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# RILEM TC 235-CTC: Corrosion Initiating Chloride Threshold Concentrations in Concrete

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*Final test report on DTI's participation  
in round robin test*

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## Foreword

Danish Technological Institut (via Henrik E. Sørensen) is a member of *RILEM Technical Committee 235 CTC: Corrosion Initiating Chloride Threshold Concentrations in Concrete*. One of the main purposes of this RILEM committee, which was established in 2009, is to develop a commonly accepted test method for determination of chloride threshold values for corrosion initiation in reinforced concrete. Presently, such a method is still lacking.

Following thorough discussions among the members, the committee decided on the experimental details of an accelerated in-lab test method for determination of chloride threshold values for initiation of reinforcement corrosion in concrete. In 2011 a round robin test was initiated with the purpose of testing the proposed method.

This report presents the results from Danish Technological Institute's participation the round robin test.



Date: 10 March 2014

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## List of contents

1	Introduction.....	4
2	Preparation of specimens.....	5
2.1	Initial treatment of rebars .....	5
2.2	Cleaning of rebars.....	6
2.3	Pre-rusting of rebars.....	6
2.4	Mixing of concrete.....	7
2.4.1	Specimen series R.....	7
2.4.2	Specimen series L .....	8
2.5	Casting and curing of concrete specimens .....	9
2.6	Cutting of specimens .....	11
2.7	Coating of specimens.....	12
3	Pre-conditioning (drying) of specimens.....	13
4	Concrete surfaces to be exposed .....	15
5	Exposure of specimens .....	18
6	Monitoring of potentials .....	20
7	Chloride ingress profiles .....	22
8	Additional initiative to promote initiation of reinforcement corrosion .....	23
9	Additional tests on dummy specimens .....	24
10	Summary of results.....	27
	Appendix A: Chloride threshold value from parallel experiments at DTI.....	28
	Appendix B: Measured chloride and calcium profiles.....	30



## 1 Introduction

RILEM TC 235-CTC has suggested an accelerated in-lab test method for determination of chloride threshold values for initiation of reinforcement corrosion in concrete. The principle of the test method is to expose a series of ten identical concrete specimens, each containing a reinforcement bar, to a 3.3 wt% NaCl solution and monitor the potential against a common standard reference electrode using a datalogger. Prior to exposure the concrete specimens are partially dried in order to facilitate capillary suction of the exposure solution, thus accelerating the chloride ingress. Onset of reinforcement corrosion is identified by a significant drop of the measured potential, and the corrosion activity is defined as being stable when the potential has dropped and remained at a potential at least 150 mV lower than the passive level for more than 7 days. At this point the specimen is disconnected from the test setup and the chloride concentration at the depth of the reinforcement bar is determined, i.e. the chloride threshold value is measured.

During autumn 2011 a round robin test (RRT) was initiated with the purpose of testing the suggested test method and Danish Technological Institute (DTI) is participating in the round robin test along with 11 other laboratories, all of which have substantial experience within production and artificial exposure of concrete specimens, as well as corrosion monitoring and quantitative analysis of concrete. A document with details on how to conduct the test was distributed to all the participating laboratories before the RRT was initiated. These instructions were followed as closely as it was possible.

For all laboratories, the RRT includes two series of test specimens and each series comprises ten concrete specimens: One series with specimens prepared with cement and reinforcement steel distributed by the RILEM committee to all the participating laboratories and another series of specimens made with locally available cement and reinforcement steel. In this report the specimen series prepared with materials distributed by RILEM is referred to as “series R”, whereas the series made with locally available materials is labelled as “series L”.

As prescribed by the RILEM instructions for the RRT, the binder of specimen series R is 100 wt% Portland cement, while the binder chosen at DTI for series L is composed of 84 wt% Portland cement, 12 wt% pulverized fly ash and 4 wt% of silica fume. This type of ternary blend has previously been used in Denmark in connection with major constructions for the infrastructure, such as the *Great Belt Fixed Link* between the Danish islands of Zealand and Funen.

Date: 10 March 2014

## 2 Preparation of specimens

### 2.1 Initial treatment of rebars

The steel bars used for both specimen series were prepared from ribbed reinforcement steel (strength class  $f_y \geq 500$  MPa) with a nominal diameter of 12 mm. Initially, the steel was cut into pieces with a length of 15 cm. Subsequently, the ribbed surface was removed from the topmost 40 mm of all rebars by grinding on a lathe. This was done in order to obtain a smooth steel surface suitable for a firm and precise mounting of the rebars in the casting moulds (Fig. 1 and 2).

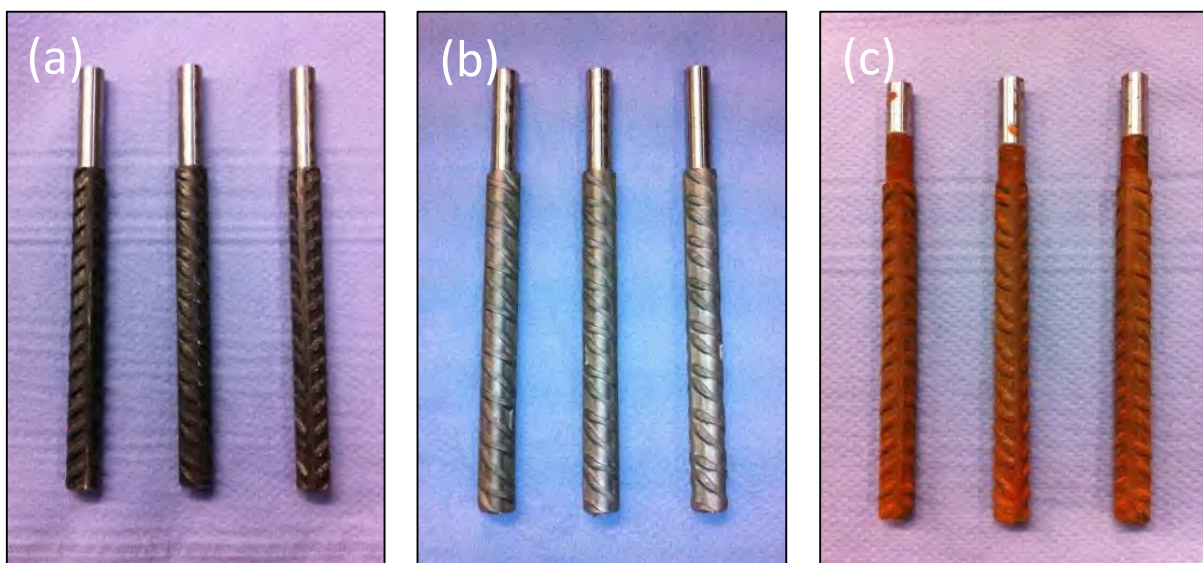


Fig. 1. Treatment of rebars from series R. (a) Example of "as-received" rebars after grinding of the topmost 40 mm (b) Rebars after chemical cleaning. (c) Rebars after pre-rusting procedure.

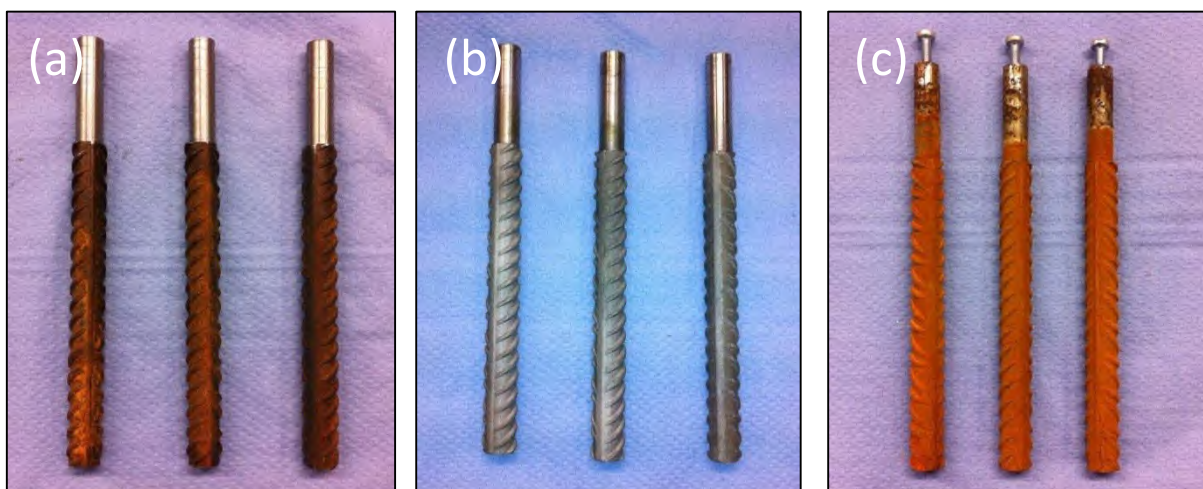


Fig. 2. Treatment of rebars from series L. (a) Example of rebars after grinding of the topmost 40 mm, but before the "as-received" rust was removed. (b) Rebars after chemical cleaning. (c) Rebars after pre-rusting procedure.

Date: 10 March 2014

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## 2.2 Cleaning of rebars

All rebars were chemically cleaned according to the procedure outlined in the RILEM directions for the RRT, i.e. the rebars were immersed in a chemical cleaner solution ( $\text{HCl}:\text{H}_2\text{O} = 1:1 + 3 \text{ g/l}$  urotropine) for 5 minutes and subsequently placed in an ultra-sonic bath for 3 minutes (Fig. 3). The results of the cleaning procedure is exemplified in Fig. 1b and 2b.



Fig. 3. Rebars were immersed in chemical cleaner solution ( $\text{HCl} 1:1 + 3 \text{ g/l}$  urotropine) for 5 minutes and subsequently placed in an ultrasonic bath for 3 minutes.

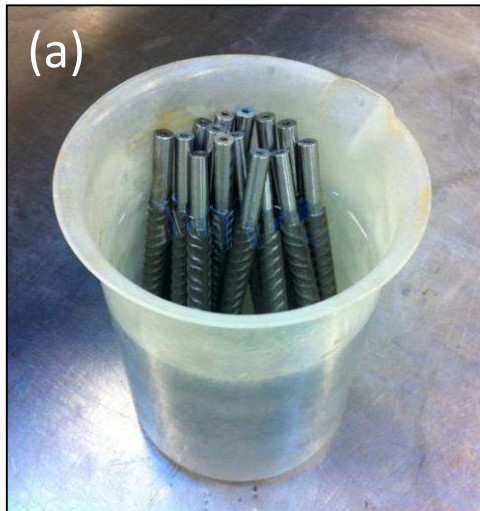
## 2.3 Pre-rusting of rebars

To simulate the surface conditions usually seen for reinforcement steel used in the construction industry, the cleaned steel bars were pre-rusted prior to casting. Firstly, the cleaned rebars were immersed in tap water for roughly 24 hours (Fig. 4a) and subsequently placed in moist chambers by means of sealed plastic buckets with water at the bottom (Fig. 4b and 4c). The rebars were hanging freely from a non-conducting wire. After 3 days the rebars were once again submerged in tap water overnight and subsequently placed in the moist chambers for 3 days.

Examples of the result of the prerusting procedure is displayed in Fig. 1c and 2c. The prerusted steel bars were kept in separate plastic bags and preserved in a desiccator (over silica gel) until casting.



Date: 10 March 2014



*Fig. 5. (a) Steel bars immersed in tap water. (b) Arrangement for pre-rusting of steel bars in a bucket. (c) Steel bars placed in sealed bucket with water at the bottom for obtaining a superficial corrosion.*

## 2.4 Mixing of concrete

### 2.4.1 Specimen series R

The concrete for specimen series R was mixed using the mixing station displayed in Fig. 6 and the slump could not be adjusted to 120-150 mm (as prescribed by the RILEM directions) without adding superplasticiser to the mixture. No air entraining agent was used and the content of air in the freshly mixed concrete was measured as 1.5% (based on the density). The achieved concrete composition is given in Table 1.

Date: 10 March 2014

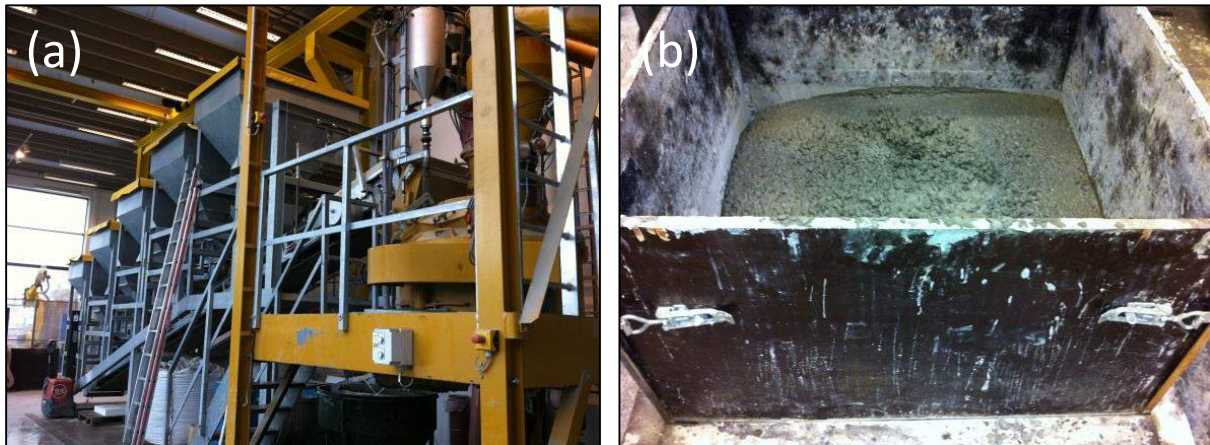


Fig. 6.(a) Full-automatic mixing station used for mixing of the concrete. (b) Fresly mixed concrete.

Table 1. Achieved concrete composition for the R series prepared with cement and steel distributed by RILEM.

<b>Specimen series ID</b>	Series R		
<b>Date of casting</b>	15 December 2011		
<b>Constituent material</b>	<b>Weight [kg/m<sup>3</sup>]</b>	<b>Volume [m<sup>3</sup>/m<sup>3</sup>]</b>	
Portland cement, CEM I 42.5 N (Aalborg low-alkali SR)	393.3	122.9	
Fine aggregates, 0/2 mm, sand (Rønne Banke)	667.8	253.9	
Medium aggregates, 4/8 mm, crushed rock (Rønne Granite)	372.3	137.4	
Coarse aggregates, 8/16 mm, crushed rock (Rønne Granite)	803.0	294.2	
Superplasticiser (Glenium SKY 532-SU)	1.5	1.4	
Water (total added)	175.2	175.2	
<i>Total</i>	2413.1	1000.0	
<b>Water/cement ratio</b>	<b>Air content [%] (measured)</b>	<b>Max. aggregate size [mm]</b>	<b>Binder content [kg/m<sup>3</sup>]</b>
0.45	1.5	16	393.3

#### 2.4.2 Specimen series L

The concrete for specimen series L was mixed using the concrete mixing station displayed in Fig. 6 and the slump was adjusted to 120 mm by adding superplasticiser to the mixture. The achieved concrete composition is given in Table 2.



Date: 10 March 2014

*Table 2. Achieved concrete composition for series L prepared with locally available cement and steel.*

<b>Specimen series ID</b>	Series L			
<b>Date of casting</b>	7 December 2011			
<b>Constituent material</b>	<b>Weight [kg/m<sup>3</sup>]</b>		<b>Volume [m<sup>3</sup>/m<sup>3</sup>]</b>	
Portland cement, CEM I 42.5 N (Aalborg low-alkali SR)	302.8		94.6	
Fly ash (Avedøre)	43.2		18.8	
Silica fume (Elkem slurry)	14.1		6.4	
Fine aggregates, 0/2 mm, sand (Rønne Banke)	684.0		260.1	
Medium aggreg., 4/8 mm, crushed rock (Rønne Granite)	380.5		140.4	
Coarse aggreg., 8/16 mm, crushed rock (Rønne Granite)	821.2		300.8	
Air entraining agent (Amex SB 22)	1.7		1.7	
Superplasticiser (Glenium SKY 532-SU)	3.0		2.7	
Water (total added)	103.6		103.6	
<i>Total</i>	2388.0		1000.0	
<b>Water/cement ratio (equiv.)*</b>	<b>Air content [%] (measured)</b>	<b>Max. aggregate size [mm]</b>	<b>Effective water content [kg/m<sup>3</sup>]</b>	<b>Binder content [kg/m<sup>3</sup>]</b>
0.40	3.7	16	144.2	360.1

\*The equivalent water/cement ratio was calculated using activity factor of 0.5 and 2.0 for fly ash and silica fume, respectively.

## 2.5 Casting and curing of concrete specimens

The concrete specimens were cast in moulds of ply-wood, and jigs, also made of plywood, were used as holders for placing the steel bar of each specimen at the correct position during casting (Fig. 7). The concrete was casted in two layers of approximately equal thickness, and each layer was compacted using a vibration table for approximately 5 seconds (Fig. 8).

The ten specimens of series R were labeled as R1, R2...R10 and the ten specimens of series L were labeled as L1, L2...L10. All specimens were demoulded after seven days and subsequently left for curing under plastic sealing for further 21 days in the laboratory (approx. 20°C) (Fig. 9).

Date: 10 March 2014

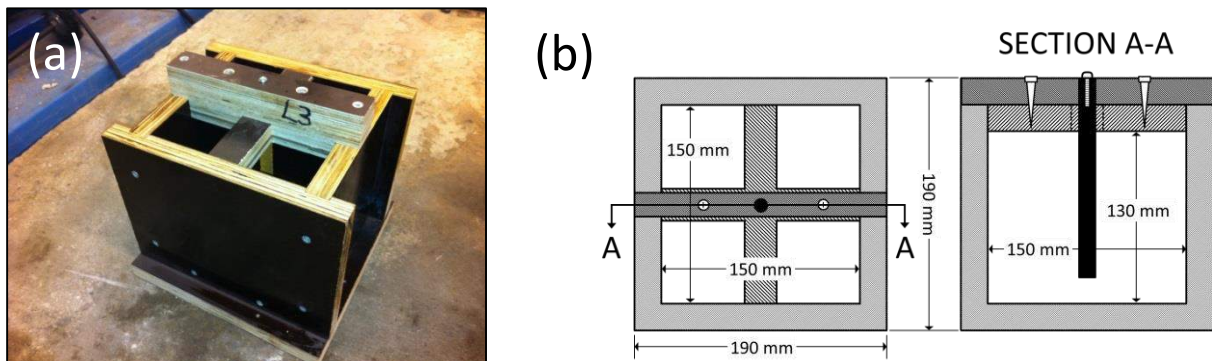


Fig. 7. (a) Mould of plywood used for casting the concrete specimens for the round robin test. A jig made of plywood is used as a holder for placing the steel bar at the correct position during casting and the bar is fixed to the jig with a screw. (b) Technical drawing of the mould.

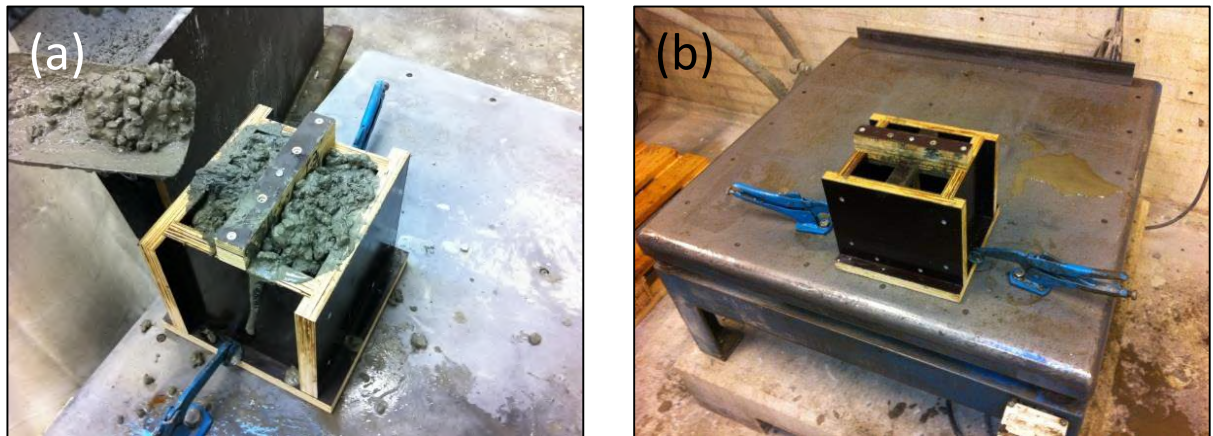


Fig. 8. (a) Casting of concrete specimen in plywood mould. (b) Vibration table used for compaction of the concrete specimens. The mould was held firmly against the table by two clamps.

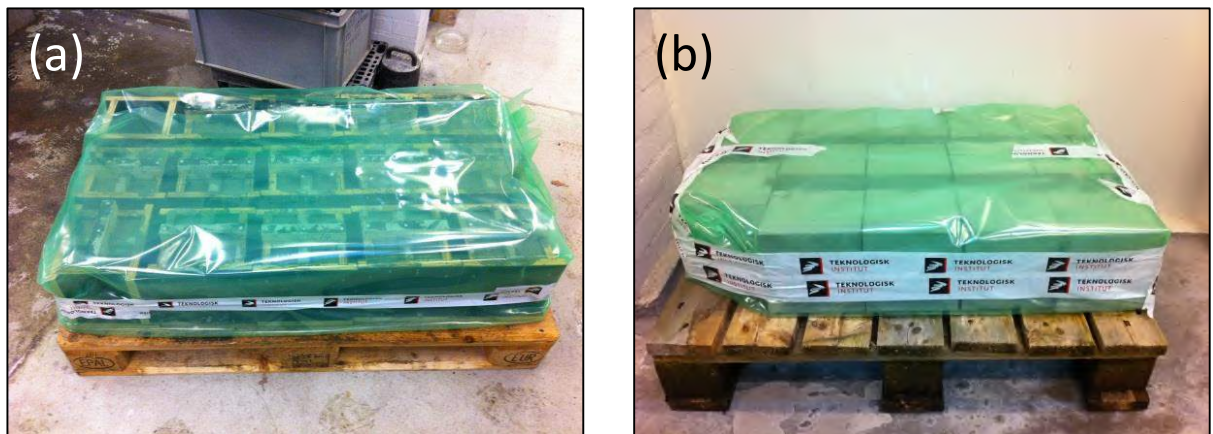


Fig. 9. (a) After casting the concrete specimens were covered with plastic to prevent evaporation. (b) After demoulding the concrete specimens were covered with plastic once again.

Date: 10 March 2014

## 2.6 Cutting of specimens

After four weeks of curing the concrete specimens were cut with a diamond saw (Fig. 10) to produce the shape displayed in Fig. 11. This was done to reduce the cover thickness to the steel bar, thus reducing the time required for the chloride ions to penetrate the concrete cover during exposure. The RILEM testing directions specifies that the cover thickness is adjusted according to the water/binder ratio of the specific concrete mix. Consequently, specimens of series R were prepared with a cover thickness of 10 mm (corresponding to a water/binder ratio of 0.45), and the specimens of series L were prepared with a cover thickness of 7 mm (corresponding to a water/binder ratio of 0.40).

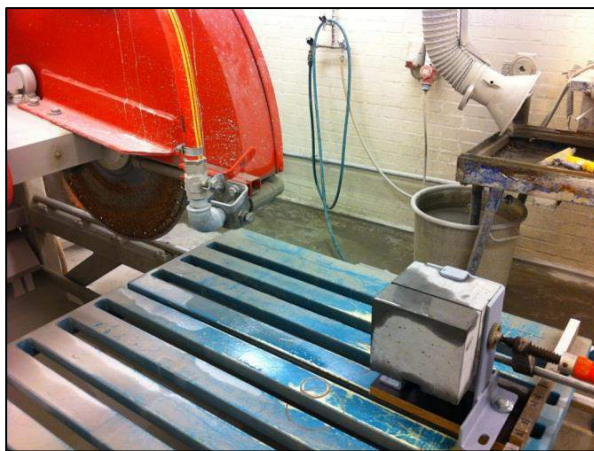


Fig. 10. The concrete specimens were cut using a diamond saw with running potable water as coolant.

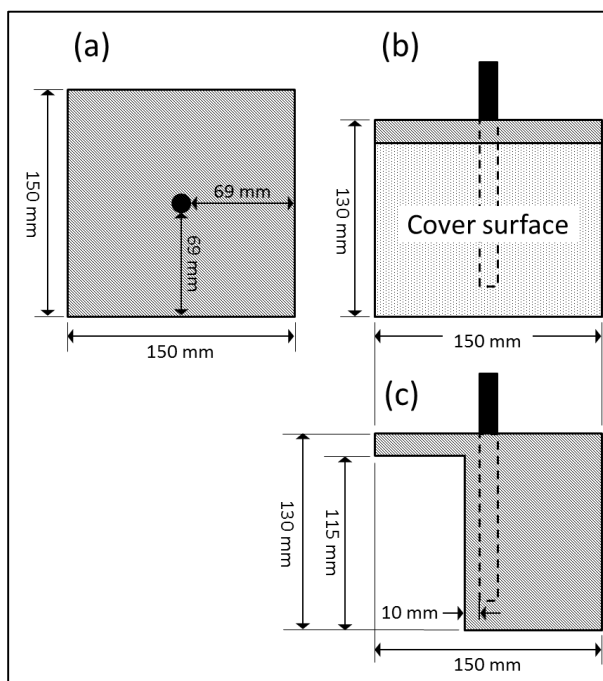


Fig. 11. Technical drawing of top view (a), front view (b), and a side view (c) of concrete specimen for the RRT. All surfaces were coated with epoxy, except the one labeled 'cover surface'. The dashed lines indicate the position of the reinforcement bar below the surface of the concrete.



Date: 10 March 2014

The excess concrete cover removed by the cutting procedure was used to produce a number of dummy specimens (Fig. 1), which was used to examine the effect of specimen drying at regular intervals.

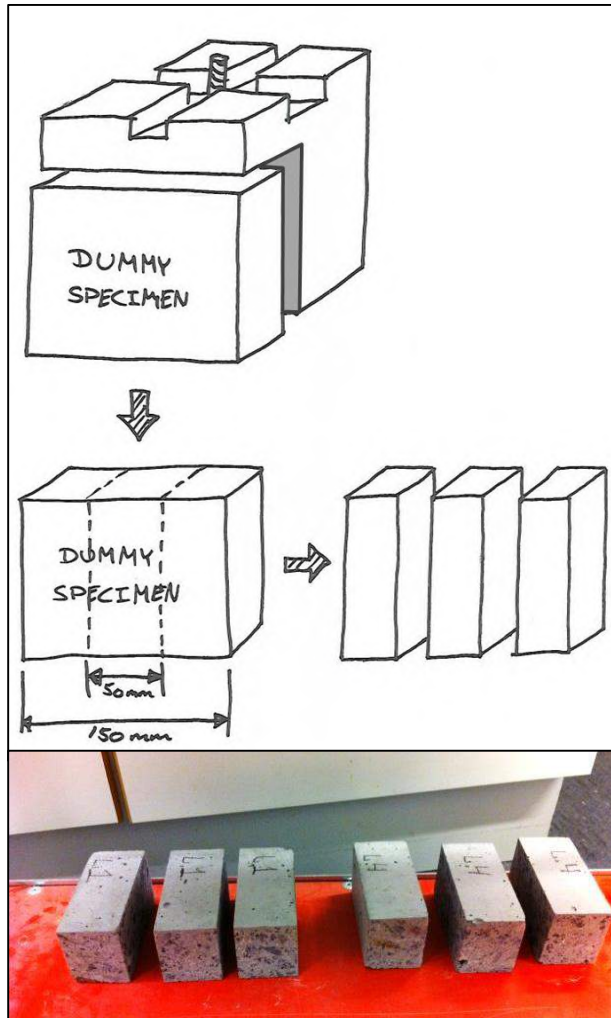


Fig. 12. Some of the cut-off pieces from the 'cubic' specimens were used to produce dummy specimens. Three specimens were produced from each of the selected cut-off pieces. Part (a) outlines the principle of the specimen cutting and examples of dummy specimens from specimen L1 and L4 are shown in part (b).

## 2.7 Coating of specimens

After cutting and surface drying (white-dry) all concrete surfaces of each specimen, except the surface to be exposed to the NaCl solution, were coated with epoxy resin (Fig. 13). This was done in order to insure a uni-directional ingress of chlorides during the exposure. Similarly, all surfaces of the dummy specimens were coated with epoxy resin, except the ones that were originally facing the 'cover surface'. All specimens were kept in the laboratory over night to allow the epoxy resin to harden. The coating procedure was repeated once more after hardening of the first layer of epoxy resin to ensure a completely tight sealing of all surfaces except the one to be exposed to the NaCl solution.

Date: 10 March 2014

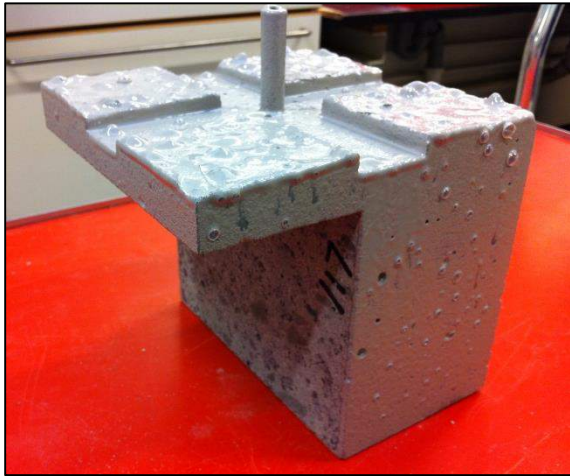


Fig. 13. Concrete specimen after coating with the first layer of epoxy resin.

### 3 Pre-conditioning (drying) of specimens

Before exposure to the chloride solution the concrete specimens were subjected to a drying regime (25% relative humidity, 25°C) to facilitate accelerated chloride ingress due to capillary suction of the exposure solution (Fig. 14). To avoid exaggerated drying, dummy specimens were taken out from the drying regime at regular intervals (every seventh day) and allowed to absorb the chloride exposure solution for 24 hours and the average chloride penetration depth was subsequently determined by spraying with a 0.1 N Ag-NO<sub>3</sub> solution on a freshly cut surface. The drying was considered as sufficient when the penetration depth was in the range of 3 mm from the rebar. In this way, the chloride penetration through the last few millimeters of concrete cover will take place by a diffusion-like process, rather than by capillary suction.



Fig. 14. The concrete specimens were placed in a climate chamber (25% RH and 20°C) in order to facilitate an accelerated ingress of chloride during exposure to NaCl solution. The dummy specimens were placed in the same chamber (not shown) and were oriented with the exposure surface (i.e. the surface without epoxy coating) facing upward. In contrast, the exposure surfaces of the specimens from series R and L were vertically oriented during the pre-conditioning as shown in the picture.



Date: 10 March 2014

Chloride penetration depths of 7 mm and 4.5 mm were measured after 21 days of drying on dummy specimens from specimen series R and L, respectively (Fig. 15). This fulfilled the abovementioned requirements for sufficient drying and the specimens were therefore removed from the drying regime at this point.

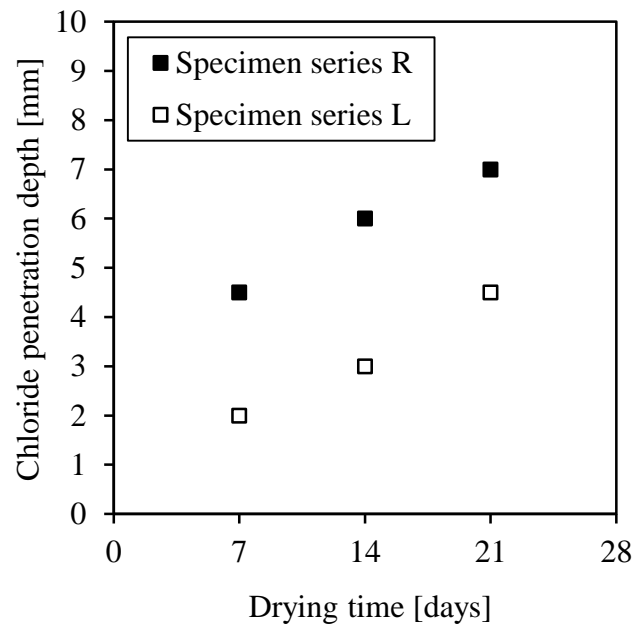


Fig. 15. Chloride penetration depths in concrete dummy specimens from specimen series L and R measured by spraying with 0.1 N  $\text{AgNO}_3$  solution on a freshly cut surface after absorption of chloride solution (3.3 wt% NaCl) for 24 hour. Sufficient penetrations depths (depending of the water/powder ratio) were measured for both specimen series after 21 of drying in a climate chamber (25% relative humidity, 20°C).

Date: 10 March 2014

#### 4 Concrete surfaces to be exposed

Before exposure to the NaCl solution a photograph was taken of the cover surface of each concrete specimen (Fig. 16 and 17).



Fig. 16 (continues on next page)



Date: 10 March 2014



Fig. 16. Photographs of the surfaces of specimens R1 to R10 to be exposed to the 3.3% NaCl solution. The epoxy resin has run down on the cover surfaces of some of the specimens. The specimens were exposed to the NaCl solution in the conditions that are presented in the pictures.



Fig. 17 (continues on next page).



Date: 10 March 2014

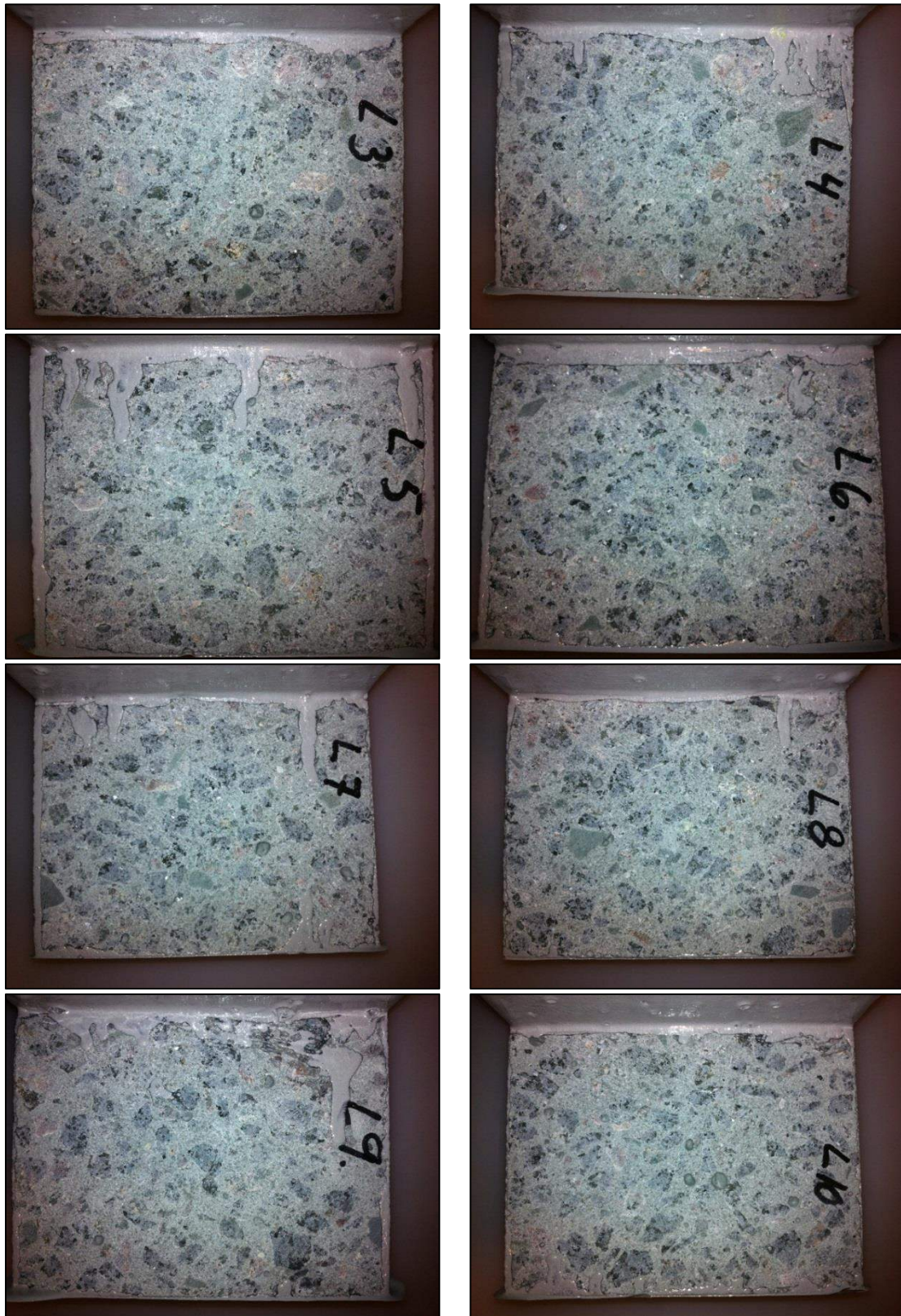


Fig. 17. Photographs of the surfaces of specimens L1 to L10 to be exposed to the 3.3% NaCl solution. The epoxy resin has run down on the surfaces of some of the specimens. The specimens were exposed to the NaCl solution in the conditions that are presented in the pictures.

Date: 10 March 2014

## 5 Exposure of specimens

At the predefined degree of drying, each series of concrete specimens was placed in a separate plastic tank (106 x 56 x 45 cm) and exposed to a 3.3 wt% NaCl solution, which was poured into the tank until the specimens were immersed to a depth of approximately 9.5 cm (Fig 18). The exposure was started on 3 February 2012 for series R and 27 January 2012 for series L.

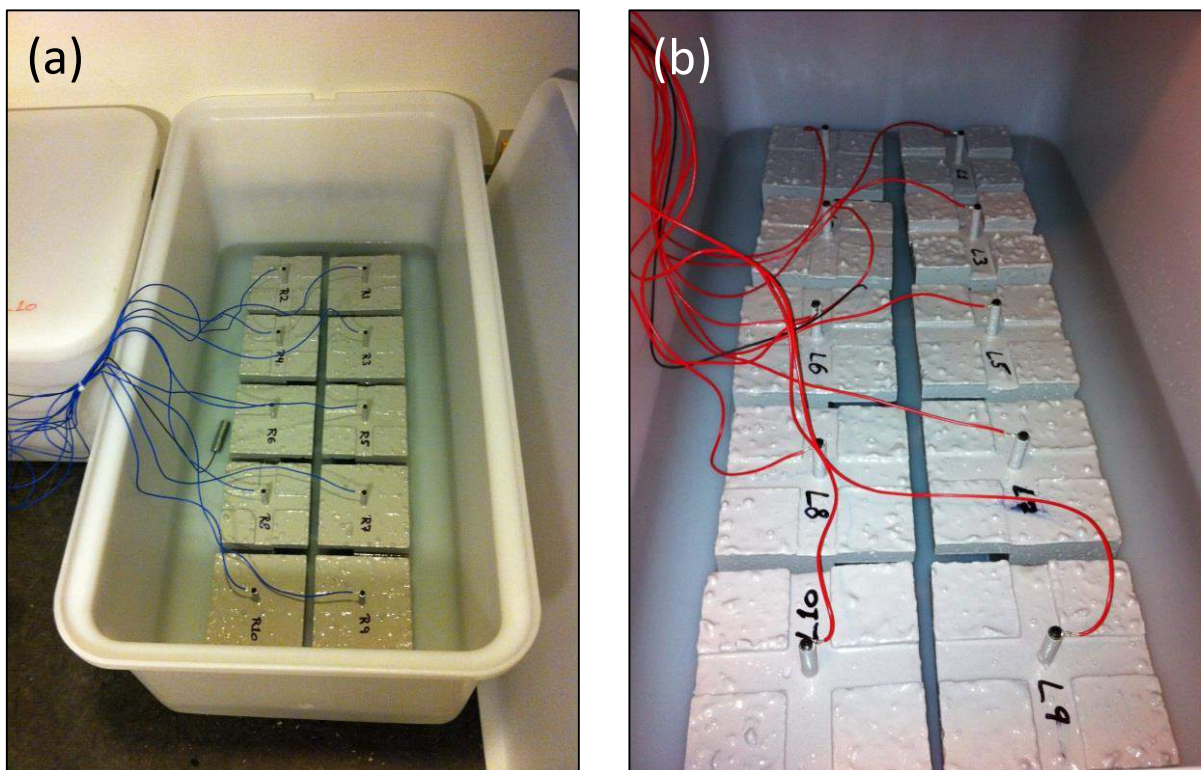


Fig. 18. Concrete specimens in exposure tanks with 3.3 wt% NaCl solution: (a) Series R and (b) Series L.

The steel bar of each concrete specimen was connected via a single-cored cable to a multiplex data-logger in order to monitor the electrochemical potential of the steel bar measured against a reference zinc electrode placed in the exposure solution (Fig. 19 and 20). The frequency of the datalogging was set to 2 hours.



Date: 10 March 2014

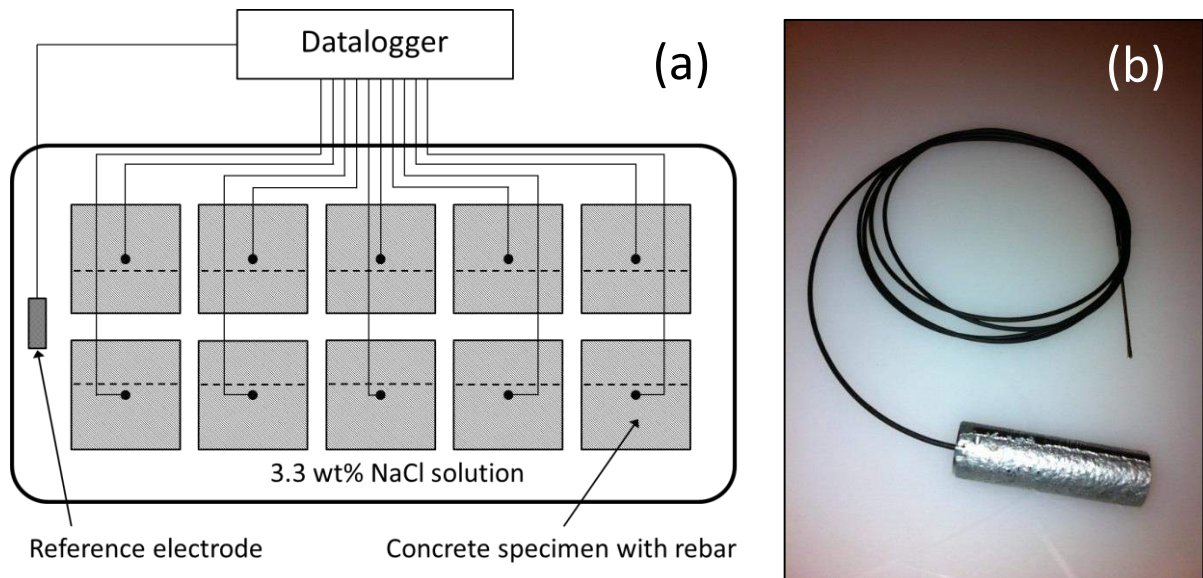


Fig. 19. (a) Arrangement of ten concrete specimens in a plastic tank containing a chloride exposure solution (3.3 wt% NaCl). The rebar of each specimen is connected to a datalogger and the electrochemical potential is measured against a reference electrode in an open circuit setup. The dashed lines indicate the position of the concrete surfaces (cover surfaces) exposed to the NaCl solution. (b) Reference zinc electrode.

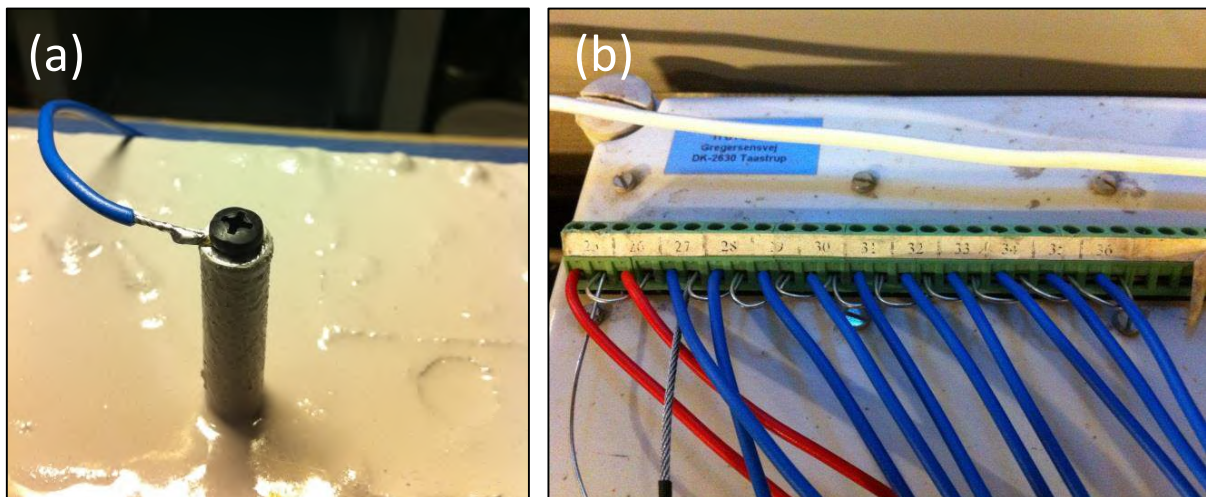


Fig. 20. (a) A cable is fixed to each rebar with a steel screw. (b) Connection of the working electrodes (rebars) to the data-logger by single-cored cables.

Date: 10 March 2014

## 6 Monitoring of potentials

The potentials measured in the open circuit setup (free potentials) are displayed in Fig. 21 and 22 for series R and series L, respectively. Despite a prolonged exposure period of more than 800 days for both series initiation of corrosion has not been observed. In a series of comparable lab experiments conducted at DTI we have encountered similar problems with very long exposure times without initiation of corrosion. A brief description of these experiments is given in Appendix A.

The potentials measured for specimen series R display values ranging between about -180 and -60 mV vs. CSE. The potentials generally display a slight and gradual increase during the exposure period, especially during the first 300 days. The only specimen of series R to show a significant decrease of the measured potential is specimen R7. However, this drop in potential (of ca. 120 mV) is gradual and extends over a period of approximately five weeks, i.e. the nature of the potential drop does not fulfill the requirements for corrosion initiation according to the RILEM RRT directions where it is specified that the potential must drop at least 150 mV within 24 hours in the case of corrosion initiation.

For series L, a wider overall span of potentials has been observed with values ranging from approximately -300 to -50 mV vs. CSE.

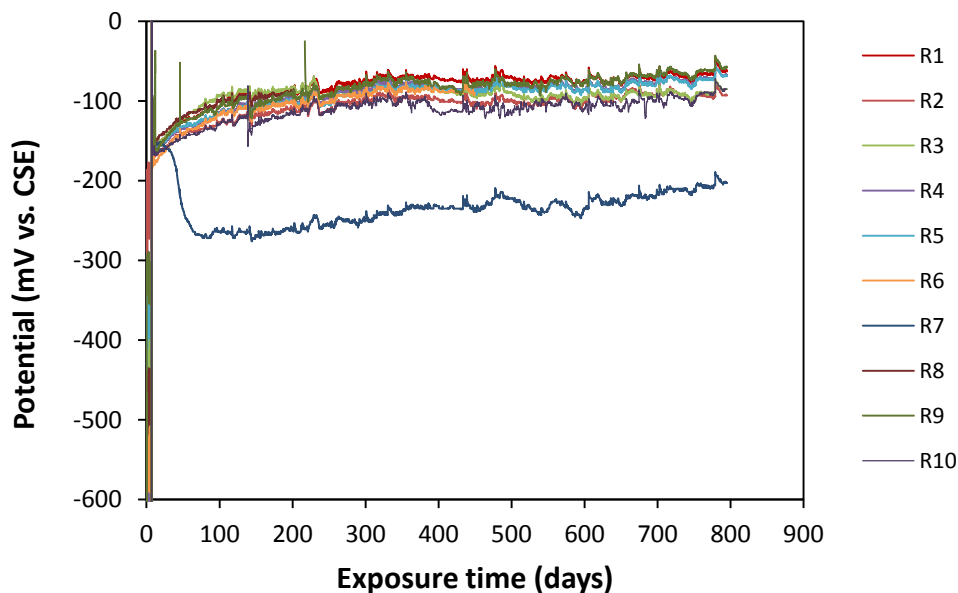


Fig. 21. Electrochemical potentials measured for specimen series R.

Date: 10 March 2014

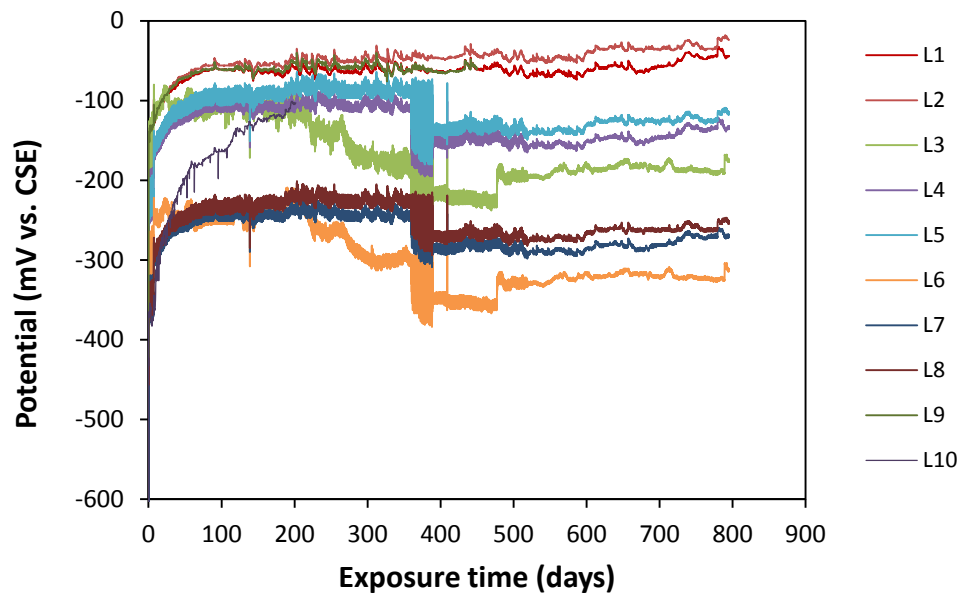


Fig. 22. Electrochemical potentials measured for specimen series L. The significant noise and sudden potential drop seen for some of the specimens after approx. 360 days of exposure is due to problems with the experimental setup, which was encountered at the transition from 2012 to 2013.

Date: 10 March 2014

## 7 Chloride ingress profiles

Generally, no indications of reinforcement corrosion have been observed during the exposure period of approximately 800 days. Since corrosion onset was expected to take place within the first few months of exposure, it was decided to disconnect two of the specimens (one from each series) from the test setup about 200 days of exposure in order to examine the status of the chloride ingress by measuring chloride profiles. This procedure was repeated for two additional specimens after about 400 days.

All chloride ingress profiles were determined by grinding off material in layers parallel to the exposed surface of the concrete specimens. The chloride content of each layer was subsequently measured according to the procedure given in *NT BUILD 208, "Concrete, hardened: Chloride content", 2nd ed., 1984*. The obtained profiles are presented in Fig. 23 and Appendix B.

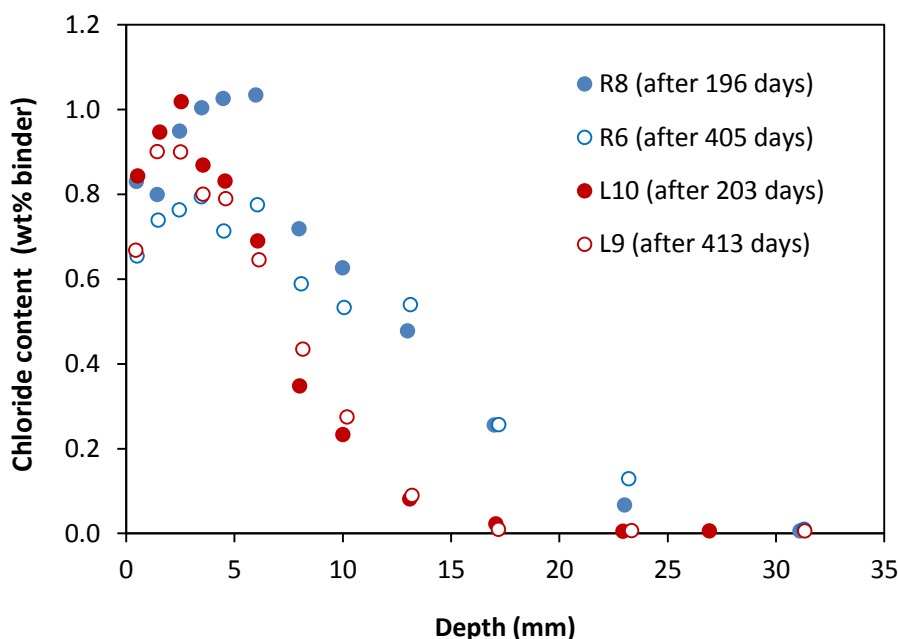


Fig. 23. Chloride ingress profiles measured on concrete specimens after roughly 200 days and 400 days of exposure to 3.3 wt% NaCl solution. The chloride profiles have been corrected for variations in calcium content (see Appendix B for explanation).

Date: 10 March 2014

## 8 Additional initiative to promote initiation of reinforcement corrosion

After about five months of exposure to the NaCl solution without initiation of corrosion it was decided to drill a hole ( $\varnothing$  30 mm) close to the rebars in specimens R9 and R10 from series R (Fig. 24). This was done with the intention of increasing the access of oxygen to the rebars, thereby perhaps promoting the initiation of reinforcement corrosion. The vertically oriented holes were placed about 1 cm from the rebars on the opposite site of the exposure surfaces. The holes were drilled to a depth of approximately 3 cm from the bottom of the specimens. However, this procedure did not result earlier corrosion start for specimens R9 and R10 within the in the observed exposure periode (~800 days). Only a minor decrease of the potentials ( $\sim 20$  mV) was observed for R9 and R10 after drilling of the holes.

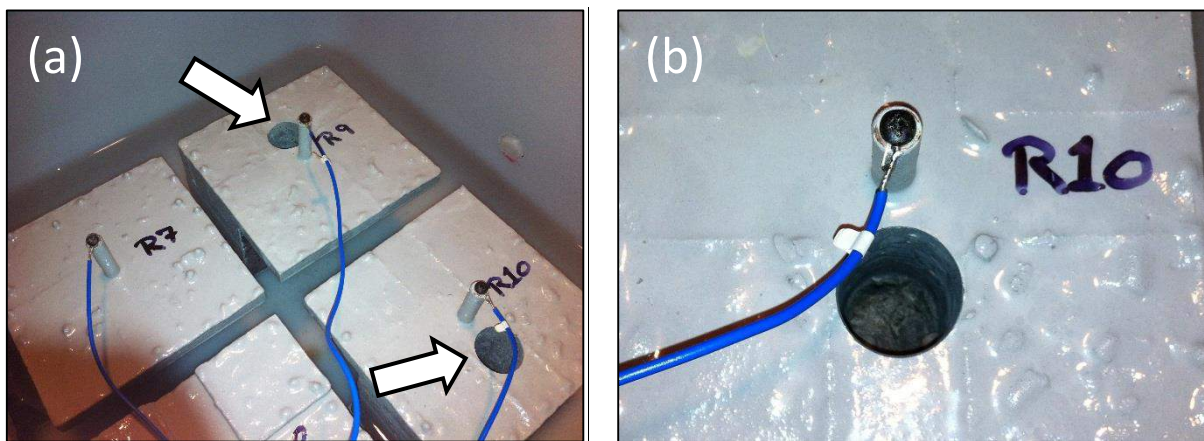


Fig. 24. (a) To enhance the access of oxygen to rebars  $\varnothing$  30 mm holes were drilled in specimens R9 and R10. The holes are indicated with arrows. (b) Close-up of hole drilled in specimen R10.



Date: 10 March 2014

## 9 Additional tests on dummy specimens

Two leftover dummy specimens (L4-1 and L4-2) from specimen series L were utilized to conduct some additional exposure experiments. The purpose of these experiments was to compare the chloride ingress in specimens in two different experimental setups: (1) A setup in which the exposure solution is replaced at regular intervals and (2) a setup where the same batch of exposure solution is used for the entire exposure period.

The following procedure was followed for these additional experiments:

- The outermost 7 mm of each dummy specimen was cut off at the surfaces that have been exposed to the drying regime (see chapter 3) (Fig. 25).
- The freshly cut surfaces were coated twice with epoxy.
- When the coatings were sufficiently dry each dummy specimen was cut in half (Fig. 26).
- One half of each dummy specimen (L4-1A and L4-2A) were placed in the same exposure tank as specimen series L (Fig. 27a).
- The other two halves (L4-1B and L4-2B) were placed in a 3.3 wt% NaCl solution (3.85 L) in a bucket covered by plastic (Fig. 27b).
- The exposure solution was replaced at intervals of two weeks. Chloride ingress profiles were measured on all four specimens after 302 days of chloride exposure (Fig. 28 and 29).

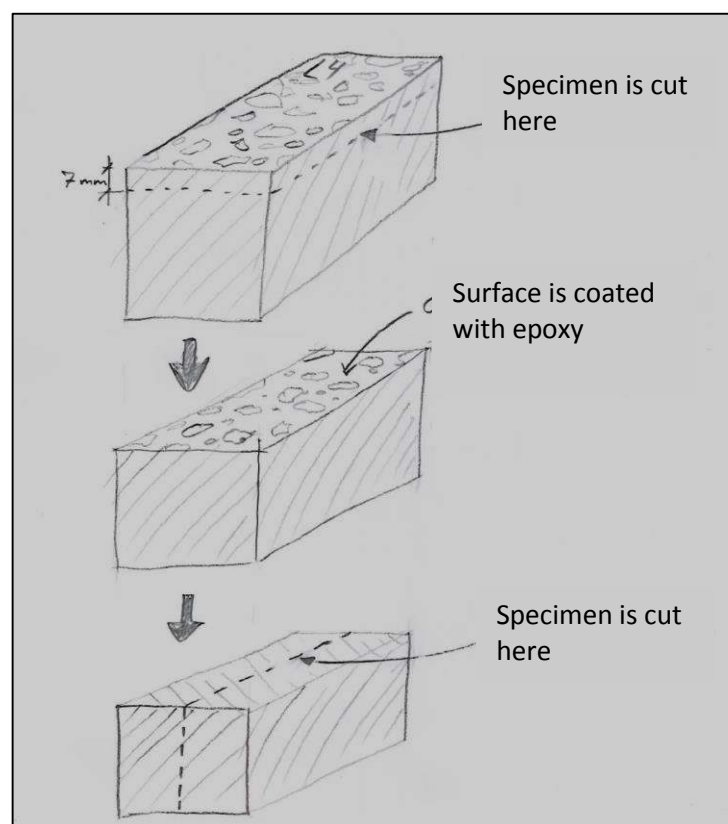


Fig. 25. Procedure for preparation of dummy specimens for additional experiments with chloride exposure.

Date: 10 March 2014

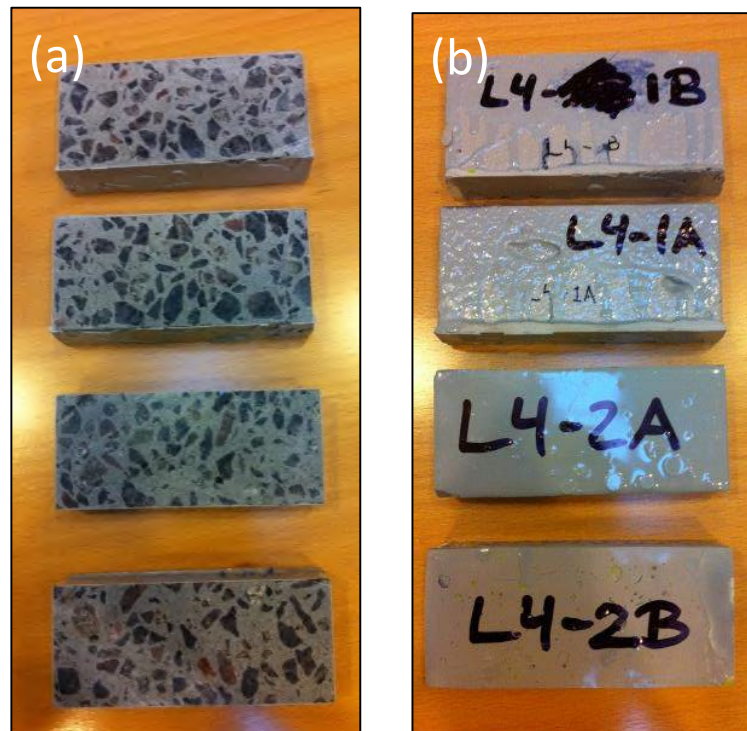


Fig. 26. Dummy specimens for additional chloride exposure experiments: (a) Surfaces to be exposed to NaCl solution and (b) epoxy coated surfaces.

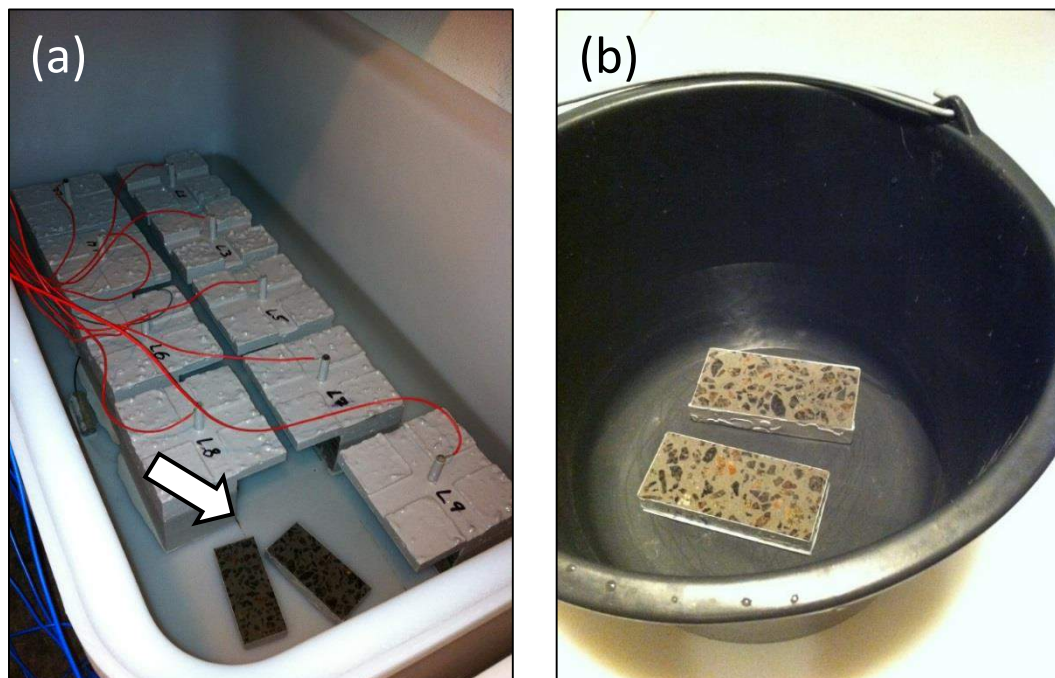


Fig. 27. (a) Dummy specimens L4-1A and L4-2A in the same exposure tank as series L. (b) Dummy specimens L4-1B and L4-2B exposed to 3.3 wt% NaCl solution in plastic bucket.

Date: 10 March 2014

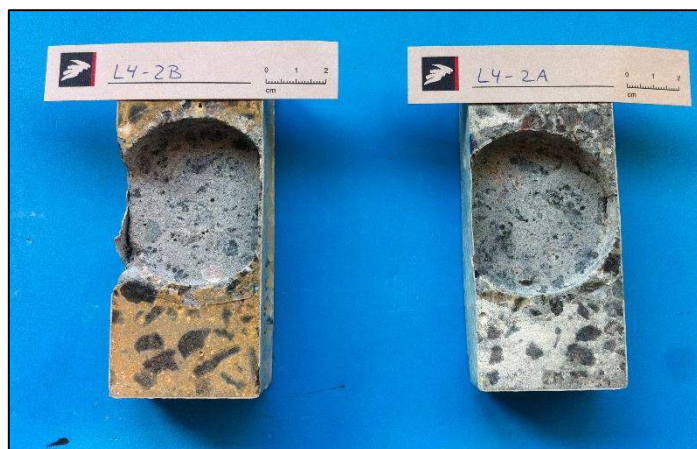


Fig. 28. Dummy specimens L4-2B and L4-2A after grinding of chloride profiles. Notice the difference in the colour of the exposed surfaces of the two specimens.

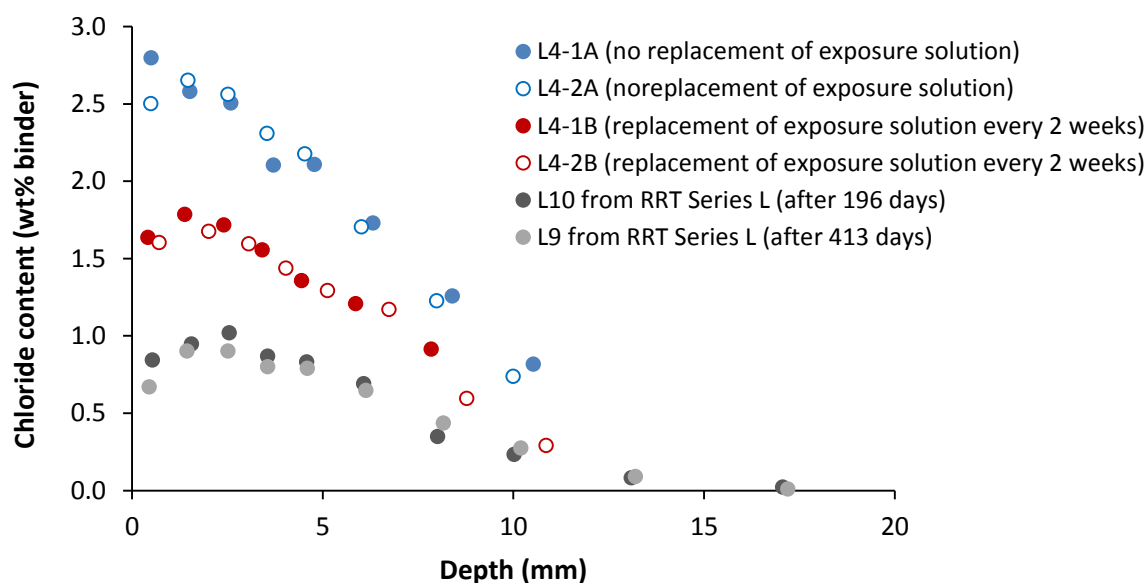


Fig. 29. Chloride ingress profiles measured on concrete specimens after 302 days of exposure to 3.3 wt% NaCl solution. For specimens L4-1A and L4-2A the exposure solution was the same for the entire exposure period, whereas the NaCl solution was replaced every two weeks for specimens L4-1B and L4-2B. Chloride profiles measured on specimens L9 and L10 from the RRT series L are also displayed for comparison. All the chloride profiles have been corrected for variations in calcium content (see Appendix B for explanation).

The measured chloride profiles clearly show a reduced ingress for specimens L4-1B and L4-2B compared to L4-1A and L4-2A, i.e. the replacement of exposure solution with regular interval must have had some retarding effect on the ingress of chlorides. Also, the concrete surfaces of the specimens from the setup with regular replacement of the solution had a noticeable yellowish colouring (Fig. 28) thus

indicating some sort of surface phenomenon, which may have a retarding effect on the ingress of chlorides.

In Fig. 29 the chloride profiles measured on specimens L10 and L9 from series L of the Round Robin test are also displayed for comparison, i.e. the profiles for L10 and L9 are the same as the ones shown in Fig. 23. Surprisingly, much higher chloride contents are observed for the specimens prepared from the dummies than for L10 and L9, despite the dummies and L10 and L9 being produced from the same batch of concrete. This is especially pronounced for L4-1A and L4-2A, which were placed in the same exposure tank as specimen series L. It is noted that the specimens prepared from the dummies had not been subjected to the pre-conditioning conditions (i.e. partial drying in climate chamber) like L9 and L10 were. We therefore speculate that the significant difference in chloride content observed between the dummies and specimens L9 and L10 might be due to some phenomenon related to the difference in sample preparation/pre-conditioning.

## 10 Summary of results

- Clear indications of corrosion initiation has not been observed for any of the specimens in series R or series L despite a prolonged exposure period of more than 800 days.
- The potentials measured for specimen series R display values ranging between about -180 and -60 mV vs. CSE. The potentials generally display a slight and gradual increase during the exposure period, especially during the first 300 days.
- For series L, a wider overall span of potentials has been observed with values ranging from approximately -300 to -50 mV vs. CSE.
- The drilling of holes behind the rebars (to increase the availability of oxygen) in two of the specimens in series R did not result in a significant change in the measured potentials, thus indicating that oxygen availability has not been a limiting factor for corrosion initiation.
- Chloride ingress profiles were measured after approximately 200 and 400 days of exposure for both specimen series. Generally, higher chloride concentrations were observed of the specimens from series R as compared to the specimens from series L. This is probably due to the difference in water/binder ratio and binder composition between the two series.
- Since corrosion onset has not been observed for any of the exposed specimens for the duration of the Round Robin test it may be concluded that the chloride threshold value for the specimens in series R and L must be higher than 0.53 and 0.44 wt% of binder, respectively, since those values are the chloride contents measured at the rebar depth after roughly 400 days of exposure, i.e. the chloride contents at the rebar depth from the “newest” determined chloride profiles.
- Additional chloride exposure experiments conducted on some leftover dummy specimens revealed that replacement of the chloride solution at regular intervals have a somewhat retarding effect on the ingress of chlorides when compared to an experimental setup in which the exposure solution is not replaced during the entire exposure period.



## Appendix A: Chloride threshold value from parallel experiments at DTI

Additional experiments have been conducted at Danish Technological Institute using a lab-method comparable to the one used for the Round Robin test carried out in connection with RILEM TC 235-CTC. The basic idea of these preliminary experiments is to expose a number of concrete specimens with cast-in rebars to a 6 wt% NaCl solution and subsequently detect the onset of reinforcement corrosion in one of two ways: (1) by observation of a significant drop in the electrochemical potential of the rebar measured against a reference electrode (open circuit test-setup) or (2) by observation of a significant increase in the current required to maintain a specific potential of the rebar measured against a reference electrode (fixed potential test-setup). When corrosion onset is observed the chloride concentration is measured at the depth of the rebars, thus giving the chloride threshold value.

The experiments were initiated in 2011 and are still running at the time of writing (March 2014). Despite a concrete cover thickness of only 5 mm for most of the specimens and a prolonged exposure time of more than 900 days, corrosion onset has only been detected for one out of 16 rebars in the open circuit setup and for two out of 16 rebars in the potentiostatic setup.

### *Preparation of concrete specimens*

A total of eight concrete specimens (P1 to P8), each containing four rebars, were cast from the same batch of concrete (P) with a water/powder ratio of 0.40 and a binder composed of 75 wt% of CEM I 52,5 N Portland cement (RAPID® cement from Aalborg Portland, Denmark) and 25 wt% of pulverized fly ash. After casting each specimen was cut with a diamond saw to produce the shape displayed in Figure 1. The cutting was performed in order to reduce the cover thickness to 5 mm (six specimens) or 15 mm (two specimens) and the epoxy coating was done to ensure a unidirectional chloride ingress during exposure. After 28 days of curing the eight concrete specimens were exposed to 6.0 wt% NaCl solutions by placing two specimens in each of four identical plastic tanks (23 x 27 x 15 cm). A more comprehensive description of the experiments can be found in reference [1].

### *Mesured chloride threshold values*

In the open circuit setup corrosion onset has been observed for only one rebar (after about 940 days of exposure) at the time of writing (March 2014). This was identified by a sudden and significant drop in the measured potential (Fig. 2). After corrosion onset the chloride concentration was measured at the depth of the rebar, thus giving a chloride threshold value of 0.73 wt% of binder.

In the fixed potential setup corrosion onset was observed for two rebar in the same specimen after 835 and 853 days, respectively. About two week after corrosion onset the rebars were disconnected from the setup and the chloride concentrations were determined at the rebar depths, which revealed unexpectedly high values (2.30 wt% of binder). Therefore, the chloride content was also measured at the depth of a third rebar in the same concrete specimen where corrosion onset had not been observed. At this rebar a much lower concentration (0.41 wt% of binder) as measured. We therefore anticipate that once the corrosion process has been initiated, the high electrical current to maintain the fixed potential in the experimental setup is capable of accelerating the ingress of chlorides into the concrete cover, much like in a chloride migration experiment. Consequently, the chloride concentration at the rebar will not represent a “true” chloride threshold value”, if the rebar is not disconnected from the experimental setup straight after registration of corrosion onset.



Date: 10 March 2014

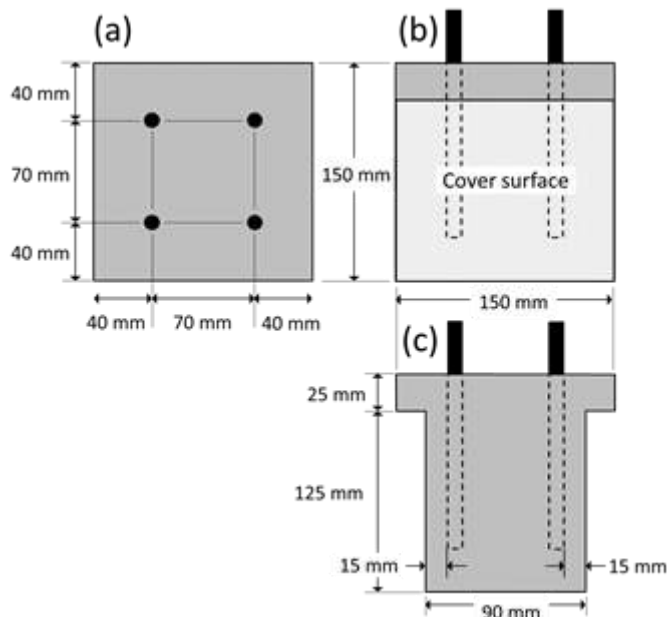


Fig. 1. Top view (a), front view (b) and side view (c) of concrete specimens from specimen series P. Each specimen of the series contains four rebars and all surfaces are sealed with an epoxy coating, except the 'cover surfaces'. The dashed lines indicate the position of the rebars below the concrete surface.

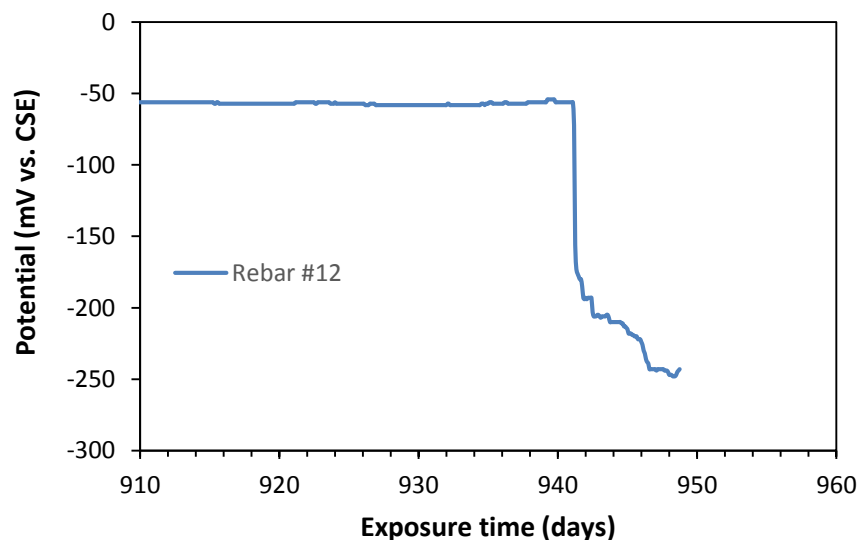


Fig. 2. Electrochemical potential measured for rebar #12 in open circuit setup. Corrosion onset is identified by the significant drop in potential at an exposure time of approximately 940 days.

## References

- [1] Henrik E. Sørensen, Søren L. Poulsen, Erik P. Nielsen. Testing of the chloride threshold values for reinforced concrete structures. In: Mark G. Alexander, Hans-Dieter Beushausen, Frank Dehn, Pilate Moyo (eds.). Concrete Repair, Rehabilitation and Retrofitting III. Proceedings of 3rd International Conference on Concrete Repair, Rehabilitation and Retrofitting, ICCRRR-3, 3-5 September 2012, Cape Town, South Africa, p. 187-188.



Date: 10 March 2014

## Appendix B: Measured chloride and calcium profiles

Chloride and calcium profiles measured in connection with the Round Robin test are given in the tables below. Chloride and calciumoxide contents were determined according to the procedure given in *NT BUILD 208, "Concrete, hardened: Chloride content", 2nd ed., 1984* and *APM 214, "Concrete, hardened: Calcium content" 1995*, respectively. The chloride contents at each profile depth in the calcium-corrected profiles were calculated using the following formula:

Chloride content (Ca-corrected) = (Chloride content / CaO content) \* theoretical average CaO content

where the "theoretical average CaO" is the total CaO content in the specific concrete mix calculated using the chemical composition of the constituent materials in the mix as well as the mixing proportions.

The "theoretical average CaO" obtained for series R and L are given here:

Series R		Series L	
Calc. CaO [wt% concr.]	Calc. CaO [wt% binder]	Calc. CaO [wt% concr.]	Calc. CaO [wt% binder]
10.69	65.61	8.36	55.42

*Data for specimen R8 from series R. Chloride and calcium profiles were measured after 196 days of chloride exposure.*

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.47	0.173	0.135	1.064	0.831	13.697	84.038
1.43	0.157	0.130	0.963	0.800	12.871	78.969
2.47	0.194	0.155	1.188	0.949	13.385	82.126
3.49	0.191	0.164	1.170	1.005	12.450	76.389
4.48	0.182	0.167	1.118	1.026	11.645	71.447
5.99	0.162	0.169	0.996	1.035	10.291	63.139
7.99	0.113	0.117	0.691	0.719	10.266	62.988
10.00	0.109	0.102	0.669	0.627	11.403	69.963
12.99	0.084	0.078	0.516	0.478	11.532	70.755
17.00	0.042	0.042	0.260	0.257	10.831	66.455
23.02	0.011	0.011	0.065	0.067	10.249	62.882
31.10	0.001	0.001	0.006	0.006	10.643	65.303

Date: 10 March 2014

*Data for specimen R6 from series R. Chloride and calcium profiles were measured after 405 days of chloride exposure.*

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.51	0.127	0.107	0.781	0.655	12.751	78.233
1.48	0.136	0.121	0.834	0.740	12.054	73.956
2.46	0.138	0.124	0.847	0.764	11.857	72.747
3.48	0.148	0.129	0.906	0.794	12.193	74.810
4.51	0.154	0.116	0.945	0.714	14.156	86.854
6.06	0.142	0.126	0.870	0.776	11.998	73.611
8.08	0.103	0.096	0.629	0.589	11.420	70.068
10.06	0.101	0.087	0.621	0.533	12.457	76.432
13.13	0.073	0.088	0.447	0.540	8.841	54.243
17.20	0.044	0.042	0.270	0.257	11.233	68.920
23.21	0.019	0.021	0.115	0.129	9.484	58.192
31.30	0.001	0.002	0.009	0.010	9.468	58.090

*Data for specimen L10 from series L. Chloride and calcium profiles were measured after 203 days of chloride exposure.*

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.53	0.166	0.127	1.104	0.844	10.928	72.468
1.56	0.170	0.143	1.126	0.947	9.935	65.886
2.55	0.184	0.154	1.223	1.019	10.030	66.514
3.55	0.148	0.131	0.985	0.869	9.465	62.765
4.57	0.140	0.126	0.929	0.832	9.333	61.890
6.07	0.115	0.104	0.761	0.690	9.217	61.120
8.01	0.057	0.053	0.376	0.348	9.012	59.763
10.01	0.034	0.035	0.223	0.234	7.981	52.928
13.09	0.012	0.012	0.079	0.082	8.069	53.508
17.06	0.004	0.003	0.025	0.023	9.210	61.075
22.94	0.001	0.001	0.007	0.006	9.628	63.849
26.93	0.001	0.001	0.007	0.006	9.253	61.364



Date: 10 March 2014

*Data for specimen L9 from series L. Chloride and calcium profiles were measured after 413 days of chloride exposure.*

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.44	0.125	0.101	0.829	0.669	10.356	68.675
1.44	0.155	0.136	1.026	0.901	9.520	63.134
2.51	0.166	0.136	1.102	0.901	10.226	67.813
3.55	0.152	0.121	1.010	0.801	10.547	69.945
4.59	0.146	0.119	0.968	0.791	10.230	67.838
6.13	0.127	0.097	0.840	0.646	10.862	72.032
8.16	0.092	0.066	0.612	0.435	11.746	77.897
10.19	0.057	0.041	0.379	0.275	11.523	76.417
13.20	0.019	0.014	0.128	0.090	11.918	79.037
17.19	0.002	0.002	0.014	0.010	11.749	77.915
23.33	0.002	0.001	0.011	0.007	12.251	81.246
31.33	0.001	0.001	0.008	0.006	11.425	75.767

Date: 10 March 2014

Data for specimen L4-1A from additional series of experiments concerned with the effect of exposure solution replacement. Chloride and calcium profiles were measured after 302 days of chloride exposure. The exposure solution was the same for the entire exposure period.

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.50	0.306	0.422	2.032	2.796	6.073	40.273
1.51	0.291	0.389	1.930	2.580	6.254	41.472
2.58	0.269	0.378	1.787	2.505	5.962	39.535
3.70	0.240	0.317	1.593	2.103	6.332	41.989
4.78	0.239	0.318	1.582	2.108	6.272	41.590
6.31	0.195	0.261	1.291	1.730	6.238	41.364
8.39	0.141	0.190	0.933	1.258	6.200	41.113
10.52	0.083	0.123	0.553	0.817	5.654	37.491

Data for specimen L4-2A from additional series of experiments concerned with the effect of exposure solution replacement. Chloride and calcium profiles were measured after 302 days of chloride exposure. The exposure solution was the same for the entire exposure period.

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.49	0.348	0.377	2.310	2.502	7.718	51.180
1.46	0.333	0.400	2.208	2.652	6.956	46.132
2.51	0.318	0.386	2.107	2.560	6.876	45.597
3.54	0.310	0.348	2.053	2.308	7.433	49.292
4.52	0.288	0.328	1.909	2.176	7.333	48.626
6.01	0.233	0.257	1.545	1.704	7.575	50.236
7.98	0.161	0.185	1.068	1.227	7.272	48.221
9.99	0.091	0.111	0.605	0.739	6.845	45.394





Date: 10 March 2014

Data for specimen L4-1B from additional series of experiments concerned with the effect of exposure solution replacement. Chloride and calcium profiles were measured after 302 days of chloride exposure. The exposure solution was replaced every two weeks during the exposure period.

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	CaO content	CaO content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.41	0.214	0.247	1.419	1.637	7.244	48.039
1.38	0.222	0.269	1.475	1.784	6.909	45.815
2.40	0.204	0.259	1.353	1.716	6.587	43.680
3.41	0.181	0.235	1.197	1.555	6.435	42.676
4.44	0.148	0.205	0.980	1.358	6.033	40.011
5.86	0.127	0.182	0.839	1.209	5.801	38.468
7.84	0.091	0.138	0.604	0.913	5.525	36.642

Data for specimen L4-2B from additional series of experiments concerned with the effect of exposure solution replacement. Chloride and calcium profiles were measured after 302 days of chloride exposure. The exposure solution was replaced every two weeks during the exposure period.

Depth	Chloride content	Chloride content (Ca-corrected)	Chloride content	Chloride content (Ca-corrected)	Calcium content	Calcium content
[mm]	[% by weight of concrete]	[% by weight of concrete]	[% by weight of binder]	[% by weight of binder]	[% by weight of concrete]	[% by weight of binder]
0.70	0.225	0.242	1.491	1.602	7.774	51.551
2.01	0.228	0.253	1.513	1.676	7.543	50.020
3.06	0.210	0.240	1.394	1.594	7.310	48.476
4.03	0.191	0.217	1.268	1.437	7.372	48.890
5.12	0.167	0.195	1.105	1.294	7.136	47.324
6.73	0.126	0.177	0.838	1.171	5.976	39.631
8.78	0.065	0.090	0.430	0.595	6.048	40.105
10.85	0.064	0.044	0.427	0.291	12.237	81.149