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Appendix 1	List of participants
Appendix 2	Workshop programme

## List of symbols and abbreviations

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RH	relative Humidity
p	the actual water vapour pressure
p <sub>s</sub>	the maximum vapour pressure or saturation pressure
DCS	degree of capillary saturation
DVS	degree of vacuum saturation
DPS	degree of pressure saturation
TDR	time domain refractometry
T	temperature
V	volume
h	hour
Ø	diameter

See also Table 4.

# 1 Introduction

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SINTEF and NTNU organized a **WORKSHOP on moisture in concrete, 6<sup>th</sup>-7<sup>th</sup> February 2012** in Trondheim. The workshop was related to and supported by the COIN Focus Areas 3.1 “Crack free concrete structures” and 3.2 “Service life”. The purpose of the workshop was to share information on

- available methods of measuring and modeling moisture in concrete and discuss pros and cons
- moisture induced volume changes.

The present report gives a brief overview over most of the methods presented and discussed at the workshop. A list of participants is given in Appendix 1; the programme is given in Appendix 2.

The moisture state of concrete may be characterized either by moisture content or by relative humidity (RH) exerted by the moisture. The two are related via the sorption isotherm (moisture content vs. RH), which is not a given relationship, but which for a given concrete depends on a large number of factors. The most important are degree of hydration, temperature history, moisture history (drying/wetting), carbonation and whether the present moisture state is one of adsorption or desorption.

The choice of RH or moisture content to characterize the moisture state in a given case depends on the purpose. For example, moisture content or degree of capillary saturation (as degree of filled pores) is obviously most relevant if the problem at hand is frost damage. For chemical reactivity of various kinds RH may be most relevant, since the RH, and not the amount, is a direct measure of the activity of the pore water.

The methods discussed are divided into two categories:

1. measuring the relative humidity (RH)
2. measuring the moisture content, and the degree of capillary, vacuum and pressure saturation (DCS, DVS and DPS)

Based on the discussions, literature and procedures earlier used at SINTEF and NTNU for measurement relative humidity, and measurement of moisture content and degree of saturation are proposed.

Table 2 and Table 5 provide an overview of respectively relative humidity sensors and methods for measurements of moisture content and degree of saturation.

## 2 Measurement of relative humidity

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### 2.1 Introduction

The relative humidity (RH) is a parameter to describe the moisture state of concrete. One measures the relative humidity of the air in equilibrium with the concrete.

The RH of air is the ratio between the actual water vapour pressure,  $p$ , in the air and the maximum vapour pressure or saturation pressure,  $p_s$ , at the actual temperature.  $RH = p/p_s$ .

In this chapter a range of RH sensors are presented and compared. The possible error sources and ways of measuring RH are discussed.

### 2.2 RH sensors

There are different types of RH sensors:

- Capacitive polymer RH sensors
- Resistive polymer RH sensors
- Electrolytic RH sensors
- Dew point RH sensors / chilled mirror sensors

A capacitive humidity sensor consists of a hygroscopic dielectrical material placed between a pair of electrodes, which form a small capacitor. The capacitance in the sensor depends on its moisture content and hence on the RH of the air it is in contact with.

In a similar way, a resistive humidity sensor contains hygroscopic material of which the resistivity changes with the RH e.g. a polymer or wood, and the electrolytic humidity sensor contains an electrolyte of which the electric conductance changes with the RH.

The chilled mirrors measure the dew or frost point temperature directly by controlling a reflective surface to an equilibrium temperature between dew/frost formation and evaporation.

A range of different RH sensors are discussed in this chapter. Table 1 gives an overview of the sensors discussed and their type.

**Table 1: The RH sensors discussed in this chapter and their type**

Sensors	Capacitive	Resistive	Electrolytic	Chilled mirrors
<b>Rotronic HygroClip 2</b>	X			
<b>Viasala HM44</b>	X			
<b>HumiGuard</b>			X	
<b>Wood samples</b>		(X)		
<b>Testo 605-H1 and 0636.9769</b>	X			
<b>Sensirion SHT75/ Rapid RH® 4.0</b>	X			

#### 2.2.1 Rotronic HygroScope DT

The Rotronic probe (HC2) measures humidity with a ROTRONIC Hygromer® IN1 capacitive sensor and temperature with a Pt100 RTD (see Figure 1) [1].

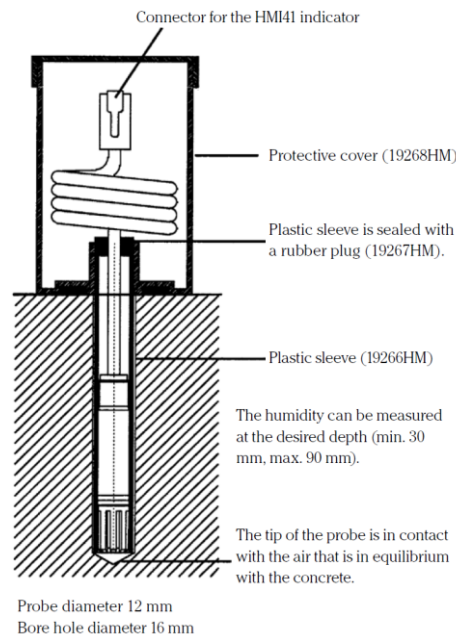
In Norway, the Rotronic sensor is sold as part of a monitoring system for concrete structures called CAMUR distributed by Protector [2].



**Figure 1: HC2 Rotronic probe [1, 3].**

### 2.2.2 Vaisala HM44 Concrete Humidity Measurement System

The Vaisala HM44 sensor measures both temperature and RH [4]. It contains a HUMICAP® 180 capacitive polymer RH sensors, and a Pt 100 IEC 751 1/3 Class B temperature sensor.<sup>1</sup>



**Figure 2: Vaisala measuring set-up [4].**

### 2.2.3 HumiGuard

The HumiGuard sensor is an electrolytic sensors [5]. The sensor is for single use only and has a limited period of use (6 months from delivery date). It is delivered with a "best before date".

The sensor characteristics are uniform within a package of sensors. The package is calibrated by the producer. The measurements are adjusted for possible drift by comparing to a small fraction of the sensors placed in a reference block with a controlled RH.

The measuring range for which the HumiGuard sensor can be used is: 75-98% RH. However, the sensor should not be submitted to RHs in the range of 95-98% for more than 1 week. Hence, the sensor should be mounted into the concrete or paste after casting and when one can expect that the RH has dropped below 95%.

<sup>1</sup> Temperature sensors are not discussed here

The HumiGuard sensor measures besides the RH also the temperature.



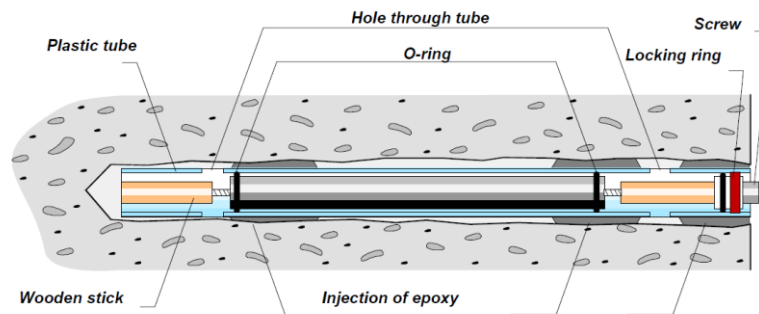
**Figure 3: The reference cell, RH sensor, sensor contact and measurement tube [5].**

### 2.2.4 Wood samples

A piece (stick) of wood is put in contact with concrete. The RH in the wood should have time to become in equilibrium with the RH in the concrete. Hence by measuring the RH of the wood, one can retrieve the RH of the concrete.

At high RH (close to 100%) the fibre solubility point of wood is reached and the sensors might rot [7]. Hence, high RH measurements can be problematic. The use of wood samples is advised in the RH interval 70-95%.

Figure 4 shows the mounting of a wood piece in a drilled hole (20 mm diameter). The RH and moisture content of the wood samples was measured.



**Figure 5: Plastic tube mounted with 2 wooden sticks. The contact between the concrete and the plastic tube is sealed with injected epoxy sealers. The diameter of the tube is 20 mm. [7]**

The moisture present in the stick can be measured using an electrical conductance moisture probe (measuring the conductivity between two needles) [7]. It is stated that the temperature only has a minor influence on the electrical conductivity of wood, which cannot be said of other RH measurements on concrete.

### 2.2.5 Testo sensor 605-H1 and 0636.9769

The Testo sensor is a capacitive sensor. In [8] two types of Testo sensors are recommended for RH measurements in concrete: Sensor 605-H1 with build-in display which shows the RH, temperature and dew point temperature and Sensor 0636.9769 combined with the Testo 635 data acquisition tool. During measuring Sensor 605-H1 should be protected with surgeon tape (e.g. scanpor). For Sensor 0636.9769 a Teflon filter is available.



**Figure 6: Testo 605-H1 [9].**

### 2.2.6 Sensirion SHT75 sensor and Rapid RH® 4.0

The Sensirion sensor is a capacitive sensor [10]. It has a length of 19.5 mm, a width of 4 mm and 2 mm thick. They are fully calibrated when purchased and log both RH and temperature. The equipment was presented by K. Kielsgaard Hansen at the moisture workshop Febr. 2012 Trondheim [11]. The Sensirion sensor should not be cast into concrete as certain parts of the sensor should not come into direct contact with the concrete. Wagner Meters offer a complete solution called Rapid RH® 4.0 EX for RH and temperature measurements in concrete floor slaps using the Sensirion sensors [12].



**Figure 7: Sensirion SHT75 sensor [10].**

### 2.2.7 Comparison

#### *RH interval covered by the sensor*

The relative humidity of concrete out in the field which is relevant for durability studies varies between approximately 70-100% RH [13-15]. The lower limit of this RH interval of interest does not pose a problem for the above named RH sensors, whereas the upper limit of this interval (95-100% RH) is not covered by some of the sensors e.g. HumiGuard, Testo and wood samples. These sensors can on the other hand be used to measure drying of concrete when one is sure that the RH has dropped below the upper RH limit of the sensors. It should be noted that even for the sensors which can be used over the entire interval (80-100% RH), the accuracy of the sensors is reduced at high RH. Based on the RH interval covered the following sensors can be used for RH measurements of field samples:

- Rotronic HygroClip 2
- Viasala HM44
- Sensirion SHT75/Rapid RH® 4.0

#### *Calibration*

The reusable sensors should be calibrated before and after each measurement. Some of the sensors are calibrated by the producer (see Table 2) and do not require calibration before use. Not having to calibrate the sensors can be an advantage when using sensors out in the field. However, one could still check the sensors before and after field measurements by exposing them to a known RH. Reusable RH sensors which can be calibrated and which cover the 80-100% RH interval are:

- Rotronic HygroClip 2
- Viasala HM44

Table 2: Comparison of relative humidity sensors

Instrument	Lab	In-situ	Principle	T range	$\Delta T$	RH range	$\Delta RH$ (*)	Calibration by producer	Cost 2012 (approx.)	Application
<b>Rotronic HygroClip 2</b>	X	X	Capacitive	-100..200°C	$\pm 0.1^\circ\text{C}$	0..100%	$\pm 0.8\%$ at 23°C 10,35,80% RH [3]	No	4100 NOK per sensor	[13, 16-18]
<b>Vaisala HM44</b>	X	X	Capacitive	0..40°C	$\pm 0.2^\circ\text{C}$ [19]	0..100%	$\pm 1.7\%$ (0-90% RH) $\pm 2.5\%$ (90-100% RH) [19]	No	2000 NOK per sensor	[8, 13, 20-22]
<b>HumiGuard</b>	X	X	Electrolytic	0..40°C		75..98%	$\pm 2\%$ at 85% RH [23]	Yes	330 SEK-RH sensor + contact 240SEK reference 120SEK control 10 000SEK start pack.	[8, 24, 25]
<b>Wood samples</b>	X	X	Resistive (?)	/	/	70..95%	$\pm 6\%$ [7]	No	/	[7, 16]
<b>Testo© 605 H1 and 0636.9769</b>	X	X	Capacitive	0..50°C	$\pm 0.5^\circ\text{C}$ [9]	5..95%	$\pm 3\%$ [9]	No		[8]
<b>Sensirion SHT75/ Rapid RH® 4.0</b>	X	X	Capacitive	-40..125°C	$\pm 0.3^\circ\text{C}$ (20-25°C) [10]	0..100%	$\pm 1.8\%$ (10-90% RH) $< \pm 4\%$ ( $> 90\%$ RH) [10]	Yes	200 NOK per Rapid sensor 2000 NOK Rapid reader	[11]

(\*) The Manual for Fuktmätning i Betong [8] gives an overview of the different possible causes of error and the associated standard errors as well as a procedure to calculate the error associated with the RH measurement.



### 2.3 Calibration

Some of the RH sensors are delivered with a calibration rig containing prepared saturated salt solutions. However, to calibrate RH sensors one can prepare one's own saturated salt solutions in sealed glass tubes (see Figure 8). Table 3 shows a selection of saturated salt solutions and their respective RH depending on the calibration temperature. A more complete list of saturated salt solutions and their respective RH depending on the temperature is given in [26].

NT Build 340 [27] gives a procedure for calibrating of RH sensors over saturated salt solutions. It describes the testing of the hysteresis and repeatability, and the calculation of the calibration constants and uncertainty.

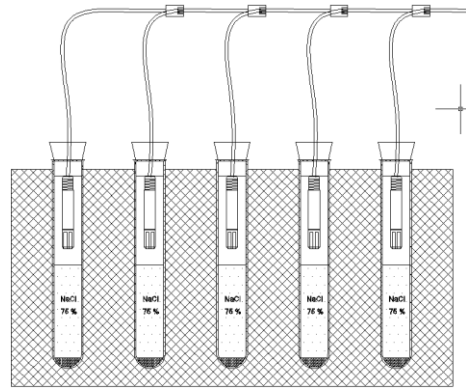
**Table 3: The RH produced by selection of saturated salt solutions for different calibration temperatures [8, 13].**

Saturated salt solutions	10 °C	15 °C	20 °C	25 °C
<b>LiCl</b>	11.3 ± 0.4	11.3 ± 0.4	11.3 ± 0.3	11.3 ± 0.3
<b>MgCl<sub>2</sub></b>	33.5 ± 0.2	33.3 ± 0.2	33.1 ± 0.2	32.8 ± 0.2
<b>Mg(NO<sub>3</sub>)<sub>2</sub></b>	57.4 ± 0.3	55.9 ± 0.3	54.4 ± 0.2	52.9 ± 0.2
<b>NaBr</b>	-	-	59	-
<b>NaCl</b>	75.7 ± 0.2	75.6 ± 0.2	75.5 ± 0.1	75.3 ± 0.1
<b>KCl</b>	86.8 ± 0.4	85.9 ± 0.3	85.1 ± 0.3	84.3 ± 0.3
<b>BaCl<sub>2</sub></b>	93 ± 2	92 ± 2	91 ± 2	90 ± 2
<b>Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O</b>	-	-	93	-
<b>KNO<sub>3</sub></b>	96 ± 1.4	95.4 ± 1.0	94.6 ± 0.7	93.6 ± 0.6
<b>K<sub>2</sub>SO<sub>4</sub></b>	98.2 ± 0.8	97.9 ± 0.6	97.6 ± 0.5	97.3 ± 0.5

RH sensors can also be calibrated using a “Two-Pressure” Humidity Generator such as a Thunder Scientific climate chamber [28]. The humidity produced with such a generator depends solely on the pressures and temperatures and does not rely on RH sensors. The uncertainty of the generated RH is about 0.5%. The University of Lund has this equipment and offers calibration of RH sensors to external customers.

For the samples where the RH is measured in a hole drilled in an existing structure or in a cast-in plastic tube, the sensors are left in place over a longer time (e.g. several weeks). The sensors should be calibrated before the start of the test and at regular intervals (e.g. each 14 days) to compensate for drifting of the sensors.

When the RH is measured on crushed samples kept in a closed test tube, the equilibrium is reached much faster (up to 6 days). Hence the sensors can be calibrated before and after the experiment.



**Figure 8: Calibration over salt solutions [22].**

## 2.4 Error sources

Performing RH measurements on concrete and interpreting the results should be done with care. Below a selection of possible error sources is discussed based on the following reports [8, 13, 21].

Inherent error sources when measuring RH:

- Sensors can drift during measurement. This is especially of importance for sensors which are kept over longer time in contact with the test material e.g. HumiGuard. Therefore, reference sensors from the same batch are tested over a reference solution to verify whether the sensors from the same batch drift.
- Hysteresis of the sensor and the tested material results in different moisture content for the same RH depending on whether moisture is taken up, absorption, or given away, desorption. This needs to be considered when using techniques where the moisture content is measured; e.g. wood samples; see 2.2.4)
- The non-linearity of sensors, meaning that the RH measured by the sensor does not relate perfectly linearly to the actual RH, results in a systematic error. To reduce this error, the sensors can be calibrated at a range of RHs within the RH interval of interest e.g. 75-95%.
- The sensors have a certain moisture capacity. Therefore, the sensors can take up moisture from the concrete or give moisture to the concrete, affecting its moisture state and the actual RH measured.
- Measuring at a different temperature than the calibration temperature.
- Error of the salt solution which is used for the calibration.

Errors in executing the RH measurements:

- Adequate sealing of the drill hole in the field or of the test tube in the lab is crucial in order to obtain relevant measurements.
- The volume of air sealed between the concrete and the sensor should be limited, as this volume of air has a moisture capacity which can affect the measured RH.
- One should wait long enough so equilibrium between the concrete and the air in contact with the sensor is obtained. The denser the concrete porosity, due to e.g. lower the w/c ratio, the longer it will take to reach equilibrium.
- Temperature difference between sensor and concrete should be avoided. Concrete contains relatively more moisture and its RH is not that sensitive to temperature variations. The air in between the concrete and the sensor on the other hand contains little moisture and the RH is much more sensitive to temperature variations. A

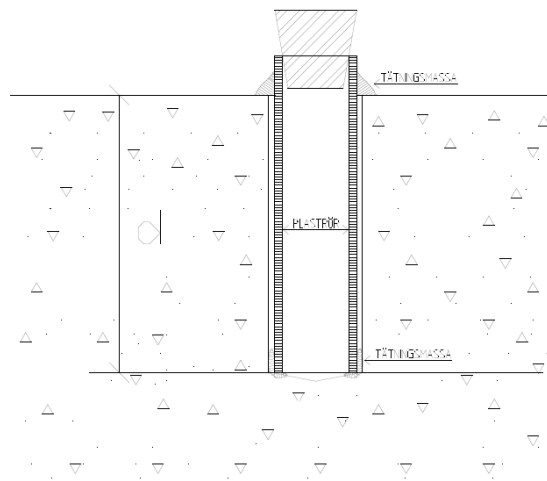
temperature difference of 1°C between the sensor and the concrete can result in a difference of  $\pm 5\%$  RH.

- Inserting a sensor at a lower temperature than the tested material might result in condensation which can result in damage to the sensor and wrong readings and a long time to reach equilibrium as the concrete needs to take up the excess moisture.
- For in field measurements one should carefully choose where to measure, e.g. avoid measuring in direct sun light, in an area where the equipment and the concrete are subjected to draft or in cracked concrete. The holes should be drilled without cooling water and cleared for dust (vacuum cleaning or blowing) to be able to seal the hole. A plastic tube which will hold the sensor is fitted into the hole and contact between the tube and the concrete is sealed in order to measure the RH at the drilled depth. One should wait with measuring the RH in the drilled holes for ca. 3 days so that the disturbance caused by the drilling does not affect the RH measurement.
- Capacitive sensors can be affected by for example polystyrene, ethanol, ammoniac, gasses from glue. These substances can make the sensors drift with 10-20% RH [21].

## 2.5 Ways of measuring RH

### 2.5.1 In a bore hole

The measuring equipment can be used in existing structures by first drilling hole and fitting in a cylindrical guiding tube which is slightly larger than the sensor (see Figure 9).



**Figure 9: Mounting of plastic tube for RH measurements in a drilled hole, from [8].**

The system - sensor, air in contact with concrete and concrete - should be sealed. For this ventilation tape [21], putty [20, 24], rubber stoppers [20, 22, 24], and Parafilm [22] have been used.

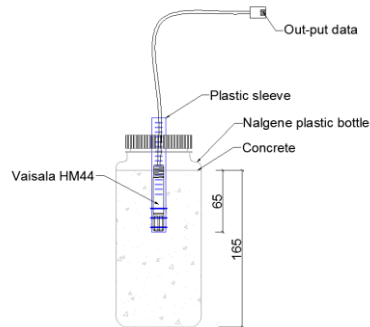
### 2.5.2 Cast-in tubes

Similarly a guiding tube with a diffusion open tape in the bottom can be cast in a concrete or cement paste sample as shown in Figure 10 [22]. To avoid possible damage of the sensor, the sensor can be entered into the tube after hardening or when the RH has dropped below the upper RH limit of the sensors.

*Duration until RH stabilizes*

Kjellmark G [22] reports that in case of cast-in plastic tubes at least 7-14 days are needed for the RH to stabilize. According to the Swedish council of the construction industry, one should at least wait 12 hours for a  $w/c > 0.4$  and 48 hours for  $w/c < 0.4$ .

When using cast-in tubes, it is advised to crush the cement paste or concrete in the bottom of the tube with a screwdriver to destroy the potentially denser surface layer which might have formed after casting of the sample.



**Figure 10: Measuring RH in a cast-in plastic tube [22].**

### 2.5.3 Crushed concrete sample

The RH can also be measured on crushed concrete or cement paste samples kept in a sealed tube as shown in Figure 11. During crushing care should be taken to avoid moisture loss.

#### *Sample size of crushed concrete*

In Lindgård J's thesis [20] the pieces of mortar obtained after crushing concrete and removing the larger aggregates have a dimension of about 2-5 mm. The pieces were not taken from the outer 15-20 mm of the concrete sample. The glass tube was filled 3/4. Kjellmark G [22] used concrete pieces with a diameter of 4-12 mm (primarily cement paste). The test tubes were put in a styrofoam mould to protect them from temperature variations.

#### *Duration until RH stabilizes*

Kjellmark G [22] reports that for the crushed concrete samples in a test tube about 4-6 days are needed for the RH to stabilize. According to Lindgård J's experiences, 3-4 days should be sufficient if the RH is  $>0.45$ .



**Figure 11: Measuring RH on a crushed concrete sample kept in a closed test tube [22].**

The samples could be crushed even finer than 2-5 mm in order to shorten the time to reach a stable RH reading. However, the finer the powder the more sensitive it is towards carbonation and changes in moisture content [29]. Concrete pieces could be sealed in a plastic bag and crushed fine with a hammer to limit the contact with the  $\text{CO}_2$  and moisture in the air.

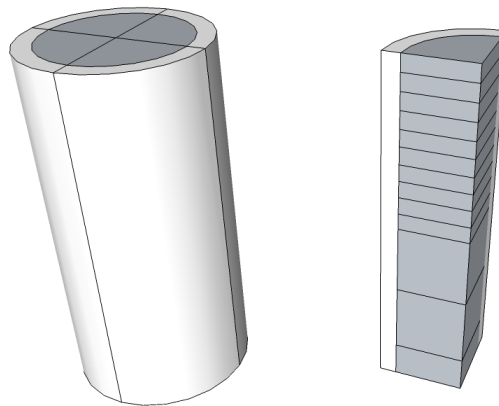
### 2.5.4 Comparison

#### *Cast-in or drilled-in cylinders:*

The set-ups with both cast-in and drilled-in cylinders require a long time to reach a stable RH reading. The long time needed to reach equilibrium is not related to the response time of the RH sensor used, but rather to the fact that the air in the cylinder needs time to equilibrate with the RH in the concrete. Thus, the sensor should preferably be left in the plastic cylinder to be able to reach equilibrium. Hence if several parallel measurements are to be performed a sensor is needed for each measurement.

#### *Crushed concrete:*

When preparing crushed concrete samples taken out in the field, one must avoid moisture loss or redistribution during the transport and storage prior to testing. The actual testing of the RH should take place in the laboratory as it is crucial to control the temperature while measuring. The concrete can be sampled by for example drilling out a concrete core or sawing and chiselling out a concrete slice. The concrete core or slice should be cleaned and dried with a moist cloth if the drilling or sawing is performed with water cooling, and it should subsequently be wrapped immediately in plastic to avoid drying. They should be transported to the laboratory as soon as possible and stored under testing conditions prior to testing. Storing at colder temperatures than the exposure temperature should be avoided as this might lead to condense formation. When sampling the core or slice for the RH measurements one should avoid taking concrete pieces from the outer surface which might have been affected by the cooling water used for drilling or sawing. Figure 12 demonstrates how concrete pieces can be taken from a concrete core for RH measurements by splitting it in four and chiselling out the inner part.



**Figure 12: Taking concrete pieces for RH measurement from drilled core.**

### 2.6 Standardisation

Using the Vaisala HM44 and Rapid RH<sup>®</sup> drilled into hardened concrete, meet the ASTM standard "F2170-02 Standard test method for determining relative humidity in concrete floor slabs using situ probes." The former can also be pre-installed into wet concrete and meet the standard.

The Manual for Fuktmätning i Betong [8] is a detailed manual for RH measurements of concrete floors either my direct measurements in a drilled hole or on taken-out samples. The Swedish Construction Federation requires that this manual shall be followed when measuring RH in concrete floors.

## 3 Measurement of moisture content and degree of saturation

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### 3.1 Introduction

The in-situ moisture content is the ratio between the mass of moisture present in the concrete and the dry mass of the concrete (or the volume of concrete).

The degree of saturation (DS) is the ratio between the volume of moisture present in the concrete and the volume of moisture in a fully saturated concrete. The degree of capillary saturation (DCS) is the ratio between the volume of moisture present and the volume which is taken up when the concrete is exposed to capillary suction from one or all sides of the sample till equilibrium. Note that capillary suction is assumed to saturate the paste i.e. gel pores, interlayer space and capillary pores.

In literature a range of methods can be found to determine the moisture content and the degree of saturation. All methods are based on the principle of weighing the samples at different moisture conditions. The difference between the methods lies in the sample size, the moisture conditions tested and equilibration time at these conditions.

### 3.2 Methods

#### 3.2.1 Applicability

The test is performed in the laboratory on samples taken in the field or produced in the laboratory.

#### 3.2.2 Principle

The sample is weighed before testing and after different conditioning steps. Based upon weight differences the following information can be obtained:

- Moisture content in weight % or vol %
- Degree of capillary saturation (DCS) in % or fraction
- Degree of vacuum or pressure saturation (DVS or DPS) in % or fraction
- Gel/capillary (suction) porosity and macro porosity (air content) in volume %
- In addition a number of density values for concrete are obtained [30]

#### 3.2.3 Test specimen

Field samples have to be wrapped in plastic and tested shortly after in the laboratory to avoid loss of moisture. Both field and laboratory concrete samples should have a maturity of no less than 28 M-days when starting the test to reduce the impact of further hydration during testing. It is important that the samples do not have loose aggregates or visible cracks.

The internal procedure used at SINTEF [31] is based on a large number of field tests, and recommends the following limitations to the dimensions of the sample: the diameter of the sample should not be larger than 50 mm or less than 10 mm, each sample should not have a volume smaller than 50 cm<sup>3</sup>, and the total volume of samples should not be less than 200 cm<sup>3</sup>.

In the thesis of Relling RH [13] DCS and PF-measurements were performed on field samples. The samples were dry sawn (slits of 40-50 mm deep, 150-200 mm long, and 20-30 mm apart). The pieces were chiseled off and wrapped immediately with plastic.

Lindgård J [20] used prisms (70×70×200 mm) wrapped and sealed in polyethylene foil to avoid moisture loss. The samples were stored at 5°C. The day prior to the measurement the sample were left in the laboratory to equilibrate to ambient temperature. The prisms were unwrapped and test samples with a thickness of about 40 mm were split from the prisms by

using a splitter. The height at which the samples were taken was noted. Before starting the test each sample was brushed with a steel brush to remove loose particles.

The pore protection ratio test involves the determination of the air/macro- and total porosities, and is used to assess concrete frost resistance. The determination of these porosities for the pore protection ratio bear some similarity with the DCS procedure. The procedure for the pore protection ratio is therefore also discussed here.

For determination of the pore protection ratio as described in the HETEK reports 93 and 97 [32, 33], a concrete core is split into discs with a thickness of approximately 20-30 mm. The number of discs required to assess the pore protection of a concrete, should be no less than 6 [33]. The test specimens described in the HETEK report 86 [34] are 5 pieces of 70×10×10 mm and one of 70×20×20 mm.

Geiker MR and Laugesen P [35] determined moisture profiles by subdividing the samples in 10 mm thick slices (see Figure 13).



**Figure 13: Subdivision of prism in 10 mm thick slices for determination of moisture profiles [35].**

### 3.2.4 Comparison of procedures

In general, all of the steps or a selection of them given in Table 4 are followed by the different methods. The differences between the methods described below are whether they include all the steps and the duration of the steps; see Table 5.

**Table 4: The different steps which can be taken to determine the degree of capillary saturation, air void content and density of concrete samples**

Step	Weight	Activity
1	$W_1$	weighing sample upon arrival
2	$W_2$	weighing after capillary water absorption (submerged or with one surface in contact with water)
3	$W_3$	weighing after oven drying at 105°C <sup>2</sup>
4	$W_4$	weighing after capillary water absorption (submerged or with one surface in contact with water)
	$W_4'$	weight under water. The volume is then given by $V = W_4 - W_4'$
5	$W_5$	weighing immediately after the air voids are filled using a pressure or vacuum <sup>3</sup>
6	$W_6$	weighing after oven drying at 105°C <sup>2</sup>

Based on the masses given in Table 4 a variety of properties of the concrete samples can be determined for example:

In-situ moisture content, weight%	$(W_1 - W_3) / W_3$
Capillary and gel porosity (=suction porosity)	$(W_2 - W_3) / V$ or $(W_4 - W_6) / V$

<sup>2</sup> It is a matter of discussion if an initial lower temperature, e.g. 50°C, should be used as drying at 105°C destroys part of the ettringite and C-S-H

<sup>3</sup> It is a matter of discussion if vacuum saturation is sufficient to fill air voids

Air voids or macro porosity	$(W_5 - W_2)/V$
Degree of capillary saturation in situ, DCS	$(W_1 - W_3)/(W_2 - W_3)$
Degree of vacuum or pressure saturation, DVS or DPS	$(W_1 - W_3)/(W_5 - W_3)$

In the thesis of Relling RH [13] all the steps given in Table 4 are followed. The capillary absorption by submersion (step 2 and 4) lasts for 1 week, the drying steps (step 3 and 6) last for 4 days and the samples are pressure saturated for 2 days at 5 MPa. Relling RH also demonstrated that drying at 105°C reduces the measured suction porosity with approx. 0.5% compared to the suction porosity measured on the sample as received [13]. This is a general conclusion, and it implies that DCS must be based on the first suction data (step 2).

The SINTEF method [31] and the method described by Sellevold and Farstad [30] are intended to characterize w/c ratio, porosity and density of a concrete. To obtain information on moisture state (DCS) for field samples, step 1, 2 and 6 must be added [14, 15]. Step 3 should last until the weight loss is less than 0.01%/hour or 7 days. Step 4 consists of 4 days with capillary suction (the sample is put 1-2 mm put in water) and subsequent 3 days submerged. The one-sided suction is done to characterize the suction rate as a measure of permeability, however this is not relevant in the present context.

Lindgård J [20] used a similar method as Relling RH except for step 3 where the samples are left to dry for 1 week at 105°C.

The method suggested by the HETEK reports 93 and 97 [32, 33] does not include step 2 and 3. The capillary saturation lasts until constant weight is reached (approx. 400 hours). It should be noted that true constant weight is never reached, especially in the presence of air voids as the air dissolves and is transported very slowly in the pore water. Pressure saturation is performed at 15 MPa for 24 hours, and the drying step (step 6) lasts until constant weight is reached (approx. 24 hours).

The method suggested in the HETEK report 86 [34] and by Geiker MR and Laugesen P in [35] is slightly different from the HETEK report 93 and 97 as the capillary suction time is reduced to 24 hours and vacuum saturation is measured instead of pressure saturation. For all HETEK procedures and the method suggested by Geiker MR and Laugesen P, boiled tap water cooled down to 20°C is used in the absorption test to eliminate the impact of air present in the tap water.

It should be noted that if immersed concrete is tested some of the air voids may be filled with moisture in-situ. This can lead to an overestimation of the suction porosity.

It should also be noted that pressure exerted on dense concrete might not be able to fill the air voids without pre-drying the concrete at 105°C [37]. Drying at 105°C renders the fine pore system coarser and makes the air voids more accessible but does not affect the volume of air voids.

### 3.2.5 Evaluation of results

The accuracy of these measurements depends on the weighing accuracy (balance with 0.01 g precision is recommended for typical samples), the sample size and the time provided for the samples to equilibrate at the different conditions.

### 3.2.6 Advantages

These methods do not require advanced laboratory equipment.



### **3.2.7 Disadvantages**

The measurements cannot be performed out in the field. The samples need to be transferred to a laboratory to be tested relatively soon after extraction.

Measured moisture contents are strongly dependent on the paste content of the concrete and hence the sample size. The degree of saturation, on the other hand, is a “normalized” property as it gives the moisture content as a fraction of the pore space.

To obtain full saturation after pressure pre-drying is required, particularly for dense concretes.

### **3.2.8 Duration**

The duration of the different test methods was determined based on the description given above is given in Table 5.

### **3.2.9 Standard methods and guidelines**

Certain research institutes have their own procedures for this kind of testing.

- SINTEF, Norway, has an internal procedure for the determination of the capillary suction rate and the air void content [31].
- EMPA, Switzerland, has similar internal procedures for Schnellporosität (1989) and Kristischer Sättigungsgrad(1989).

This test method is also described in detail by Fagerlund G in a tentative recommendation for frost resistance testing [36].

### 3.3 Comparison of the methods

Table 5: The sample size, weighing steps and duration of the described methods for measurement of the degree of saturation.

Test method	Sample size	Step 1: upon arrival	Step 2: capillary suction / absorption	Step 3: drying at 105°C	Step 4a: capillary suction / absorption	Step 4b: vacuum saturation	Step 5: pressure saturation	Step 6: drying at 105°C	Test duration	Results					Ref.
										Profiles	Moisture content	DCS	DYS	Air voids	
<b>SINTEF</b>	10 <math>\phi</math> < 50 mm $V_{\text{sample}} > 50 \text{ cm}^3$ $V_{\text{tot, sample}} > 200 \text{ cm}^3$	No	No	to constant weight (<math>< 0.01\%/h</math>) or 7 days	4 days capillary suction + 3 days submerged		2 days at 5 MPa pressure	4 days	20 days			X		X	[31]
<b>Relling RH</b>	20×40×150 mm	Yes	1 week submerged	4 days	1 week submerged		2 days at 5 MPa pressure	4 days	24 days		X	X		X	[13]
<b>Lindgård J</b>	40×100×100 mm	Yes	1 week submerged	1 week	1 week submerged		2 days at 5 MPa pressure	4 days	27 days		X	X		X	[20]
<b>Sellevoid and Farstad</b>	disk of core with height 20-40 mm	No	No	to constant weight	4 days submerged		overnight at 5MPa	No	9 days (?)			X		X	[30]
<b>HETEK No.93/97</b>	disk of core with height 20-30 mm	Yes	No	No	to constant weight (approx. 400 h)		24 h at 15 MPa pressure	24h	19 days		X	X		X	[32] [33]
<b>HETEK No. 86</b>	70×10×10 mm and 70×20×20 mm	Yes	No	No	24 hours	24 h at 20 bar		24h	3 days		X	X	X		[34]
<b>Geiker and Laugesen</b>	slice of prism with height min 10 mm	Yes	No	No	24 hours	24 h at 20 bar		to constant weight (~ 24 h)	3 days	X	X	X	X	X	[35]

## 4 Recommendations for moisture measurements

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The following procedures are based on the information given in this report and the discussions held during the workshop.

### 4.1 Field samples

#### 4.1.1 Sample extraction in the field

Equipment needed:

- core drilling equipment
- drying cloth
- plastic to wrap the cores
- tape to seal the wrapping of the cores
- permanent marker to mark the samples
- closed boxes to put the wrapped samples in for transportation
- form to note all extracted cores

Procedure:

1. Sample extraction from an existing structure can be performed by for example core drilling with water cooling. The diameter of the core should be at least 100 mm. The following steps, 2-4, should be performed each time after drilling a core. One should not drill several cores and perform each step for a series of cores together.
2. After the drilling the core has to be cleaned and dried immediately with a moist cloth.
3. The core has to be named using a permanent marker.
4. Immediately after marking the core has to be sealed with several layers of plastic tightened with tape to prevent moisture loss.
5. The cores should be transported to the laboratory the same day as they were extracted. The cores should preferably be stored sealed at conditions similar to the original exposure conditions. They should not be put in a cold room with a temperature considerably lower than the temperature of the examined structure, to avoid condensation which will lead to moisture loss and erroneous results.

Reporting:

- Date of extraction
- Weather conditions during core extraction.
- Amount and the name of samples extracted.
- Description where the sample were taken.
- Temporary storage time and conditions before transport.
- Transport time and conditions of the samples.
- Arrival date at the laboratory.
- If deviations of the above-named procedure have occurred, they should be noted.

#### 4.1.2 RH measurements in the laboratory on field samples

Measuring RH in the field is quite problematic due to temperature variations during measuring, differences in temperature between the sensor and the concrete, challenges to tighten the space between the sensor tube and the concrete, long equilibration time etc. Therefore, extraction of samples in the field and measuring in the laboratory is recommended. All activities in the laboratory should be performed in an acclimatized room to limit the influence of temperature variations on the RH measurements. If data for the temperature of the concrete at the site is available the RH measured at 20°C in the laboratory could be back calculated to the RH in the existing structure in the field.

##### Equipment needed:

- 20°C conditioning room
- RH sensors preferably as many as the number of samples that need to be analysed.
- data logging system for the RH sensors
- newly prepared saturated salt solutions with RH in the estimated RH range of the samples for calibration of the sensors
- core splitter to split the core in four
- chisel to chisel off concrete pieces from the centre of the core
- hammer to crush the concrete pieces to a diameter of 2-5 mm and to remove large aggregates
- plastic bag to keep the samples in while crushing with the hammer to limit the contact with air
- glass test tubes with a diameter which accommodates the RH sensor (e.g. larger than 12 mm for a Viasala RH sensor)
- putty to seal the tube – sensor connection
- plugs for glass tubes with samples that cannot be immediately measured due to lack of sensors
- form to note all tested samples

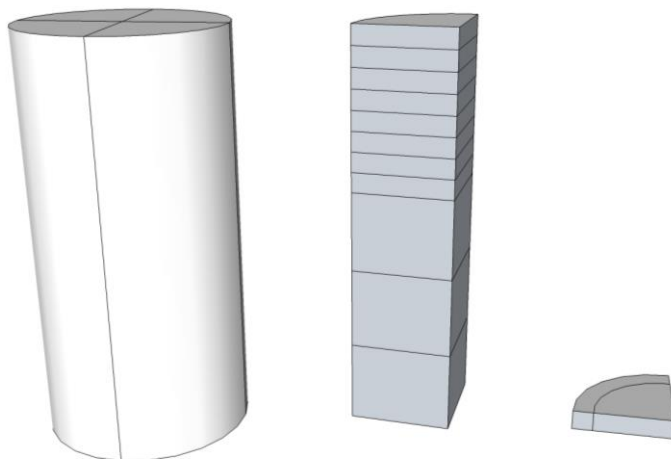
##### Procedure:

1. The RH sensors should be calibrated before each measurement by testing known saturated salt solutions with RH values in the range of interest (e.g. KCl 85.1%RH and KNO<sub>3</sub> 94.6%RH) at 20°C.
2. The sealed cores should be tested within 1-2 days after extraction to avoid moisture redistribution. The cores should be stored at the testing temperature prior to testing (e.g. 20°C). The handling of the samples e.g. transport, arrival date and storage time should be reported in detail.
3. The cylindrical core should be cracked twice along its axes so that a quarter of the cylinder is obtained (see Figure 14 ) using a core splitter.
4. Two quarters of the core will be used for RH measurements. The quarters are split into 10 mm thick sections inwards from the exposed surface.
5. The inner part of the quarter pieces is chiselled off and used for the measurement. (The outer part might be affected by the cooling water while drilling.)
6. The inner part of each section is put into a plastic bag and labelled from which cylinder and at which depth it is taken from. There should be two duplicates of each section, one of each quarter of the cylinder.
7. These sample should be crushed with a hammer in the closed plastic bag to pieces with a diameter of 2-5 mm. Large aggregate particles should be taken out.

8. The concrete pieces are filled into a glass test tube. The glass tube has to be labelled according to the concrete core the pieces originate off and the depth in the core they are taken of. The excess air in the glass tubes should be limited by filling the tube until a 10 mm gap is left between the material and the sensor. If there is not sufficient concrete powder available, the tube can partly be filled with glass beads.
9. An RH sensor (e.g. Viasala) is put in the top of the tube and the whole system is sealed with putty (see Figure 11). If there are not enough RH sensors available at the time of sample extraction, the glass tubes should be sealed and kept in the acclimatized room until an RH sensor becomes available again.
10. The system should be left for equilibration at a 20°C acclimatized room and the RH should be logged during 4 days. If a steady value is obtained (e.g.  $\Delta RH \pm 0.5$ ) the RH sensor can be removed.
11. After completing the measurement, the RH sensor should measure two known salt solutions in the range of the measured RH value to check whether the sensor has not drifted.

Reporting:

- The calibration results of the RH sensors before the measurement.
- Date of arrival of the cores in the laboratory, time of storage (1 or 2 days), storage conditions (temperature and RH).
- RH testing conditions (temperature and RH).
- The different steps of how the samples are extracted from the core (splitting, chiselling, crushing, size of resulting concrete powder).
- List of names of samples, from which core they are taken as well as the depth.
- The RH values measured on the samples during 4-6 days until a stable RH value is reached. For each sample 2 duplicates are measured.
- Report the calibration results of the RH sensors over known saturated salt solutions with RH values in the range of interest after the measurement.
- If deviations of the above-named procedure have occurred, they should be noted.



**Figure 14: Extraction of the samples from the core for RH measurements**

### 4.1.3 Moisture content and degree of capillary and pressure saturation measurements in the laboratory

These measurements need to be done in the laboratory. Hence samples are extracted in the field and transported to the laboratory for testing.

Equipment needed:

- core splitter to split the core in slices
- steel brush
- permanent marker to mark the samples
- weight which can weigh samples in air and submerged
- plastic boxes filled with water
- oven 105°C<sup>4</sup>
- desiccator with silica gel
- pressure tank going up to 5 MPa
- form to note all tested samples

Procedure:

1. The sealed cores should be tested within 1-2 days after extraction to avoid moisture redistribution. The cores should be stored at the testing temperature prior to testing (e.g. 20°C). The handling of the samples e.g. transport, arrival date and storage time should be reported in detail.
2. The cylindrical core should be cracked twice along its axes so that a quarter of the cylinder is obtained (see Figure 14) using a core splitter.
3. Two quarters of the core will be used for the measurement of the moisture content and degree of capillary saturation. The quarters are split into 10 mm thick sections inwards from the exposed surface.
4. The quarters of the cylinder are split into a series of 1-2 cm thick disks if one wants to obtain profile [35] or in 4 cm thick disk if one is interested in a value for the bulk concrete [31]. For the moisture content measurements on concrete the sample size needs to be large enough or several samples need to be taken as the measurement is sensitive to the paste content of the sample.
5. The extracted samples are brushed with a steel brush to remove loose particles. Two duplicates, one of each quarters of the cylinder, should be made of each sample.
6. All samples are marked with permanent marker.
7. The samples are weighed ( $W_1$ ).
8. The samples are put into a water bath for 1 week in water after which they are weighed in air ( $W_2$ ).
9. The samples are subsequently put to dry at 105°C<sup>4</sup> until constant weight ( $W_3$ ), hourly difference <0.01% [31], by monitoring the weight twice daily after the first day for approx. 4-7 days. Prior to weighing the hot samples they should be cooled to room temperature in a desiccator with silica gel. The time needed for the cooling depends on the size and amount of samples.
10. The samples are put into a water bath for 1 week in water after which they are weighed in air ( $W_4$ ) and submerged for volume determination ( $W_4'$ ).
11. The samples are then put for 2 days at 5 MPa in a pressure tank and are weighed immediately after removing the pressure to prevent the loss of water ( $W_5$ ).
12. Finally the samples are dried for 7 days at 105°C and weighed ( $W_6$ ).

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<sup>4</sup> An initial lower temperature could be selected to limit decomposition of ettringite and C-S-H.

Reporting:

- Date of arrival of the cores in the laboratory, time of storage (1 or 2 days), storage conditions (temperature and RH).
- Moisture content and degree of capillary saturation testing conditions (temperature and RH).
- The different steps of how the samples are extracted from the core (splitting and brushing).
- List of names of samples, from which core they are taken as well as the depth.
- The weights of all of the samples at the different steps of the procedure ( $W_1$ ,  $W_2$ ,  $W_3$ ,  $W_4$ ,  $W_4'$ ,  $W_5$  and  $W_6$ ).
- Calculation of :
  - o Moisture content  $(W_1 - W_3) / W_3$
  - o Suction porosity = Gel + Capillary porosity  $(W_2 - W_3) / V$
  - o Air voids  $(W_5 - W_2) / V$
  - o Degree of capillary saturation (DCS)  $(W_1 - W_3) / (W_2 - W_3)$
  - o Degree of pressure saturation (DPS)  $(W_1 - W_3) / (W_5 - W_3)$
- If deviations of the above-named procedure have occurred they should be noted.

## 4.2 Laboratory samples

### 4.2.1 Relative humidity

For laboratory samples, it is recommended to measure RH on crushed samples rather than on cast in samples. The time needed to reach RH equilibrium is much shorter for crushed samples than for cast-in sensors. A short equilibrium time is important as sensors can start to drift.

The same recommendations regarding the measuring procedure are valid as for the field samples.

### 4.2.2 Moisture content and degree of capillary saturation

The samples (cores or cubes) are split into the required size (e.g. 1 cm thick for profiling and max 4 cm for bulk). As mentioned for the field samples care has to be taken regarding the sample size when determining the moisture content.

The same procedure as for the field samples is recommended.

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**Appendix 1****COIN WORKSHOP MOISTURE IN CONCRETE**6<sup>th</sup>-7<sup>th</sup> February 2012, NTNU, Trondheim**List of participants**

Jan-Erik	Jonasson	Luleå University	Sweden
Peter	Fjellström	Luleå University	Sweden
Lars-Olof	Nilsson	LTH	Sweden
Kurt Kielsgaard	Hansen	DTU	Denmark
Alexander	Michel	NTNU	Norway
Anja	Klausen	NTNU	Norway
Claus	Larsen	SVV	Norway
Erik	Sellevoid	NTNU	Norway
Gunrid	Kjellmark	Sintef	Norway
Jan	Lindgård	Sintef	Norway
Jan-Arve	Øverli	NTNU	Norway
Klaartje	De Weerd	Sintef/NTNU	Norway
Knut	Kjellsen	NORCEM	Norway
Marius	Kvalvik	Sintef	Norway
Mette	Geiker	NTNU	Norway
Stefan	Jacobsen	NTNU	Norway
Terje	Kanstad	NTNU	Norway
Øyvind	Bjøntegaard	SVV	Norway

**Appendix 2**

## COIN WORKSHOP MOISTURE IN CONCRETE

6<sup>th</sup>-7<sup>th</sup> February 2012, NTNU, Trondheim**Programme**

12:30	<b>Welcome and refreshment</b> , Mette Geiker	
13:00	Moisture in concrete, theoretical background and overview of measuring methods <ul style="list-style-type: none"> <li>• Lars-Olof Nilsson</li> </ul>	
	Measurement of relative humidity, laboratory <ul style="list-style-type: none"> <li>• Peter Johansson</li> <li>• Kurt Kielsgaard Hansen</li> <li>• Gunrid Kjellmark</li> <li>• Jan Lindgård</li> </ul>	
	Measurement of moisture content and degree of saturation in concrete, laboratory, traditional methods <ul style="list-style-type: none"> <li>• Jan Lindgård</li> <li>• Mette Geiker</li> </ul>	
	Discussion	
15:30	<b>Break</b>	
16-18	Measurement of moisture in concrete, in-situ Moderator: Mette Geiker	CrackFree og COIN DP3.1, Moderator: Knut Ose Kjellsen
19:00	<b>Dinner</b>	

08:15	Modelling of early age moisture state and transport in concrete <ul style="list-style-type: none"> <li>• Lars-Olof Nilsson</li> </ul>	
	Temperature induced moisture transport <ul style="list-style-type: none"> <li>• Stefan Jacobsen</li> </ul>	
	Moisture induced volume changes, autogeneous conditions <ul style="list-style-type: none"> <li>• Øyvind Bjøntegaard</li> </ul>	
	Drying induced volume changes, drying conditions <ul style="list-style-type: none"> <li>• Jonasson Jan-Erik</li> </ul>	
	Measurement of autogeneous deformation at isothermal conditions, comparison of methods <ul style="list-style-type: none"> <li>• Gunrid Kjellmark</li> </ul>	
	Discussion	
11:45	<b>Lunch</b>	
12:45	Measurement of moisture content, TDR <ul style="list-style-type: none"> <li>• Alexander Michel</li> <li>• Kurt Kielsgaard Hansen</li> </ul>	
13:15	Summary discussion, moisture measurements; Mette Geiker	
13:45	<b>Closure</b>	