ROUND ROBIN TEST
CHLORIDE MIGRATION

WORKING INSTRUCTION

MEASUREMENT PROTOCOL

QUESTIONNAIRE

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Date: 24 april 2009

WORKING INSTRUCTION

STAGE 1 of ROUND ROBIN TEST on CHLORIDE MIGRATION

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Date: 16 april 2009

In Stage 1 of this Round Robin Test (RRT) each participating laboratory will receive a pre-packed set of 3 cubes 150mm, made of mortar, and labelled with the name of the laboratory.

The finished face (i.e. the top side) of each cube is provided with a sticker. The information on the sticker includes the (unique) cube number, the date of casting, the date of testing, and the reference *TOP* (see Figure 1).

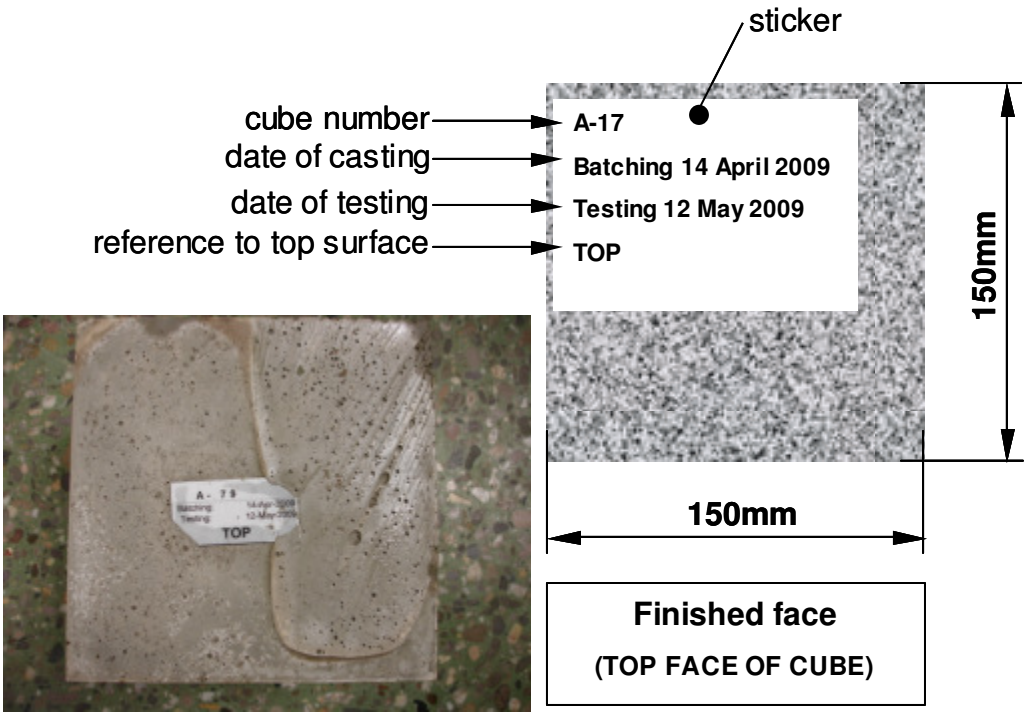


Figure 1 *Information provided on the finished face (top surface) of a cube and photo giving an example of a demoulded cube*

All cubes have been cast on Tuesday April 14 (2009); this has been achieved by homogeneous mixing of a single batch of mortar in an excess amount. All cubes have been prepared in the same way (fixed vibration time on the same vibration table; one type of mould). The mixture proportions and the type of cement used will not be given to the participating laboratories prior to migration testing.
After demoulding (1 day after casting) all cubes have been immersed completely in a saturated calcium hydroxide solution.

Immediately after receipt of the cubes at the participating laboratory the cubes have to immersed in water until drilling of the cores .

All participating laboratories will perform the migration tests according to their prevailing standard procedure.
Most laboratories will follow NT Build 492 whereas most German laboratories will follow the BAW Merkblatt.
Simply said: follow the procedure you normally follow.

On **Monday May 11** or if this is not possible at the earliest thereafter, the testing samples (disc ∅100mm; height 50mm) will receive pre-treatment (i.e. if the procedure in use at a laboratory indicates pre-treatment). From each cube a core ∅100mm is drilled perpendicular to the finished face of a cube (see Figure 2).

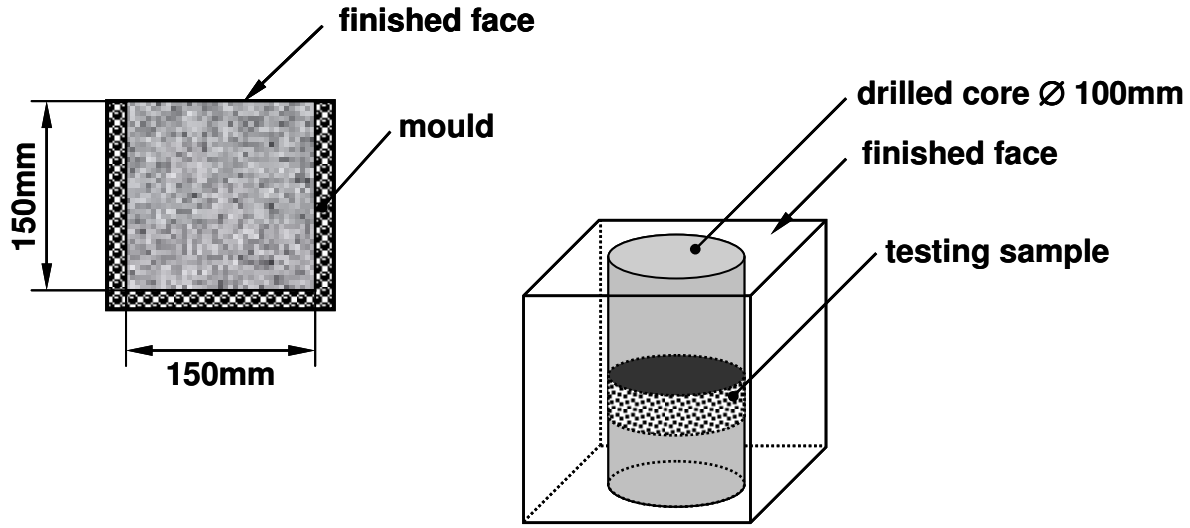


Figure 2 Position of core and testing sample in cube

Thereupon, from each core a testing sample ∅100mm, height 50mm is sawn. For all testing samples the surface to be exposed to chloride is positioned at a depth of 75mm from the finished surfaced of a cube (i.e. at half cube height), see Figure 3.

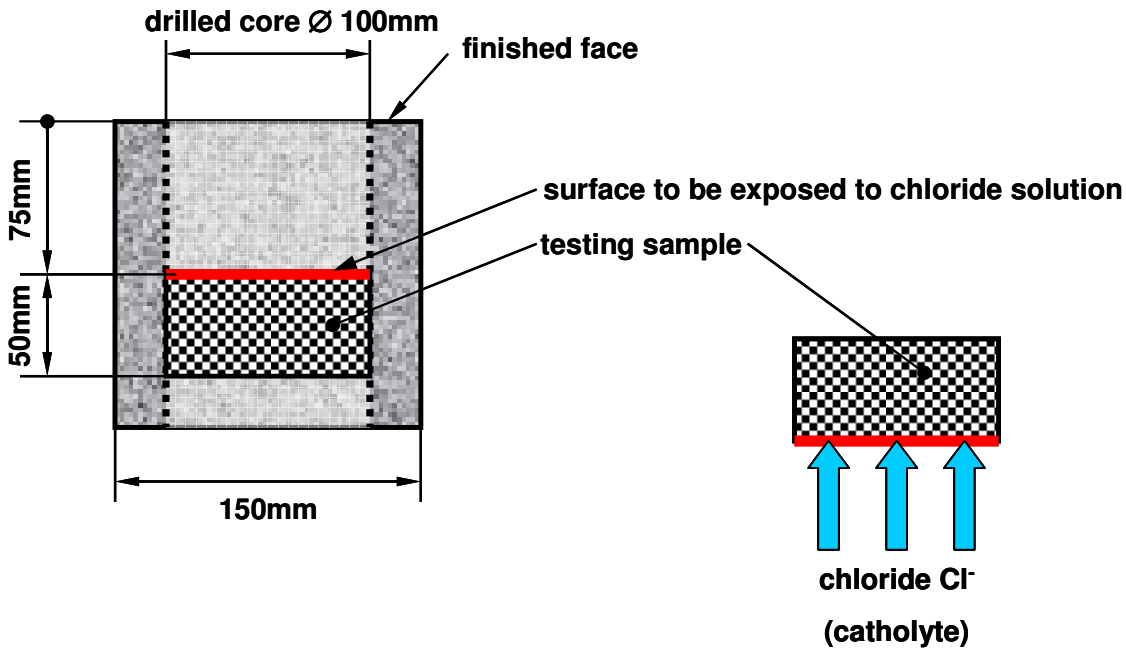


Figure 3 *Position of the testing sample in a drilled core and position of the surface to be exposed to chloride in the migration test (catholyte side)*

If the participating laboratory considers this testing sample inappropriate, then as an alternative a testing sample may be taken at the mirrored position (see Figure 4). However, the surface at a depth of 75 mm from the finished surface will remain the surface to be exposed to the chloride solution.

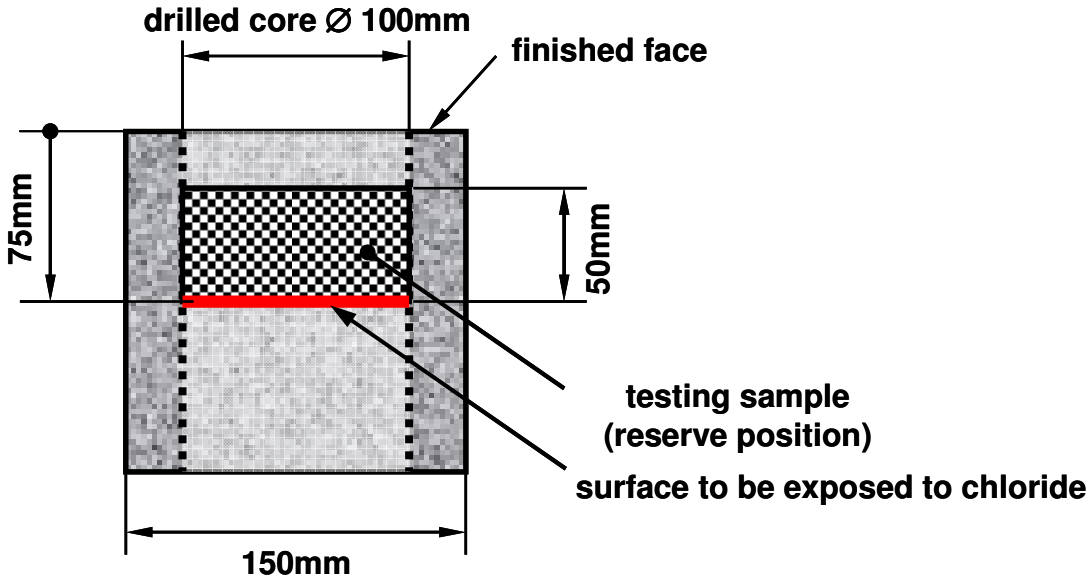


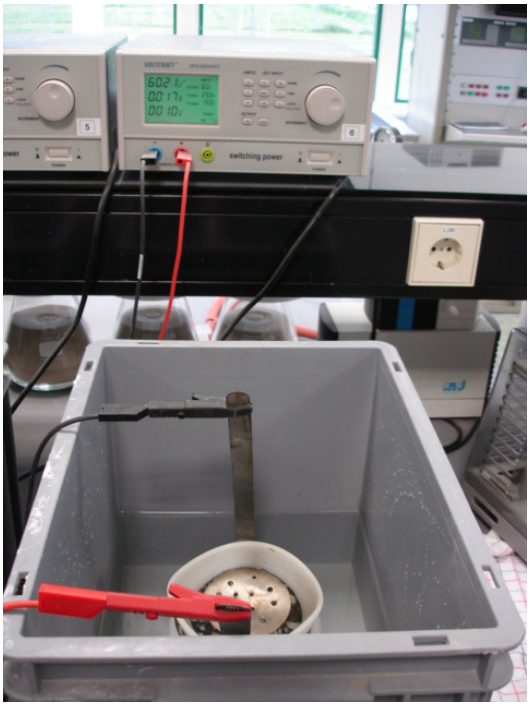
Figure 4 *Alternative position of the testing sample in a drilled core and position of the surface to be exposed to chloride*

Laboratories that prefer to test more samples can use this alternative position. In the report the position of each testing sample in the cube shall be mentioned explicitly.

After preparation of the testing samples (from each cube at least 1 and in total a minimum of 3) these samples will be completely water saturated by following the procedure in current use at the participating laboratory (e.g. NT Build 492 or BAW Merkblatt).

On **Tuesday May 12** (2009), i.e. at an age of 28 days, the testing samples (at least 3) will be subjected to the migration test according to the procedure in use at the participating laboratory .

For the evaluation of the test arrangements a number of additional measurements to be performed at the start, during and at the end of the test is suggested (provided that the participating laboratory has the appropriate equipment available); for details see the *Measaurement Protocol*. These additional measurements are not compulsory, however, it is (urgently) suggested to perform most of these measurements. The information gained from these measurements increases the opportunity to identify possible causes of deviation in the results of the migration test. This will give the participating laboratory the opportunity to modify it's migration test arrangement. In addition, the inclusion of representative digital pictures of the test arrangement of the participating laboratory in the report is appreciated; at least one picture of the overall arrangement (all testing facilities) and one of a single testing facility (preferably during operation with a testing sample); as an example see Figures 5a and 5b.



Figures 5a and 5b "Representative" pictures of the migration arrangement

NOTE:
For all laboratories the test conditions are equal:
Impressed voltage **U = 30.0V; test duration = 48.0h**
(it is known and appreciated that this is not according to NT Build 492).

On **Thursday May 14** (2009) the migration tests will be completed after a test duration of 48h; the penetration depth is measured on split testing samples according to the prevailing procedure. It is allowed to use both halves of the split testing sample for measurements of the penetration depth. Pictures of the split samples after spraying with silver nitrate solution are included in the report.
After completion of the migration test a full test report is drafted and as soon as possible this is sent to the contact of this RRT (preferably by e-mail). The full information for correspondence of the contact is:

joost.gulikers@rws.nl

Rijkswaterstaat Centre for Infrastructure
P.O. Box 20000
3502 LA Utrecht
Netherlands

Mobile +31 88 797 2305 or 31 6 20 54 12 51

The contact will "anonymise" all the information that is provided by the participating laboratories and thus it will not be possible to directly relate the reported data to a laboratory. This is preferred by some Dutch laboratories. Later, after agreement of all laboratories involved, the names can be given. Of course, for the non-Dutch laboratories a different agreement can be made.

The anonymised measurement data will be discussed by a "RRT-committee", however, it is suggested that the contact will distribute all the data (made anonymous) freely among the non-Dutch laboratories for expert comment. After discussion laboratories that have reported measurement results that significantly deviate from the average result of all reporting laboratories, will be addressed personally by the contact within 2 weeks after submission of it's report. If possible suggestions will made to these "deviating" laboratories to modify the test arrangement and/or the test procedure within 4 weeks. Modification is of course not obligatory, however it is considered important that the basic arrangement is correct before starting with Stage 2 of the RRT (realcrete).

MEASUREMENT PROTOCOL
STAGE 1 of ROUND ROBIN TEST on CHLORIDE MIGRATION

Author: Joost Gulikers
Rijkswaterstaat – Centre for Infrastructure
Date: 20 April 2009

General information

For each laboratory it's standard procedure for execution of the chloride migration test is applicable. N.B. For the Dutch laboratories this is the translation of NT Build 492 including modifications (this is included in the set of documents sent to the Dutch laboratories); for the German laboratories this is (probably) the BAW Merkblatt; for the other laboratories this will probably be NT Build 492.

All participating laboratories will perform the migration tests according to their own prevailing standard procedure.
Simply said: follow the procedure you normally follow.

For simplicity and clarity: in this Round Robin Test NT Build 492 is assumed to be the reference;
therefore all laboratories indicate differences in test set-up, concentrations etc with respect to NT Build 492
(see measurement file: RRT.exe; tab: procedure)

If things are not sufficiently clear:
contact the "contact"

joost.gulikers@rws.nl
+31 88 797 2305

Period May 6 – May 12 2009
Immediately after receipt of the cubes, remove the packaging and immerse the cubes completely in water at a temperature of approximately 20°C until pre-treatment (NT Build 492) or preparation for testing (BAW Merkblatt).
Report the date of receipt to the contact and mention the date of testing.

If the cubes are received later than Monday May 11, the planning may be adjusted; inform the contact and suggest/discuss how to act.

REMARK: in the measurement file: **RRT measurement.xls** a more detailed chronological overview of the suggested (required and optional) measurements is given.

Monday May 11 2009 (applicable to pre-treatment):

- retrieve 3 mortar cubes 150mm from the storage room;
- *optional:* measure the electrical AC resistance R_c of each cube according to the Two Electrode Method (TEM). To this end an LCR-meter is required to produce an AC voltage between 2 opposite faces of the cube and using a measurement frequency $f > 100$ Hz (see Figure 6). A wetted sponge will act as the intermediate between the metal (stainless steel) plate electrodes and the mortar. In addition, measure the AC resistance between top and bottom face of the mortar cube. Report the frequency applied and possibly the type of LCR-meter.
  *N.*B. One Dutch laboratory has recommended to apply a weight of 5kg on the top electrode as to achieve reproducible results.
  *N.*B. It is (of course) allowed to report resistance values for a number of frequencies (if the LCR meter allows this).

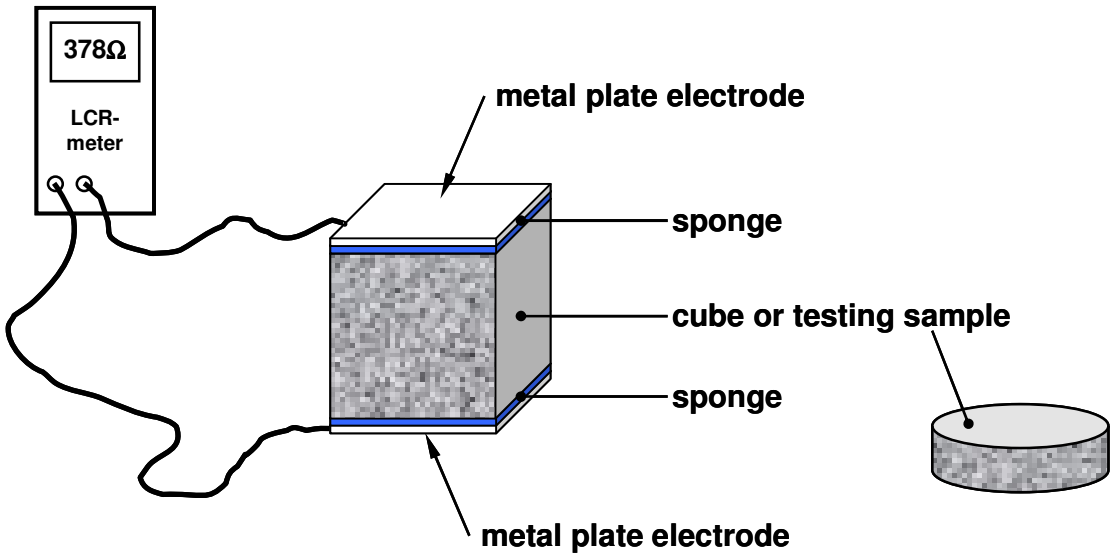


Figure 6: Test arrangement for resistance measurement on cube and/or testing sample

- From each cube drill a cylinder ∅100mm, i.e a diameter that is fit for the migration set-up of the laboratory; the direction of drilling is perpendicular to the TOP face (i.e. the face where the information sticker is attached);

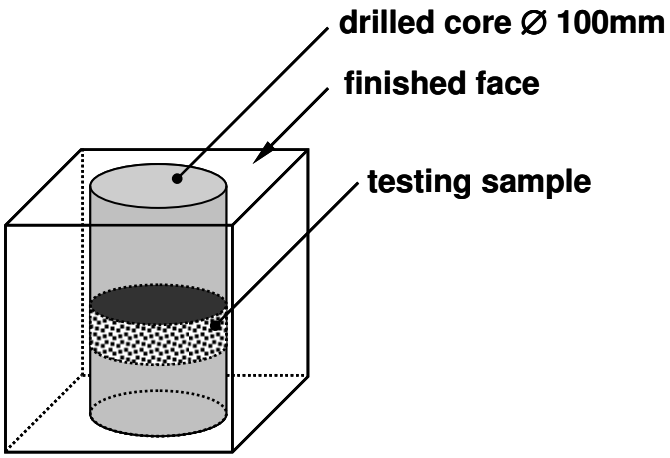


Figure 2 *Position of the drilled core and testing sample in a 150mm cube*

- From each drilled cylinder saw the testing sample required fro the migration test; the testing sample (thickness 50mm) is located a depth of 75 to 125 mm from the finished (TOP) surface (see Figure 8); if needed the alternative position of the testing sample may be chosen;

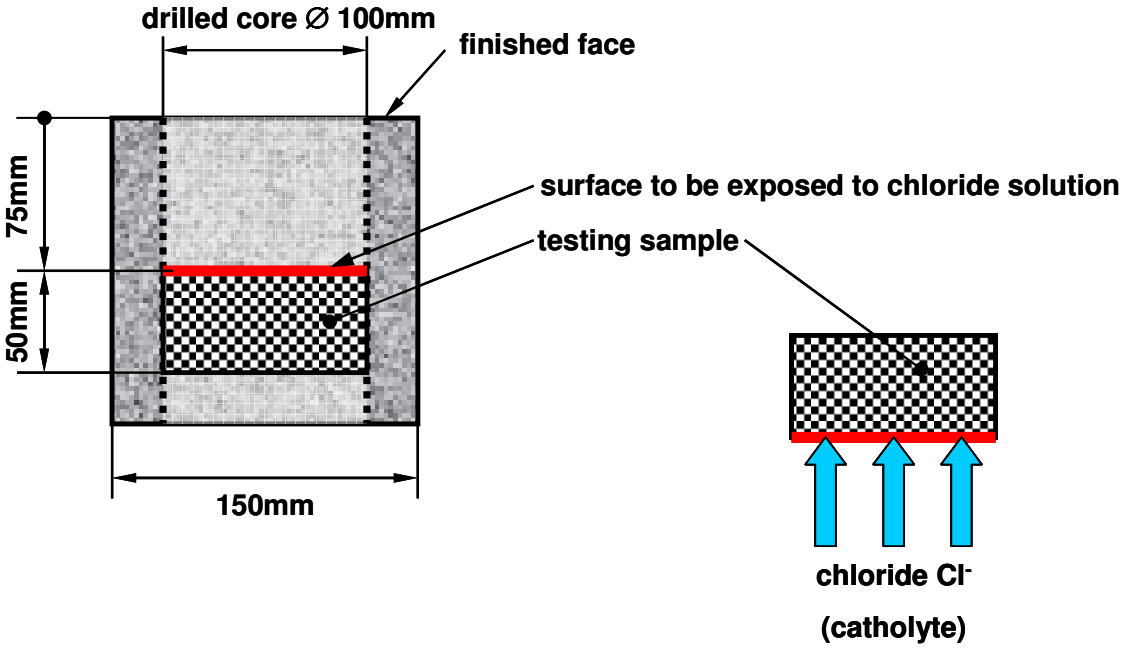


Figure 8 *Position of the testing sample in the drilled core and position of the surface to be exposed to chloride*

- Water saturate each testing sample with water according to the procedure in use at the laboratory, e.g. vacuum treatment according to NT Build 492;
- For all testing samples maintain storage under water until the migration test will start;

Tuesday 12 May 2009

- *Optional*: prior to adjustment in the rubber sleeve, measure the electrical AC resistance R_m of the water saturated testing sample using the Two Electrode Method;
- NOTE: The cathode and anode are **NOT** coupled to the DC voltage source;
- NOTE: Use freshly made solutions for both the anolyte and the catholyte;
- Place the testing samples as well as the anode and cathode in the test arrangement and fill the reservoirs with the freshly-made chloride-solution and hydroxide solution, respectively;
- *Optional*: measure the chloride concentration of the catholyte with a chloride-ion selective electrode (or an equivalent method);
- *Optional*: after approximately 5 minutes measure the voltage U_o between anode and cathode for each test facility/testing sample using a multimeter (cathode connected to COM); NOTE: the DC voltage source is NOT connected; see Figure 9.

NOTE: for reasons of simplicity in the figures the position of the testing samples in the set-up is suggested to be horizontal. This could be misleading for the reader: in reality the sample is to be placed under a slope of approximately 32° as indicated in both NT Build 492 and the BAW Merkblatt. The reader could also regard the figures to represent the front view.

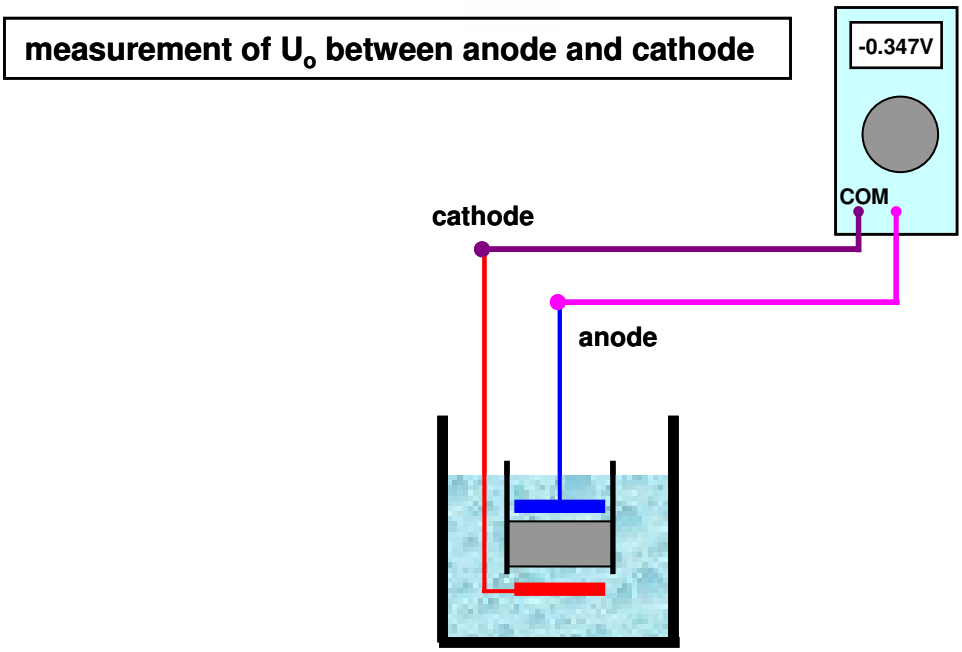


Figure 9 *Measurement of the initial voltage U_o between anode and cathode prior to application of the impressed voltage (the voltage source is NEITHER connected to the anode nor to cathode).*

- *Optional*: measure the AC electrical resistance R_{ac} between anode and cathode for each test facility/testing sample using an LCR-meter; NOTE: the voltage source is not connected; see Figure 10.
  NB **NEVER** perform resistance measurements when anode and cathode are connected to the DC voltage source.

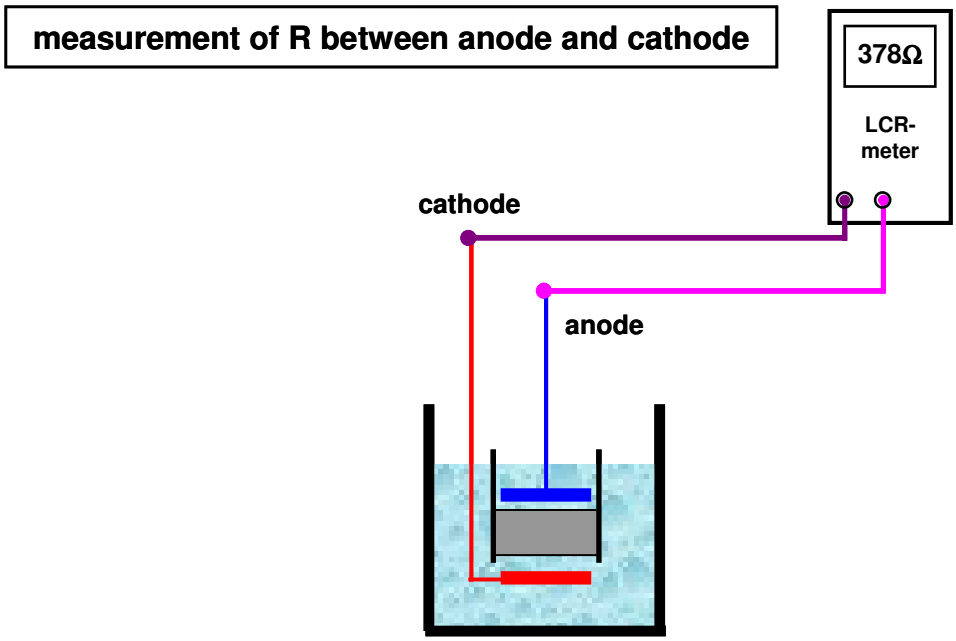


Figure 10 *Measurement of the electrical AC resistance between anode and cathode prior to the application of an impressed voltage. The voltage source is **NEITHER** connected to the anode nor to the cathode).*

- *Optional*: Insert a **precision resistor** R = 10Ω (or else R < 50Ω) with a dissipating power of 10W into the measuring circuit between the positive terminal of the DC voltage source and the anode (see Figure 11); report the magnitude of the precision resistor; NB for a lower dissipating power level there is a greater risk that the heat production will result in "burning" of the resistor; alternatively a **Zero Resistance Ammeter** (ZRA) may be used (see Figure 11)
- *Optional*: measure the pH and chloride concentration of the catholyte with an appropriate method; report the measurements results and the methods used;
- *Optional*: measure the pH of the anolyte with an appropriate method; report the measurements results and the method used;
- Measure and record the temperature of the anolyte and the catholyte;
- Connect the anode(s) and cathode(s) to the voltage source;
- Adjust the DC voltage source to an output voltage of 30.0V;
- Measure the resulting current according to the procedure generally used by the laboratory (e.g. from the display of the voltage source or using a multimeter inserted in the circuit); NOTE a multimeter may introduce an extra resistance in the circuit; check this internal multimeter resistance en report the resistance value (if known);
- *Optional*: measure and record the voltage between anode and cathode for each test facility/testing sample using a multimeter (see Figure 11);
- *Optional*: measure and record the voltage over the precision resistor for each test facility/testing sample using a multimeter (see Figure 11);

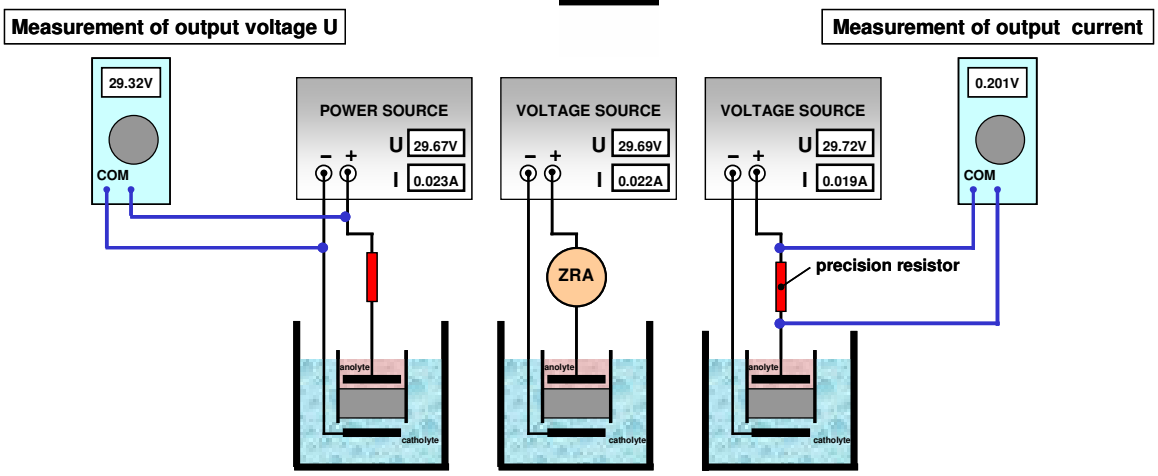


Figure 11 *Measurement of the output voltage U between anode and cathode, the voltage U_R over the precision resistor R (check of the resulting current), and the resulting current using an external ZRA.*

- Maintain the output voltage U = 30V during 48.0h;
- *Optional*: measure and record frequently or continually the voltage over the precision resistor;
- Alternatively measure the current flowing through ach testing sample frequently or continually using a Zero Resistance Ammeter (ZRA);

Tuesday 12 May – Thursday 14 May

- Check for excessive accumulation of bubbles at both anode and cathode. Check the current output and compare this with the initial level. If needed, stir the solution or move the electrodes gently (take care of the voltage between the electrodes) and check if the current output changes to a higher level.
- *Optional*: Regularly measure the temperature of the anolyte and the catholyte
- *Optional*: Regularly measure the output voltage with a multimeter
- *Optional*: Regularly measure voltage drop over external resistor; **and or**
- *Optional*: Regularly measure the output current using a ZRA (Zero Resistance Ammeter).
- *Optional*: Regularly measure the pH of the anolyte and the cathloyte
- *Optional*: Regularly measure the chloride concentration of the catholyte

Thursday 14 May 2009

- Measure and record the temperature of the anolyte and the catholyte at the end of the migration test;
- *Optional*: measure the pH and chloride concentration of the catholyte with an appropriate method;
- *Optional*: measure the pH of the anolyte with an appropriate method;
- *Optional*: measure and record the voltage over the precision resistor using a multimeter;
-

- Measure and record the voltage between anode en cathode using a multimeter (cathode connected to COM);
- Maintain the multimeter between anode en cathode;
- Switch of the voltage source or adjust the voltage source to an output level U = 0.0V;
- Uncouple anode and cathode from the voltage source by pulling out the cables;
- Measure as soon as possible after this physical uncoupling the voltage $U_{ac,end1}$ between anode and cathode by using the multimeter;
- Maintain the testing sample in the testing facility;
- Approximately 5 minutes after physical uncoupling measure the voltage $U_{ac,end2}$ between anode and cathode using the multimeter;
- *Optional*: Approximately 5 minutes after physical uncoupling measure the AC electrical resistance $R_{ac,end}$ between anode and cathode using an LCR-meter (with the testing sample still placed in the testing facility);
- Remove the testing sample from the testing facility;
- Split or break the testing sample;
- Spray the freshly split or broken surface with a silver nitrate solution;
- Within **5** minutes after spraying measure the penetration depth at 6 equidistant locations at a minimum;
  N.B. The requirement of 5 minutes is indicated by experiences gained from pre-testing. For longer waiting times a "halo" has been reported to develop which may obscure the measurement of the actual penetration depths.
- Make a clear photo of each split and sprayed testing sample (full width);
- Record any peculiarities and/or deviations;
- Draft a detailed report mentioning all individual measurement results (including penetration depths) and calculations and comment if required;

QUESTIONNAIRE – ROUND ROBIN TEST CHLORIDE MIGRATION

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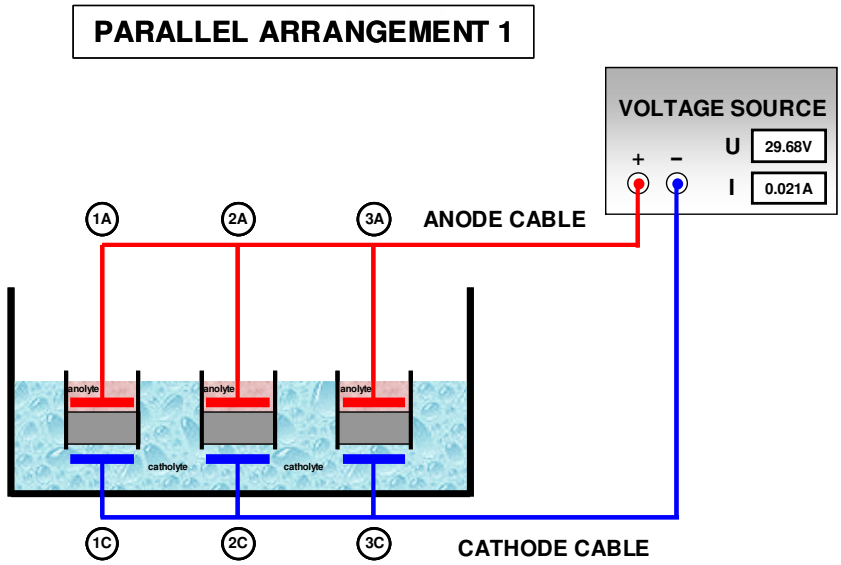
Date: 16 April 2009

These questions apply to the <u>normal</u> test procedure adopted by the participating laboratory.

1. Which electrical arrangement is used: a parallel arrangement of a number of individual test facilities with one common voltage source either using one common catholyte or separate catholytes, or one power source for each testing facility (choose from Figures 20, 21 en 22 or include a sketch if your arrangement is different)?
2. What is the (average) amount/volume of catholyte (chloride solution) that is available per testing facility at the start of the migration test (or: how much catholyte is prepared on average for each testing sample)?
3. How frequent a freshly-made <u>anolyte</u> solution is introduced?
    a. After each test the anolyte solution is replaced by a freshly-made solution
    b. The anolyte solution remaining after a test is re-used and regularly freshly-made solution is added; on average after …..tests a freshly-made solution is used;
4. How frequent a freshly-made <u>catholyte</u> solution is introduced?
    a. After each test the catholyte solution is replaced by a freshly-made solution
    b. The catholyte solution remaining after a test is re-used and regularly freshly-made solution is added; on average after …..tests a freshly-made solution is used;
5. On average how many migration tests are performed per year
    a. More than 1 test per week;
    b. Less than 1 test per week;
6. Is the chloride concentration of the catholyte frequently measured, e.g. with a chloride ion selective electrode, and if yes when (prior, during and/or after the test) and what is the concentration at the start relative to the concentration at the end of the test (much higher, lower etc)?
7. What is the catholyte composition, e.g. 100g NaCl dissolved in 900 g of tap water?
8. What is the anolyte composition, e.g. 12 g NaOH dissolved in 1 litre of tap water?
9. How many times has a “breakthrough” been observed after the test (chloride has penetrated deeper than the thickness of the testing sample/ the penetration depth lies “beyond” the testing sample/ the complete cross sectional area shows discoloration)?
10. If “breakthroughs” have been observed what was then mostly the (likely) cause?
11. What type of stainless steel is used for the electrodes (if known)?
12. What is the (normal) variation (minimum and maximum) in the average penetration depths (measured after the migration test); this question does not refer to the variation as measured in <u>one single testing sample</u>.
    a. 10-30mm
    b. ……

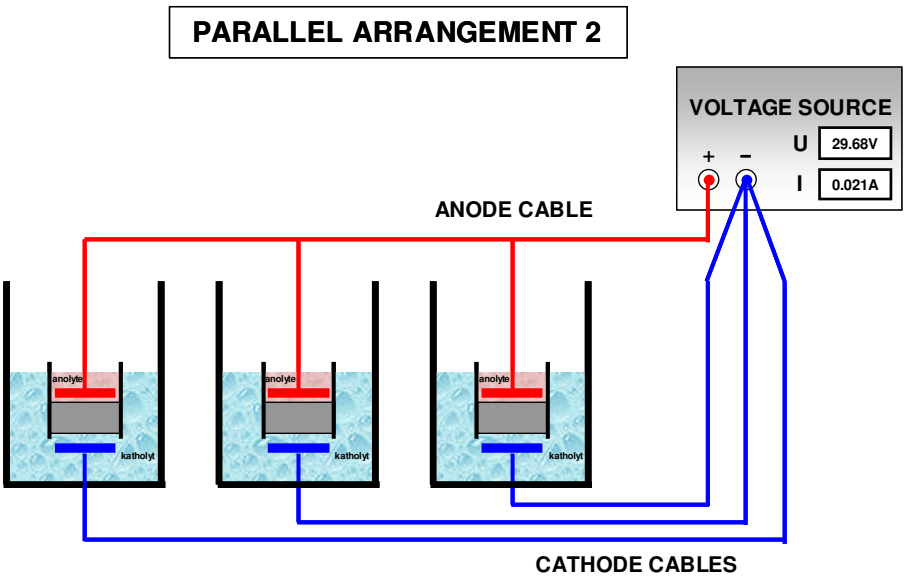
13. What is in general the average penetration depth for all the migration tests performed until now? N.B. The implicit assumption is that the test conditions (voltage and duration) are such that the penetration front will be at a depth of 25mm.

14. How is the applied voltage been measured?
    a. As the value given on the display of the voltage source?
    b. Through a (possibly temporarily connected) multimeter introduced in the circuit?
    c. Another way ……
15. How is the resulting current been measured:
    a. From the display of the voltage source?
    b. Through a (possibly temporarily connected) multimeter introduced in the circuit?
    c. Another way ……
16. Is the migration test performed in a conditioned room (temperature and RH)?
17. Have there been occasions that the testing is warm or hot at the end of the migration test? Does this happen frequently and what temperature level occurs?
18. Own comments/remarks/questions



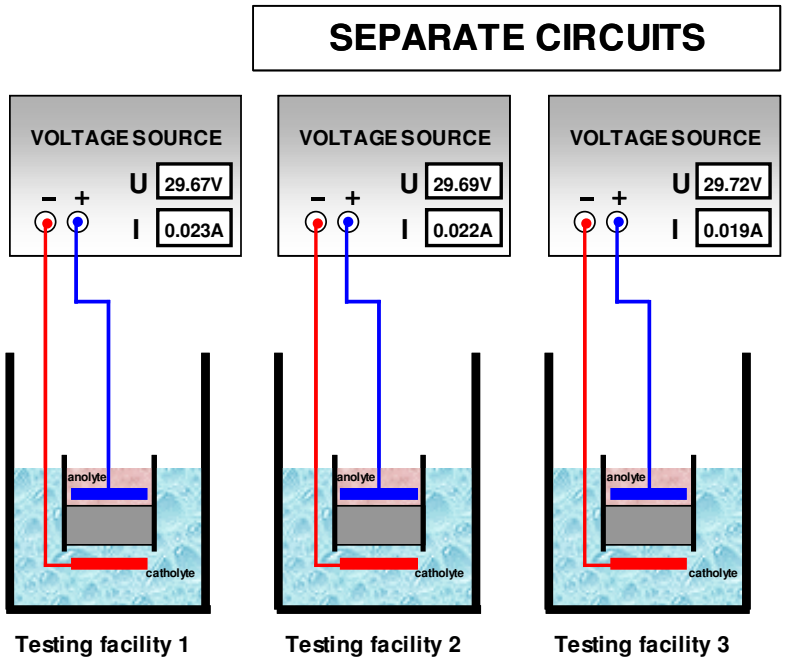
- ALL ANODES ARE COUPLED
- ALL CATHODES ARE COUPLED
- ONE SINGLE VOLTAGE SOURCE
- ONE COMMON CATHOLYTE

Figure 20 Test set-up with parallel arrangement of the test facilities



- ALL ANODES ARE COUPLED
- ALL CATHODES ARE COUPLED
- ONE SINGLE VOLTAGE SOURCE
- FOR EACH TEST SET FACILITY A SEPARATE CONTAINER

Figure 21 Test set-up with an alternative parallel arrangement of the test facilities



•EACH TESTING FACILITY HAS IT'S OWN VOLTAGE SOURCE

•EACH TESTING FACILITY HAS IT'S OWN CATHOLYTE AND ANOLYTE COMPARTMENT

Figure 22 Test arrangement with a voltage source for each individual test facility