

CONCRETE, HARDENED: ACCELERATED CHLORIDE PENETRATION

UDC 622.43

Key words: Test method, hardened concrete, chloride penetration, non-steady state diffusion

1 SCOPE

This Nordtest method specifies a procedure for the determination of penetration parameters for estimating the resistance against chloride penetration into hardened concrete or other cement-based materials.

The resistance against chloride penetration is determined by accelerated testing.

2 FIELD OF APPLICATION

The method is applicable to test specimens from existing structures and to new samples older than 28 maturity-days. The concrete test specimens must be free from construction faults such as cavities and visible cracks.

It is important to keep in mind that the values for the chloride penetration parameters are dependent on concrete maturity. Especially concretes containing pozzolans will not have reached optimum maturity after a period of 28 maturity-days, which is the specified minimum curing time before exposure.

Deviations from the requirements of the method concerning exposure temperature, exposure time, together with the composition and the chloride concentration of the exposure liquid, can be made where required by the purpose of the test. In case of any deviations, it must be stated in the test report that the results are obtained from a modified test and the deviations must be specified.

Parameters of importance for the resistance against chloride penetration are e.g. composition, workmanship, surfacing, curing, age.

3 REFERENCES

NT BUILD 202, 2nd ed. Approved 1984-05. Concrete, hardened: Sampling and treatment of cores for strength tests.

NT BUILD 208, 2nd ed. Approved 1984-05. Concrete, hardened: Chloride content.

4 DEFINITIONS

Chloride penetration: The ingress of chlorides into concrete due to exposure to external chloride sources.

Exposure temperature: The temperature of the exposure liquid while the test specimen is submerged in it.

Exposure time: The time from immersion of the test specimen in the exposure liquid to profile grinding.

Profile grinding: Grinding off concrete powder in thin successive layers from a test specimen using a dry process.

Maturity-day: A concrete of 28 maturity-days has developed a maturity corresponding to curing for 28 days at 20 °C.

Surface-dry condition: Is achieved by drying the water-saturated test specimen with a clean cloth or similar leaving the test specimen damp but not wet. This is achieved by wetting the cloth with the liquid in which the test specimen has been immersed and then wringing it out sufficiently to absorb any liquid adhering to the surface of the specimen.

5 SAMPLING

This method requires drilled cores or cast cylinders as test specimens. They must be representative of the concrete and/or structure in question. The concrete must be hardened to minimum 28 maturity-days. At least three test specimens should be used in the test. The diameter should be at least $\varnothing 75$ mm, but not less than 3 times the maximum aggregate size. The length should be minimum 100 mm.

6 METHOD OF TEST

6.1 Principle

A water-saturated concrete specimen is on one plane surface exposed to water containing sodium chloride. After a specified exposure time thin layers are ground off parallel to the exposed face of the specimen and the chloride content of the layers, C_x , is measured. The original (initial) chloride content of the concrete, C_i , is measured at a suitable depth below the exposed surface. The effective chloride transport coefficient, D_e , and the boundary condition of the chloride profile at the

exposed surface, C_s , are calculated. This is done by using the related values of measured depth below the exposed surface, x , and measured chloride content, C_x .

The penetration parameter, K_{C_r} , is calculated for a selected chloride concentration, C_r . The influence of D_e , C_s , C_i and C_r is combined in the calculation of K_{C_r} , which facilitates comparison of the results.

6.2 Reagents and apparatus

6.2.1 Reagents

- Redistilled or demineralised water.
- Calcium hydroxide (Ca(OH)_2), technical quality.
- Sodium chloride (NaCl), technical quality.
- 2-component (chloride-ion diffusion-proof) polyurethane or epoxy-based paint (membrane).
- Chemicals for chloride analysis according to applied test method.

6.2.2 Apparatus

- Water-cooled diamond saw.
- Balance, accuracy better than ± 0.01 g.
- Thermometer, accuracy better than ± 1 °C.
- Temperature controlled cupboard.
- Plastic container with tight-fitting lid.
- Equipment for grinding off and collecting concrete powder from thin concrete layers (less than 2 mm).
- Equipment for crushing concrete.
- Standard sieve, mesh width 1.0 mm.
- Equipment for chloride analysis according to applied test method.
- Slide caliper, accuracy better than ± 0.1 mm.

6.3 Preparation of test samples

From each of the concrete cones or concrete cylinders, the parts 6.3.1 and 6.3.2 specified below are cut off by means of a water-cooled diamond saw.

6.3.1 Test specimen for exposure in NaCl solution

If a drilled core is used, the test specimen is prepared by cutting off the outermost approx. 70 mm of the core. A test specimen is thus obtained, of which one end face is the original surface and the other is a sawn face. The outermost approx. 10 mm is then cut off the original concrete surface (note 1), and the resulting sawn surface is exposed in the NaCl solution.

Note 1: It is very important that the test is made on the concrete between the surface and the layer of reinforcement because it is here that the protection against chloride penetration is needed. Furthermore, the quality of the concrete in this particular area can deviate from the rest of the concrete. The outermost approx. 10 mm of concrete is removed to ensure that the measurement is made in an area with an approximately constant cement matrix content.

If a cast cylinder is used, the test specimen is prepared by dividing the cylinder into halves by a cut perpendicular to the axis of the cylinder. One half is used as test specimen, with the sawn surface exposed in the NaCl solution.

The test specimen is immersed in a saturated Ca(OH)_2 solution at about 23 °C in a tightly closed plastic container. The container must be filled to the top to minimize carbonation of the liquid. The next day the mass in surface-dry condition (m_{sd}) is determined by weighing the test specimen.

The storage in the saturated Ca(OH)_2 solution continues until m_{sd} does not change by more than 0.1 mass % per 24 hours.

All faces of the test specimen except the one to be exposed are then dried at room temperature to a stable white-dry condition and given an approx. 1 mm thick epoxy or polyurethane coating. Precautions must be taken to ensure that no coating material gets onto the surface to be exposed. It must be ensured that the method of application and hardening prescribed by the supplier of the coating material is observed.

When the coating has hardened, the test specimen is immersed in the Ca(OH)_2 solution until m_{sd} stabilizes as described above.

6.3.2 Slice of at least 20 mm thickness

From the remainder of the drilled core or cast cylinder a slice of at least 20 mm thickness is cut in extension of test specimen 6.3.1.

6.4 Procedure

6.4.1 Exposure liquid

An aqueous NaCl solution is prepared with a concentration of $165 \text{ g} \pm 1 \text{ g NaCl per dm}^3$ solution. This exposure liquid is used for 5 weeks and then replaced by a new pure NaCl solution. The NaCl concentration of the solution must be checked at least before and after use.

6.4.2 Exposure temperature

The temperature of the water bath must be 21–25 °C with a target average temperature of 23 °C. The temperature must be measured at least once a day.

6.4.3 Exposure

The Ca(OH)_2 solution in the container used for water saturation is replaced with the exposure liquid and the test specimen 6.3.1 is immersed in surface-dry condition in the saline solution. It is important that the container is completely filled with the exposure liquid and closed tightly. The ratio between the exposed area in cm^2 and the volume of exposure liquid in dm^3 shall be minimum 20 and maximum 80. The container is placed in the temperature controlled cupboard during exposure. The exposure shall last for at least 35 days, and the container is

shaked once every week. The date and time of exposure start and exposure stop is recorded.

6.4.4 Profile grinding

The chloride profile is measured immediately after the exposure by grinding off material in layers parallel to the exposed surface. The grinding is performed within a diameter approx. 10 mm less than the full diameter of the core. This obviates the risk of edge effects and disturbances from the coating.

At least eight layers must be ground off. The thickness of the layers must be adjusted according to the expected chloride profile, so that minimum 6 points covers the part of the profile between the exposed surface and the depth with a chloride content of $C_i + 0.03$ mass %. However, the outermost layer must always have a thickness of minimum 1.0 mm.

It must be ensured that a sample of at least 5 g of dry concrete dust is obtained from each layer. For each sample of concrete dust collected, the depth below the exposed surface is calculated as the average of five uniformly distributed measurements using a slide caliper.

6.4.5 Chloride analysis

The acid-soluble chloride content of the samples is determined to three decimals in accordance with NT BUILD 208 or by a similar method with the same or better accuracy. The accuracy must be documented.

6.4.6 Initial chloride content

From the concrete slice 6.3.2, a representative subsample of approx. 20 g is prepared, e.g. by crushing until the material passes a 1 mm standard sieve, followed by splitting. The acid-soluble chloride content of the subsample is determined to three decimals by using the method described in 6.4.5. The measured chloride content is the initial chloride content of the specimen, C_i .

6.5 Expression of results

6.5.1 Test results

The values of C_s and D_e are determined by fitting the equation (1) to the measured chloride contents by means of a non-linear regression analysis in accordance with the method of least squares fit. The first point of the profile determined from the sawn face is omitted in the regression analysis. The other points are weighted equally.

$$C(x,t) = C_s - (C_s - C_i) \cdot \operatorname{erf} (x\sqrt{4 \cdot D_e \cdot t}) \quad (1)$$

where

$C(x,t)$ [mass %] is the chloride concentration, measured at the depth x at the exposure time t

C_s [mass %] is the boundary condition at the exposed surface

C_i [mass %] is the initial chloride concentration measured on the concrete slice 6.3.2

x [m] is the depth below the exposed surface (to the middle of a layer)

D_e [m²/s] is the effective chloride transport coefficient

t [s] is the exposure time (with an accuracy better than 5 hours)

erf is the error function defined in (2)

$$\operatorname{erf} z = 2\sqrt{\frac{1}{\pi}} \cdot \int_0^z \exp(-u^2) du \quad (2)$$

Tables with values of the error function are given in standard mathematical reference books.

The penetration parameter, K_{C_r} is calculated using the values of C_i [mass % of concrete], C_s [mass % of concrete], D_e [m²/s], and C_r [mass % of concrete] (note 2) according to (3). The C_r -value is set to 0.05 mass % unless another value is required.

$$K_{C_r} = 2\sqrt{D_e} \cdot \operatorname{erf}^{-1} \left(\frac{C_s - C_r}{C_s - C_i} \right) \quad (3)$$

Note 2: In (3) C_r is a selected reference chloride concentration. Note that K_{C_r} only is defined when $C_s > C_r > C_i$.

The test results are:

- The initial chloride concentration, C_i , stated to three decimals in mass % of dry concrete.
- The boundary condition at the exposed surface, C_s , stated to three decimals in mass % of dry concrete.
- The effective chloride transport coefficient, D_e , stated to two significant digits in m²/s.
- The penetration parameter, K_{C_r} , stated to two significant digits in mm/√year. The C_r -value used to calculate K_{C_r} must be clearly stated in the test report.

Please note that the values of C_s and D_e should not be directly used for prediction of chloride penetration under conditions other than those used in the test. (If K_{C_r} is calculated in the unit mm/√s it is multiplied by $5.6157 \cdot 10^6$ to translate the unit to mm/√year.)

6.5.2 Other information of importance

- The measured chloride contents at all points are plotted versus the depths below the exposed surface. The curve for the optimized mathematical model (1) is plotted on the same graph (See Figure 1).
- The correlation between the measured chloride contents and the corresponding chloride contents calculated according to (1) is determined by means of a linear regression analysis.
- The average exposure temperature is calculated. The variation must be illustrated, e.g. by giving the measured temperature curve.
- The average chloride concentration of the exposure liquid is calculated.

6.6 Accuracy

The following variation coefficients (the standard deviation divided by the mean value) can be expected:

$$C_s = 20 \%, D_\theta = 15 \% \text{ and } K_{0,05} = 10 \%$$

6.7 Test report

The test report shall include the following information, if relevant:

- a) Name and address of the test laboratory, and the place at which the tests were performed if different from the laboratory address.
- b) Date and identification number of the test report.
- c) Name and address of the organisation or person who ordered the test.
- d) Purpose of the test.
- e) Method of sampling and other circumstances (date and person responsible for the sampling).
- f) Name and address of the manufacturer or supplier of the tested object.
- g) Name or other identification marks of the tested object.
- h) Description of the tested object including the age of the test specimen.
- i) Date of supply of the tested object.
- j) Date of the test.
- k) Test method.
- l) Conditioning of the test specimens, environmental data during the test (temperature, chloride concentration in exposure liquid, etc.)
- m) Identification of the test equipment and instruments used.
- n) Any deviations from the test method together with other information of importance for judging the result.
- o) Test results.
- p) Inaccuracy or uncertainty of the test result.
- q) Date and signature.

A.1 NON-LINEAR REGRESSION ANALYSIS

The regression analysis is performed by minimising the sum given in (A.1)

$$S = \sum_{n=2}^N \Delta C^2(n) = \sum_{n=2}^N (C_m(n) - C_d(n))^2 \quad (A.1)$$

where

- S [(mass %)²] is the sum of squares to be minimized
- N [-] is the number of concrete layers ground off
- $\Delta C(n)$ [mass %] is the difference between the measured and the calculated chloride concentration of the *n*'th concrete layer
- $C_m(n)$ [mass %] is the measured chloride concentration of the *n*'th concrete layer

$C_d(n)$ [mass %] is the calculated chloride concentration in the middle of the *n*'th concrete layer

Refer to Figure 1 for clarification.

A.2 BACKGROUND INFORMATION

Frederiksen, J. M.: "APM 302 - dansk målemetode for chloridindtrængning i beton" (APM 302 - Danish test method for chloride penetration into concrete). Dansk Beton, No. 2, p. 22-27, 1992. (In Danish.)

Sørensen, H. E.: "Determination of chloride penetration parameters for concrete". Nordtest project 1154-94, Nordtest, SF-02151 ESPOO.

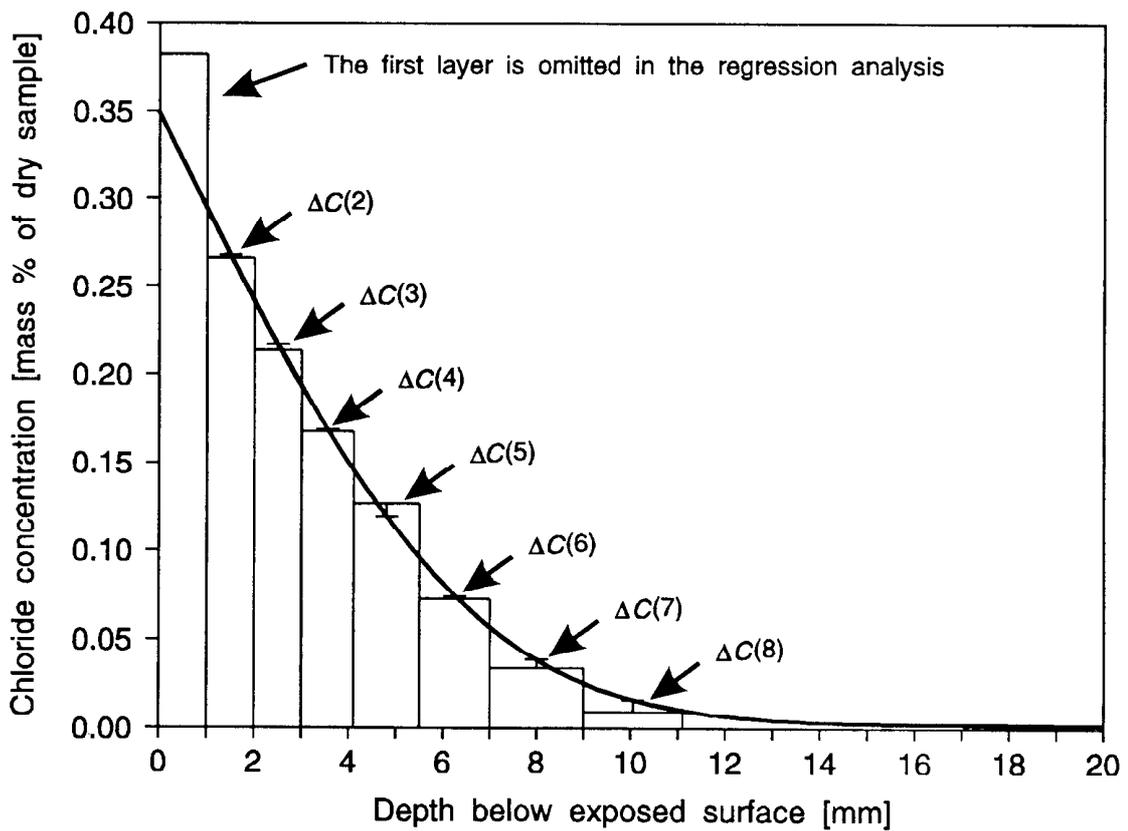


Figure 1. The regression analysis. The bars represent the measured chloride contents. The curve represents the optimized mathematical model.

7 Discussions and Suggestions

7.1 Some suggestions to the improvement of the immersion test

For the immersion test, the possible error sources include powder grinding and chloride analysis. According to the results from another Nordtest project /15/, the repeatability and reproducibility of chloride analysis could be limited to less than COV 10% if a titration method, especially potentiometric titration method, is employed. In the current version of NT BUILD 443, the depth intervals for powder grinding are not well specified. Because in most cases it is impossible to re-grind a specimen, the depth intervals must be pre-decided before the grinding. Different laboratories may grind the specimen at different depth intervals, resulting in different statistic weights in the curve-fitting. This error may be minimised by specifying the depth intervals according to the quality of concrete. An example is given in Table 7.1.

Table 7.1. Recommended depth intervals (in mm) for powder grinding.

water/binder	0.25	0.30	0.35	0.40	0.50	0.60	0.70
Depth #1	0~1	0~1	0~1	0~1	0~1	0~1	0~1
#2	1~2	1~2	1~2	1~3	1~3	1~3	1~5
#3	2~3	2~3	2~3	3~5	3~5	3~6	5~10
#4	3~4	3~4	3~5	5~7	5~8	6~10	10~15
#5	4~5	4~6	5~7	7~10	8~12	10~15	15~20
#6	5~6	6~8	7~9	10~13	12~16	15~20	20~25
#7	6~8	8~10	9~12	13~16	16~20	20~25	25~30
#8	8~10	10~12	12~16	16~20	20~25	25~30	30~35

Note: For concrete with pozzolanic additions such as fly ash, slag, and silica fume, the depth intervals in the column one place left should be applied, e.g. for slag cement concrete with $w/b = 0.4$, the depth intervals in the column for $w/b = 0.35$ should be applied.