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Comparison of Diffusion and Migration Coefficients Determined from Tests on Concrete in Prestressed Floor Slabs HC-500

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Abstract. Protective properties of concrete for steel can be assessed with a diffusion coefficient. This method refers to steel exposed to chloride ions present in sea water or deicing agents. However, the aggressive environment with chlorides hinders the determination of diffusion coefficient as they enter into electrostatic interaction with other ions present in concrete pore water, adsorb hardened cement grout on the surface of hydrated minerals, and react with concrete components. But Fick's laws can be used to express chloride ion flow in concrete regardless of complex physical, chemical and electrostatic processes. Fick's laws are predominantly used to calculate values of that coefficient values with reference to methods defined by standards AASHTO T 259 and NT BUILD 443. The main disadvantage of such methods is their duration. Therefore, an electric field is used to speed up a flow of chloride ions. On the basis of obtained results, we can either calculate a passed charge or a migration coefficient. The determined coefficient of migration according to NT BUILD 492 characterized chlorides ingress into concrete when exposed to the electric field. But this method did not specify the time, after which the critical value of chloride ions was achieved at the reinforcement. Also, the method described in the standard ASTM C 1202-97 is only useful for comparing protective properties of concrete. However, the migration of chlorides could be analysed with the theoretical model, which was the base to derive the equation of migration. This paper presents experimentally determined averaged results and values of diffusion coefficients determined from migration tests. It also compares results from diffusion tests performed in accordance with standards AASHTO T 259 and NT BUILD443, and results from migration tests conducted in accordance with standards ASTM C 1202-97 and NT BUILD 492.

1. Introduction

Diffusion coefficient is the main criterion for assessing protective properties of concrete for steel in the environment exposed to chloride ions originating from the sea water or deicing agents. Unambiguous determination of diffusion coefficient in the aggressive environment containing chlorides is difficult due to electrostatic interaction with other ions in concrete pore water, adsorption of hardened cement grout on the surface of hydrated minerals, and reaction with concrete components. Despite such complex physical, chemical and electrostatic processes, Fick's laws are usually used to describe chloride ion flow in concrete. In most cases, Fick's laws are the base for calculating coefficient values and standard methods ASTM C 1556 – 03 [1]; NT BUILD443 [2]. The most common present practice to measure the chloride penetration depth in an immersion test is the measuring of the chloride profile. The fitting of this profile to the so called error function equation



enables the calculation of a diffusion coefficient. A long testing time is disadvantageous, thus the flow of chloride ions is accelerated by the electric field. Results from such tests are used to determine either the value of passed charge according to ASTM C 1202 – 97 [3] or the value of migration coefficient according to NT BUILD 492 [4]. Another practice that is easier and quicker to perform is the colourimetric method, based on spraying a silver nitrate aqueous solution (0.1 M AgNO₃) on the cross section perpendicular to the penetrated surface. In the absence of chloride, the concrete becomes dark-coloured, while in presence of chlorides the colour obtained is white. This method has been used also to calculate the diffusion coefficient from migration experiments [10]. The use of this colourimetric method poses some questions because the colourimetric front seems to correspond to free chlorides in solution, and there is no agreement concerning the limit at which the colour change takes place (0.15% and 0.01% free chlorides by weight of cement [10]. Migration coefficient determined according to NT BUILD 492 [4] quite satisfactory describes the process of chloride penetration into concrete under the impact of the electric field. However, we are unable to determine time, after which concentration of chloride ions reaches the critical value at the reinforcement surface under the structure normal operation, as chloride ion penetration into concrete is described by the diffusion process. Similarly, tests performed according to ASTM C 1202-97 [3] can be only used to compare protective properties of concrete. ASTM C 1202-97 standard [3] states that the accelerated method of charge measurement can be in practice used for concrete, for which quite close correlations were established between the test procedure and results from the test performed according to ASTM C 1556 – 03 [1]. In concrete with an additive of calcium nitrate, some discrepancies were observed between results from the accelerated procedure. The reason was the fact that higher electric charge flowing through concrete with Ca(NO₃)₂ was measured with additional ions present in the additive. Some inaccuracies between the measured charge and the amount of chlorides penetrating the specimen indicated the possible occurrence of discrepancies in evaluating permeability of concrete with fly ash, metallurgical slag or with other additives reducing water content in concrete [3]. The test results showed that the values of diffusion coefficient determined from the previous test procedures differed in values and the order of their magnitude depending on the applied test procedure and the measuring setup. Differences in the values of diffusion coefficient determined on the basis of chloride concentrations changing over time, indicated the need to use theoretical models of thermodynamics for analysing the process. Such models are used to determine the migration equation and express the converse tasks of that equation [5]. The theoretical thermodynamic model was used in the paper [5], to analyse chloride migration. As a result, the equation of migration was obtained and the converse task of the migration equation was expressed. Therefore, experimentally determined results were averaged on theoretical grounds and values of diffusion coefficients were determined from migration tests. In the paper [6], the accelerated tests performed on chloride ion migration into cylindrical elements drilled directly from the top surface of prefabricated and prestressed floor slabs HC500-19/R120, were used for defining the diffusion coefficient of chloride ions in concrete, from which those slabs were produced. The obtained value of diffusion coefficient $\bar{D}_s = 0,72 \cdot 10^{-12} \text{ m}^2/\text{s}$ classifies the tested concrete to the group of very low chloride permeability [5, 6, 7, 8]. This paper describes how cylindrical specimens drilled directly from floor slabs, were subjected to diffusion tests carried out in accordance with ASTM C 1556 – 03 [1] and NT BUILD443 [2] standards, and migration tests in accordance with to ASTM C 1202-97 [3] and NT BUILD 492 [4] standards. The value of diffusion coefficient determined from diffusion tests and standard methods was close to the diffusion coefficient determined from the thermodynamic migration model in the non-steady state [9].

2. Test methods and diffusion coefficient calculation

Investigated specimens were sampled directly from the upper part of pre-stressed concrete slabs using a diamond core drilling machine with diameter 8 cm - figure 1(left). Considered precast concrete with w/c = 0.32 and average compressive strength of concrete $f_{ctm} = 65 \text{ MPa}$ was made of C50/C60 concrete based on Portland cement CEM II 52.5 R: 550 kg/m³ and crushed BASALT: 1304 kg/m³ [12].

The test was carried out according to standards NT BUILD 443 [8] and ASTM C 1556 – 03 [9]. Three cylindrical samples with a diameter of 80 mm and a height of 50 mm were insulated at all of their surfaces with resin, except the top, which at the same time was the top surface of prestressed HC-500 concrete slabs. Samples were soaked in limewater as much as possible and then stored in a closed container immersed in 16.5% NaCl solution for 5 weeks. According to ASTM C 1556 – 03 [1], the coefficient D was determined like in the standard NT BUILD 443 [2], by adjusting the chloride concentration diagram which was determined on the basis of calculated distribution of chloride ions to cement weight according to the known diffusion equation with chloride ion concentrations obtained from tests.

$$c_{cal}(x,t) = c_{0,cal} \left(1 - \operatorname{erf} \frac{x}{2\sqrt{Dt}} \right), \quad s = \sqrt{\frac{\sum_{i=1}^n [c_{cal}(x,t) - c(x,t)]^2}{n-1}} \quad (1)$$

where $c_{0,cal}$ is the calculated mass density of chlorides at the edge of the element, erf – the Gauss error function, t- time. To find the most convergent results from calculations and tests, the following data were calculated, s - the mean square error, and n - the number of concrete layers, for which chloride concentration was determined.

The next test was carried out following the standard ASTM C1202-97 [3]. This standard recommends the application of a voltage of 60 V across specimens during a 6-hour period and the measurement of passed electrical charge. Triplicate specimens were tested. Tests were performed using Chloride Penetrability PROOVE'it System instruments of German Instruments company - Figure 1 (right). They were carried out on concrete discs with the thickness of 50 mm and the diameter of 80 mm, drilled out directly from the top surface of HC-500 floor slabs, which were trimmed at both sides to even the surface. Specimens were placed in the migration chamber between two (copper) electrodes. One part of the chamber was filled with 3% NaCl solution, and the second part with 0.3N NaOH solution. The specimen was stored under dry air conditions for one hour, under vacuum conditions for three hours (pressure < 1 mm Hg), then it was soaked with limewater under the same vacuum conditions for one hour. After that time, the specimen was finally placed in the test chamber. Prior to commencement of tests, specimens were kept in water for 18 hours. Concrete was tested depending on the charge passing through the specimen. The charge Q flowing through the tested specimen was measured on the basis of current intensity recorded every 30 minutes for 6 hours of the test procedure.

$$Q = 900(I_0 + 2I_{30} + 2I_{60} + \dots + 2I_{300} + 2I_{330} + I_{360}), \quad (2)$$

where I_0 is current intensity measured directly after applying voltage, I_{30} , ..., I_{360} – current intensity after time $t = 30$ min., ..., $t = 360$ min. from the moment of voltage application.



Figure 1. (Left) Sampling of concrete specimens from HC500 floor slabs. (Right) Migration laboratory setup for tests ASTM C 1202-97 [3] of concrete specimens [15].

The method by Tang and Nilson [11] for determining the diffusion coefficient has been adopted in NT BUILD 492 [4] as the standard test. Tests were performed on the test stand shown in Figure 1 (left). Three cylindrical samples having 80 mm in diameter and 50 mm in height were tested; side surfaces of the specimen were covered with resin to ensure unidirectional flow of chloride ions. Cylinders containing 10% NaCl solution were tightly attached to the top surface of cylindrical concrete samples and cathodes made of stainless steel were placed inside and adjusted to the cross-section of cylindrical elements. Three samples connected in parallel in an electric circuit were simultaneously tested on a damp sponge, under which there was a platinum-coated titanium anode in solution of 0.3 M NaOH. After saturating samples with limewater, the system was connected to a DC voltage source $U = 30$ V.



Figure 2. (Left) Migration laboratory setup for tests NT-Build 492 [4] of concrete specimens. (Right) Depth of chloride penetration x_d determined with the colourimetric method

The duration of the test and the value of applied voltage depended on current intensity specified at the beginning of tests. That value was adopted in accordance with the standard table [4]. Chloride migration studies were measured at 96 hours with a DC voltage source $U \approx 50$ V. After switching off the electrical field, one of three specimens were split in two halves and the penetration of chlorides x_d was measured over one of the halves using the colourimetric method – Figure 1(right). According to the standard [4], the migration coefficient was calculated from the following expression:

$$D_T = 1,189 \cdot 10^{-11} \left(\frac{x_d - \alpha \sqrt{x_d}}{t_d} \right), \quad \alpha = 2 \sqrt{\frac{RTL}{zFU}} \operatorname{erf}^{-1} \left(1 - \frac{2c_d}{c_0} \right), \quad (3)$$

where c_0 and c_d are chloride concentrations in the chamber and in concrete at the depth, x_d , t_d – test duration, hour, L – thickness of the specimen, mm, U – absolute value of the applied voltage.

The thermodynamical model thoroughly described in [5] is based on the theoretical extraction of a representative volume element of concrete. This volume element contains a concrete skeleton, pores, and the aqueous solution. In the model, the skeleton and water particles (solvent) are assumed to be the inert component $\alpha = 0$, which is not directly involved in the process. Anions $\text{Cl}^- - \alpha = 1$ and $\text{OH}^- - \alpha = 2$ and cations $\text{Na}^+ - \alpha = 3$, $\text{K}^+ - \alpha = 4$, and $\text{Ca}^{2+} - \alpha = 5$ are components involved in this process. Six specimens were exposed to the electric field, inducing the migration of chloride ions. During the test, containers with 3% solution of NaCl were tightly attached to the top surface of specimens. The level of NaCl solution was maintained at 150 mm over the surface of a specimen. Before the migration test, specimens were kept in distilled water for 72 hours to improve the electrical conductivity of concrete. Specimens were connected to the source of direct current (18 V). The test was conducted in two time intervals: $t_1 = 24$ h and $t_2 = 48$ h. NaCl solution was replaced every 24 hours. During the whole period of measurements, the temperature of the solution was constant and equal to $20^\circ\text{C} \pm 1^\circ\text{C}$.

Quantification results were determined for the mass density ρ^1 of chloride ions migrating in concrete specimens exposed to the electric field. Only free chlorides (soluble in water) were of interest during tests.

After completing migration and diffusion, samples were left in room conditions for 2 days. Then, layers of crumbled concrete having a thickness $g = 2$ mm were sampled from the slab surface at 10 consecutive depth increments using the “Profile Grinding Kit” from German Instruments AS. Crushed concrete from two similar samples was mixed together. Then, distilled water was added in a 1:1 weight ratio. The thick solution was stirred every few hours for one day and then filtered through a medium filter. The leaching process was repeated twice. Model solutions were subjected to chemical analysis by determining chloride ions in the liquid according to PN-ISO 9297 [13] by means of an argentometric titration according to Mohr concentration c^1 [g/m³]. The volume V_s of the experimental liquid was estimated on the basis of water volume weight. During the accompanying tests, the volume weight of concrete was quantified. Quantification was performed for the mass density ρ^1 of chloride ions migrating in concrete specimens exposed to the electric field after 96 hours. For establishing the concentration $c^1(t)$ [%] of chloride ions by weight of cement, the mass density of chloride ions ρ^1 was a point of reference for a constant mass density of cement ρ_{cem} in tested concrete

$$c^1(t) = \frac{\rho^1(x_i, t)}{\rho_{cem}} 100\% \quad (4)$$

Based on the expression determining the diffusion coefficient of chloride ions $D^1 = I/Q$ was the inverse of the diffusion resistance of the entire tested concrete zone within the range a

$$D^1 = \frac{\bar{j}^1(a) a \Delta t}{\frac{z^1 F U g}{R T h} [\bar{\rho}_1^1 + \bar{\rho}_2^1 + \dots + \bar{\rho}_n^1] \Delta t - B}, \quad B = \int_0^a Q_x [\rho^1(x, t + \Delta t) - \rho^1(x, t)] dx. \quad (5)$$

In this expression, $\bar{j}^1(a)$ is the value of the mass flow of chloride ions passing through the plane situated at a distance $x = a$, $\bar{\rho}_1^1, \bar{\rho}_2^1, \dots, \bar{\rho}_n^1$ are mass densities of ion Cl^- at midpoints of consecutive intervals $[0, g], [g, 2g], \dots, [(n-1)g, a]$ averaged in time Δt . The first component of the denominator defines the steady part of chloride ion flows, while the second component B – defines the non-steady part. In this expression, z^1 is the ion valence, $R = 8.317$ J/mol·K – universal gas constant, $F = 96\,487$ C/mol – the Faraday constant, U – the voltage between electrodes, h – the specimen height [5].

3. Results and discussions

The value of diffusion coefficient $D_{ns}^1 = 1.44 \cdot 10^{-12}$ m²/s was determined according to ASTM C 1556 – 03 [1] and NT BUILD 443 [2] by adjusting the chloride concentration diagram. It was obtained from the calculated distribution of chloride ions to cement weight according to the diffusion equation (1) with chloride ion concentrations determined from 35-day long tests on the basis of the lowest value of the mean square error – Figure 3 (left).

Tests were conducted in accordance with the standard [3], thus the current intensity was recorded every 30 minutes. Values of measured I and the charge Q flowing through the specimens calculated from the expression (2) are shown in Table 1. On the basis of the charge value and in accordance with the standard [3], the tested concrete can be considered as having very low permeability. According to the standard [3], concrete with very low permeability is characterized by the value of charge flowing through concrete within the range $(100 \div 1000)$ C, and it is usually latex-modified concrete with internal sealing.

Table 1. Results from measured current intensity I and calculated charge Q for chloride migration tests according to the standard [3]

Time	Specimen 1 I mA	T °C	Specimen 2 I mA	T °C	Specimen 3 I mA	T °C
0	16.5	21	18.0	19	16.8	21
0:30	16.4	21	17.9	20	16.9	21
01:00	16.3	21	17.4	20	16.8	21
01:30	16.0	22	16.6	20	16.6	21
02:00	16.1	22	16.5	20	16.5	21
02:30	16.1	22	16.4	20	16.6	21
03:00	16.0	22	16.4	21	16.5	21
03:30	15.9	22	16.2	21	16.4	22
04:00	15.9	22	16.0	21	16.4	22
04:30	15.8	22	16.1	21	16.3	22
05:00	15.7	23	16.0	21	16.2	22
05:30	15.6	23	16.0	21	16.2	22
06:00	15.6	23	16.0	21	16.1	22
Q (C)	352		366		364	

The value of migration coefficient $D_T = 0.6 \cdot 10^{-12} \text{ m}^2/\text{s}$ was calculated according to the equation (3) NT BUILD 492 [4] after defining the depth of chloride penetration $x_d = 10 \text{ mm}$ using the colourimetric method – Figure 1 (right). The diagram of chloride concentrations from 96-hour migration tests shows that the depth of chloride penetration $x_d = 10 \text{ mm}$ corresponds to chloride concentration by cement weight equal to 0.62% – Figure 2.

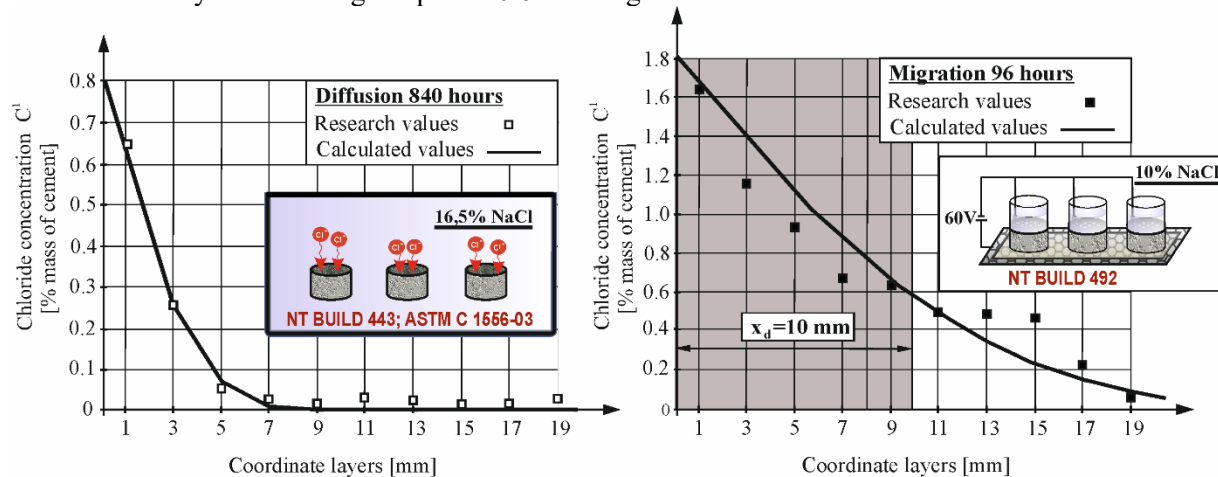


Figure 3. (Left) Comparison of the profile of measured and calculated chloride concentration – measurements carried out 840 hours after the diffusion experiment. Diffusion laboratory setup for tests ASTM C 1556 – 03 [1] and NT BUILD 443 [2] of concrete specimens. (Right) Comparison of the profile of measured and calculated chloride concentration – measurements carried out 96 hours after migration experiment. Migration laboratory setup for tests NT-Build 492 [4].

Moreover, the value of migration coefficient $D_{ns}^1 = 1.45 \cdot 10^{-10} \text{ m}^2/\text{s}$ was determined by adjusting the diagram of chloride concentration. It was obtained from the calculated distribution of chloride ions to cement weight according to the diffusion equation (1) with chloride ion concentrations determined from 96-day long migration tests on the basis of the lowest value of the mean square error – Figure 3 (right). Similarly, the value of migration coefficient $D_{mig}^1 = 1.2 \cdot 10^{-11} \text{ m}^2/\text{s}$ was determined from 24- and

48-hour migration tests. Differences between obtained values can be explained as the result of different concentrations of the reference solution, different durations of the experiment and different values of the current applied to accelerate ion flow – Figure 4.

On the basis of the measured mass distribution ρ^1 of chloride ions migrating in concrete under the electric field, the reliable value of diffusion coefficient was determined from the relationship (5). Values of diffusion coefficient achieved for the theoretical model (based on migration results) ranged from $D = 0.72 \cdot 10^{-12}$ m²/s for steady state to $1.45 \cdot 10^{-12}$ m²/s, taking into account the non-steady course of the migration, classified the tested concrete to the group with very low chloride permeability [5, 6, 7,8].

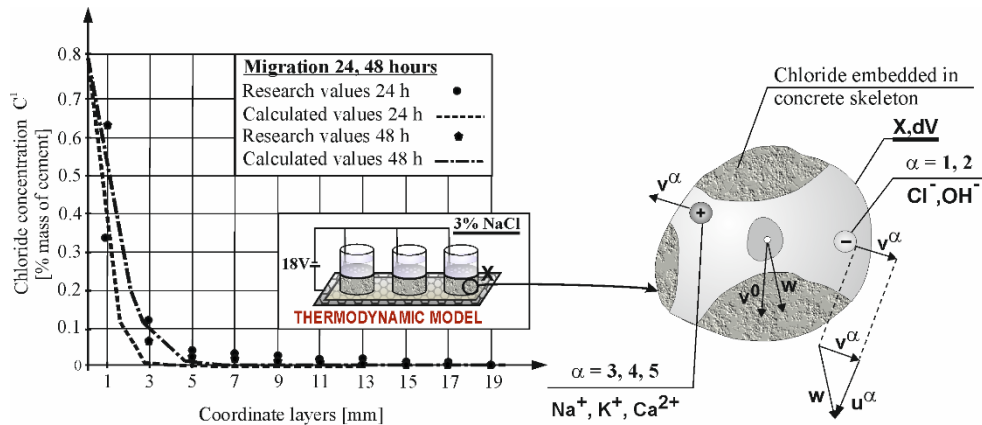


Figure 4. (Left) Comparison of the profile of measured and calculated chloride concentration – measurements carried out 24 and 48 hours after the migration experiment. Migration laboratory setup for tests of the thermodynamic model [5] of concrete specimens. (Right) The model of ion penetration into concrete and the process components in the representative volume element.

Table 2 presents values determined according to standards [1, 2], the thermal diffusion model [5], coefficients of diffusion and migration [4], values of the mean square error, and the charge measured in accordance with [3].

Table 2. Values of determined coefficients of diffusion and migration, values of the mean square error and the measured charge

Test procedure	Measuring method	Mean square error s	Coefficient of diffusion, migration D (m ² /s)	Electric charge Q (C)
ASTM C 1556 – 03 [1],				
NT BUILD 443 [2]	Equation (1)	2.65×10^{-2}	1.45×10^{-12}	-
Diffusion 840 h				
NT BUILD 492 [4]	Equation (1)	0.14×10^{-2}	1.45×10^{-10}	-
Migration 96 h				
NT BUILD 492 [4]	Equation (3)	-	0.61×10^{-12}	-
Migration 96 h				
Thermodynamical model [5]	Equation (5)	-	$(0.72 \div 1.45) \times 10^{-12}$	-
Migration 24 h, 48 h				
Thermodynamical model [5]	Equation (1)	$(3.95 \text{ and } 2.65) \times 10^{-2}$	1.2×10^{-11}	-
Migration 24 h, 48 h				
ASTM C1202-97 [3]	Equation (2)	-	-	361
Migration 6 h				

At present, there are many applicable standard procedures for determining coefficients of diffusion or migration [1, 2, 3, 4]. Each method should be applied separately to compare the resistance of

different types of concrete against chloride ion ingress. Because each method requires different test conditions (concentration of reference solution of chlorides, type of the target solution, different durations of tests, the value of applied voltage in migration tests), coefficients of migration or diffusion determined by different methods, are not identical for the same type of concrete. Thus, the question can arise which method and value we should choose for determining the diffusion coefficient while making a numerical forecast for durability of the structure made of the tested type of concrete. This issue is very important for the predicted durability of structures made of prestressed floor slabs regarding corrosion risk at the interface between steel and concrete. Corrosion can result in a rapid loss in adhesion between steel wires and concrete, and consequently can lead to the sudden destruction of the structure [15]. Because tests accelerated by the electric field are easy and quick to perform, there is a tendency to apply such rapid methods of measuring charge flow, concrete resistance or conductance and converting the obtained values into the diffusion coefficient. However, the need for confirming the above tests with tests conducted under diffusion conditions (as recommended by the standard [3]) is often neglected. And diffusion tests should be conducted, particularly if they are to forecast the durability taking into account determined coefficients [14].

4. Conclusions

This paper describes how diffusion and migration coefficients were determined using the selected methods. Those coefficients were calculated for concrete specimens drilled directly from precast prestressed floor slabs HC500 made of C50/C60 concrete. Values of migration coefficient in accordance with the test procedure specified in NT BUILD 492 were determined using two methods. The first method consisted in determining colourimetrically the maximum depth of chloride penetration and adjusting the chloride concentration diagram prepared on the basis of migration tests. There was a significant difference in values of the migration coefficient. It can be stated that even the same test procedure gives very different values of the migration coefficient when dissimilar calculation methods are used. Like for test procedures performed in accordance with ASTM C 1202-97, we can only compare protective properties of tested types of concrete. These methods cannot be used to determine the time, after which the concentration of chloride ions reaches its critical value on the reinforcement surface in the operational conditions because the ingress of chloride ions to concrete is described by the diffusion process.

However, diffusion coefficient determined in accordance with ASTM C 1556 – 03, NT BUILD 443 quite well describes the process of chloride ion diffusion in the tested concrete. Similar values of diffusion coefficient were obtained both on the basis of standards and the thermodynamic model of migration. The method on the basis of the thermodynamic model of migration is shorter than specified in standards. The reason for shorter time is that this method uses results obtained from the accelerated method and can be applied for standard concrete and concrete with different types of cement, high performance concrete HC and self-compacting concrete SCC with ISP slag.

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