



HETEK

Chloride Penetration into Concrete
Relevant Test Methods



Report No.94
1997



Road Directorate Denmark
Ministry of Transport

IRRD Information

Title: HETEK, Chloride Penetration into Concrete, Relevant test methods

Editor: J.M. Frederiksen

Subject classification: Field 32: Concrete

Key words:	Concrete	4755
	Chloride	7314
	Durability	5910
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	In-situ	6226
	Denmark	8028

Abstract: This report is part of a series of reports generated in the research project HETEK headed by the Danish Road Directorate. The present sub task is concerning chloride transport into concrete and this report contains several detailed test methods relevant to the subject.

Some of the test methods are generally accepted standards (Nordtest NT BUILD) while some have a lower status, i.e. they are detailed descriptions of often used procedures developed by laboratories that are experienced in the actual fields of testing.

Most of the test methods are in English language but some were only present in Danish versions.

UDK: 691.32
ISBN: 87-7491-804-4
ISSN: 0909-4288

1 Preface

HETEK is covering 8 topics. The present project is dealing with chloride penetration into concrete. The sub task is performed by the consortium ACCE consisting of AEC, Chalmers and Cementa.

1.1 Background

The Road Directorate in Denmark has launched a number of research projects in 1995 to be performed and completed during 1996. The package of projects has been given the name HETEK, which is short, in Danish, for High quality concrete, the Contractors TEChnology. The projects cover eight topics:

1. Test methods for chloride resistance of high quality concrete
2. Test methods for freeze/thaw durability of high quality concrete
3. Self-desiccation
4. Curing Technology
5. Casting and compaction
6. Curing treatment
7. Guidance in trial castings
8. Remedial measures during the execution phase

The projects are to give a state-of-the-art report, identify the need for further research, perform some of that research and finally give guidelines for the contractor.

From this sub task a number of reports were published. The state-of-the-art report on Chloride penetration was first completed. The first research report describes the experimental study on the effect of w/c ratio on chloride penetration. The second report describes the road environment from field investigations of chloride and moisture conditions in a number of road bridges. The third report summarises the theoretical background to the final result of the project: A system for estimation of the service-life of concrete structures exposed to chloride from sea water or de-icing salt. This system was published in a manual comprising a complete description of how to perform a service life design whether dealing with design of new structures, construction or maintenance and surveying.

1.1.1 About HETEK-1

The research consortium ACCE was given the first project HETEK-1 on chloride resistance of high quality concrete. The task for this project is to re-evaluate existing methods, and develop new ones, for determination of chloride penetration in high quality concrete. The methods must consider the differences in environmental actions on the concrete structure. Quantitative criteria for approval shall be laid down to ensure compliance with the durability requirements and the economy of the methods shall be estimated.

The research consortium ACCE consists of the three partners: AEC, Chalmers University of Technology and Cementa AB.

1.1.2 About the research consortium

AEC Consulting Engineers (Ltd.) A/S is a private consultant company in Denmark. AEC mainly works in the field of concrete structures and topics related to the repair, durability and maintenance of those. The typical clients of AEC are other consultants, contractors, building owners, insurance companies, cement producers and suppliers and also producers of materials for concrete repair and maintenance. The company has two departments: a structural department and a materials department. The structural department offers consultancy regarding specialised construction problems and conventional consultancy in civil engineering. The materials department, the AEC laboratory, assesses deterioration of concrete structures, prescribes and develops repair methods and evaluates repair materials. In addition, research and development regarding concrete durability tasks are solved for clients and/or financed by fundings.

Chalmers University of Technology educate civil engineers and researchers and do research in a number of basic and applied sciences and technologies. The department of Building Materials at the School of Civil Engineering, is participating in HETEK-1. The main research area is transport processes in porous building materials, mainly cement-based and wooden-based materials and surface materials on such materials. Examples of concrete research are: Moisture binding and flow properties of concrete, self-desiccation and drying of hardening high performance concrete, plastic shrinkage and early age cracking, chloride penetration into structures exposed to sea water and de-icing salts. The relationships between mix design, micro and pore structure and properties are experimentally studied and the behaviour in different environments are modelled and verified on concrete structures.

Cementa AB is a cement producer in Sweden. The activities of Cementa regarding concrete research are as follows: High Performance Concrete, i.e. high strength, low water content and low permeability. Concrete and environment, i.e. problems regarding moisture in concrete and emissions from concrete. Durable Concrete, i.e. long time experiments regarding chloride ingress, permeability, strength evolution and carbonation. No Slump Concrete, i.e. rheological aspects of making pre-cast concrete products.

1.2 Scope

The scope of this report is to provide reliable test methods in order to ensure a uniform way of collecting data to be used in one way or the other when building experience about chloride ingress into concrete and corrosion initiation in concrete and concrete structures exposed to chloride laden environments, i.e. marine environments and road environments.

1.3 Structure

The report is structured into two parts: a short report and an appendix. The report part consists of an introduction and a short description of the test methods including the intended use of each method. The appendix contains copies of the full test methods.

J M Frederiksen edited and H. Sørensen performed the quality assurance.

1.4 Limitations

The test methods presented here are those that existed as reliable test methods in the Spring of 1997. More test methods are needed in order to complete the collection of methods needed when analysing concrete structures exposed to chloride.

2 Short description of the test methods

This Chapter gives a short description of each of the test methods presented in the Appendix. In addition there is given a short comment of the intended use of each of the test methods.

NT BUILD 208, Edition 2, Approved 1984-05

Concrete, hardened: Chloride content

This test method is used to determine the total content of chloride in hardened concrete by dissolving the chloride in a solution of nitric acid and measure the chloride content by Volhard titration. The method does not state in which form the chloride exists in the concrete.

The method provides sufficient accuracy for chloride contents in concrete if the sample size is as prescribed (the sample should contain about 2g of cement (note the correction made in the test method in the Appendix)) and when used by experienced laboratories.

NT BUILD 357, Approved 1989-11

Concrete, repairing materials and protective coating: Carbonation resistance

This method is used to determine the resistance against CO₂-penetration into concrete repairing materials and concrete protective coatings. The carbonation resistance is measured as the carbonation depth by the phenolphthalein method on laboratory prepared samples from an accelerated laboratory test.

The principle and definitions in the measurement of the carbonation depth are recommended for use before measurements of chloride profiles in concrete after natural exposure.

NT BUILD 443, Approved 1995-11

Concrete, hardened: Accelerated chloride penetration

This method is used to determine the resistance against chloride penetration into hardened concrete or other cement-based materials. The resistance against chloride penetration is measured as the chloride profiles in concrete samples after exposure to a 10 %(w/w) chloride solution at 23 °C. The chloride profiles are measured by profile grinding and subsequent Volhard titration. The chloride profiles are transformed into descriptive penetration parameters by curve-fitting of a model based on the mathematical “error” function. The method can be used to compare both concrete, mortar and other cement-based materials.

The method can be used for research purposes, for pre- and inspection testing of cement-based materials.

APM 207, 1st edition, September 1992

Concrete testing, hardened concrete: Micro chloride profile

This method is used to determine the variation of the total content of chloride in hardened concrete. The variation of chloride content in the concrete is determined as the chloride profile perpendicular to the exposed surface. The chloride profile is measured by profile grinding, analysis of the chloride content in each layer of the profile by dissolving in a solution of nitric acid and measurement of the chloride content by Volhard titration. The test method produces a chloride concentration profile down to a so-called micro scale based on measurements in steps of approx. 0.5 mm up to 5 mm. The method does not state in which form the chloride exists in the concrete.

The method can be used on naturally or laboratory exposed concrete.

APM 214, 2nd edition, May 1995

Concrete testing, hardened concrete: Calcium content

This method is used to determine the total content of calcium in hardened concrete by dissolving the calcium in a solution of nitric acid and measure the calcium content by titration using a calcein indicator. The method does not state in which form the calcium exists in the concrete. The method is based on BS 1881: Part 124:1988.

The method can be used on naturally or laboratory exposed concrete.

APM 219, 1st edition, May 1996

Concrete testing, hardened concrete: Electrical resistivity

This method is used to determine the electrical resistivity of hardened concrete by measuring the electrical resistance of a concrete specimen having a well-defined geometry. The electrical resistance is recorded as direct readings on a sample subjected to an alternating electrical current field over a well-defined area. Knowing the dimensions of the test specimen the electrical resistivity is calculated from the electrical resistance. Presupposing the existence of a relationship between quality of concrete and electrical resistivity the test result is often used as a uniformity parameter.

The method may be used to evaluate different mix designs and as a uniformity measurement in a concrete production.

APM 303, 2nd draft, September 1993

Concrete testing, hardened concrete: Chloride threshold value for reinforcement corrosion

This method is used to determine the initiating chloride content (the threshold value) for reinforcement corrosion in concrete at different electrochemical potentials. The resistance against chloride penetration acc. to NT BUILD 443 is determined as well. The test principle is exposure of specimens to a 10 %(w/w) chloride solution and registration of corrosion initiation on embedded test electrodes kept potentiostatically at different electrical potentials. The corrosion initiation is measured by monitoring the electrical current to maintain a potentiostatically fixed potential on each test electrode. The chloride threshold value is determined by measuring the chloride content of the concrete at the steel surface of the test electrode at the time of corrosion initiation.

APM 321, 1st edition, May 1991

Concrete testing, hardened concrete: Qualitative 2D chloride penetration

This method is used to produce a qualitative evaluation of the chloride penetration into concrete or other cement-based materials. The test principle is cutting a chloride exposed specimen perpendicular to the exposed face, spraying indicator on the freshly cut surface and observing the colour change in the chloride contaminated areas on the surface.

The test method is very applicable for studies of chloride penetration into concrete with cracks, construction joints or defects with extension to the exposed concrete surface. In addition the method serves as a good indicator of the depth of the chloride ingress when planning the position of sub samples in a chloride profile.

APM 402, 1st edition, January 1994

Concrete testing, concrete repair: Chloride penetration resistance of paint on concrete

This method is used to determine the protective effect of paints or paint systems against ingress of chloride into concrete exposed to spray of water containing chloride. The test principle is based on comparative measurements of the penetration depths of chloride in unpainted concrete references as well as painted samples. References and samples are exposed to cyclic alternating immersion in 10 %(w/w) chloride solution for 2 hours and subsequent air-drying for 4 hours. After 30 weeks the chloride profiles are determined in the references and samples acc. to APM 207 and the "equivalent concrete thickness" for the paint is calculated.

CTH Rapid Method, 2nd draft, April 1996

Recommended procedure for determination of chloride diffusion coefficient by using CTH Rapid Method

This method is used to determine the chloride diffusion coefficient in aged ordinary concrete specimens. The chloride diffusion coefficient is calculated from the measured penetration depth of chloride in a specimen after exposure in a 10 %(w/w) sodium chloride solution and application of an electrical potential of 30 V for approx. 24 hours. The penetration depth is determined by application of a silver nitrate solution on a split surface of the specimen. The test result can be correlated to the calculated transport coefficient determined according to NT BUILD 443.

Appendix

The test methods are presented in complete versions in the same order as the short descriptions were given above.

CONCRETE, HARDENED: CHLORIDE CONTENT

1. SCOPE AND FIELD OF APPLICATION

This NORDTEST method is intended for the determination of the total content of chloride in the hardened concrete by dissolving it in a solution of nitric acid and by titration.

The method does not state in which form the chlorides exists in the concrete or if they are easy or difficult to dissolve in water.

The method can be used in well-equipped laboratories.

2. REFERENCES

NT BUILD 202 CONCRETE, HARDENED: SAMPLING AND TREATMENT OF CORES
FOR STRENGTH TESTS

3. SAMPLING

If the intention is to determine the average content of chlorids in the concrete, one should select one or several test specimens so that the estimated amount of concrete exceeds about 1 kg. The test specimens may be taken out in connection with the cooling

published by

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key words

concrete
hardened concrete
chlorides
analysis
test method

classification

UDC 666.97/691.32

in water, for instance acc. to NT BUILD 202, with the condition that this is stated in the test report.

If the intention is to determine the content of chlorides in a certain place in the concrete, e.g. the content of chlorides in a concrete cover layer, then one should use a reciprocating drilling machine and then collect so much of the drill cuttings from the actual place in the concrete that one estimates that the amount of ~~concrete~~^{Cement} in it exceeds about 2 ~~kg~~.

4. METHOD OF TEST

4.1 Apparatus

Equipment for the collection of test specimens acc. to 3., e.g. core drilling machine or a reciprocating drilling machine, the latter should have a diameter of at least 18 mm.

Grinder for the grinding of concrete down to a particle size of less than 0.1 mm.

Apparatus for dividing up the concrete sample.

Oven thermostatically controlled for a range of 105 ± 5 °C.

Balance with an accuracy within ± 0.01 g.

Glass bottle with a ground glass stopper, holding about 300 ml.

Distilled or de-ionized water.

Burettes 2 of them, each with a volume of 25 ml. One to be used for the silver nitrate solution and the other for the ammonium-tiocyanate solution.

Gas burner, or similar equipment.

Nitric acid (HNO_3) concentrated.

Filter, either made from paper or from a material with similar capacities and 1 per cent nitric acid solution which may be needed

in certain cases.

Silver nitrate solution (AgNO_3), about 0.1 N

Benzyl alcohol ($\text{C}_7\text{H}_8\text{O}$) or nonanol-(1), ($\text{C}_9\text{H}_{20}\text{O}$).

Ammoniumthiocyanate solution (NH_4SCN), about 0.1 N

Saturated ammonium ferri-sulphate solution ($\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$)
with 10 volume per cent nitric acid.

4.2 Preparation of Test Samples

The drill cuttings do not need to be prepared for a test analysis.

The selected test specimens should be crushed with a hammer or similar tool to a size that suits the mill or grinder and in such a manner that no material is lost.

The crushed material is then ground in the grinder until a particle size less than 0.1 mm is obtained.

Several ground test samples are then homogenized and at least 100 g is then divided in the sample divider so that a sample for test analysis containing about 2 g of concrete is obtained.

The test sample is dried at 105 ± 5 °C until no reduction in the weight can be seen.

4.3 Procedure

Weigh the glass bottle. Insert the sample for analysis in the glass bottle. Determine the weight of the sample for analysis.

Add about 20 ml of distilled water and shake the bottle so that the particles separate.

Add 10 ml concentrated nitric acid, shake the bottle and add about 50 ml hot water. Let the mixture cool down until it reaches ambient temperature.

Filter the solution if it is dark in colour. Rinse the filter with 1 per cent strong nitric acid. Permit the filtrate to flow down into the glass bottle.

Add distilled water until the volume of 100 ml is reached.

Add the silver nitrate solution in excess from the burette (about 20 ml is usually sufficient).

Add 2 - 3 ml benzyl alcohol or nonanol and 1 ml saturated ammonium ferri-sulphate solution. Insert the stopper in the bottle and shake so vigorously that the silver nitrate separates.

Titrate the remaining amount of silver nitrate with the ammonium-tiocyanate solution. Shake the bottle vigorously when the turning point of the content is near. Continue the titration at a slower rate during a continuously intensive mixing of the content until the solution obtains a permanent, weakly red colour.

The content of chlorides Cl^- is then calculated according to the formula:

$$\text{Cl}^- = 3.545 (V_1 N_1 - V_2 N_2) / m, \text{ weight in per cent, where}$$

V_1 = the added silver nitrate solution in ml

N_1 = the normality of the silver nitrate solution

V_2 = used amount in ml of the ammoniumtiocyanate solution during the titration

N_2 = the normality of the ammoniumtiocyanate solution

m = the weight of the sample used in the analysis expressed in g.

4.4 Expression of Results

The chloride-content is expressed in weight in per cent with two decimal figures.

4.5 Test Report

If a test report is submitted, it should contain at least the

following information:

- a) Name and address of the testing laboratory
- b) Date and identification symbols of the report
- c) Test method (number and title)
- d) Any deviation from the test method
- e) Name and address of the person or institution who ordered the test
- f) Name and address of the person performing the test
- g) Name and address of the manufacturer of the concrete
- h) The identification symbols of the concrete. The composition, age and temperature of the concrete should also be noted
- i) Date when the test was performed, and when the sample was received
- j) Test results
- k) Any other information of importance for the evaluation of the test results
- l) Evaluation of the test results, if this is required in the request for the test.

CONCRETE, REPAIRING MATERIALS AND PROTECTIVE COATING: CARBONATION RESISTANCE

 UDC 691.32:658.588

Key words: Repairing materials, concrete, protective coating, test method

SUMMARY

The resistance against CO₂-penetration through concrete repairing materials and concrete protective coating is determined by an accelerated laboratory test on laboratory prepared samples by exposing the samples to carbon dioxide enriched air in order to evaluate the effect of concrete repairing materials.

The depth of CO₂-penetration is measured by the phenolphthalein method.

1 SCOPE

This method specifies an accelerated laboratory test procedure in which the resistance against CO₂-penetration through concrete repairing materials or concrete protective coating can be determined in order to evaluate the effect of concrete repairing materials. It consists of monitoring the rate of carbonation of specimens exposed to an atmosphere of 3 % carbon dioxide and 55 – 65 % relative humidity.

2 FIELD OF APPLICATION

The method can be used to compare both concrete, mortar or concrete protective coatings.

3 REFERENCES

RILEM draft recommendation CPC-18. Measurement of hardened concrete carbonation depth. Materials and structures. Nov/Dec 1984, Vol 17, No 102.

4 DEFINITION

Carbonation depth: The average distance, measured in mm, from the surface of concrete or mortar where the carbon dioxide has reduced the alkalinity of the hydrated cement to an extent such that the phenolphthaleine indicator is not colored red. That happens in the pH-range 8.3–10.0.

5 SAMPLING

The test may be carried out on laboratory cast concrete or mortar specimens of various shapes. Specimen may, in the case of concrete, be either of the cylinder type with diameter 100 mm and length 200 to 300 mm, or prisms with cross section 100 x 100 mm and length 300 – 500 mm. In the case of mortar, the RILEM prism type of 40 x 40 x 160 mm size may be used. The test is carried out with 2 or 3 parallel samples.

The specimens to be tested are compared with a reference concrete made of 300 – 350 kg Ordinary Portland Concrete (or equivalent), per m³ concrete, water to cement ratio 0.60 ± 0.01 , slump 120 ± 20 mm and a well graded aggregate with d_{max} 16 mm. A melamine based plasticizer may be used if necessary. No air entraining admixture should be used. The moulds are placed on a vebe table and worked until the mix is properly placed and compacted.

The specimens are stripped 1 day after casting, and cured in water, $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ until 14 days of age, then cured in air, $50 \pm 5 \text{ } \%$ RH, $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ until 28 days of age before they are subjected to the test.

Ready mixed repairing mortar should be cured according to the specifications from the producer.

When a concrete protective coating is to be tested, it is applied on cured reference concrete. The coating is allowed to cure according to the specification from the producer. The reference specimens should be kept in air, $50 \pm 5 \text{ } \%$ RH, $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ while the coated specimens are cured.

Polymer latex modified mortar or concrete is stripped 2 days after casting, and then cured in air, $50 \pm 5 \text{ } \%$ RH, $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ until 28 days of age before testing.

6 METHOD OF TEST

6.1 Principle

The specimens are exposed to an atmosphere containing 3 % carbon dioxide and with a relative humidity between 55 and 65 %.

The carbonation depth is measured by applying phenolphthalein color indicator on the fracture surface of a freshly broken-off fragment of the specimens.

Thus the same specimens may be used several times in order to map the development of the carbon dioxide attack. Sound concrete or mortar is colored red by the indicator while the color of carbonated concrete or mortar remains unchanged.

6.2 Apparatus and equipment

Compartment with gas inlet and outlet for specimen exposure.

Gas supply (3 % CO₂ in air).

Humidity controller (55 – 65 % RH) inside compartment.

Phenolphthalein solution (1 g phenolphthalein in a solution of 500 ml distilled/ion exchanged water and 500 ml ethanol).

6.3 Preparation of test samples

Laboratory cast specimens are exposed to the test atmosphere after curing according to chapter 5.

6.4 Procedure and accuracy

After sampling, the specimens are placed inside the compartment, and exposed to the carbonation atmosphere. The carbonation depth is measured at least once a month.

If the carbonation front does not run as a straight line parallel to the surface (Fig 1), the depth of carbonation must be determined in the following way:

In the case of the carbonation front running as in Fig 2, a graphical average and the maximum should be recorded. If the carbonation front runs parallel to the surface, apart from isolated deeper carbonated areas, as in Fig 3, then the maximum depth of carbonation must be given as well as the normal depth. In this case no average is to be calculated.

Greater depths of carbonation in the corner areas of laboratory specimens, where carbon dioxide has penetrated from two sides at once, should be ignored. The carbonation depth on duplicate specimens from the same concrete should not differ more than 20 % of the carbonation depth. If the difference is > 20 %, both values should be reported. If the difference is < 20 %, it is sufficient to report the average value.

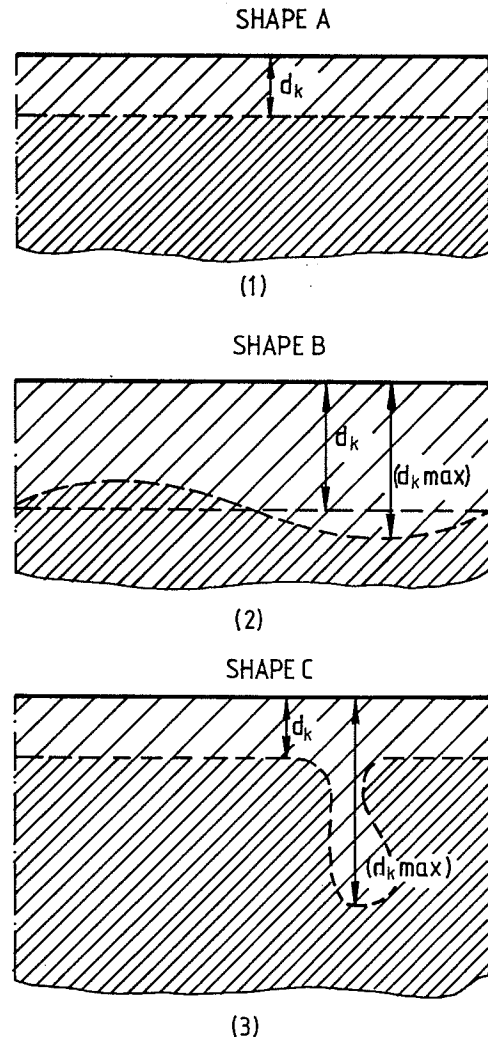


Fig 1, 2, 3. Defining the depth of carbonation according to shape (a), (b), (c)

6.5 Expression of results

The selected parameters may be expressed as follows:

Parameter	Symbol	Dimension
Relative humidity	RH	%
Temperature	T	° C
Time	t	month (depends on material quality)
CO ₂ contents of the atmosphere in the test chamber	CO ₂	%
Carbonation depth	d	m

The carbonation depth may be expressed in tabular form including specimen dimensions, carbonation depth, exposure time. It may also be expressed as a photograph of the phenolphthalein sprayed fracture surface.

6.6 Test report

The test report shall include the following information, if relevant

- a) Name and address of the testing laboratory
- b) Identification number of the test report
- c) Name and address of the organization or the 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 manufacturer or supplier of the tested object
- g) Name or other identification marks of the tested object
- h) Description of the tested object
- 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, time, RH, etc)
- m) Identification of the test equipment and instruments used
- n) Any deviations from the test method
- o) Test results (use SI units)
- p) Inaccuracy or uncertainty of the test result
- q) Date and signature.

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_p , 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_{er} , 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 past 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.

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

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)

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

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:

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

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)

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

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

$C_c(n)$ [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.

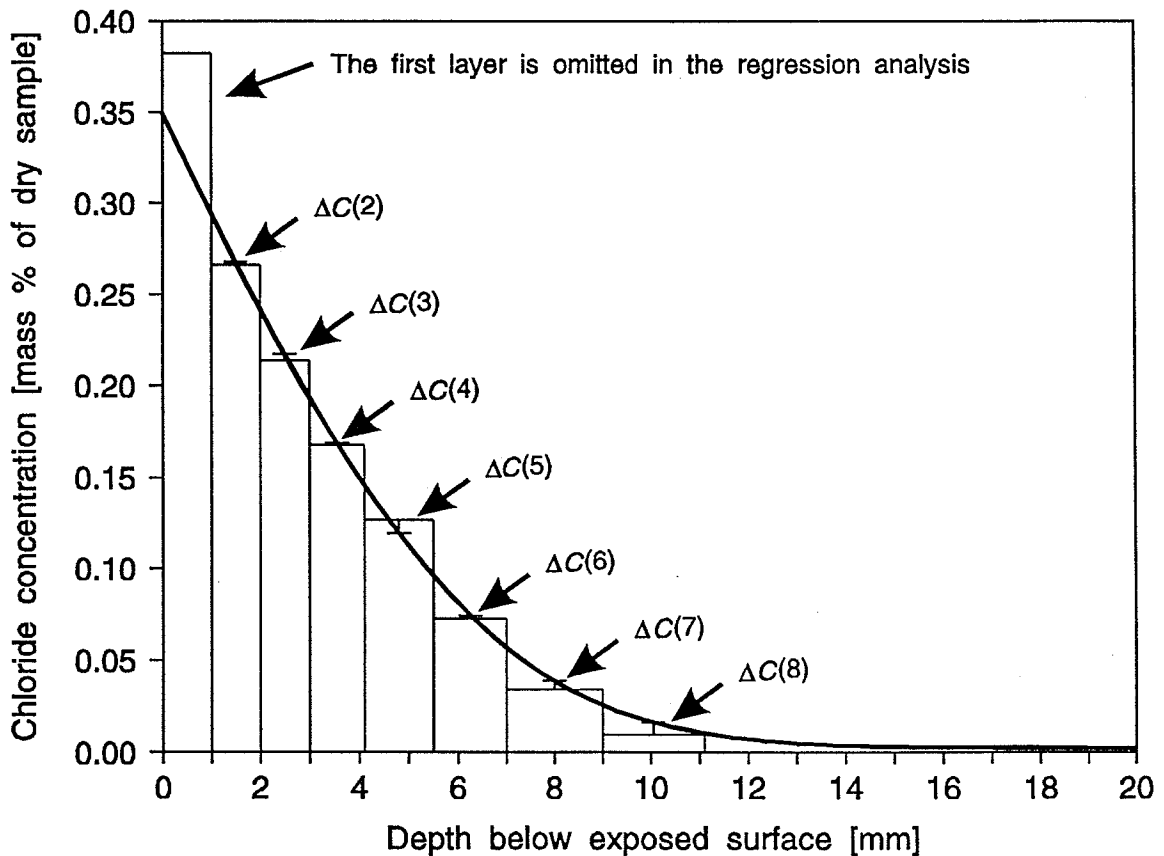


Figure 1. The regression analysis. The bars represent the measured chloride contents. The curve represents the optimized mathematical model.

AEC laboratory	Concrete testing	APM 207
Staktoften 20 DK-2950 Vedbæk	Hardened concrete	1st edition September 1992
Tel:+45 45661266	Micro chloride profile	Init: HS

1. Object and application

This test method describes a procedure for determining the variation of the chloride content of hardened concrete. The test method produces a concentration profile at a so called micro scale based on measurements in steps less than 5 mm.

2. References

- DS 423.22. Concrete testing. Hardened concrete. (In danish).
Drilling of cylinders for determination of compressive strength. (Identical to NT Build 202).
- DS 423.28. Concrete testing. Hardened concrete. (In danish).
Chloride content. (Identical to NT Build 208).

3. Sampling

The samples are typical drilled cores taken according to DS 423.22. The diameter of the cores shall be 75 mm at least.

4. Short description of the test procedure

Thin layers of concrete powder are ground off the concrete sample. The chloride content of the layers are determined according to DS 423.28 (Volhard titration).

5. Chemicals

- 5.1 Redistilled or demineralized water.
- 5.2 Nitric acid (HNO₃), concentrated, GR.
- 5.3 Silver nitrate standard solution (AgNO₃), approx. 0.1 N.
- 5.4 Ammonium thiocyanate standard solution (NH₄SCN), approx. 0,1 N.
- 5.5 Ferric ammonium sulphate solution (NH₄Fe(SO₄)₂·12H₂O), saturated, in 10 vol% nitric acid.

6. Apparatus

- 6.1 Equipment for grinding off and collecting concrete powder from thin concrete layers (less than 2 mm).
- 6.2 Scales, accuracy better than ± 0.01 g.
- 6.4 Drying cupboard, ventilated and thermostat regulated for $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$.
- 6.5 Erlenmeyer flasks, about 300 ml.
- 6.6 Burettes, 2 nos., about 25 ml, for titration according to DS 423.28.
- 6.7 Bunsen burner or other heat source.
- 6.8 Glass filtering funnel with filter of paper or similar.
- 6.9 Slide gauge or similar.

7. Preparation of specimen

The face opposite to the face of interest is cut and/or ground perpendicular to the profile direction.

8. Test procedure

The sample is analyzed for acid-soluble chloride content as a function of the depth in the profile direction of interest. The grinding is performed within a diameter approx. 10 mm less than the full diameter of the sample. This obviates the risk of disturbing contamination from the cylinder face. 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 surface is calculated as the average of five uniformly distributed measurements with an accuracy of at least 0.1 mm. The acid-soluble chloride content of the samples is determined in accordance with DS 423.28 or by a similar method with an equal or better accuracy.

9. Presentation of result

The chloride contents are given in mas% of dry concrete to two decimal places. It is recommended to visualize the results by producing a figure showing the chloride content as a function of the depth below the surface.

AEC laboratory	Micro chloride profile	APM 207
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10. Test report

A test report must at least contain the following information:

- 10.1 Name and address of the test laboratory, and the place at which the tests were performed if different from the laboratory address.
- 10.2 Date and clear identification of the report.
- 10.3 Name and address of the client.
- 10.4 Description and marking of the specimen.
- 10.5 Date of receipt of the specimen and performance of the test.
- 10.6 Specification of the test method.
- 10.7 Description of the sampling, if relevant.
- 10.8 All deviations from or additions to the test method, and any omissions, together with other information of importance for judging the result.
- 10.9 The test results.
- 10.10 Signature and title or other identification of person(s) responsible for the technical content of the report.
- 12.11 Date of release of the report.

AEC laboratoriet	Betonprøvning	APM 214
Staktoften 20 DK-2950 Vedbæk	Hærdnet beton	2. udgave Maj 1995
tlf 45 66 12 66	Calciumindhold	Init: HS

1. Formål og anvendelsesområde

Denne prøvningsmetode beskriver fremgangsmåden ved bestemmelse af hærdnet betons calciumindhold ved opløsning i salpetersyre og analyse ved titrering med calcein indikator. Metoden er udarbejdet på grundlag af BS 1881:Part 124 [1].

2. Referencer

- [1] British Standard 1881, Testing concrete. Part 124. Methods for analysis of hardened concrete. 1988.
- [2] Dansk Standard 423.22, 2. udgave. Marts 1984. Betonprøvning. Hærdnet beton. Udboring af cylindre til styrkebestemmelse.
- [3] Dansk Standard 423.28, 1. udgave. Marts 1984. Betonprøvning. Hærdnet beton. Chloridindhold.

3. Prøveudtagning

Prøven til calciumanalyse kan være udboret eller udfræset betonpulver. Alternativt kan prøven være et stykke beton. Prøvestykket kan udtages med kernebor, eventuelt med brug af vandkøling, fx efter [2].

4. Kort prøvningsbeskrivelse

Salpetersyre og kogende vand tilsættes den tørrede prøve. Efter afkøling filtreres opslemningen, og filtratets calciumindhold bestemmes ved titrering med EDTA og fluorescerende calcein indikator.

5. Kemikalier

- 5.1 Redestilleret vand.
- 5.2 Salpetersyre (HNO_3), koncentreret, analysekvalitet.
- 5.3 Saltsyre (HCl), koncentreret, analysekvalitet.
- 5.4 Calciumcarbonat (CaCO_3), analysekvalitet.
- 5.5 Triethanolamin (TEA), analysekvalitet.
- 5.6 Natriumhydroxid (NaOH), analysekvalitet.
- 5.7 Ethylen-diamin-tetra-eddikesyre (EDTA), analysekvalitet.
- 5.8 Calcium-standardopløsning, ca. 1,00 g CaO /liter.
Afvej ca. 1,78 g calciumcarbonat (5.4), tørret ved 150-200 °C, og notér massen med 4 decimaler. Opløs pulveret i let overskud af fortyndet saltsyre (1 del HCl (5.3) til 4 dele vand (5.1)), afkog kuldioxid, nedkøl og fortynd til 1 liter i en målekolbe.
- 5.9 TEA-opløsning, 1 del TEA (5.5) til 4 dele vand (5.1).
- 5.10 Calcein (screenet) indikator, 0,2 g calcein, 0,12 g thymolphthalein og 20 g kaliumchlorid blandes og formales til fint pulver.
- 5.11 NaOH -opløsning, 200 g NaOH (5.6) opløses i 1 liter vand (5.1).
- 5.12 EDTA-standardopløsning, 0,1 N EDTA. 0,1 mol EDTA (5.7) afvejes og opløses i varmt vand (5.1). Filtrering foretages om nødvendigt. Opløsningen nedkøles og fortyndes til 1

liter i en målekolbe og opbevares i en polyethylenflaske (6.11). Standardisering foretages mod 50 ml af calcium-standardopløsningen (5.8) ved titrering som beskrevet under punkt 8.2.

6. Apparatur

- 6.1 Udstyr til udtagning af prøve, fx kerneboremaskine eller slagboremaskine.
- 6.2 Udstyr til nedknusning af beton eller fræsning af pulverprøver.
- 6.3 Standardsigte, maskevidde 1,0 mm.
- 6.4 Analysevægt, nøjagtighed bedre end $\pm 0,0001$ g.
- 6.5 Vægt, nøjagtighed bedre end $\pm 0,01$ g.
- 6.6 Tørreskab, ventileret og termostatreguleret for $105\text{ °C} \pm 5\text{ °C}$.
- 6.7 Gasbrænder eller anden varmekilde.
- 6.8 Glaskolbe, konisk, ca 300 ml.
- 6.9 Pipetter, 5 ml og 10 ml.
- 6.10 Målekolbe, 300 ml og 1000 ml.
- 6.11 Polyethylenflasker, 1 liter.
- 6.12 Glasfiltertragt med filter af papir eller tilsvarende (vacuumfiltrering er ofte nødvendig).
- 6.13 Plastflasker med tætsluttende skruelåg, 100 ml.
- 6.14 Burette, ca. 25 ml, til titrering.
- 6.15 Ultraviolet lyskilde (UV-lampe).

7. Prøvetilberedning

Betonpulver skal ikke tilberedes yderligere før analysen.

Udtagne prøvestykker nedknes uden tab af materiale til gennemfald på 1,0 mm standardsigte. Større prøver kan nedknes delvist, homogeniseres og neddeles, således at der fremkommer en repræsentativ delprøve, som nedknes til gennemfald på 1,0mm standardsigte.

Der skal opnås en analyseprøve på mindst 10 g.

8. Fremgangsmåde ved prøvning

- 8.1 Syreopløsning af calcium

Pulverprøven tørres, vejes og syreopløses med salpetersyre foretages iht DS 423.28 [3]. Filtratet fortyndes med redestilleret vand til et kendt volumen (V_{tot} , fx 300ml) i en målekolbe.
- 8.2 Udtagning af prøve og titrering

Det fortyndede filtrat omrystes, og en delprøve udtages med pipette (V_a , fx 10 ml). Delprøven fortyndes med vand til ca. 150 ml i en konisk kolbe. 5 ml TEA-opløsning og 5 ml NaOH-opløsning samt 0,15 g calcein indikator tilsættes under omrøring. UV-lampen tændes, og der titreres med EDTA-standardopløsning (5.12) under omrøring til omslagspunktet nås, hvor indikatoren skifter fra fluorescerende grøn til violet uden rest af fluorescens. Den anvendte mængde EDTA-standardopløsning (V_{EDTA}) noteres til nærmeste 0,01 ml.

AEClaboratoriet	Calciumindhold	APM 214
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- 8.3 Alternative analysemetoder
Calciumanalysen kan også foretages med alternative metoder, fx potentiometrisk titrering eller atomabsorptionsspektrofotometri, såfremt metoden har samme eller bedre nøjagtighed.

9. Beregning af resultater

Prøvens calciumoxidindhold beregnes af f9.1:

$$(f9.1) \quad C_{CaO} = \frac{5,608 \cdot V_{EDTA} \cdot N_{EDTA}}{M_p} \cdot \frac{V_{tot}}{V_a} \cdot F$$

hvor

C_{CaO} [mas%] er prøvens CaO-indhold

V_{EDTA} [ml] er anvendt mængde EDTA-standardopløsning ved titreringen

N_{EDTA} [g/mol] er normaliteten af EDTA-standardopløsningen

M_p [g] er massen af den tørrede pulverprøve

V_{tot} [ml] er volumenet af målekolben anvendt til fortynding af filtratet

V_a [ml] er volumenet af den udtagne analyseprøve til titrering.

F [-] er en titreringsfaktor, der korrigerer for anvendelse af salpetersyre i stedet for saltsyre ved syreopløsningen (værdien 1,04 kan anvendes, hvis den ikke er bestemt for den aktuelle prøve)

10. Angivelse af resultat

Prøvens calciumindhold angives som prøvens CaO-indhold i mas% med 2 decimaler.

11. Prøvningsrapport

Eventuel prøvningsrapport skal indeholde mindst følgende information:

- 11.1 Navn og adresse på prøvningslaboratoriet og stedet for prøvningens udførelse, såfremt dette er forskellig fra prøvningslaboratoriets adresse.
- 11.2 Dato og entydig identifikation af rapporten.
- 11.3 Navn og adresse på rekvirenten.
- 11.4 Beskrivelse og mærkning af prøven, samt eventuelt angivelse af prøvens placering i konstruktionen.
- 11.5 Dato for prøvens modtagelse og prøvningens udførelse.
- 11.6 Angivelse af prøvningsmetoden.
- 11.7 Beskrivelse af prøveudtagningsmetoden.
- 11.8 Alle afvigelser, tilføjelser eller udeladelser fra prøvningsmetoden og øvrige oplysninger af betydning for bedømmelsen af resultatet.
- 11.9 Prøvningsresultat.
- 11.10 Underskrift og titel eller anden markering af person(er), der er teknisk ansvarlig(e) for rapporten.
- 11.11 Dato for rapportens frigivelse.

AEClaboratory	Concrete testing Hardened concrete Electrical resistivity	APM 219
Staktoften 20		1. edition
DK-2950 Vedbæk		May 1996
tlf +45 45 66 12 66		Init: JMF

1. **Field of application**

The method presupposes the existence of a relationship between quality of concrete and electrical resistivity. The electrical resistivity is mainly depending on the water/cement ratio, the moisture content, the temperature, the concrete maturity and the composition. Due to this electrical resistivity of hardened concrete can be used as a uniformity parameter. The scope of this test method is to provide a detailed description of how to measure the resistivity of concrete.

It is assumed that the concrete test specimens are free of construction faults such as cavities and visible cracks and no reinforcement are present.

2. **References**

NT BUILD 201, 2. ed., Concrete: Making and curing of moulded test specimens for strength test.

NT BUILD 202, 2. ed., Concrete, hardened: Sampling and treatment of cores for strength tests.

3. **Test specimens**

This method requires drilled cores or cast cylinders as test specimens. They must be representative for the concrete and/or structure in question. At least three test specimens should be used in the test.

4. **Short description of the test**

A water saturated concrete specimen of well-defined thickness is subjected to an alternating current field over a well-defined area. The test result is provided as directly readings of the electrical resistance (in $k\Omega$) of the concrete specimen. Knowing the dimensions of the test specimen, the electrical resistivity (in $k\Omega\text{cm}$) is calculated from the electrical resistance.

5. **Chemicals**

5.1 A calcium hydroxide solution is prepared by adding $\text{Ca}(\text{OH})_2$ to distilled or demineralized water in the ratio of 2 g per dm^3 . This solution is referred to as the »wetting solution«.

6. **Apparatus**

6.1 Stone saw

6.2 Balance, capacity/accuracy better than 1000 ± 1 g

6.3 Four point resistance meter, e.g. the commercial product: »Norma«, Erdungsmesser D3950, which is able to supply a stable alternating current of approx. 40 mA at the frequency of 108 Hz, quartz-stabilized. This apparatus is provided with a 4 digit digital

*

display for readings of ohmic resistance. The electrical resistance is given in Ω or in $k\Omega$ (maximum reading is 199.9 $k\Omega$).

Alternatively a programmed datalogger can be used if many measurements shall be performed at the same time.

- 6.4 Two stainless steel plates with the minimum dimensions corresponding to the exposed surface of the concrete specimen. The stainless steel plates shall be connected through insulated wires to the resistance meter.
- 6.5 Two sponges to be placed between the stainless steel plates and the test specimen.
- 6.6 Pressure spray can containing the wetting solution for prewetting of sponges before the measurements.
- 6.7 Weight with a mass of 1.5 kg.

7. Specimen preparation

Three test specimens are made from three drilled cores or cast cylinders ($\varnothing 100 \pm 1$ mm). Preparing specimens from drilled cores: After cutting of the outer 10 mm from the original end face test specimens with a thickness of 50 ± 1 mm shall be cut. Preparing specimens from cast cylinders: The specimens can be three 50 mm slices cut around the middle of the core.

The test specimens are kept under water in order to saturate the specimens and to prevent any drying. In order to avoid leaching as little water as possible should be used. The specimens are kept under water until the change in mass per day is less than 0.1% and the time for performing the measurements is due.

8. Test procedure

The electrical resistivity measurements shall be performed according to the following procedure:

- 8.1 The test setup shall follow the principles shown in Figure 1.
- 8.2 The sponges are wetted using the pressure spray can containing the »wetting solution«, cf. Section 5.1 and wringed a little to remove excess water on the sponges. The sponges are placed in the test setup and the total resistance of both sponges, R_{sp} is recorded when stability is achieved (normally after 5 to 10 seconds). The electrical resistance of the sponges shall not exceed 100 Ω .

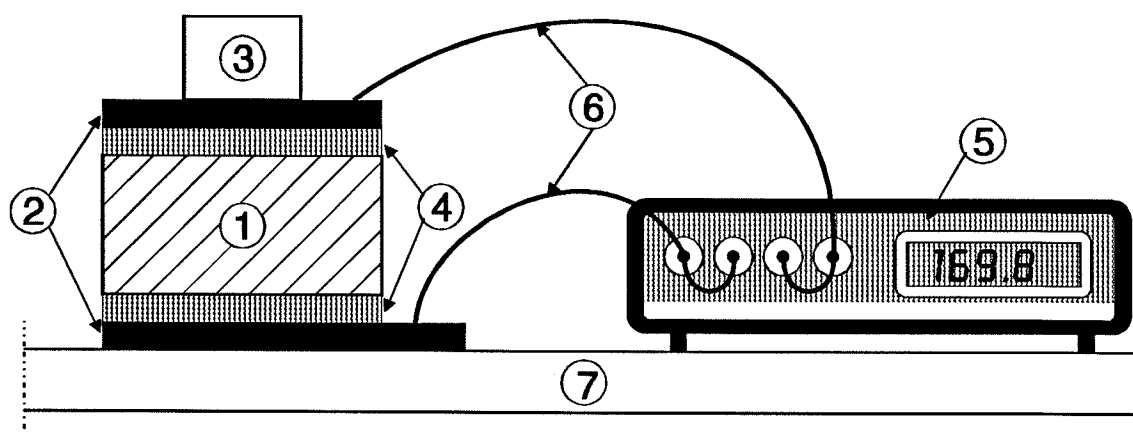


Figure 1. Sketch of the test set-up for measuring the resistivity of concrete. The numbers refer to: 1. Test specimen; 2. Stainless steel plate; 3. 1.5 kg weight; 4. Sponge; 5. Four point resistance meter; 6. Connecting wires; 7. Non-metallic table.

- 8.3 Each test specimen shall carefully be wiped dry with a tissue especially on the curved surface on which the sponges are not attached. The temperature of the test specimens shall be maintained close to the room temperature, $23 \pm 2^\circ\text{C}$.

Each test specimen is then placed between the sponges in the test setup. The total resistance of both sponges and the test specimen, R_{s+sp} is recorded when stability is achieved.

The measurement on each test specimen is repeated after 2 to 3 minutes. If the difference between the obtained resistance values in two measurements on the same test specimen is less than 5% the test is concluded. If the difference between two measurements is bigger than 5%, the procedure is repeated until the obtained results are within the acceptable scatterband.

9. Calculation of results

The electrical resistance of each of the specimens is appearing from (1):

$$R_s = R_{s+sp} - R_{sp} \quad (1)$$

where

R_s is the electrical resistance of each test specimen

R_{s+sp} is the measured mean electrical resistance of each test specimen and the sponges

R_{sp} is the measured electrical resistance of the sponges

The electrical resistance of the test specimens will normally be given in kΩ. The electrical resistivity, ρ , is calculated from (2):

$$\rho = A \times R_s / \delta \quad (2)$$

where

ρ is the electrical resistivity of each test specimen	[kΩcm]
A is the cross sectional area of each test specimen	[cm ²]
δ is the thickness of each test specimen	[cm]

10. Presentation of the test result

The test result is given as the resistivity value for each test specimen and their mean value. The latter is taken as the resistivity of the particular concrete. The resistivity values are all given with three significant digits in kΩcm.

11. Test report

A test report must at least contain the following information:

- 11.1 Name and address of the test laboratory, and the place at which the tests were performed if different from the laboratory address.
- 11.2 Date and clear identification of the report.
- 11.3 Name and address of the client.
- 11.4 Description and marking of the sample.
- 11.5 Date of receipt of the sample and performance of the test.
- 11.6 Specification of the test method.
- 11.7 Description of the sampling, if relevant.
- 11.8 All deviations from or additions to the test method, and any omissions, together with other information of importance for judging the result.
- 11.9 Test result.
- 11.10 Signature and title or other identification of person(s) responsible for the technical content of the report.
- 11.11 Date of release of the report.

AECLaboratoriet	Betonprøvning	APM 303
Staktoften 20 DK-2950 Vedbæk	Hærdnet beton	2. udkast September 1993
45 66 12 66	Chloridtærskelværdi for armeringskorrosion	Init: HA/JMF/HS

1. Formål og anvendelsesområde

Denne prøvningsmetode beskriver en procedure til at bestemme det korrosionsinitierende chloridindhold (tærskelværdien) i beton ved forskellige elektrokemiske potentialer. Desuden bestemmes parametre til estimering af indtrængningshastigheden af chloridioner i vandmættet hærdnet beton.

I metoden forudsættes, at prøveemnerne er af høj kvalitet; dvs specialfremstillede til formålet og uden udførelsesmæssige fejl som fx lunger og synlige revner.

Der kan afviges fra metodens krav til eksponeringstemperatur og eksponeringsvæskens sammensætning og/eller chloridkoncentration, såfremt dette begrundes af prøvningens formål.

2. Referencer

- [1] Arup, Hans: Bestemmelse af chloridtærskelværdi. Artikel i "Marina betongkonstruktioners livslængde", CEMENTA AB, side 125-132, 1993.
- [2] APM 302, 2. udgave. Maj 1991, Betonprøvning, Hærdnet beton, Chloridindtrængning.
- [3] DS 423.28, 1. udgave. Marts 1984. Betonprøvning. Hærdnet beton. Chloridindhold.
- [4] DS 423.21, 2. udgave. Marts 1984. Betonprøvning. Fremstilling og lagring af støbte prøvelegemer til styrkebestemmelse.
- [5] Basisbetonbeskrivelsen for bygningskonstruktioner, Byggestyrelsen, 1986.

3. Prøveemner

Den beton, der skal prøves, skal være hærdnet til minimum 28 modenhedsdøgn med mindre prøvningens formål betinger andet.

Prøvningen udføres på en specialfremstillet prøve af beton med indstøbte U-bøjleformede armeringsemner (4.2.4), en referen-
 * ceelektrode (4.2.8), to modelektroder (4.2.9) samt de nødvendige

lednings- og stikforbindelser. Chloridindtrængningen i betonen bestemmes på dertil indrettede prøveklodser, der fremstilles og eksponeres sammen med prøveemnet.

Afhængig af prøvningens formål kan der anvendes forskellige typer og dimensioner af armeringsjern.

4. Fremgangsmåde ved prøvning

4.1 Metodens princip

Et vandmættet betonprøveemne eksponeres på en plan savskåren flade med natriumchloridholdigt vand. Det elektrokemiske potentiale af indstøbte armeringsemner holdes i forsøgsperioden under potentiostatisk kontrol på definerede potentialer. Korrosionsaktiviteten følges ved måling af den fornødne strøm til opretholdelsen af potentialet for hvert armeringsemne. Initiering af korrosion regnes at være sket, når den anodiske strøm til et armeringsemne stiger markant.

Med passende mellemrum bestemmes chloridindtrængningen i betonen ved måling af chloridprofilet i parallelt eksponerede prøveklodser.

Chloriddiffusionskoefficienten, D , og chloridprofilets randbetingungelse mod den eksponerede overflade (beregnet overfladekoncentration), C_s , beregnes for hver af de målte profiler.

Til hver af de registrerede tider for korrosionsinitiering udregnes chloridkoncentrationen i en dybde svarende til dæklaget. Til denne beregning anvendes de målte chloridprofiler som udgangspunkt.

4.2 Forbrugsvarer

- 4.2.1 Redestilleret eller demineraliseret vand.
- 4.2.2 Calciumhydroxid ($\text{Ca}(\text{OH})_2$), teknisk kvalitet.
- 4.2.3 Natriumchlorid (NaCl), teknisk kvalitet.
- 4.2.4 Blankt \emptyset 8 mm komprimeret stål 37-2 DIN 1652.
- 4.2.5 Massiv tin-belagt kobberledning ca \emptyset 1 mm (fx nr 44/7004-18 fra Raychem).
- 4.2.6 2-komponent (chloridiondiffusionstæt) polyurethan- eller epoxy-baseret maling (membran).
- 4.2.7 Eldåse til stikforbindelse (fx h·b·l = 20·50·100 mm).
- 4.2.8 Referenceelektrode, egnet til varig indstøbning i beton, (fx ERE 10, BAC Bergsøe Anti Corrosion A/S).

- 4.2.9 Titannet med oxidbelægning, bredde 15-20 mm, maskevidde ca 2·2 mm, (fx Titananodegitter fra BAC Bergsøe Anti Corrosion A/S).
- 4.2.10 Acetone eller lign. til affedtning af armeringsemner.
- 4.2.11 Indstøbningsmasse (4.7), fx Densitop T1.
- 4.2.12 Salpetersyre (HNO_3), koncentreret, analysekvalitet.
- 4.2.13 Sølvnitrat standardopløsning (AgNO_3), ca 0,1 N.
- 4.2.14 Ammoniumthiocyanat standardopløsning (NH_4SCN), ca 0,1 N.
- 4.2.15 Ferriammoniumsulfatopløsning ($\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$), mættet, i 10 vol% salpetersyre.

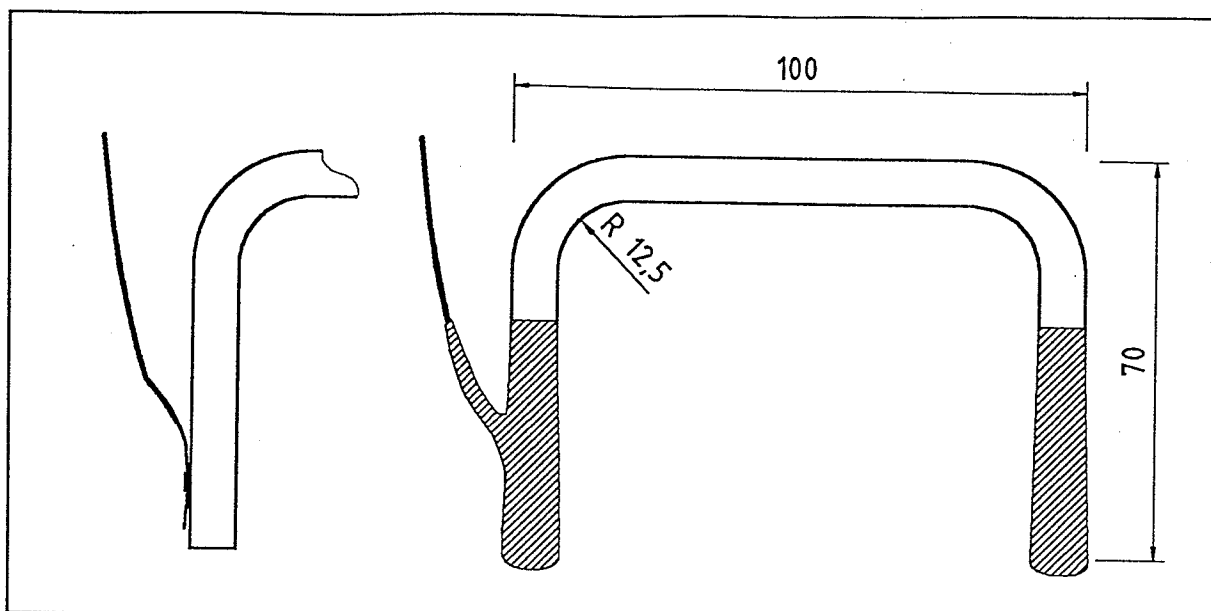
4.3 Særligt udstyr

- 4.3.1 Udstyr til modstandssvejsning.
- 4.3.2 Forme af betonbestandigt materiale til fremstilling af indstøbte modelektroder (4.5) og referenceceller (4.6). Indvendigt mål (h·b·l) 40·40·280 mm.
- 4.3.3 Forme af betonbestandigt materiale med fastholdelsesanordninger til sammenstøbning af de præfabrikerede komponenter til korrosionscellerne (4.7). Indvendigt mål (h·b·l) 40·330·344 mm.
- 4.3.4 Forme af betonbestandigt materiale til fremstilling af betonemnerne (4.8). Indvendigt mål (h·b·l) ca 200·330·344 mm.
- 4.3.5 Udstyr til betonfremstilling.
- 4.3.6 Vandkølet diamantsav, der kan gennemsæve et snit på ca 350 mm · 350 mm.
- 4.3.7 Termometer, nøjagtighed på $\pm 1^\circ\text{C}$.
- 4.3.8 Eksponeringskar med væskeomrøring.
- 4.3.9 Potentiostat, spændingsdeler og konstantstrømforsyning.
- 4.3.10 Datalogger eller mV-meter med hukommelse, autorange, 10 μV følsomhed samt mulighed for dataoverførsel til PC.
- 4.3.11 Udstyr til affræsning og opsamling af betonpulver fra tynde betonlag (under 2 mm).
- 4.3.12 Udstyr til chloridanalyse iht [3].

4.4 Fremstilling af armeringsemner

Til hver prøvning anvendes 20 stk armeringsemner. Armeringen (4.2.4) slibes med smergellærred og afklippes i længder af 210 mm. Emnerne bukket med bukkediameter på 25 mm til åbne U-formede bøjler med de udvendige mål 70·100·70 mm. På hver bøjles ene ende modstandssvejses en ca 0,3 m lang ledning (4.2.5), der over svejsningen fastgøres med et plastspændebånd eller lignende. De

yderste ca 40 mm af bøjleenderne påføres en epoxy- eller polyurethanbelægning (4.2.6). Se figur 1.

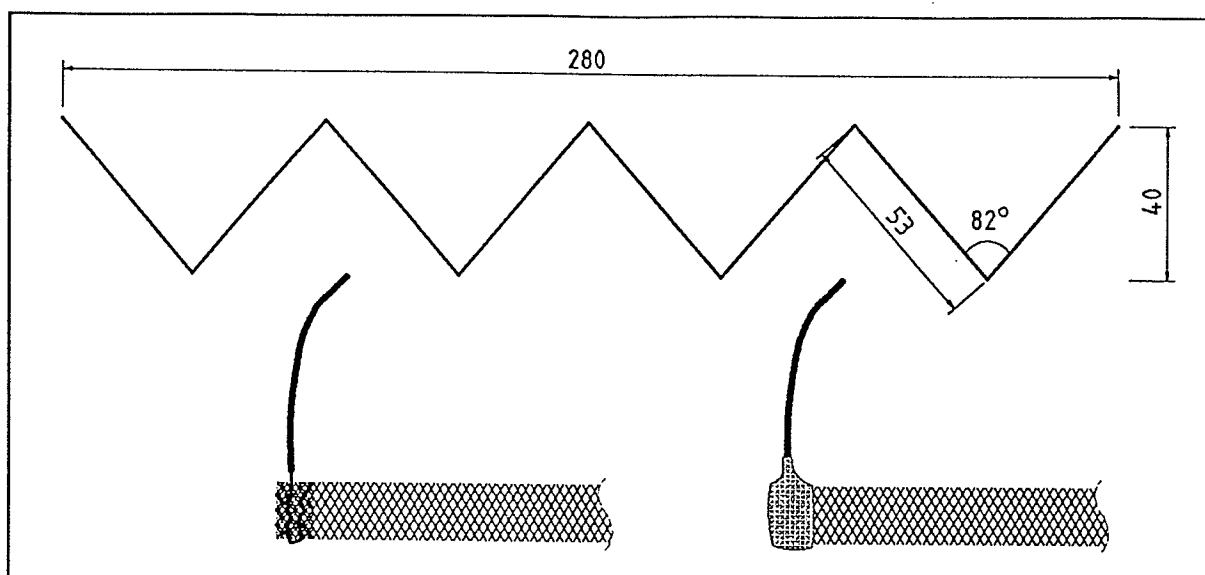


Figur 1: Skitse af bøjleformet armeringsemne med påmonteret punktsvejst ledning og epoxy- eller polyurethanbelagte ender. Mål er angivet i mm. Efter Hans Arups anvisninger.

4.5 Indstøbning af modelektroder

Til hver prøvning anvendes 2 stk titannet (4.2.9) afklippet i længder á ca 450 mm. I hvert stykkes ene ende indflettes den ene ende af en ca 300 mm lang ledning i nettet, som derefter bukkes og bankes sammen omkring den indflettede ledning. Det sammenbukkede område dækkes med epoxy- eller polyurethan (4.2.6), der skal hærde før arbejdet fortsættes. Titannettene bukkes nu i zig-zag form med 8 modsatvendte knæk, hver med en sidelængde på ca 53 mm. Se figur 2. De tildannede stykker titannet placeres herefter liggende i rene forme (4.3.2) med ledningerne stikkende ud. Titannettene indstøbes herefter i en letbearbejdelig cementmørtel med bindemiddel af en flyveaskefri cement og tilslag af kvarts-sand af klasse A iht [5] og med d_{max} på 4 mm. Der må ikke tilsættes mikrosilica. Der anvendes et v/c-forhold på 0,50 og et cement/sand-forhold på 1:3. Mørtelen udstøbes i to lige tykke lag og det sikres, at titannettet er placeret i det nederste lag. Mørtelen skal komprimeres ved vibrering for hvert lag. Oversiden

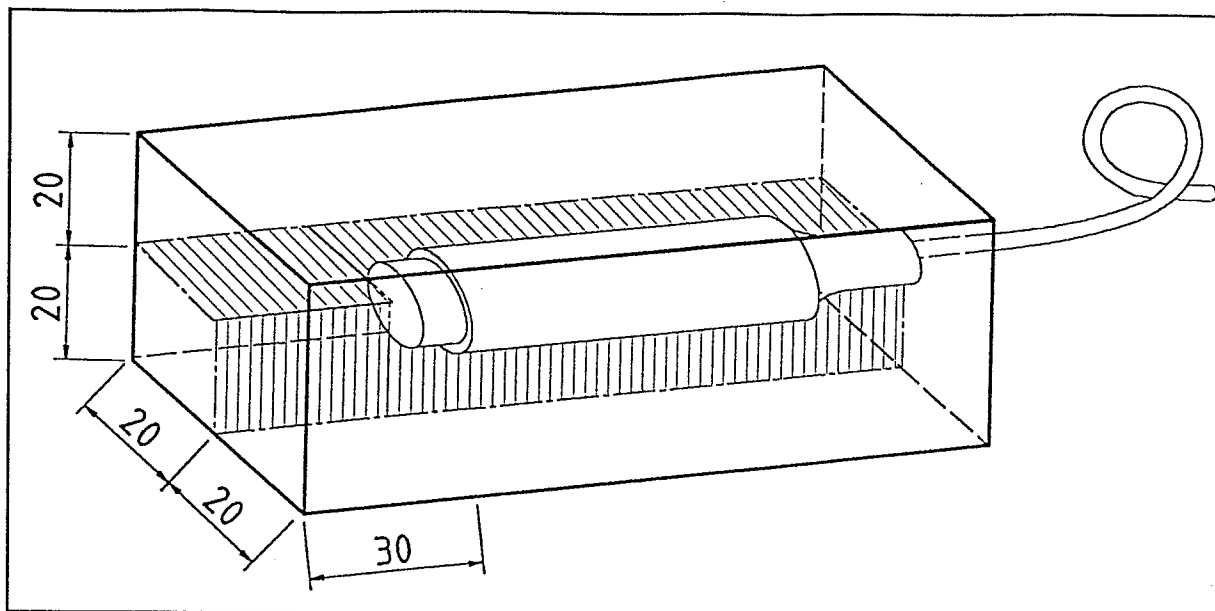
afrettes og tildækkes med plastfolie. Når mørtelen er hærnet, afformes prismerne. Herefter opbevares emnerne beskyttet mod udtørring og carbonatisering.



Figur 2: Skitse af titannet med påmonteret ledning og tildannet til et zig-zag-formet emne, klar til indstøbning i cementmørtel. Mål er angivet i mm. Efter Hans Arups anvisninger.

4.6 Indstøbning af referenceelektroder

En form (4.3.2) fyldes halvt med mørtel (4.5) og komprimeres ved vibrering. En referenceelektrode (4.2.8) lægges oven på mørtelen i den ene ende af formen med forbindelsesledningerne ført ud af formens endestykke. Se figur 3. Formen fyldes med mørtel, der komprimeres ved vibrering. Oversiden afrettes og tildækkes med plastfolie. Når mørtelen er hærnet, afformes prismet. Prismet knækkes i to halvdele, der hver indeholder en referenceelektrode. Herefter opbevares emnerne beskyttet mod udtørring og carbonatisering.

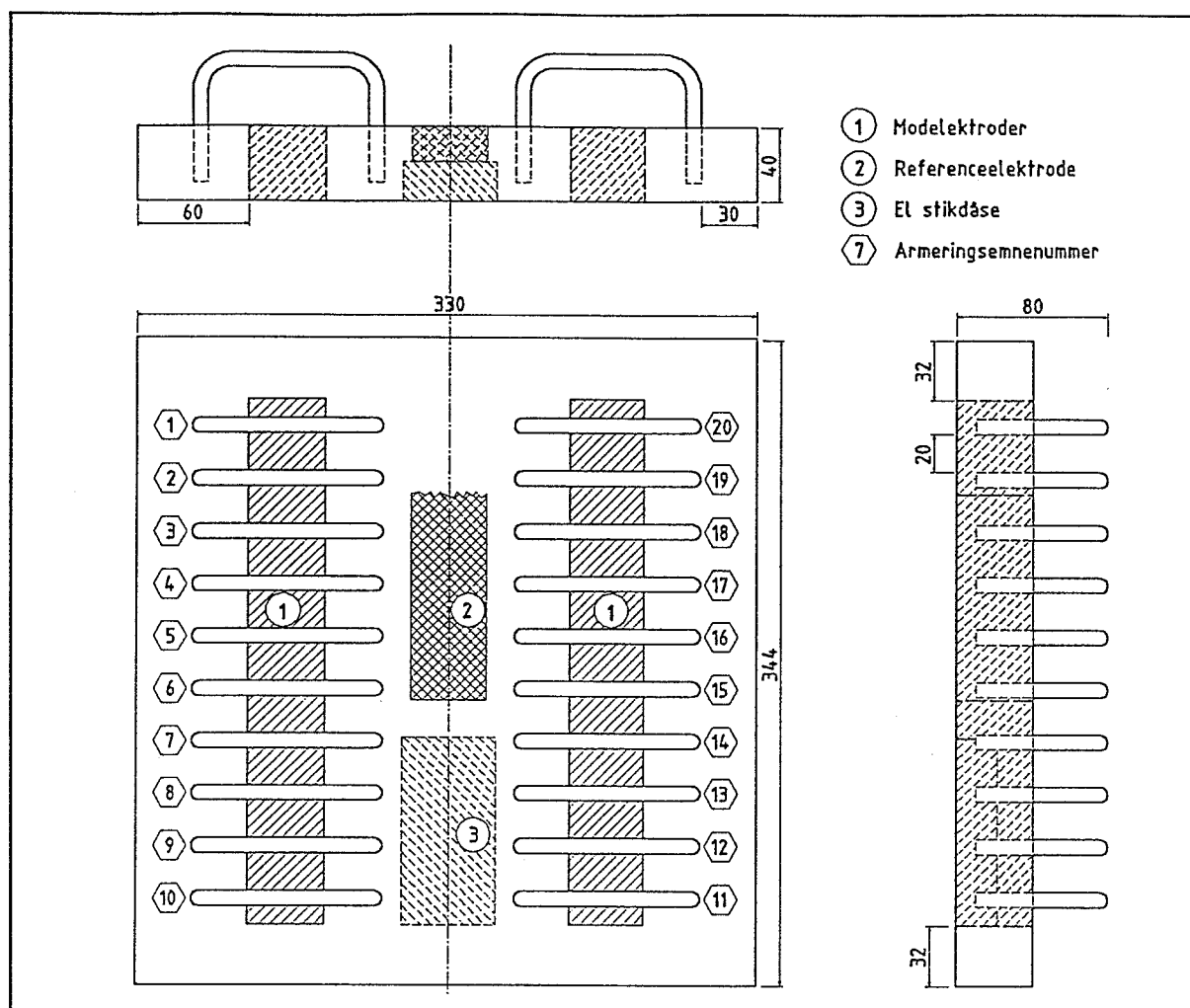


Figur 3: Skitse af emne med referenceelektroder efter indstøbning i cementmørtel. Mål er angivet i mm. Efter Hans Arups anvisninger.

4.7 Sammenstøbning af korrosionscelle

Til hver prøvning fremstilles en korrosionscelle, der består af 20 stk armeringsemner (4.4), 2 stk indstøbte modelektroder (4.5), 1 stk indstøbt referenceelektrode (4.6) samt 1 stk el-stikdåse (4.2.7). De enkelte komponenters indbyrdes placering i formen (4.3.3) fremgår af figur 4.

Ledningsforbindelserne fra armeringsemner, modelektroder og referenceelektrode indføres i el-stikdåsen. Alle komponenter og specielt armeringsemnerne skal være fastholdt under støbning og vibrering i deres rette positioner, jvf figur 4. Til sammenstøbningen af komponenterne anvendes en indstøbningsmasse (4.2.11), der giver en solid indstøbning fri for gennemgående revner og samtidig sikrer en god vedhæftning til den senere påstøbte beton. Armeringsemnerne skal renses for eventuelle stænk fra indstøbningsmassen. Dette foretages senest i forbindelse med afformningen af korrosionscellen.



Figur 4: Skitse, der viser placeringen af de enkelte komponenter i korrosionscellen. Mål er angivet i mm. Efter Hans Arups anvisninger.

4.8 Fremstilling af betonprøveemner

Til hver prøvning fremstilles et betonprøveemne indeholdende en korrosionscelle (4.7). Inden udstøbningen af prøveemnet renses armeringsemnerne med acetone (4.2.10). Korrosionscellen placeres i bunden af formen (4.3.4).

Der fremstilles en beton med den ønskede betonsammensætning (betonen bør være afprøvet tidligere for at sikre, at sammensætningen og den hærtnede beton er som forventet). Betonen udstøbes i tre lige tykke lag, og der komprimeres ved vibrering mellem udstøbning af hvert lag. Til sidst afrettes oversiden, og

der tildækkes med plastfolie. Korrosionscellen skal påstøbes så meget beton, at armeringsemnerne er dækket med det ønskede dæklag og yderligere 80 mm til udskæring af betonklodser (4.9.2).

Prøveemnet afformes ved en alder på 1-3 døgn og opbevares derefter indpakket i kraftig plastfolie.

4.9 Prøvetilberedning

Ved en betonalder på 14 døgn gennemskæres det udstøbte prøveemne ved brug af vandkølet diamantsav, således at det ønskede dæklag opnås. Prøveemnets savskårne flade må højst have en planhedsafvigelse på $\pm 0,5$ mm af hensyn til fastlæggelse af dæklagstykkelsen. Det kan være nødvendigt at planslibe snitfladen ved brug af vandkølet slibeudstyr.

4.9.1 Prøveemne med korrosionscelle.

Prøveemnet afrenses for skære-/slibeslam ved skrubning under rindende vand. Det tørres derefter til en stabil hvidtør tilstand og belægges med en ca 1 mm tyk epoxy- eller polyurethanbelægning (4.2.6) på de fire sideflader. Der skal drages omsorg for, at intet belægningsmateriale påføres fladen, der skal eksponeres. Det skal sikres, at de af materialeleverandøren foreskrevne påførings- og hærdeforhold bliver overholdt. Under forarbejdningen skal prøveemnet beskyttes mod udtørring og carbonatisering.

Når belægningen er hærdnet, og senest ved en betonalder på 21 døgn, neddyppes prøveemnet halvt (med den savskårne flade neddykket) i en mættet $\text{Ca}(\text{OH})_2$ -opløsning ved $23 \pm 2^\circ\text{C}$ i mindst 7 døgn.

4.9.2 Betonklodser til bestemmelse af chloridprofiler.

Af den afskårne betonplade fremstilles mindst 3 betonklodser på ca 70·100·100 mm. Disse forarbejdes og lagres nøjagtigt på samme måde som prøveemnet med korrosionscellen. Alle sider forsegles med undtagelse af den savskårne flade, der er modstykke til prøveemnets eksponeringsflade. Denne flade skal eksponeres sammen med prøveemnet indeholdende korrosionscellen.

4.10 Fremgangsmåde ved prøvning

Der fremstilles en vandig NaCl-opløsning med en koncentration på $165 \text{ g} \pm 1 \text{ g}$ NaCl pr liter opløsning. Eksponeringsvæskens tempera-

tur skal være $23 \pm 2^\circ\text{C}$. Temperaturen skal måles mindst en gang dagligt.

Når prøveemnet og prøveklodserne har været vandlagret i mindst 7 døgn og har opnået en alder på 28 mindst døgn, skal de nedlægges i vandmættet, overfladetør tilstand i saltopløsningen. Opløsningens NaCl-koncentration kontrolleres mindst en gang før, en gang pr uge under og en gang efter eksponeringen. Hvis koncentrationen afviger mere end ± 3 g NaCl pr liter opløsning, skal eksponeringsvæsken justeres til $165 \text{ g} \pm 1 \text{ g}$ NaCl pr liter opløsning.

De indstøbte armeringsemner (20 stk) holdes under potentiostatisk kontrol i hele forsøgsperioden, idet dog 2 stk armeringsemner korroderer frit. Der anvendes 9 potentialer (i forhold til referenceelektroden ERE 10) fordelt på armeringsemnerne som anvist i tabel 1.

Armeringsemne nr	1 + 11	2 + 12	3 + 13	4 + 14	5 + 15
Potential [mV]	-450	frit	-350	50	-250
Armeringsemne nr	6 + 16	7 + 17	8 + 18	9 + 19	10 + 20
Potential [mV]	0	-200	-50	-150	-100

Tabel 1: Oversigt over potentialværdier for armeringsemner under potentiostatisk kontrol. Nummerering af armeringsemner er vist på figur 4.

Korrosionsaktiviteten følges ved daglige målinger af den fornødne strøm ($1\text{-}5 \mu\text{A}$) til opretholdelsen af potentialet til hvert armeringsemne. Korrosionsinitiering regnes at være sket når den anodiske strøm til prøverne er over $30\text{-}50 \mu\text{A}$, hvorefter det pågældende armeringsemne frakobles potentialstyringen.

Når antallet af indstøbte emner med konstateret korrosionsinitiering er hhv 1, 5 og 20 stk, måles chloridindtrængningen i den pågældende beton ved at analysere for syreopløseligt chloridindhold som funktion af dybden under den eksponerede overflade iht principperne i APM 302 [2] i en af de tilhørende prøveklodser. Der skal affræses mindst 8 lag til et profil. Lagenes tykkelse skal afpasses efter det forventede chloridprofil og det aktuelle dæklag (profilet bør nå dybere end dæklagets tykkelse). Det

yderste lag skal dog altid have en tykkelse på mindst 1,0 mm. Det skal endvidere sikres, at der for hvert lag opnås en pulverprøve på mindst 5 g tørt materiale. For hver udtaget pulverprøve af betonstøv bestemmes dybden under den eksponerede overflade som gennemsnittet af 5 jævnt fordelte målinger med en nøjagtighed på mindst 0,1 mm.

Prøvernes syreopløselige chloridindhold bestemmes iht [3] eller tilsvarende metode med samme eller bedre nøjagtighed.

4.11 Beregning af resultater

De målte chloridindhold optegnes som funktion af dybden under den eksponerede overflade.

Værdierne af C_s og D bestemmes ved at tilpasse funktionen (f4.1) til de målte chloridindhold ved ikke-lineær regressionsanalyse efter mindste kvadraters metode. Profilets første punkt bestemt fra den savskårne flade udelades ved regressionsanalysen. De øvrige punkter vægtes ens.

$$(f4.1) \quad C_x = C_s - (C_s - C_i) \operatorname{erf}(x/\sqrt{4Dt})$$

hvor C_x [mas%] er chloridkoncentrationen, målt i dybden x til eksponeringstiden t ,

C_s [mas%] er randbetingelsen mod den eksponerede overflade (beregnet overfladekoncentration),

C_i [mas%] er initialchloridkoncentrationen,

x [m] er dybden under den eksponerede overflade (til midten af et lag),

D [m²/s] er chloriddiffusionskoefficienten,

t [s] er eksponeringstiden (med en nøjagtighed på ± 1 time),

erf er fejlfunktionen bestemt ved (f4.2)

$$(f4.2) \quad \operatorname{erf} z = 2\pi^{-1/2} \int_0^z \exp(-u^2) du$$

Tabeller med værdier for fejlfunktionen findes i almindelige matematiske opslagsværker.

Korrelationen mellem de målte chloridindhold og de tilsvarende beregnede chloridindhold iht (f4.1) beregnes, fx ved lineær regression.

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Den gennemsnitlige eksponeringstemperatur beregnes. Variationen skal anskueliggøres, fx ved afbildning af den målte temperaturkurve.

Den gennemsnitlige chloridkoncentration af eksponeringsvæsken beregnes. Variationen skal anskueliggøres, fx ved angivelse af de målte værdier.

4.12 Angivelse af resultat

Initialchloridkoncentrationen, C_i , og den beregnede overfladekoncentration, C_s , angives i mas% af tør beton med 2 decimaler.

Diffusionskoefficienten, D , angives i m^2/s med 2 betydnende cifre.

For hvert armeringsemne angives tiden til korrosionsintiering regnet fra eksponeringens start.

Det aktuelle betondæklag målt fra den savskårne overflade angives med en nøjagtighed på 1 mm.

Ved anvendelse af de beregnede chloridindtrængningsparametre C_s og D , til hver af de aktuelle eksponeringstider samt den målte værdi for C_i , beregnes værdien af chloridkoncentrationen $C_{cr,E}$ for hvert af armeringsemnerne. Værdierne $C_{cr,E}$ angives for hvert armeringsemne med 2 decimaler sammen med de tilhørende potentialer i mV og sammenhængen illustreres grafisk ved afbildning af $C_{cr,E}$ som funktion af potentiallet.

Prøvningsresultaterne kan evt. angives i form af et rapportskema, se afsnit 6.

5. Prøvningsrapport

Eventuel prøvningsrapport skal indeholde mindst følgende information:

- 5.1 Navn og adresse på prøvningslaboratoriet og stedet for prøvningens udførelse, såfremt dette er forskellig fra prøvningslaboratoriets adresse.
- 5.2 Dato og entydig identifikation af rapporten.
- 5.3 Navn og adresse på rekvirenten.
- 5.4 Dato for betonfremstillingen og prøvningens udførelse.
- 5.5 Angivelse af prøvningsmetoden.

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- 5.6 Alle afvigelser, tilføjelser eller udeladelser fra prøvningsmetoden og øvrige oplysninger af betydning for bedømmelsen af resultatet.
- 5.7 Angivelse af de gennemsnitlige værdier for temperaturen og chloridkoncentrationen i eksponeringsvæsken.
- 5.8 Prøvningsresultat.
- 5.9 Underskrift og titel eller anden markering af person(er), der er teknisk ansvarlig(e) for rapporten.
- 5.10 Dato for rapportens frigivelse.

6. Rapportskema

Prøvningens resultat kan evt. angives på et rapportskema som vist på næste side.

AEClaboratoriet

Chloridtærskelværdi

APM 303

Chloridtærskelværdi for armeringskorrosion

AEClaboratoriet
Staktoften 20
DK-2950 Vedbæk
Tlf. 45 66 12 66

iht. APM 303

Lab nr:
Prøve nr:
Init:
Udskrift:

Materialedata

Armeringsemner	
Referenceelektrode	
Kalibreringspotential	
Modelektrode	
Betonrecept	Støbedato

Eksponeringsdata

Dækklag

Prøveemne (mål i mm)

Dato for start	/ /	Tilstræbt
----------------	-----	-----------

Eksponeringsvæske

Temperatur [°C]

Chloridkonc. [g Cl/l]

Spændingsdelers ID-kode

Kode 10 [mV]

Kode 1 [mV]

De aktuelle dækklag er angivet på skitsen af prøveemnet.
(Prøveemnet er set fra den eksponerede side).

Armeringsemne nr									
10	9	8	7	6	5	4	3	2	1
←-elektronik									
Armeringsemne nr									
11	12	13	14	15	16	17	18	19	20

Måleværdier

Potentialer måles mod referenceelektroden

Emne nr	Eo [mV]	Ec [mV]	Tj [dato]	Ti [døgn]	Ccr [mas%]
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Symbolforklaring

Eo : Potential før eksponering

Ec : Påtrykt potential under eksponering

Tj : Dato for korrosionsstart (afmontering af jumper)

Ti : Tid til korrosionsstart

Ccr : Beregnet chloridkoncentration ved korrosionsstart

AEClaboratoriet	Betonprøvning	APM 321
Staktoften 20 DK-2950 Vedbæk	Hærdnet beton	1. udgave Maj 1991
45 66 12 66	Kvalitativ 2D-Chloridindtrængning	Init: JMF

1. Formål og anvendelsesområde

Denne metode beskriver fremgangsmåden ved kvalitativ vurdering af chloridindtrængning i hærdnet beton eller andre cementbundne materialer, der har været eksponeret i chloridholdig væske.

Metoden er udviklet for at muliggøre kvalitative studier af chloridindtrængning i revner, støbeskel eller andre defekter med udstrækning fra en betonoverflade og (fortrinsvis vinkelret) ind i betonen. Metoden kan også anvendes til formål, hvor man fx blot ønsker et kvalitativt billede af chloridindtrængningen omkring sten mv.

Metoden beskriver fremgangsmåden ved kunstig eksponering for natriumchlorid i laboratoriet. En tilsvarende fremgangsmåde kan benyttes ved undersøgelser af prøver udtaget fra konstruktioner udsat for virkelig chloridpåvirkning.

2. Referencer

- DS 423.22, 2. udgave. Marts 1984. Betonprøvning. Hærdnet beton.
Udboring af cylindre til styrkebestemmelse.
- DS 423.28, 1. udgave. Marts 1984. Betonprøvning. Hærdnet beton.
Chloridindhold.

3. Prøveudtagning

Prøvelegemerne kan være udborede kerner eller støbte cylindre, der er repræsentative for den aktuelle beton og/eller konstruktion. Betonen skal være hærdnet til minimum 28 modenhedsdøgn. Der bør indgå mindst tre prøvelegemer i prøvningen. Størrelsen bør være mindst $\varnothing 75 \cdot 100$ mm.

4. Kort prøvningsbeskrivelse

En vandmættet betonprøve udsættes på en plan flade for ensidig påvirkning af natriumchloridholdigt vand. Efter en kendt eksponeringstid skæres en eller flere skiver af prøven vinkelret på prøvens eksponerede flade. De friske snitflader påsprøjtes

indikatorvæske, hvorefter indtrængningen af chloridioner kan ses via et rødt farveomslag.

5. Kemikalier

- 5.1 Redestilleret eller demineraliseret vand.
- 5.2 Calciumhydroxid ($\text{Ca}(\text{OH})_2$), teknisk kvalitet.
- 5.3 Natriumchlorid (NaCl), teknisk kvalitet.
- 5.4 Salpetersyre (HNO_3), koncentreret, analysekvalitet.
- 5.5 Sølvnitrat standardopløsning (AgNO_3), ca 0,1N.
- 5.6 Kaliumdichromatopløsning ($\text{K}_2\text{Cr}_2\text{O}_7$), 5%.
- 5.7 2-komponent (chloridiondiffusionstæt) polyurethan- eller epoxybaseret maling (membran), fx Irete IR 3360.

6. Apparatur

- 6.1 Vandkølet diamantsav.
- 6.2 Vægt, nøjagtighed bedre end $\pm 0,01\text{g}$.
- 6.3 Termometer, nøjagtighed bedre end $\pm 1^\circ\text{C}$.
- 6.4 Eksponeringskar med væskeomrøring.
- 6.5 Forstøverflaske, 2 stk, ca 100ml.

7. Prøvetilberedning

For hvert af de udtagne emner udføres følgende:

Emnet nedlægges i en mættet $\text{Ca}(\text{OH})_2$ -opløsning ved ca 23°C i en tæt lukket beholder. Beholderen skal fyldes helt op for at minimere carbonatisering af væsken. Efter et døgn bestemmes m_{ot} ved at veje emnet i overfladetør tilstand (note 7.1). Lagringen i den mættede $\text{Ca}(\text{OH})_2$ -opløsning fortsættes, indtil m_{ot} ikke ændres med mere end 0,1mas% pr døgn. Derefter tørres emnet til en stabil hvidtør tilstand og belægges med en ca 1 mm tyk epoxy- eller polyurethanbelægning på alle overflader undtagen den flade, der skal eksponeres. Der skal drages stor omhu for, at intet belægningsmateriale påføres fladen, der skal eksponeres. Det skal sikres, at de af materialeleverandøren foreskrevne påførings- og hærdeforhold bliver overholdt. Når belægningen er hærnet, nedlægges emnet atter i $\text{Ca}(\text{OH})_2$ -opløsningen til m_{ot} stabiliseres som beskrevet ovenfor.

Note 7.1: m_{ot} skal opnås ved at aftørre emnet med en ren serviet eller tilsvarende, således at overfladen netop ikke står blank. Dette gøres ved at væde servietten i væsken, som emnet har ligget i, og derefter vride servietten så meget, at den er i stand til at absorbere al væske på emnets overflade.

8. Fremgangsmåde ved prøvning

8.1 Eksponering

Der fremstilles en vandig NaCl-opløsning med en koncentration på $165\text{g} \pm 1\text{g}$ NaCl pr liter opløsning. Opløsningens NaCl-koncentration kontrolleres mindst en gang før, en gang under og en gang efter eksponeringen. Hvis koncentrationen afviger mere end $\pm 3\text{g}$ NaCl pr liter opløsning, skal eksponeringsvæsken justeres til $165\text{g} \pm 1\text{g}$ NaCl pr liter opløsning. Eksponeringsvæskens temperatur, der skal være 20°C - 25°C med en tilstræbt middeltemperatur på 23°C . Temperaturen skal måles mindst en gang dagligt.

Emnet nedlægges i overfladetør tilstand (note 7.1) i saltopløsningen.

8.2 Fremstilling af chloridindikator

Der fremstilles to væsker, der tilsammen udgør en såkaldt chloridindikator, på følgende måde:

Af 5.4 og 5.5 fremstilles en blandingsvæske med pH-værdi på ca 2; herved haves indikatorens komponent A. Blandingen overføres til en forstøverflaske. Flasken mærkes tydeligt med et A.

Væsken 5.6 er indikatorens komponent B. Den overføres ligeledes til en forstøverflaske. Flasken mærkes tydeligt med et B.

8.3 Måling af chloridindtrængning

Efter mindst 35 døgn eksponering skæres en eller flere ca 10mm tykke skiver af emnet vinkelret på den eksponerede overflade indenfor en diameter, der er ca 10mm mindre end kernens fulde diameter. Herved undgås eventuelle randeffekter og forstyrrelser fra den påførte belægning.

For hver snitflade gøres følgende:

Snitfladen renses for skæreslam med en let vædet ren serviet. Fladen aftørres med en serviet, så den i løbet af få minutter fremstår mat. Komponent A påsprøjtes den vandretliggende flade,

og det tilses vha skråt indfaldende lys, at fladen er belagt med en ubrudt væskefilm. Komponent A påsprøjtes supplerende i nødvendigt omfang. Fladen betragtes nøje, mens væsken dækker den. Når der ses et svagt og jævnt udbredt brunligt omslag på fladen, aftørres væskefilmen, så fladen igen fremstår mat. Komponent B påsprøjtes umiddelbart herefter, så en ny ubrudt væskefilm dækker fladen. Det brunlige område farves herved rødligt i en skarpere kontrast til det ufarvede område. Fladen aftørres atter.

Det nu rødligt farvede område på fladen angiver et område, hvor indikatoren ikke er påvirket af fladens chloridindhold. Skillelinien mellem det farvede og det ufarvede repræsenterer en grænse, hvor chloridindholdet anses for værende konstant. Den absolutte værdi for chloridindholdet i denne grænselinie afhænger af en række faktorer. Såfremt et skøn for denne værdi ønskes i et konkret tilfælde, anbefales det at måle et chloridprofil vinkelret på overfladen, fx efter princippet angivet i APM 302, i umiddelbar nærhed af den analyserede flade.

9. Angivelse af resultat

Resultatet angives med et farvefoto, hvorpå en målestok er vist i samme plan som de(n) påsprøjtede flade(r). På fotoet skal grænsen for farveomslaget kunne ses.

10. Prøvningsrapport

Eventuel prøvningsrapport skal indeholde mindst følgende information:

- 10.1 Navn og adresse på prøvningslaboratoriet og stedet for prøvningens udførelse, såfremt dette er forskellig fra prøvningslaboratoriets adresse.
- 10.2 Dato og entydig identifikation af rapporten.
- 10.3 Navn og adresse på rekvirenten.
- 10.4 Beskrivelse og mærkning af prøven, samt eventuelt angivelse af prøvens placering i konstruktionen.
- 10.5 Dato for prøvens modtagelse og prøvningens udførelse.
- 10.6 Angivelse af prøvningsmetoden.

- 10.7 Beskrivelse af prøveudtagningen, hvis det er relevant.
- 10.8 Alle afvigelser, tilføjelser eller udeladelser fra prøvningsmetoden og øvrige oplysninger af betydning for bedømmelsen af resultatet.
- 10.9 Prøvningsresultat.
- 10.10 Underskrift og titel eller anden markering af person(er), der er teknisk ansvarlig(e) for rapporten.
- 10.11 Dato for rapportens frigivelse.

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Staktoften 20 DK-2950 Vedbæk	Concrete repair	1st edition January 1994
+45 45 66 12 66	Chloride penetration resistance of paint on concrete	Init: JMF

1. **Object and application**

This test method describes a procedure for measuring the protective effect of paints or paint systems (cf. definition in note 1) against ingress of chlorides into concrete exposed to spray of water containing chlorides.

The measuring principle is based on comparative measurements of the ingress of chlorides into unpainted references as well as painted samples.

Note 1: In this test method, a paint or a paint system is either a product which forms a thin layer or a product which acts as an impregnant. The product must be able to protect concrete surfaces for 10 or more years as a final treatment. The product must be applied with a brush, roller or sprayer. The product is called a paint system if the treatment is a combination of two or more separately applied layers, possibly of different composition (eg smoothing materials based on portland cement and paint based on polymer dispersion).

2. **References**

- [1] DS 423.28, 1st edition. March 1984. Concrete testing. Hardened concrete. Chloride content. (Danish and English).
- [2] APM 302, 2nd edition. May 1991. Concrete testing. Hardened concrete. Chloride penetration. (Danish and English).
- [3] Udbuds- og anlægskrav. Betonbroer. »Vejledning i overfladebeskyttelse af betonbroer« med tilhørende kontrolmetoder, Vejdirektoratet Broområdet, april 1993. (Eng: Tendering and Construction Regulations. Concrete Bridges. »Guidelines for Surface Protection of Concrete Bridges« with attached Inspection Methods, The Danish Road Directorate, April 1993).
- [4] DS 423.21, 2nd edition. March 1984. Concrete testing. Making and curing of moulded test specimens for strength tests. (Danish and English).
- [5] DS 423.23, 2nd edition. March 1984. Concrete testing. Hardened concrete. Compressive strength of test specimens. (Danish and English).
- [6] Basic Concrete Specification for Building Structures, National Building Agency, 1986.
- [7] J. Crank, Mathematics of Diffusion, 2nd edition, Oxford University Press, 1975.

3. **Sampling**

The specimens (samples and references) shall be cut parts of concrete prisms of minimum dimensions $w \cdot h \cdot l = 90 \cdot 100 \cdot 400$ mm (cf. 7.2) made in the laboratory. The shape is chosen to allow the specimens to be painted using a

method of application suitable for buildings. At least four unpainted references and four painted samples are necessary.

4. Short description of test procedure

Specially manufactured painted and unpainted concrete specimens are conditioned and exposed to cyclically alternating immersion in water containing sodium chloride and air-drying. The immersion time is 2 hours and the drying time is 4 hours.

After 30 weeks of exposure, thin layers of concrete are ground off parallel to the exposed face of the painted samples as well as the unpainted references. The chloride content of the layers is then measured. The original (initial) chloride content of the concrete is measured in an unexposed, unpainted reference. The dry thickness of the paint layers is measured on an unexposed, painted sample.

The apparent chloride diffusion coefficient and the boundary condition of the chloride profile at the exposed surface are calculated for the unpainted references, using the related values of measured depth below the exposed surface and measured chloride content.

The distance (in depth) which the measured chloride profile in the painted samples has to be moved to obtain the best fit with the chloride profile in the references is calculated. This distance is called the »equivalent concrete thickness« of the product.

5. Chemicals

- 5.1 Redistilled or demineralized water.
- 5.2 Sodium chloride (NaCl), technical quality.
- 5.3 Nitric acid (HNO₃), concentrated, GR.
- 5.4 Silver nitrate standard solution (AgNO₃), 0.1N or 0.02N (cf. note 4 page 6).
- 5.5 Ammonium thiocyanate standard solution (NH₄SCN), 0.1N or 0.02N (cf. note 4 page 6).
- 5.6 Ferric ammonium sulphate solution (NH₄Fe(SO₄)₂·12H₂O), saturated, in 10 vol% nitric acid.
- 5.7 2-component (chloride-ion diffusion-proof) polyurethane or epoxy-based paint.
- 5.8 Acrylic polymer-modified mortar with a maximum particle size of 1/2 mm.

6. Apparatus

- 6.0 Equipment to produce concrete prisms.
- 6.1 Water-cooled diamond saw.
- 6.2 Scales, capacity/accuracy better than 25 kg/± 0.01 kg and 600 g/± 0.01 g.
- 6.3 Climate chamber, where air humidity and temperature are kept constant at 50 ± 5% RH and either 23 ± 2°C or 38 ± 2°C.
- 6.4 Drying cupboard, ventilated and thermostat-regulated for 105°C ± 5°C.
- 6.5 Erlenmeyer flasks, about 300 ml.
- 6.6 Burettes, 2 nos, about 25 ml, for titration according to [1].

- 6.7 Bunsen burner or other heat source.
- 6.8 Glass filtering funnel with filter of paper or similar.
- 6.9 Thermometer, accuracy better than $\pm 1^\circ\text{C}$.
- 6.10 Exposure tank with automatically regulated cyclic variation of water level, enabling the samples to be exposed to alternate immersion into water (containing sodium chloride) and drying by air. An example of a test setup is shown in appendix 1, figure A1.1. The given exposure conditions can be achieved by placing the test setup in a chamber with controlled and adjustable climate (i. e. heating, cooling, wetting and drying).
- 6.11 Equipment for grinding off and collecting concrete powder from thin concrete layers (between approx. 0.5 mm and 2 mm).

7. Preparation of specimens

Unless otherwise stated, it is assumed that the preparation of specimens is carried out at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ RH.

7.1 Preparation of concrete prisms (concrete age 0-7 days)

The concrete prisms shall be made of a (non-air-entrained) concrete with following characteristics:

Cement type:	PC(A/HS/EA/G) (Danish classification)
Cement content:	480 kg/m ³ concrete
w/c-ratio:	0.42
d _{max} :	8 mm
Filler content:	5-10 mas% of total aggregate
Fine aggregate content:	45-50 mas% of total aggregate
Fine aggregate type:	Class A acc. to [6] with a low chloride content
Coarse aggregate content:	800-900 kg/m ³ concrete
Coarse aggregate type:	Crushed rock materials (with a low chloride content). One of the following commodities are suitable:
	Rønne Granite
	Glensanda Granite
	Falkenberg Granite
	Walhamn Granite

The total amount of water shall be determined as the total of added water, water content in additives and water content in aggregates adjusted for absorption.

The actual concrete composition used and information concerning the composition of aggregates shall be stated in the test report in the shape of completed forms acc. to page 1 and 2 of the concrete form in [6].

The concrete shall be mixed in a pan mixer. The concrete shall be cast in two layers of equal thickness in moulds made of a concrete-resistant, non-water-

absorbent material with minimum inside dimensions $w \cdot h \cdot l = 90 \cdot 100 \cdot 400$ mm. The concrete shall be compacted between casting of each layer using vibration. The concrete surface shall be levelled off and covered immediately with an 0.2-0.35 mm thick plastic film. In addition 3 nos. $\text{Ø}100 \cdot 200$ mm cylinders acc. to [4] shall be made.

The concrete prisms shall be kept covered until they are 7 days old. The prisms shall be demoulded at the age of 1-3 days.

At the age of 7 days the compressive strength shall be tested acc. to DS 423.23. (The expected value of the measured compressive strength is 38-46 MPa.)

7.2 Preparation of specimens (concrete age 7 days)

At the age of 7 days the concrete prisms shall be cut using a water-cooled diamond saw. The prisms shall first be cut in the longitudinal, horizontal, central plane and then in the transverse, vertical, central plane, thus producing 4 specimens with minimum dimensions: $w = 90$ mm, $h = 50$ mm - cutting loss, $l = 200$ mm - cutting loss.

To remove all cutting slurry the surfaces of the specimens shall be carefully scrubbed with a brush under running water.

Each specimen shall be judged visually with respect to homogeneous distribution of aggregate and content of encapsulated air. Specimens with abnormal separation and many air bubbles shall be rejected.

The cut surfaces from the longitudinal, horizontal, central plane shall be designated »face A«, while the other surfaces shall be designated »face B«. All 4 specimens originating from the same concrete prism shall be kept together and numbered acc. to figure 1.

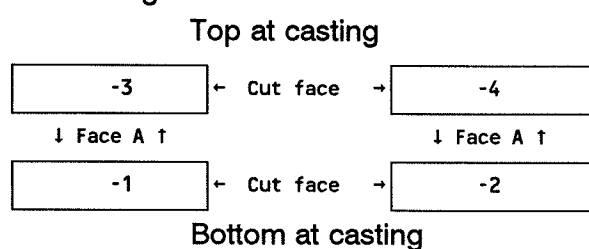


Figure 1: Elevation of concrete prism. Sketch showing the division of one prism into specimens after cutting. The given numbering shall be used.

7.3 Coating of sides and bottom (concrete age 7-14 days)

After fabrication of the specimens, face A shall be covered in such a way that it is protected against drying and contamination from the coating material during the subsequent sealing of faces B. Any pores in faces B shall then be filled, using a suitable material (5.8), and faces B of the specimens shall be covered by more than one application, using an epoxy or polyurethane coating (5.7). Face B must be dried to a stable white-dry condition before application of coating commences. An approx. 1 mm thick layer without pin-holes shall be applied. It must be

ensured that the method of application and hardening prescribed by the supplier of the coating material is observed.

The specimens shall be stored as prescribed until a concrete age of 14 days.

7.4 Application of paint to specimens (concrete age 14-21 days)

It must be ensured that there is good ventilation around each specimen. The specimens shall be left uncovered for 1 day before commencement of paint application.

When the concrete is 15 days old, at least 4 samples shall be prepared by applying paint to specimens according to the method prescribed by the supplier.

Each operation shall be registered separately (cf. note 2 page 5). During the application and drying the samples shall be left in an upright position. The following parameters shall be registered:

- Method of application (unless otherwise prescribed by the supplier, a 20 mm broad flat brush shall be used)
- Prewetting and/or dilution, if any
- Mixing proportions (by mass) for multicomponent products, if any.
- Consumption per m² for each sample (by recording the mass of tools, containers and material, respectively, before and after every operation).
- Time interval between operations.
- Temperature, relative humidity and other conditions during the application.

Note 2: Normally 4-5 operations are necessary to obtain the correct composition and layer thickness. The paint application is often composed as follows: Two applications of pore-filling material, perhaps priming, and finishing with 2-3 applications of final paint.

At the same time as paint application commences, references shall be made by sealing at least 4 specimens using close-fitting plastic film.

After application, the samples shall be left to dry until a concrete age of 21 days.

7.5 Conditioning of paint (concrete age 21-49 days)

At a concrete age of 21 days, all samples and references shall be placed in a climate chamber with ventilated air and exposed as follows:

- 14 days in air.
- The temperature shall then be gently increased for 4 hours to $38 \pm 2^\circ\text{C}$.
- 7 days at a temperature of $38 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ RH.
- The temperature shall then be gently reduced for 4 hours to $23 \pm 2^\circ\text{C}$.

The plastic film shall be removed from 3 references, which shall be transferred together with 3 samples to the exposure tank (concerning use of the last reference, see 8.3). The specimens shall be placed upright in the exposure tank

(6.10) and positioned in such a way that no shadow effect occurs during the drying periods. The test setup (6.10) shall then be filled with pure drinking water. There must be sufficient water to ensure that all the specimens are just completely immersed at the same time. At a concrete age of 42 days all the specimens in the exposure tank shall be exposed as follows:

- 7 days, alternating between 4 hours drying and 2 hours immersion in water.
- The period shall end with 2 hours immersion in water.

The specimens shall then be removed from the exposure tank and dried to surface-dry condition (cf. note 3), whereupon their mass shall be measured ($m_{ot,start}$).

Note 3: m_{ot} is achieved by drying the specimen with a clean cloth or similar leaving it damp but not wet. This is obtained by wetting the cloth with the liquid in which the specimen has been immersed and then wringing it out sufficiently to absorb any liquid adhering to the surface of the specimen.

8. Test procedure

8.1 Exposure conditions

An aqueous NaCl-solution shall be prepared with a concentration of $165 \text{ g} \pm 1 \text{ g}$ NaCl per litre solution (e.g. by using the liquid from the conditioning of the specimens (7.5)).

Not later than the day on which the conditioning (7.5) is completed, the specimens and the references shall be placed as previously in the exposure tank (6.10).

After this the exposure conditions shall be as follows:

State	Duration	Exposure conditions
Wetting period	2 hours (filling must take place in less than 15 minutes)	Liquid temperature $23 \pm 2 \text{ }^\circ\text{C}$ NaCl-concentration $165 \pm 1 \text{ g/litre}$ at start and after adjustment, otherwise $165 \pm 9 \text{ g/litre}$
Drying period	4 hours (emptying must take place in less than 15 minutes)	Air temperature $23 \pm 2 \text{ }^\circ\text{C}$ Air humidity $50 \pm 5 \text{ \%RH}$ Wind velocity $1.3 \pm 0.7 \text{ m/s}$

The NaCl-concentration of the solution must be checked at least once before the exposure, once every week during it, and once after it. If the concentration deviates more than $\pm 9 \text{ g}$ NaCl per litre solution, the exposure liquid must be adjusted to $165 \text{ g} \pm 1 \text{ g}$ NaCl per litre solution.

The temperature of the exposure liquid must be measured at least once a day. The air temperature and humidity must be measured at least 6 times every

day. The wind velocity in front of the samples shall be checked once during the test period.

The exposure of the specimens shall be stopped after 30 weeks \pm 1 week duration. The specimens shall then immediately be conditioned to surface-dry condition (cf. note 3), whereupon their mass shall be measured ($m_{ot,stop}$). The chloride profile in three samples and three references shall be measured.

8.2 Measuring the chloride profiles

The analysis is carried out immediately after the exposure by grinding material off in layers parallel to the exposed surface. The grinding shall be performed within an area of approx. 25 · 140 mm positioned at least 25 mm from the edge of the samples.

On the references at least 8 layers shall be ground off. The first layer ground off must always have a thickness of approx. 1 mm. A thickness of approx. 5 mm is recommended for the other layers.

The paint layer on the samples shall be ground off until the concrete is visible. Then 4-6 layers shall be ground off. The thickness of the layers shall be adjusted according to the expected chloride profile (a layer thickness of approx. 1 mm is recommended as a starting point).

It must be ensured that a powder sample of at least 5 g of dry concrete dust is obtained from each layer. For each powder sample of concrete dust collected, the depth below the exposed surface shall be calculated as the average of five uniformly distributed measurements with an accuracy of at least 0.1 mm.

The acid-soluble chloride content of the powder samples shall be determined according to [1] or by a similar method with the same or better accuracy. The measured chloride contents shall be plotted as a function of the depth below the exposed surface.

Note 4: The expected values of chloride content of surface protected concrete are below 0.05 mas% after exposure as prescribed in this method. For that reason, a higher accuracy of measurement is needed than can be achieved with the given sample sizes and chloride analysis by Volhard titration according to [1]. It is therefore recommended that the normality of AgNO_3 and NH_4SCN used in the titration shall be reduced from 0.1 N (as prescribed in [1]) to 0.02 N.

8.3 Measuring the initial chloride content

A representative sub-sample of approx. 20 g shall be made from the non-exposed reference, e.g. by grinding. The acid-soluble chloride content of the sub-sample shall be measured according to [1] (cf. note 4) or by a similar method with the same or better accuracy. The chloride content found is the initial chloride content of the specimen, C_i .

8.4 Measuring the thickness of the paint system

The thickness of each layer of the product shall be measured on the non-exposed sample by microscopy according to VDPRØV.11/93 in [3].

9. Calculation of results

9.1 Exposed references

The values of C_s and D_{app} shall be determined by fitting the equation (eq1) 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 shall be omitted from the regression analysis. The other points shall be weighted equally.

$$C_x = C_s - (C_s - C_i) \cdot \operatorname{erf} \left(\frac{x}{2\sqrt{D_{app}t}} \right) \quad (\text{eq1})$$

where

C_x	[mas%]	is the chloride concentration, measured at the depth x , at the exposure time t
C_s	[mas%]	is the boundary condition at the exposed surface (calculated surface concentration)
C_i	[mas%]	is the initial chloride concentration measured on the concrete slice (8.3)
x	[m]	is the depth below the exposed surface (to the middle of a layer)
D_{app}	[m ² /s]	is the apparent chloride diffusion coefficient
t	[s]	is the exposure time (with an accuracy better than 5 hours)
erf		is the error function determined by means of (eq2)

$$\operatorname{erf}(z) = \frac{2}{\sqrt{\pi}} \int_0^z e^{-u^2} du \quad (\text{eq2})$$

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

The correlation between the measured chloride contents and the corresponding chloride contents calculated according to (eq1) shall be determined, e.g. by means of a linear regression analysis.

The initial chloride concentration, C_i , and the calculated surface concentration, C_s , shall be given in mas% of dry concrete to three decimal places. The apparent chloride diffusion coefficient, D_{app} , shall be given in m²/s (or in mm²/yr, as 1 m²/s = 3,1536 · 10¹³ mm²/yr) to two significant digits. (The expected values for C_s and D_{app} of the actual concrete quality are 0.5-0.6 mas% and 3 · 10⁻¹² - 4 · 10⁻¹² m²/s (95 - 130 mm²/yr), respectively, at an exposure time of 30 ± 1 weeks.)

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9.2 Exposed samples

The chloride penetration resistance of the product shall be stated as the equivalent concrete thickness, L', calculated according to equation (eq3) by means of a regression analysis in accordance with the method of least squares fit:

$$L' = 2\sqrt{D_{app} \cdot t} \cdot \operatorname{erf}^{-1} \left(\frac{C_s - C_x}{C_s - C_i} \right) - x \quad (\text{eq3})$$

When calculating L' for each measured profile (x,C_x) in the samples, the mean values of C_s and D_{app} (determined as given above for the references) shall be used.

9.3 Climate data

Mean values shall be calculated for the temperature of the exposure liquid and the temperature and humidity of the air during the exposure time, and the variation shall be illustrated by means of, for example, a graph of the measured values.

The mean chloride concentration of the exposure liquid shall be calculated and the variation illustrated, e.g. by giving the measured values.

10. Presentation of result

The equivalent concrete thickness, L', shall be given in mm to one decimal point. The thickness of the product shall be given in mm to two decimal points. These results can be given in a form as shown below:

References Subject no	Identification	C _s [mas%]	C _i [mas%]	t [days]	D _{app} [m ² /s]	
R1						
R2 R3 R4						
Mean						
Samples Subject no	Identification	Thickness [mm] Mortar Primer Paint			t [days]	L' [mm]
1						
2 3 4						
Mean						

*

11. Test report

A test report must at least contain the following information:

- 11.1 Name and address of the test laboratory, and the place at which the tests were performed if different from the laboratory address.
- 11.2 Date and clear identification of the report.
- 11.3 Name and address of the client.
- 11.4 Description and marking of the tested product.
- 11.5 Date of receipt of the product and performance of the test.
- 11.6 Specification of the number, title and edition of the test method.
- 11.7 Description of the sample preparation, including information concerning the concrete composition used and the measured 7-day strength (7.1). In addition, the procedure used to apply the paint, if different from the given method (7.4).
- 11.8 All deviations from or additions to the test method, and any omissions, together with other information of importance for judging the result.
- 11.9 Indication of the recorded air temperature and humidity plus NaCl-concentration and temperature of the exposure liquid during the exposure (9.3).
- 11.10 Test result (10).
- 11.11 Signature and title or other identification of person(s) responsible for the technical content of the report.
- 11.12 Date of release of the report.

Appendix 1

Example of test setup layout

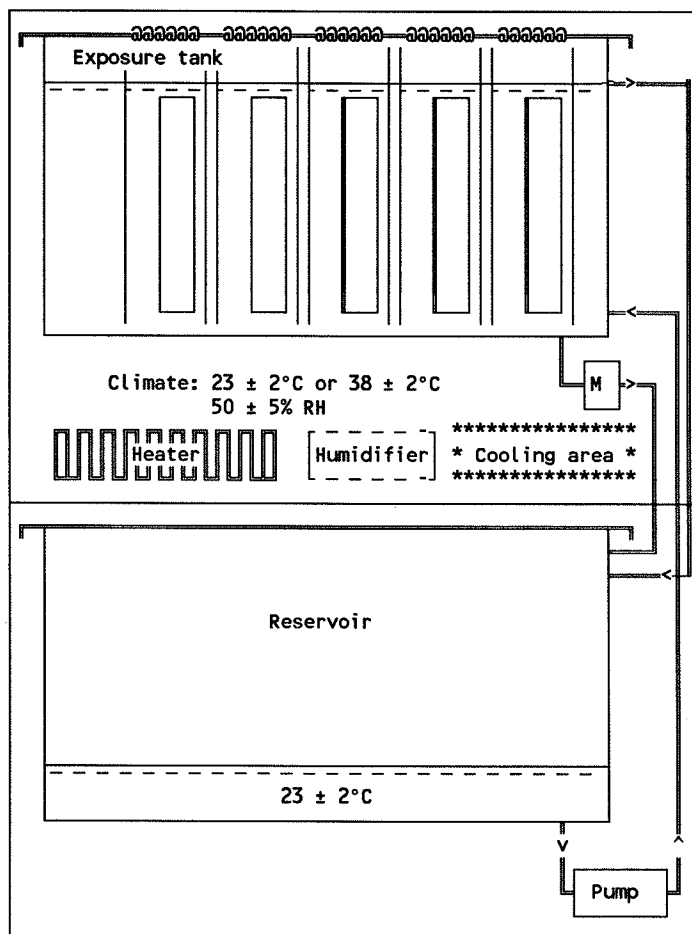


Figure A1.1: Example of test setup layout. In the exposure tank it must be possible to alternate between a water level as shown in the figure and a dry climate at $23 \pm 2^\circ\text{C}$, $50 \pm 5\% \text{ RH}$, and a wind velocity of $1.3 \pm 0.7 \text{ m/s}$. The situation shown is the wet situation. The following symbols are not self-explanatory:

M denotes magnet valve (of the type »normally open« which ensures that the specimens will not be immersed in case of power failure)

@@@@@ denotes ventilator

The exposure tank is equipped with a grating in the bottom which allows air circulation below the samples. The outlet is level with the bottom so that large quantities of water not are left in the tank in the dry periods.

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Recommended Procedure for Determination of Chloride Diffusion Coefficient by Using CTH Rapid Method

(April 1996, 2 nd draft)

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1. SCOPE

- 1.1 This procedure is for the determination of chloride diffusion coefficient in aged ordinary concrete specimens.

2. APPARATUS

2.1 Vacuum Saturation Apparatus

- 2.1.1 Vacuum container: capable of containing at least one piece of rubber sleeve assembled with specimen and clampers (Ref. 2.2.1, 2.2.2 and 4.1.1).
- 2.1.2 Vacuum pump: capable of maintaining a pressure of less than 10 mmHg (1.33 kPa) in container.

2.2 Potential Supply Apparatus

- 2.2.1 Rubber sleeve: 150 mm long, cut from a $\text{Ø}115/100$ mm (outer/inner) multi-layer silicon rubber pipe.
- 2.2.2 Clamper: $\text{Ø}115 \times 20$ mm stainless steel (see Fig. 1).
- 2.2.3 Catholyte reservoir: $370 \times 270 \times 280$ mm (L \times W \times H) plastic box.
- 2.2.4 Plastic support: (see Fig. 2).
- 2.2.5 Cathode: 0.5 mm thick stainless steel plate (see Fig. 2).
- 2.2.6 Anode: 0.5 mm thick stainless steel plate (see Fig. 3).
- 2.2.7 Power supply: capable of supplying a voltage of 60 VDC and a current of 1 A.

2.3 Specimen Splitting Equipment

- 2.3.1 Any hydraulic splitting device with a capacity of 10 tons.

3. REAGENTS

3.1 Catholyte

3.1.1 10% NaCl in 0.1 M Na/K(OH) solution (NaOH/KOH = 1).

3.2 Anolyte

3.2.1 0.3 M Na/K(OH) solution (NaOH/KOH = 1).

3.3 Silver nitrate solution

3.3.1 0.1 N AgNO₃ solution, stored in a spray bottle.

4. SPECIMENS AND PRECONDITIONING

4.1 Specimens

4.1.1 Shape and size: cylinder samples with a diameter of 100 mm and thickness of 50 mm, drilled from the structure or cast in a special mould.

4.2 Conditioning

4.2.1 Assemble specimen in rubber sleeve and tight it with two clampers to prevent the leakage through the curved surface (see Fig. 3).

4.2.2 Place specimen in vacuum container and fill both container and the top side of specimen with anolyte up to a certain level.

4.2.3 Seal container and start vacuum pump. Pressure should decrease to less than 10 mmHg (1.33 kPa) within a few minutes.

4.2.4 Maintain vacuum for 6 hours and then allow air to reenter container.

4.2.5 Keep both sides of specimen suck anolyte for about 18 hours more.

5. TEST PROCEDURE

5.1 Potential applying

5.1.1 Empty the solution from the top side of specimen, transfer specimen to catholyte reservoir and set it on plastic support (see Fig. 5).

5.1.2 Put anode and fill 500 ml anolyte in the top side of specimen.

5.1.3 Connect cathode to negative pole and anode to positive pole of power supply. Turn on the power, set to 30 ± 0.2 V, and record initial current with a 3½-digit multimeter.

5.1.4 Chose a proper test duration according to initial current (see Table 1).

5.1.5 Record final current before terminate test.

5.2 Penetration depth measuring

5.2.1 Take away anode and empty the solution from the top side of specimen.

5.2.2 Take off specimen from rubber sleeve by means of wooden rod, and wipe away solution on the surfaces of specimen.

5.2.3 Axially split specimen into two pieces with a hydraulic splitting device.

- 5.2.4 Spray silver nitrate solution on the split surfaces, store specimen pieces in a dark place for one hour, and then expose them under a fluorescent light for a few hours.
- 5.2.6 Measure the average front of white zone in the central part of specimen and take a mean value of two pieces of specimen. This mean value with a precision of 0.5 mm stands for penetration depth of chlorides.

6. CALCULATION

6.1 Calculate the diffusion coefficient by using the following equation

$$D = \frac{RTL}{zFU} \cdot \frac{x_d - \alpha\sqrt{x_d}}{t}$$

where:

$$\alpha = 2\sqrt{\frac{RTL}{zFU}} \cdot \operatorname{erf}^{-1}\left(1 - \frac{2c_d}{c_0}\right)$$

D : diffusion coefficient, m^2/s ;

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

F : Faraday constant, $F = 9.648 \times 10^4 \text{ J}/(\text{V}\cdot\text{mol})$;

U : absolute value of potential difference, V ;

R : gas constant, $R = 8.314 \text{ J}/(\text{K}\cdot\text{mol})$;

T : solution temperature, K ;

L : thickness of the specimen, m ;

x_d : penetration depth, m ;

t : test duration, second, $t = t_{\text{CTH}} \times 3600$;

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

c_d : chloride concentration at which the colour changes, $c_d \approx 0.07 \text{ N}$;

c_0 : chloride concentration in the upstream cell, $c_0 \approx 2 \text{ N}$;

Let $\xi = \operatorname{erf}^{-1}\left(1 - \frac{2 \times 0.07}{c_0}\right)$, the values of ξ are given in *Table 2*.

Table 1—Test Duration for $\varnothing 100 \times 50$ mm specimen

Initial Current I_0 (mA)	Test Duration t_{CTH} (hour)
$I_0 < 5$	$t_{CTH} = 168$
$5 \leq I_0 < 10$	$t_{CTH} = 96$
$10 \leq I_0 < 30$	$t_{CTH} = 48$
$30 \leq I_0 < 60$	$t_{CTH} = 24$
$60 \leq I_0 < 120$	$t_{CTH} = 8$
$I_0 > 120$	$t_{CTH} = 4$

Table 2—The values of ξ

c_0	0.5 N	1.0 N	1.5 N	2.0 N	2.5 N	3.0 N	3.5 N	4.0 N	5.0 N
ξ	0.764	1.044	1.187	1.281	1.351	1.407	1.452	1.491	1.554

EXPLANATORY NOTES

- 1) For very dense concrete, a voltage of 40 V may be applied instead of 30 V (Ref. 5.1.3).
- 2) The experimental set-up can be used for a steady-state procedure. In this case, the curved surface of specimen may be coated with epoxy, a platinum anode may be used and a lid for the top end of rubber cleve should be added to prevent anolyte from evaporation.
- 3) The experimental set-up can also be used for AASHTO test, if its procedure is followed (Ref. AASHTO T 277-83, "Standard Method of test for determination of the chloride permeability of concrete").

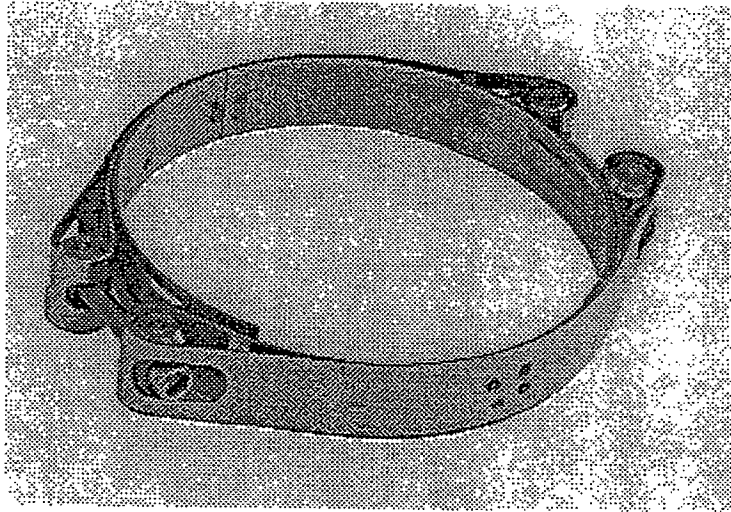


Fig. 1—Stainless steel clasper.

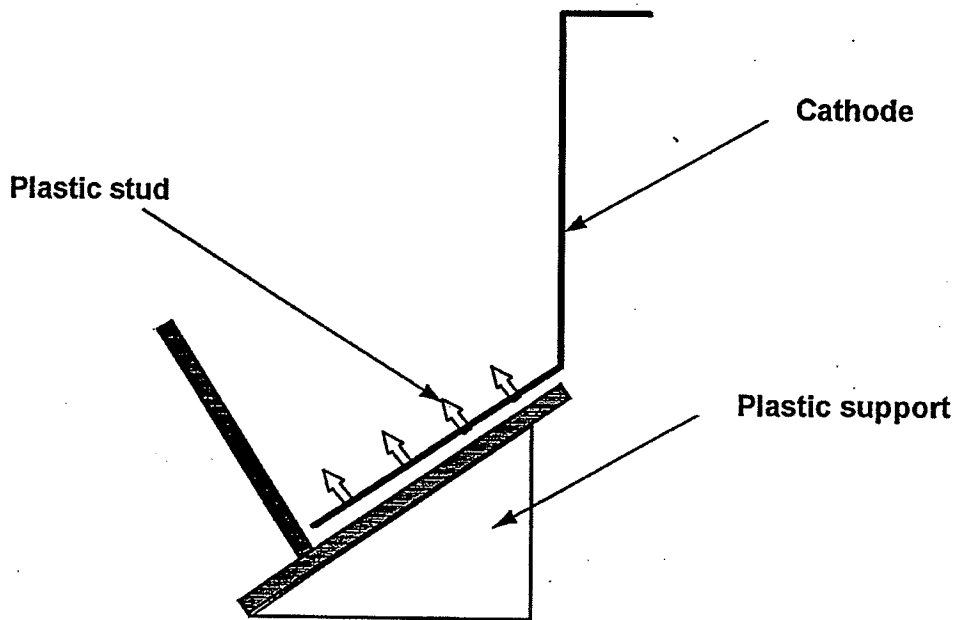


Fig. 2—Plastic support and cathode.

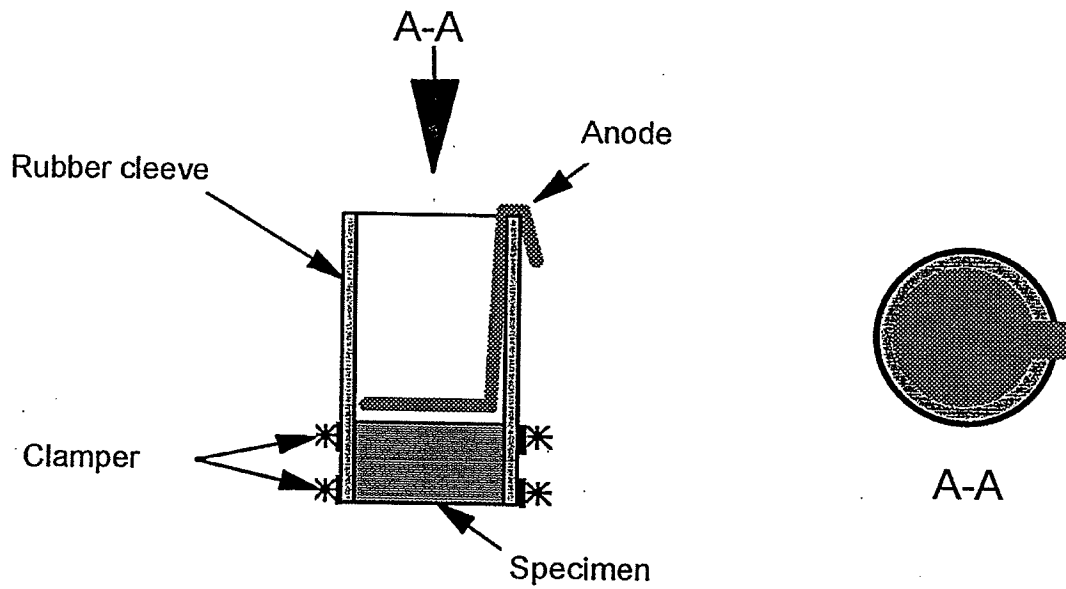


Fig. 3—Rubber sleeve assembled with specimen, clampers and anode.

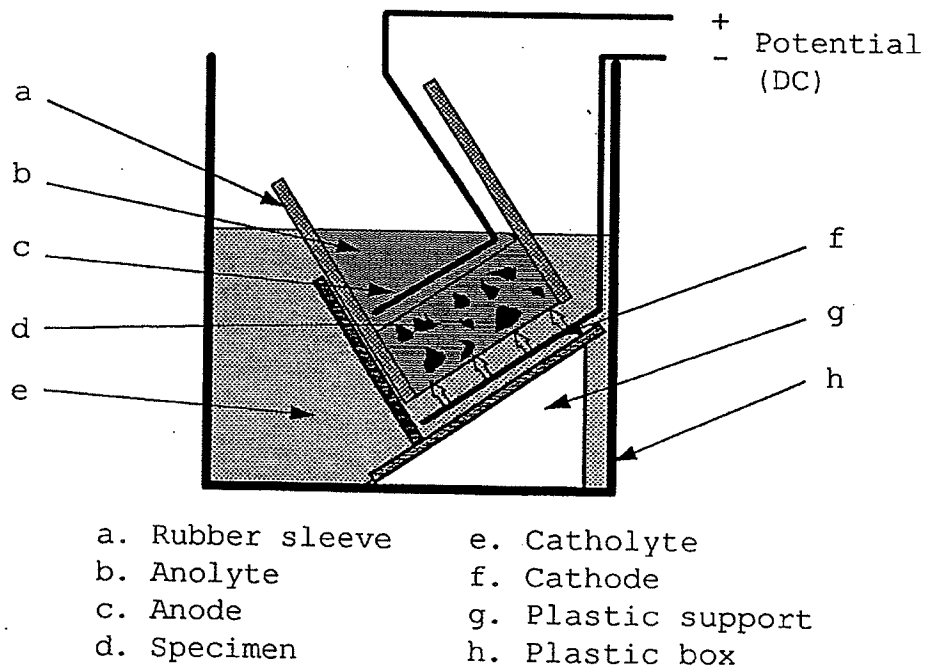


Fig. 5—Experimental arrangement for potential applying.