nordtest method

NT BUILD 492

Approved 1999–11

1(8)

CONCRETE, MORTAR AND CEMENT-BASED REPAIR MATERIALS:
CHLORIDE MIGRATION COEFFICIENT FROM NON-STEADY-STATE MIGRATION EXPERIMENTS

UDC 691.32/691.53/691.54

Key words: Chlorides, concrete, diffusion, mortar, repair materials, migration, test method

1 SCOPE

This procedure is for determination of the chloride migration coefficient in concrete, mortar or cement-based repair materials from non-steady-state migration experiments.

2 FIELD OF APPLICATION

The method is applicable to hardened specimens cast in the laboratory or drilled from field structures. The chloride migration coefficient determined by the method is a measure of the resistance of the tested material to chloride penetration. This non-steady-state migration coefficient cannot be directly compared with chloride diffusion coefficients obtained from the other test methods, such as the non-steady-state immersion test or the steady-state migration test.

3 REFERENCES

/1/ NT BUILD 201, "Concrete: Making and curing of moulded test specimens for strength tests", 2nd ed., Approved 1984-05.

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

/3/ NT BUILD 208, "Concrete, hardened: Chloride content", 2nd ed., Approved 1984-05.

/4/ Tang, L and Sørensen, H.E., "Evaluation of the Rapid Test Methods for Chloride Diffusion Coefficient of Concrete, NORDTEST Project No. 1388-98", SP Report 1998:42, SP Swedish National Testing and Research Institute, Borås, Sweden, 1998.

4 DEFINITIONS

Migration: The movement of ions under the action of an external electrical field.

Diffusion: The movement of molecules or ions under a concentration gradient or, more strictly, chemical potential, from a high concentration zone to a low concentration zone.

5 SAMPLING

The method requires cylindrical specimens with a diameter of 100 mm and a thickness of 50 mm, sliced from cast cylinders or drilled cores with a minimum length of 100 mm. The cylinders and cores should meet the requirements described in NT BUILD 201 and NT BUILD 202 respectively. Three specimens should be used in the test.

6 TEST METHOD

6.1 Principle

An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration, the specimen is axially split and a silver nitrate solution is sprayed on to one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth.

6.2 Reagents and apparatus

6.2.1 Reagents

- Distilled or de-ionised water.
- Calcium hydroxide: $Ca(OH)_2$, technical quality.
- Sodium chloride: NaCl, chemical quality.
- Sodium hydroxide: NaOH, chemical quality.
- Silver nitrate: $AgNO_3$, chemical quality.
- Chemicals for chloride analysis as required by the test method employed (optional, see 6.4.6).

6.2.2 Apparatus

- Water-cooled diamond saw.
- Vacuum container: capable of containing at least three specimens.
- Vacuum pump: capable of maintaining a pressure of less than 50 mbar (5 kPa) in the container.

- Migration set-up: One design (see Appendix 1) includes the following parts:
  - Silicone rubber sleeve: inner/outer diameter 100/115 mm, about 150 mm long.
  - Clamp: diameter range 105 ~115, 20 mm wide, stainless steel (see Figure 2 in Appendix 1).
  - Catholyte reservoir: plastic box, 370 x 270 x 280 mm (length x width x height).
  - Plastic support: (see Figure 3 in Appendix 1).
  - Cathode: stainless steel plate (see Figure 3 in Appendix 1), about 0.5 mm thick.
  - Anode: stainless steel mesh or plate with holes (see Figure 4 in Appendix 1), about 0.5 mm thick.

  Other designs are acceptable, provided that temperatures of the specimen and solutions during the test can be maintained in the range of 20 to 25 °C (see 6.4.2).
- Power supply: capable of supplying 0 ~60 V DC regulated voltage with an accuracy of ±0.1 V.
- Ammeter: capable of displaying current to ±1 mA.
- Thermometer or thermocouple with readout device capable of reading to ±1 °C.
- Any suitable device for splitting the specimen.
- Spray bottle.
- Slide calliper with a precision of ±0.1 mm.
- Ruler with a minimum scale of 1 mm.
- Equipment for chloride analysis as required by the test method employed (optional, see 6.4.6).

6.3 Preparation of the test specimen

6.3.1 Test specimen

If a drilled core is used, the outermost approximately 10 ~20 mm thick layer should be cut off (Note 1) and the next 50 ±2 mm thick slice should be cut as the test specimen. The end surface that was nearer to the outermost layer is the one to be exposed to the chloride solution (catholyte).

If a Ø100 x 100 mm cast cylinder is used, cut a 50 ±2 mm thick slice from the central portion of the cylinder as the test specimen. The end surface that was nearer to the as-cast surface is the one to be exposed to the chloride solution (catholyte).

If a Ø100 x 200 mm cast cylinder is used, prepare the test specimen by first cutting the cylinder into two halves (i.e. into two Ø100 x 100 mm cylinders), and then cutting a 50 ±2 mm thick slice from one half. The end surface that was nearer to the first cut (the middle surface) is the one to be exposed to the chloride solution (catholyte).

Measure the thickness with a slide calliper and read to 0.1 mm.

Note 1: The term 'cut' here means to saw perpendicularly to the axis of a core or cylinder, using a water-cooled diamond saw.

6.3.2 Preconditioning

After sawing, brush and wash away any burrs from the surfaces of the specimen, and wipe off excess water from the surfaces of the specimen. When the specimens are surface-dry, place them in the vacuum container for vacuum treatment. Both end surfaces must be exposed. Reduce the absolute pressure in the vacuum container to a pressure in the range of 10–50 mbar (1–5 kPa) within a few minutes. Maintain the vacuum for three hours and then, with the vacuum pump still running, fill the container with the saturated $Ca(OH)_2$ solution (by dissolving an excess of calcium hydroxide in distilled or de-ionised water) so as to immerse all the specimens. Maintain the vacuum for a further hour before allowing air to re-enter the container. Keep the specimens in the solution for 18 ± 2 hours.

6.4 Procedure

6.4.1 Catholyte and anolyte

The catholyte solution is 10 % NaCl by mass in tap water (100 g NaCl in 900 g water, about 2 N) and the anolyte solution is 0.3 N NaOH in distilled or de-ionised water (approximately 12 g NaOH in 1 litre water). Store the solutions at a temperature of 20–25 °C.

6.4.2 Temperature

Maintain the temperatures of the specimen and solutions in the range of 20–25 °C during the test.

6.4.3 Preparation of the test

- Fill the catholyte reservoir with about 12 litres of 10 % NaCl solution.
- Fit the rubber sleeve on the specimen as shown in Figure 4 in Appendix 1 and secure it with two clamps. If the curved surface of the specimen is not smooth, or there are defects on the curved surface which could result in significant leakage, apply a line of silicone sealant to improve the tightness.
- Place the specimen on the plastic support in the catholyte reservoir (see Figure 1 in Appendix 1).
- Fill the sleeve above the specimen with 300 ml anolyte solution (0.3 M NaOH).
- Immerse the anode in the anolyte solution.
- Connect the cathode to the negative pole and the anode to the positive pole of the power supply.

6.4.4 Migration test

- Turn on the power, with the voltage preset at 30 V, and record the initial current through each specimen.
- Adjust the voltage if necessary (as shown in Table 1 in Appendix 2). After adjustment, note the value of the initial current again.

- Record the initial temperature in each anolyte solution, as shown by the thermometer or thermocouple.
- Choose an appropriate test duration according to the initial current (see Table 1 in Appendix 2).
- Record the final current and temperature before terminating the test.

6.4.5 Measurement of chloride penetration depth

- Disassemble the specimen by following the reverse of the procedure in 6.4.3. A wooden rod is often helpful in removing the rubber sleeve from the specimen.
- Rinse the specimen with tap water.
- Wipe off excess water from the surfaces of the specimen.
- Split the specimen axially into two pieces. Choose the piece having the split section more nearly perpendicular to the end surfaces for the penetration depth measurement, and keep the other piece for chloride content analysis (optional).
- Spray 0.1 M silver nitrate solution on to the freshly split section.
- When the white silver chloride precipitation on the split surface is clearly visible (after about 15 minutes), measure the penetration depth, with the help of the slide calliper and a suitable ruler, from the centre to both edges at intervals of 10 mm (see Figure 5 in Appendix 1) to obtain seven depths (notes 2, 3 and 4). Measure the depth to an accuracy of 0.1 mm.

Note 2: If the penetration front to be measured is obviously blocked by the aggregate, move the measurement to the nearest front where there is no significant blocking by aggregate or, alternatively, ignore this depth if there are more than five valid depths.

Note 3: If there is a significant defect in the specimen which results in a penetration front much larger than the average, ignore this front as indicative of the penetration depth, but note and report the condition.

Note 4: To obviate the edge effect due to a non-homogeneous degree of saturation or possible leakage, do not make any depth measurements in the zone within about 10 mm from the edge (see Figure 5 in Appendix 1).

6.4.6 Surface chloride content (optional, Note 5)

- From the other axially split specimen, cut an approximately 5 mm thick slice (Note 6) parallel to the end surface that was exposed to the chloride solution (catholyte).
- Determine the chloride content in the slice in accordance with NT BUILD 208 or by a similar method with the same or better accuracy.

Note 5: Information on chloride binding capacity of the tested material can be estimated from the surface chloride content.

Note 6: The thickness of the slice should always be less than the minimum penetration depth.

6.5 Expression of results

6.5.1 Test results

Calculate the non-steady-state migration coefficient from equation (1):

$$D_{nssm} = \frac{RT}{zFE} \cdot \frac{x_d - \alpha\sqrt{x_d}}{t} \quad (1)$$

where:

$$E = \frac{U-2}{L} \quad (2)$$

$$\alpha = 2\sqrt{\frac{RT}{zFE}} \cdot \mathrm{erf}^{-1}\left(1 - \frac{2c_d}{c_0}\right) \quad (3)$$

D_{nssm}: non-steady-state migration coefficient, m^2/s;

z: absolute value of ion valence, for chloride, $z = 1$;

F: Faraday constant, $F = 9.648 \times 10^4$ J/(Vmol);

U: absolute value of the applied voltage, V;

R: gas constant, $R = 8.314$ J/(Kmol);

T: average value of the initial and final temperatures in the anolyte solution, K;

L: thickness of the specimen, m;

x_d: average value of the penetration depths, m;

t: test duration, seconds;

erf^{-1}: inverse of error function;

c_d: chloride concentration at which the colour changes, $c_d \approx 0.07$ N for OPC concrete;

c_0: chloride concentration in the catholyte solution, $c_0 \approx 2$ N.

Since $\mathrm{erf}^{-1}\left(1 - \frac{2 \times 0.07}{2}\right) = 1.28$, the following simplified equation can be used:

$$D_{nssm} = \frac{0.0239(273+T)L}{(U-2)t}\left(x_d - 0.0238\sqrt{\frac{(273+T)L\,x_d}{U-2}}\right) \quad (4)$$

where:

D_{nssm}: non-steady-state migration coefficient, $\times 10^{-12}$ m^2/s;

U: absolute value of the applied voltage, V;

T: average value of the initial and final temperatures in the anolyte solution, °C;

L: thickness of the specimen, mm;

x_d: average value of the penetration depths, mm;

t: test duration, hour;

6.6 Accuracy

6.6.1 Repeatability

The coefficient of variation of repeatability is 9 %, according to the results from the Nordic round-robin test between six laboratories /4/.

6.6.2 Reproducibility

The coefficient of variation of reproducibility is 13 % for Portland cement concrete or for concrete mixed with silica fume, and 24 % for concrete mixed with slag cement, according to the results from the Nordic round-robin test between six laboratories /4/.

6.7 Test report

The test report should, if known, include the following information:

a) Name and address of the test laboratory.

b) Date and identification number of the test report.

c) Name and address of the organisation or person who ordered the test.

d) Name and address of the manufacturer or supplier of the material or object tested.

e) Date of arrival of the material or object tested.

f) Description of the material or object tested, including sampling, composition, and curing age.

g) Purpose of the test.

h) Test method.

i) Any deviation from the test method.

j) Name and address of the person who performed the test.

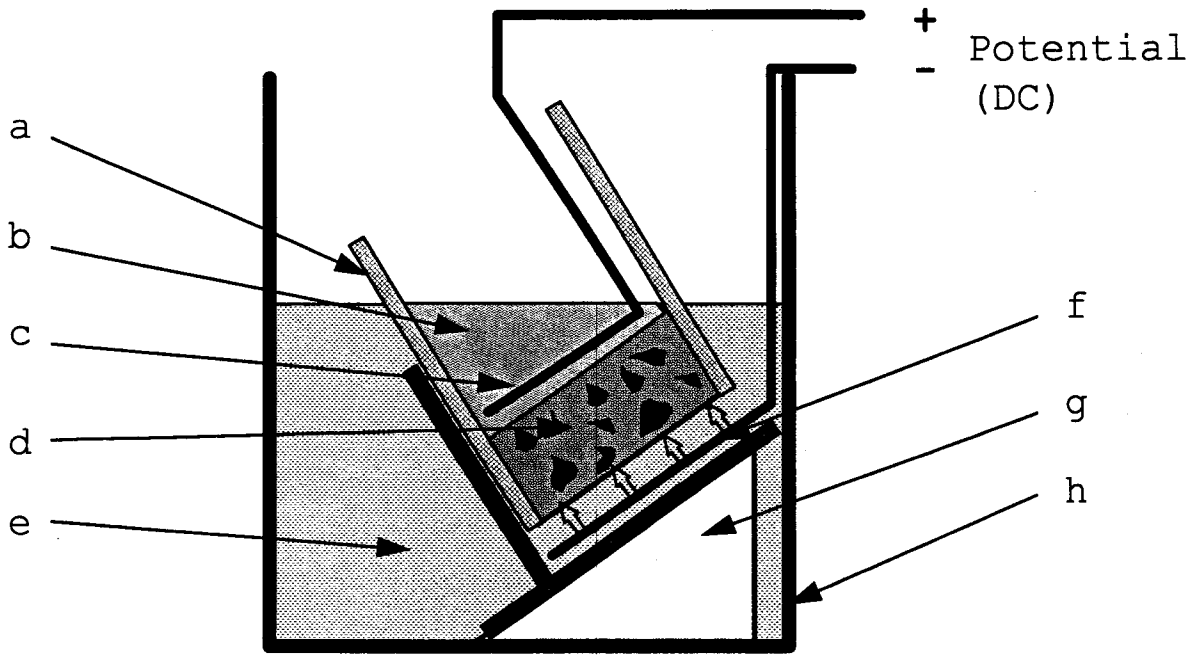
k) Date of the test.

l) Test results, including the specimen dimensions, applied voltage, initial and final currents, initial and final temperatures, average and individual data of penetration depth and migration coefficient.

m) Any observation of an abnormal penetration front due to a defect in the specimen.

n) Optional information about surface chloride content.

o) Date and signature.



a. Rubber sleeve
b. Anolyte
c. Anode
d. Specimen
e. Catholyte
f. Cathode
g. Plastic support
h. Plastic box

Fig. 1. One arrangement of the migration set-up.

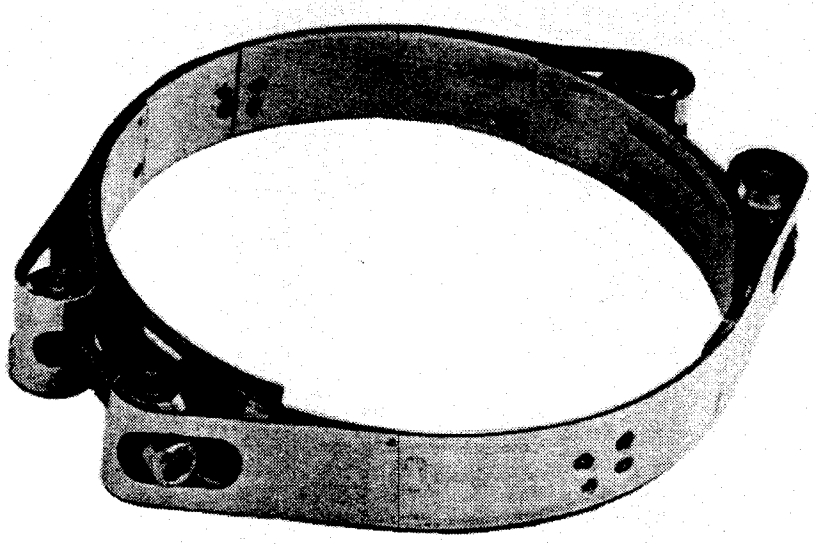


Fig. 2. Stainless steel clamp.

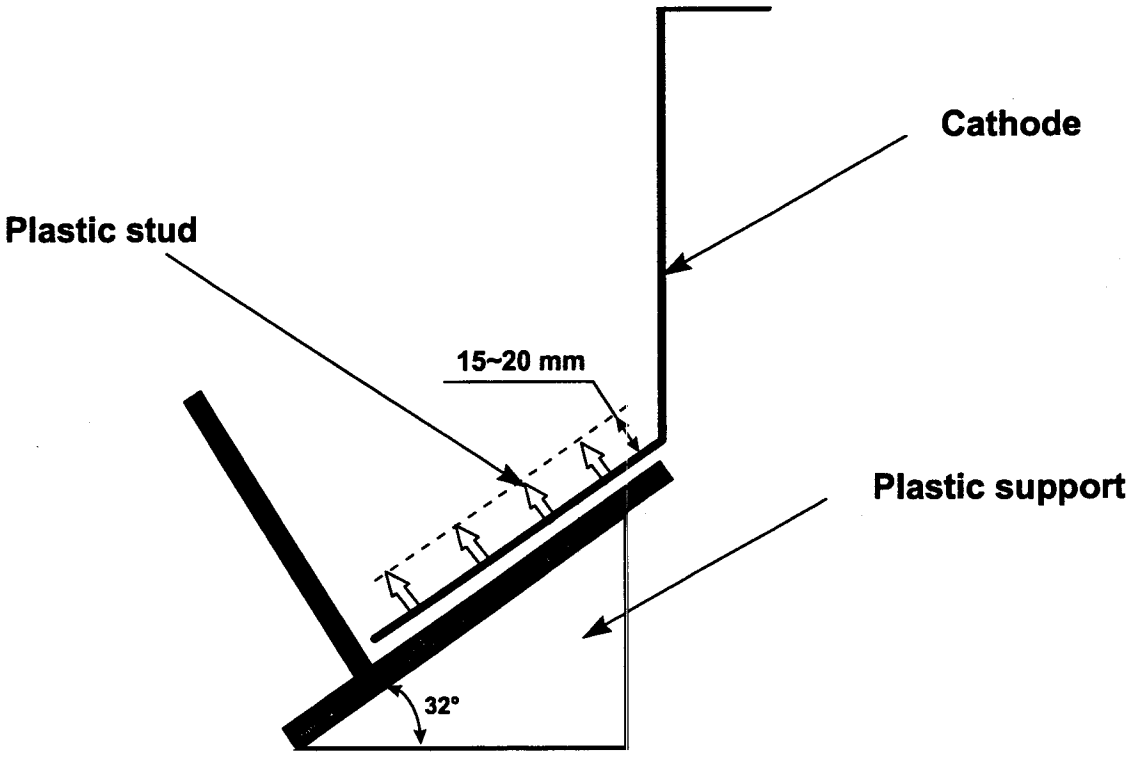


Fig. 3. Plastic support and cathode.

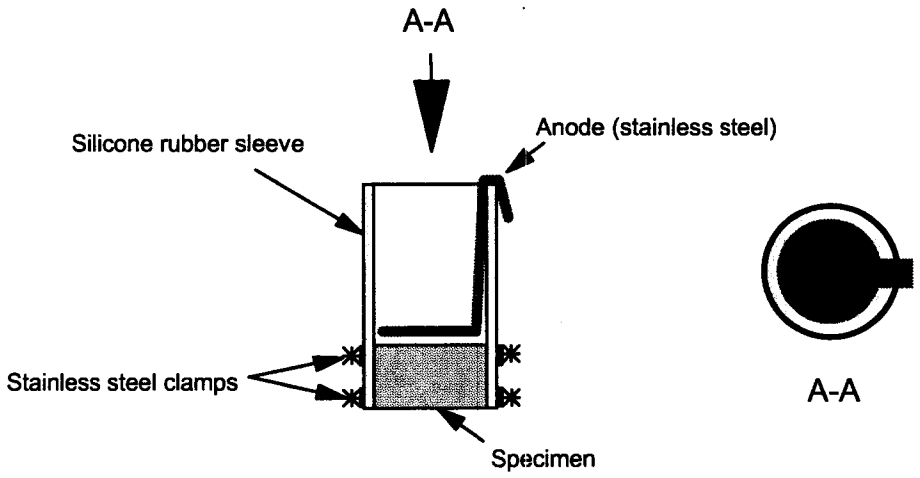


Fig. 4. Rubber sleeve assembled with specimen, clamps and anode.

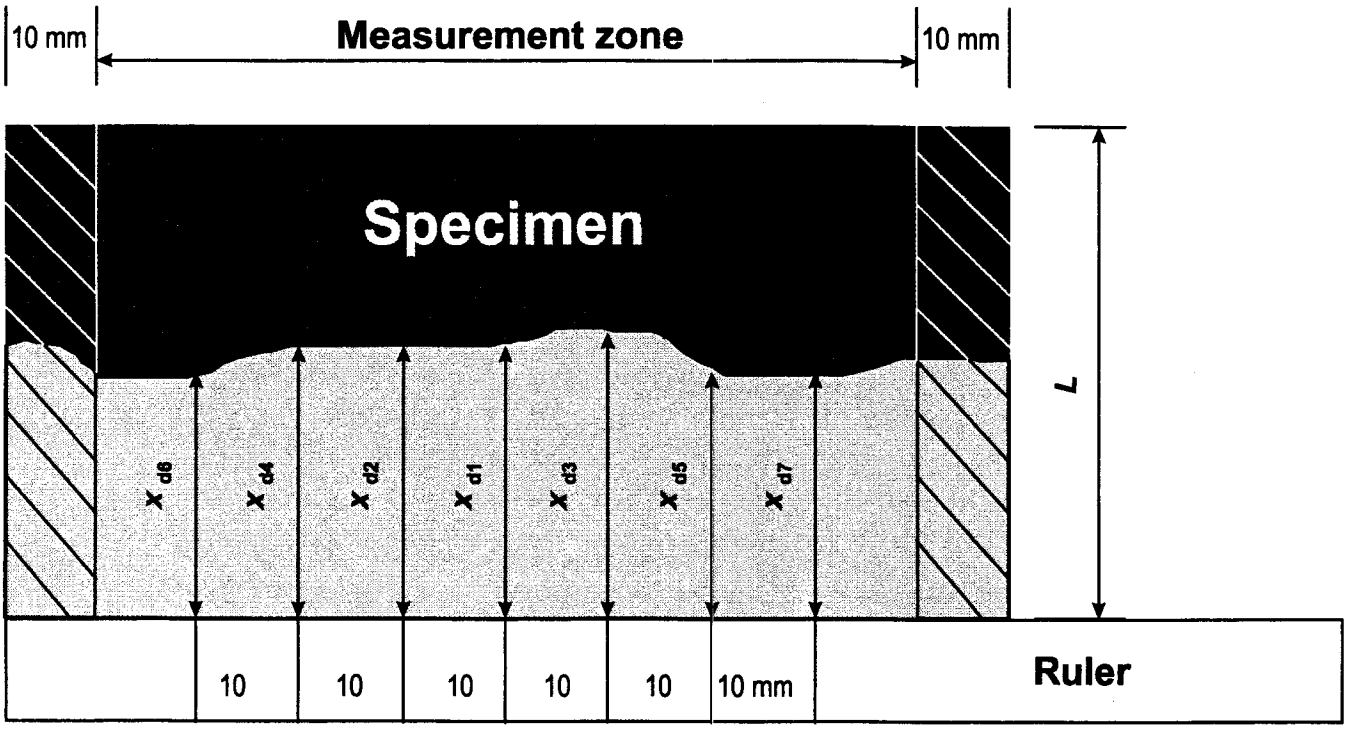


Fig. 5. Illustration of measurement for chloride penetration depths.

Table 1. Test voltage and duration for concrete specimen with normal binder content.

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
$5 \leq I_0 < 10$	60	$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$	60	$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	24
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	24
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	24
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	24
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	24
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	24
$120 \leq I_0 < 180$	15	$60 \leq I_0 < 90$	24
$180 \leq I_0 < 360$	10	$60 \leq I_0 < 120$	24
$I_0 \geq 360$	10	$I_0 \geq 120$	6

Note: For specimens with a special binder content, such as repair mortars or grouts, correct the measured current by multiplying by a factor (approximately equal to the ratio of normal binder content to actual binder content) in order to be able to use the above table.