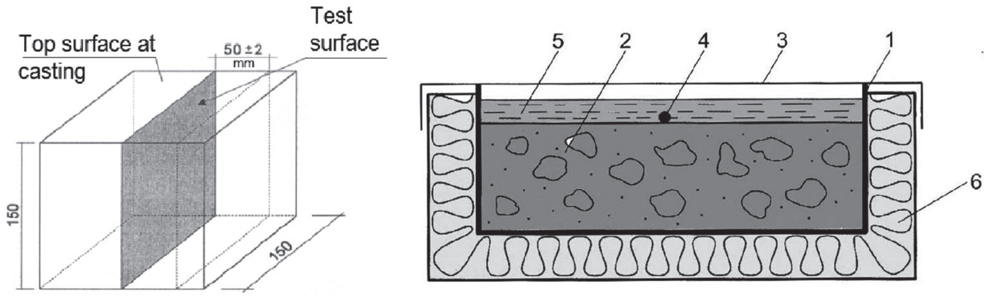
5. Concrete scaling caused by frost and de-icing salts – Standard test methods

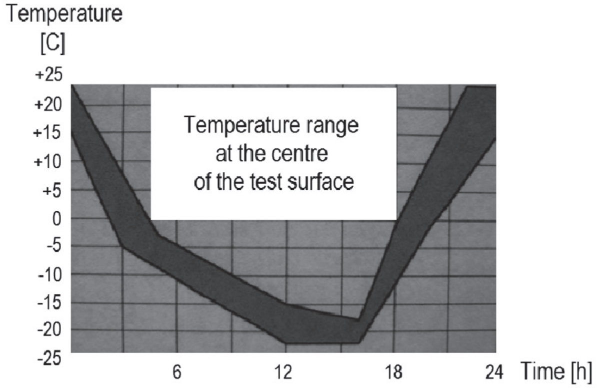
When concrete freezes in the presence of a salt solution, **scaling** (the loss of material from the surface of concrete) can occur. Scaling does not occur if there is no free liquid on the surface of concrete, i.e. saturated concrete does not scale. Small particles of paste and mortar break away from the concrete, and the mass of broken particles increases with the number of cycles, gradually exposing the coarse aggregate [7, 17, 19]. Concrete roads and pavements exposed to deicing salts during winter or marine structures exposed to saline sea water are examples of applications in which salt-frost resistance concrete is required in order to prevent damage. To determine the concrete resistance to scaling, several standardized methods can be used [14, 32, 34]. The European standard **CEN/TS 12390-9** [34] presents three test methods: the **Slab** Test (reference method) and the alternative methods – the **Cube** Test and the **CDF** Test (**C**apillary suction of **D**e-icing solution and **F**reeze thaw Test). In the USA, the **ASTM C 672** method [32] is applied.

5.1. CEN/TS 12390-9 test method (Slab Test)

The standard **CEN/TS 12390-9**, **Slab** Test (**reference** method) [34] is based on the criteria in the Swedish standard SS 137244 – the Boraas method. The concrete durability (resistance) during freezing and thawing in the presence of de-icing salts (3% NaCl solution) is evaluated on the basis of the **mass** of scaled material **related to the concrete surface** subjected to the specified number of cycles of freezing and thawing.

Four **150 × 150 × 50 mm** specimens should be used in the test. A 50 mm thick specimen is sawn from a 150 mm concrete cube (perpendicular to the top surface so that the saw cut for the test surface is located in the centre of the cube – Fig. 9) after 21 days of concrete curing (1 day in the mould, protected against drying by use of a polyethylene sheet, 6 days in tap water at +20(±2)°C and 14 days in the climate chamber /+20(±2)°C and 65(±5)% of relative humidity/). When the concrete is 25 days old, a rubber sheet is glued to all surfaces of the specimen except the test surface. The edge of the rubber sheet should reach 20 mm above the test surface (Fig. 9). After fixing the rubber sheet the specimen is returned





SLAB TEST

Points specifying the shaded area

Upper limit		Lower limit	
t [h]	T [C]	t [h]	T [C]
0	+24	0	+16
5	-3	3	-5
12	-15	12	-22
16	-18	16	-22
18	-1	20	-1
22	+24	24	+16

Fig. 9. Test of the concrete resistance to freezing and thawing in the presence of 3% NaCl. CEN/TS 12390-9 (Slab Test) method – Test set-up: 1 – rubber sheet, 2 – concrete specimen (150 × 150 × 50 mm), 3 – polyethylene sheet, 4 – temperature measuring device in contact with the test surface, 5 – freezing medium (3% NaCl solution), 6 – thermal insulation [15, 34, 40]

to the climate chamber. When the concrete is 28 days old, all surfaces of the specimen with the exception of the test surface are thermally insulated and the top exposed surface is covered with de-mineralized water for 72 hours. Directly before placing the specimen in the freezing chamber, water is replaced by **3% NaCl** solution (the freezing medium). The freezing medium is prevented from evaporating by applying a polyethylene sheet. Then the cooling and thawing cycles (each of 24 hours) are applied. During the test, the temperature (measured at the centre of the test surface) in the freezing medium shall remain within the shaded area shown in Fig. 9 (in the range from +20(±4)°C to –20(±2)°C). The scaled material is collected, dried and weighed after **56** freeze-thaw **cycles**; in some cases the test should end after 28 cycles, e.g. for precast concrete products (paving blocks [36], paving flags [37], kerb units [38]) or after 112 cycles in order to obtain the results after longer exposure. The mean mass of scaled material per unit area after 28, 56 and 112 cycles is used for evaluating the scaling resistance, according to the criteria presented in Tab. 4.

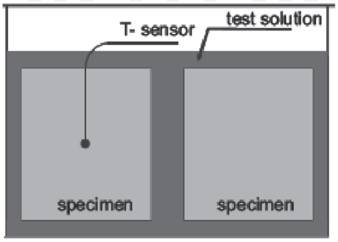
Table 4

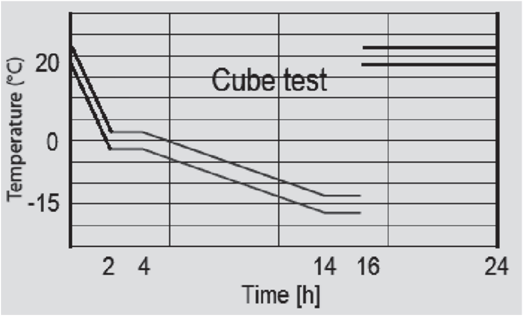
Criteria of the scaling resistance evaluation [15]

Requirements	**Scaling resistance**
$m_{56} < 0.1$ kg/m^2	very good
$m_{56} < 0.2$ kg/m^2 or $m_{56} < 0.5$ kg/m^2 and $m_{56}/m_{28} < 2$ or $m_{112} < 0.5$ kg/m^2	good
$m_{56} < 1.0$ kg/m^2 and $m_{56}/m_{28} < 2$ or $m_{112} < 1.0$ kg/m^2	admissible
$m_{56} > 1.0$ kg/m^2 and $m_{56}/m_{28} > 2$ or $m_{112} > 1.0$ kg/m^2	inadmissible

5.2. CEN/TS 12390-9 test method (Cube Test)

In the **Cube** Test, described in standard **CEN/TS 12390-9** [34], cube specimens immersed in **3% NaCl solution** are subjected to freeze-thaw attack and the **mass loss** of the cubes after 56 freeze-thaw cycles is evaluated.





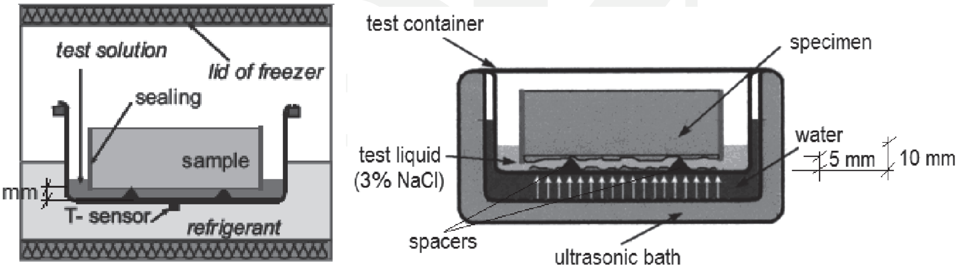
CUBE TEST			
Points specifying the shaded area			
t [h]	T [C]		
	Upper limit	Nominal value	Lower limit
0	+22	+20	+18
2	+2	0	-2
4	+2	0	-2
14	-13	-15	-17
16	-13	-15	-17

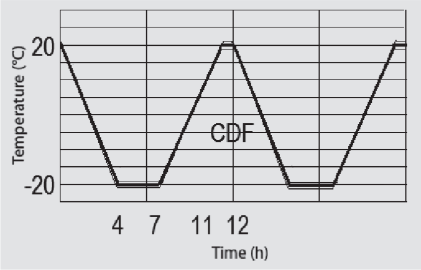
Fig. 10. Test of the concrete resistance to freezing and thawing in the presence of 3% NaCl. CEN/TS 12390-9 (Cube Test) method – Test set-up [34, 40]

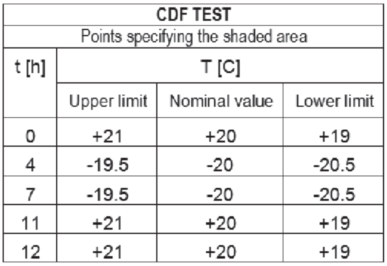
The test requires four **100 mm** cubes. After 27 days (1 day in the moulds, covered by a polyethylene sheet, 6 days in tap water at +20(±2)°C and 20 days in the climate chamber /+20(±2)°C and 65(±5)% of relative humidity/) the specimens are placed in two containers (2 containers with 2 cubes each = 4 cubes). The face which was uppermost during casting should be perpendicular to the base of the container. Each of two containers is filled with the freezing medium for 24 hours. The mass of freezing medium absorbed by each specimen in 24 hours is measured and the mean value of the four cubes is calculated. After 28 days the containers containing the cubes immersed in the freezing medium are placed in the freeze-thaw chests and freeze-thaw cycles are applied. After **56 cycles** of freezing and thawing (or 7, 14, 28, 42 cycles) (16 h air freezing between +20(±2)°C and -15(±2)°C and 8 h thawing in a water bath at +20(±2)°C /the range of temperature: within the shaded area shown in Fig. 10/) the percentage of mass loss is calculated for every two cubes in each container and the mean value of the two containers is determined to evaluate the scaling resistance. The tested concrete may be regarded as scaling resistant if the percentage loss of mass after 56 freeze-thaw cycles is less than 5%, as suggested by Boos and Giergiczny (who tested a range of concretes containing various cement types) [5].

5.3. CEN/TS 12390-9 test method (CDF test)

In the **CDF** Test (**C**apillary suction of **D**e-icing solution and **F**reeze thaw Test) (Fig. 11), presented in standard **CEN/TS 12390-9**, the scaling resistance is evaluated by the measurement of concrete mass scaled from specimens after 28 freeze-thaw cycles. The **mass** per 1 m^2 of the **test surface** is determined.





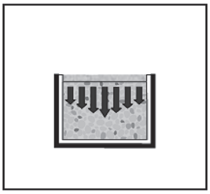
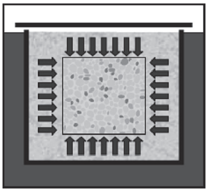
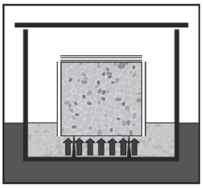


CDF TEST			
Points specifying the shaded area			
t [h]	T [C]		
	Upper limit	Nominal value	Lower limit
0	+21	+20	+19
4	-19.5	-20	-20.5
7	-19.5	-20	-20.5
11	+21	+20	+19
12	+21	+20	+19

Fig. 11. Test of the concrete resistance to freezing and thawing in the presence of 3% NaCl. CEN/TS 12390-9 (CDF Test) method – Test set-up [34, 40, 43]

Table 5

Overview of test methods according to CEN/TS 12390-9 [4, 5, 15, 34, 40]

Test parameter	**SLAB Test (reference method)**	**CUBE Test (alternative method)**	**CDF Test (alternative method)**
			
Curing conditions	1 day in the mould W – 6 days A – 21 days H – 3 days L – without contact	1 day in the mould W – 6 days A – 20 days H – without contact L – 1 day	1 day in the mould W – 6 days A – 21 days H – without contact L – 7 days
	W – under water A – in standard climate of 20°C/65% relative humidity H – in contact with water L – in contact with the freezing medium (3% NaCl) before freezing and thawing		
Number of specimens	4		5
Specimen dimensions [mm]	150 × 150 × 50	100 × 100 × 100	150 × 140 × 70
Test surface	cut (centre of the cube)	formed	
Tested specimen age	min 31 days	28 days	min 35 days
Test direction	one surface of the specimen tested from the top downwards	all surfaces of the specimen	one surface of the specimen tested from the bottom upwards
Tmax/Tmin	+20(±4)°C/-20(±2)°C	+20(±2)°C/-15(±2)°C	+20(±1)°C/-20(±0.5)°C
	measured in the test solution at the centre of the test surface	measured in the centre of the cube	measured below the test vessel
Freezing medium (test liquid/ solution)	3% NaCl		
Duration of one freeze-thaw cycle	24 hours		12 hours
Number of freeze-thaw cycles	56 cycles		28 cycles
Collecting the scaled material			ultrasonic bath for 3 minutes
Test criterion	mass of scaled material per 1 m^2 of concrete surface [kg/m^2]	the percentage loss of weight (mass) [% by mass]	mass of scaled material per 1 m^2 of concrete surface [kg/m^2]
Limit value [4, 5, 15]	< 1 kg/m^2 after 56 cycles	< 5% by mass after 56 cycles	< 1,5 kg/m^2 after 28 cycles

The **CDF** Test requires 5 specimens with the dimensions of **150 × 140 × 70 mm** (prepared by splitting a 150 mm cube mould with plates which have hydrophobic surfaces and a thickness of less than 5 mm; see the standard). The specimen are stored during 28 days (1 day in the mould, covered by a polyethylene sheet, 6 days in tap water at +20(±2)°C and 21 days in the climate chamber /+20(±2)°C and 65(±5)% of relative humidity/). After 21–26 days of storing, the side surfaces are either covered with aluminum foil glued with butyl rubber or sealed with epoxy resin (after this treatment the specimens are returned to the climate chamber). After 28 days each specimen is placed in the test container on the 5 mm high spacers with the test surface downwards (see Fig. 11). The freezing medium (**3% NaCl solution**) is poured into the container to the height of 10 mm. The pre-saturation by capillary suction lasts 7 days at the temperature of +20(±2)°C. Then, a number of 12 hour freeze-thaw cycles are applied. Two freeze-thaw cycles are carried out once a day. Starting at +20(±1)°C, the temperature is lowered to –20(±0.5)°C in 4 h and kept constant for 3 h. Then the temperature is increased to +20(±1)°C in 4 h and kept constant for 1 h (Fig. 11). After **28** freeze-thaw **cycles** (or 14 cycles), when the temperature is above 15°C, the specimen is placed in a ultrasonic bath for 3 minutes to remove any loose particles from the test surface. The scaled material is collected using a paper filter (see the standard). The mass of dried scaled material after 28 freeze-thaw cycles is determined in relation to the concrete surface exposed to the NaCl solution [kg/m^2]. The mean value of the five specimens is calculated to evaluate the scaling resistance. The tested concrete is considered to be scaling resistant, as proposed by Boos and Giergiczny [5], and also Beblacz and Kaminski [4], if the mass of scaled material per unit area after 28 freeze-thaw cycles is less than 1.5 kg/m^2.

The overview of the test methods presented in CEN/TS 12390-9 [4, 5, 34] is shown in Tab. 5.

5.4. ASTM C 672 test method

The ASTM C 672 method [32] is similar to the CEN/TS 12390-9 – Slab test.

ASTM C 672 requires two specimens (the surface area of at least 0.045 m^2 and the thickness of at least 75 mm, e.g. 300 mm × 200 mm × 90 mm [14]). The specimen is demoulded after 24 hours and subjected the standard moist curing for 13 days. Thereafter, the specimen is stored in air for 14 days (at standard room temperature of 23(±2)°C and 45–55% relative humidity). When the concrete is 28 days old, it is covered with **4% $CaCl_2$ solution** (cover thickness of 6 mm) [15].

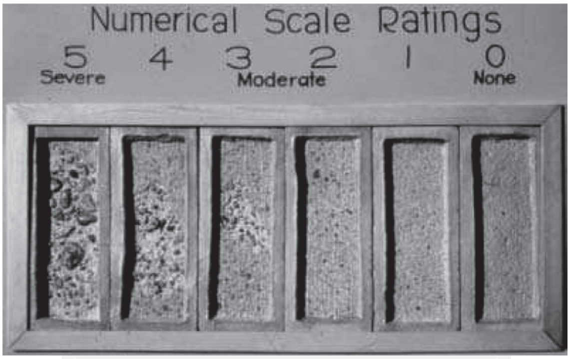
The specimen is subjected to **50 freeze-thaw cycles**. Each cycle lasts 24 hours (16 to 18 h of freezing at around **-18**(±2)°C and 6 to 8h of thawing at **+23**(±2)°C and 45–55% relative humidity). Every five cycles the salt solution is washed off and replaced. A **qualitative** surface assessment (**visual evaluation** from 0 to 5, where “0” indicates “no scaling” and “5” indicates “severe scaling”) is done in accordance with the classification presented in Tab. 6. In many laboratories loose scaled material is collected, dried and weighed at regular intervals during the test for a **quantitative** assessment of scaling apart from the subjective judgment of visual rating.

The ASTM C 672 exposure regime is reported to be harsh (4% $CaCl_2$ solution!) [4, 15].

Table 6

Test of concrete scaling – ASTM C 672 visual assessment [6, 15]

Rating	Condition of surface
0	No scaling
1	Very slight scaling (max 3 mm in depth, no coarse aggregate visible)
2	Slight to moderate scaling
3	Moderate scaling (some coarse aggregate visible)
4	Moderate to severe scaling
5	Severe scaling (coarse aggregate visible over entire surface)



6. Conclusions

Chloride ingress in concrete structures induces corrosion and consequent concrete cracking, spalling, scaling, etc. The long service life of coastal structures, highways, bridges, pavements, etc. requires the concrete used to build them to be resistant to chlorides.

The paper provides details of the test methods (laboratory and applicable in-field) used to determine the chloride permeability of concrete, chloride ion content in concrete and concrete scaling due to cyclic freezing and thawing in the presence of NaCl solution (de-icing salts, saline sea water, etc.).

The most common laboratory test methods used to determine the resistance of concrete to the penetration of chloride ions: the NT Build 492 (NordTest) – the Rapid Chloride Migration Test (RMT) (popular in Europe, including the Polish laboratories) and the ASTM C 1202/ AASHTO T 277 Test – the Rapid Chloride Permeability Test (RCPT) (widespread in the USA and Canada) are described in the paper, specifying the major differences between them (in concentration of the NaCl solution: 3% – RMT, 10% – RCPT, in voltage: 30 V – RMT, 60 V – RCPT and in the test duration: 6 – 96 hours, usually 24 hours – RMT, 6 hours – RCPT), and emphasizing the fact that the tests are non-steady state tests (in reality, chloride penetration into concrete is a non-steady-state process). The paper also points out to certain disadvantages of the Rapid Chloride Permeability Test: the charge passed is related not only to chloride ions, but to all the ions present in the pore solution (e.g. Cl^-, OH^-, SO_4^{2-}, Na^+, Ca^{2+}, etc.) whose concentration may vary in different pozzolanic concretes and the high voltage applied (60 V) leads to an increase in temperature, which affects the amount of charge measured in the test. Nevertheless, the Rapid Chloride Permeability Test is widely used because it is so quick. The special testing set-up (e.g. the Proove'it system) applied for a quick and easy "in situ" diagnosis of the resistance of concrete to chloride ion penetration (in existing and newly constructed concrete bridges, pavements, marine structures, etc.) is based on the procedures presented in the RCPT. The Proove'it system can be used either to measure the total charge passed (in the RCPT) or the depth of chloride penetration (in the RMT). Besides, the paper describes the "in situ" test (fast and easy to perform) for chloride ion content in concrete which can be carried out with e.g. the Rapid Chloride Test (RCT) kit.

Moreover, the paper reviews the test methods for estimation of concrete resistance to scaling during freezing and thawing in the presence of de-icing salts: the Slab Test (based on the criteria in the Swedish standard SS 137244 – the Boraas method) – as the reference method and the Cube Test and the CDF Test (Capillary suction of De-icing solution and Freeze thaw Test) as alternative methods described in the European standard CEN/TS 12390-9 as well as the ASTM C 672 method. All the above-mentioned methods are applicable to the specimens cast in a laboratory or drilled from field structures. It should be noted that there is no correlation between the results obtained from these test methods, because the test procedures are different.

In all the three European standardized methods, the temperature range of freezing and thawing (from +20°C to –20°C) is identical. Another factor that is comparable are curing conditions. The methods differ in all the other parts of the procedure regarding concrete preservation before the test, the properties of test specimens (age, number, shape, dimensions, production/casting, etc.), the way in which the test surface is prepared, the characteristics of freeze-thaw cycles (number, duration, upper and lower limits for temperature, evolution of temperature, etc.), the surface of the test specimen in contact with the test liquid (NaCl) (upper or lower face or fully immersed test specimen), the type of thaw salt (NaCl or $CaCl_2$; most typically 3% NaCl solution is used as it is most critical for the degree of surface scaling), the position of the temperature sensor (at the centre of the test surface, in the centre of the specimen, below the test vessel), the way in which the scaled material is collected (by rinsing or ultrasonic cleaning), the way in which the test specimens are cooled off (with air or liquid), etc. (Tab. 5) [40]. In all the three tests there is no recommendation for limit values. However, the paper specifies the suggested limit values presented in the literature on the subject [4, 5, 15].

The paper also attempts to provide a brief analysis of each test method. The Slab Test is used to test e.g. concrete paving elements (the freezing in this method progresses verically from top to bottom, i.e. in the way that resembles the naturally occurring freezing cycles). The main disadvantage of the Slab Test and the Cube Test methods is the long duration of the study (56 days). In the case of the Slab Test the short duration of action of the lowest temperature entails too mild testing conditions (in cold regions, the temperature of a concrete structure may remain at sub-freezing levels for longer periods of time). In the CDF Test method the scaling resistance is evaluated quite soon – after 14 days (28 freeze-thaw cycles, each of 12 hours). In all three European standardized methods the process of sample preparation is laborious. The paper also illustrates several shortcomings of the ASTM C 672 method. The ASTM C 672 method has been criticized because the salt solution is not covered with a plastic sheet and thus not protected against evaporation, the time of freezing and thawing is not precisely specified, the exposure regime is harsh (50 freeze-thaw cycles, the presence of 4% $CaCl_2$ solution), and the visual, qualitatively evaluation of scaling is a subjective assessment (the rating scale can only be used for a relative comparison of concretes whereas the assessment of scaling in the European standard CEN/TS 12390-9 methods is quantitative). However, the tests have also been criticized for the short period of pre-saturation, which can be insufficient for concrete with e.g. slag cement (because it can produce the osmotic effect).

A deterioration process takes years in the field, but in the laboratory it may take only weeks. Natural conditions are varied, complex and extremely difficult to reproduce. Tests usually amplify the severity of exposure conditions and consequently the damage of the material. It is hard to represent true field conditions in a laboratory test. The basic reason for the severity of most laboratory tests is unlimited access to water at all time, which is not always the situation in the field. The moisture content of the specimens and also the maturity of concrete at the test beginning are important factors related to concrete durability.

A comparative analysis of the test results derived from both laboratory and field studies may indicate the most suitable method for a given type of concrete. The choice of method should depend, on the one hand, on the environmental conditions in which the material works and, on the other hand, it should be based on the expertise obtained in the course of concrete laboratory testing [24–26]. The more experience is gained with the tests, the more appropriate procedures for determining chloride permeability are invented. So, modified and new test methods are undoubtedly to be expected in the nearest future.

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