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# **ALKALI -SILICA REACTION (ASR) – PERFORMANCE TESTING: INFLUENCE OF SPECIMEN PRE-TREATMENT, EXPOSURE CONDITIONS AND PRISM SIZE ON CONCRETE POROSITY, MOISTURE STATE AND TRANSPORT PROPERTIES**

Jan Lindgård<sup>1\*</sup>, Erik J. Sellevold<sup>2</sup>, Michael D.A. Thomas<sup>3</sup>, Bård Pedersen<sup>4</sup>, Harald Justnes<sup>2</sup> and Terje F. Rønning<sup>5</sup>

<sup>1</sup> SINTEF Building and Infrastructure, NO-7465 TRONDHEIM, Norway

<sup>2</sup> Norwegian University of Science and Technology, NO-7491 TRONDHEIM, Norway

<sup>3</sup> University of New Brunswick, FREDERICTON, Canada

<sup>4</sup> Norwegian Public Roads Administration, NO-0033 OSLO, Norway

<sup>5</sup> Norcem Heidelberg Cement, NO-3950 BREVIK, Norway

## **Abstract**

Whether or not concrete prism tests (CPTs) developed for assessment of alkali-silica reactivity of aggregates might be suitable for general ASR performance testing of concrete has been evaluated. This paper presents the background for the choice of test procedures and results on how variations in specimen pre-treatment, ASR exposure conditions and prism size influence concrete porosity, moisture state and transport properties. Results from measurements of alkali leaching and prism expansions during the ASR exposure are presented in a separate paper, together with discussion of consequences for ASR test procedures.

For ordinary Portland cements and with water-to-cementitious-materials ratio (w/cm) 0.45 and higher it was found that the internal moisture state is sufficiently high in all the assessed procedures to produce ASR expansion. However, for less permeable concretes lack of internal moisture and lower rate of diffusion can significantly reduce the rate and extent of ASR expansion during laboratory performance testing.

**Keywords:** Alkali-Aggregate Reaction, performance testing, moisture, porosity, permeability.

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Correspondence to: [jan.lindgard@sintef.no](mailto:jan.lindgard@sintef.no), phone +47 93 05 86 89

# 1 INTRODUCTION

## 1.1 Background

Since ASR was recognised as a concrete durability problem more than 70 years ago by Stanton [1], several comprehensive research projects have focused on test methods for determining the alkali reactivity of aggregates and corresponding acceptance criteria. In Europe, only national ASR aggregate tests are available today. As part of the international harmonisation of such aggregate tests, two previous RILEM technical committees (TC 106-AAR, 1998–2000 and TC 191-ARP, 2001–2006) have proposed and validated several RILEM aggregate test methods for classifying the alkali reactivity of aggregates: petrographic method (AAR-1, 2003 [2]), accelerated mortar bar tests (AAR-2, 2000 [3] and AAR-5, 2005 [4]) and concrete prism tests, CPTs (AAR-3, 2000 [3] and AAR-4.1, 2006 [5]), in addition to recommendations for how to use these test methods and interpret the results (RILEM AAR-0, 2003 [6]). These draft RILEM methods have been developed further by RILEM TC 219-ACS (2007–2012), partly based on findings in the EU funded "PARTNER" research project where all the RILEM aggregate test methods were evaluated [7]. In USA and Canada, corresponding ASTM and CSA test methods exist (ASTM C 1260-07 [8]; ASTM C1293-08b [9]; ASTM C 295-08 [10]; CSA A23.2-14A-04 [11]; CSA A23.2-25A-09 [12]; CSA A23.2-15A [13]).

Internationally, various ways of controlling ASR are suggested (in addition to use of non-reactive aggregates): utilization of low-alkali cement, limiting the alkali content of the concrete, incorporation of supplementary cementing materials (SCMs; e.g. silica fume, fly ash, ground granulated blast furnace slag (ggbfs), metakaolin and other pozzolans) or use of lithium salts. For example RILEM TC 219-ACS has prepared a document with recommendations on how to ensure durable non-reactive concrete (RILEM AAR-7.1, 2008 [14]). This document is scheduled for publication together with the RILEM aggregate test methods in a special issue of *Materials and Structures* during 2012.

SCMs control expansion due to ASR by binding alkalis and limiting their availability for reaction with alkali–silica reactive aggregates [15]. The efficiency of the SCMs depends on their characteristics and amount, the nature of the reactive aggregate and the availability of alkali in the concrete. Chappex and Scrivener [16] also showed that the aluminium present in certain SCMs (e.g. metakaolin) may limit the

dissolution of silica from reactive aggregates. Consequently, to be able to utilise alkali–silica reactive aggregates for production of durable concretes, the effects of various measures must be correctly identified by accelerated laboratory performance tests (or ideally by relevant long-term field experience). Several performance tests have been used worldwide for at least 15 years. In principle two groups of ASR performance test methods exist, one using mortar bars and the other using concrete prisms. However, the test conditions (e.g. pre-curing, temperature, alkali content, humidity) differ from one test method to another. Thus, the results and conclusions from different test methods may vary widely.

The most frequently used concrete performance test is ASTM C1293-08b [9], where concrete prisms are stored at high humidity over water in sealed containers at 38°C. Other examples of concrete performance tests are the French 60°C CPT [17] and the Norwegian 38°C CPT [18].

## **1.2 Development of reliable ASR performance test methods**

### *1.2.1. Main challenges*

In 2006, Thomas et al. [19] provided a critical evaluation of different ASR performance test methods. The authors concluded that none of the currently available or commonly used test methods meet all the criteria for an ideal performance test. For example, the main shortcoming of the Canadian 38°C CPT [11] (similar to ASTM C-1293-08b [9]) is the duration of the test (2 years) and that addition of alkalis is required to compensate for alkali leaching effects, i.e. the fact that alkalis are leached out of the prisms during exposure in the humid environment. Thus, the authors concluded that the method neither can be used to determine the “critical” alkali content for an alkali–reactive aggregate, nor to determine how the level of a SCM required to control expansion varies with the concrete alkali content.

In 2010, Lindgård et al. [20] assessed about 15 years of experience with use of the Norwegian 38°C CPT [18]. This method is similar to ASTM C 1293-08b [9], but larger prisms are used (100 mm cross section compared with 75 mm in the ASTM method). The method has been specified in the Norwegian guidelines [21] for performance testing of concrete mixes and/or binders since 1996.

Despite the long testing time required (1-2 years), the Norwegian system for performance testing has proven to be an advantageous and flexible tool to document critical alkali limits for binders and aggregates. However, even though the extent of alkali leaching is less for the Norwegian CPT with larger prisms than used in the ASTM C-1293 CPT, Lindgård et al. [20] recommended that the influence of alkali leaching on the measured expansions in the Norwegian CPT should be investigated further.

The development of accurate and reliable performance test methods for the production of durable concretes is a challenge. Several requirements must be fulfilled, some being somewhat contradictory. On the one hand the test methods should be inexpensive and rapid, calling for extremely accelerated test conditions. On the other hand a performance test should mirror the field performance of the actual concrete for more than 50 years lifetime. Another important requirement is the possibility to test job mixes identical to the concrete composition that will be used on actual projects. Use of mortar bars is in conflict with this latter requirement. According to Thomas et al. [19], other important requirements for an ideal performance test for ASR are:

- The test should be capable of determining the “critical” alkali content for specific aggregates, i.e. the alkali leaching problem must be solved.
- The test should be capable of assessing all types of SCMs, lithium compounds and combinations of SCM and lithium, with cements of different alkali levels.

### *1.2.2. RILEM TC 219-ACS*

Today, research is on-going in several countries with the aim to improve current ASR performance test methods and develop alternative tests. As part of the international harmonisation of ASR performance test methods, the "Performance testing" task group of RILEM TC 219-ACS is working on a performance testing concept aiming to develop one or more reliable ASR concrete performance test methods that might cover several applications/areas, ranging from combination of various aggregates with a standard CEM I binder up to the "ultimate goal" to document the alkali reactivity of any concrete mix design ("job mix").

### 1.3 PhD study on ASR

The main objective of the PhD study by Jan Lindgård, being part of the Norwegian COIN program (2007-2014, [www.coinweb.no](http://www.coinweb.no)), has been to evaluate whether concrete prism tests developed for assessment of alkali-silica reactivity of aggregates might be suitable for general ASR performance testing of concrete. This paper is one of several from the PhD study, which has been performed in close co-operation with the "Performance testing" task group of RILEM TC 219-ACS (all authors of this paper, except one, are members of this RILEM task group).

#### *1.3.1. Literature review – assessment of parameters influencing laboratory performance testing*

As stated by Thomas et al. [19], the only suitable benchmarking of a laboratory performance test is against real concrete structures or, as a surrogate, against large concrete blocks exposed outdoors to natural weathering conditions. However, long-term field experience is available only for a limited number of commercial SCMs, e.g. some class F fly ashes and some slag cements. When developing an accelerated performance test method, it is thus crucial as a first step to evaluate fundamental questions theoretically in order to ensure a satisfactory laboratory/field correlation.

As a collaborating work between the PhD study and the work within the task group "Performance testing" in RILEM TC 219-ACS, a comprehensive literature review has recently been performed. In total, 12 authors contributed to the report that included about 250 references [22]. The main objective was to assess how various parameters might influence the laboratory/field correlation with respect to ASR performance testing, either directly or indirectly. The most important findings in the literature survey and recommendations for performance testing have recently been summarised by Lindgård et al. [23]. These recommendations include precautions when testing various aggregates and binders, important factors to take into account during mix design, as well as possible influences on ASR expansion of various conditions during pre-storage and the ASR exposure. Additionally, the literature survey has identified several issues that need further research in order to develop a reliable performance test procedure.

### *1.3.2. Parameters focused on in the PhD study*

Based on the most important findings from the literature review ([22], [23]), the PhD study has focused on the effect of various specimen "pre-treatments", "ASR exposure conditions" as well as prism size on:

- Porosity and internal moisture state of the concrete prisms.
- Concrete transport properties (with respect to mobility of water and ions).
- Alkali leaching (rate and extent) from the concrete prisms during the ASR exposure.
- Concrete prism expansion (rate and final expansion).

Additionally, the effect of water-to-cementitious-materials ratio (w/cm) and type of binder is assessed.

The specimen "pre-treatment", defined as the moisture conditions during pre-storage and the length of the pre-storage period at ambient temperature (up to the point of the initial (zero) length comparator reading), vary for different concrete prism tests used in the different countries. After casting, most test methods prescribe storage of the moulds at 18–23°C and minimum 90–95 % RH in the surroundings, while others prescribe more humid storage of the moulds, e.g. in a fog room with 100 % RH. After de-moulding the day after casting, some test methods prescribe direct exposure of the prisms to the actual exposure temperature, e.g. ASTM C-1293-08b [9]. Other methods prescribe 0.5 h submersion of the prisms in water after de-moulding, before further preparation for final exposure. Finally, the length of the pre-storage period at 18–23°C normally varies from 1 day (e.g. as in ASTM C-1293-08b [9] and RILEM AAR-4.1 [5]) to 7 days (e.g. as in RILEM AAR-3 [3]). Some laboratories use an even more prolonged pre-storage period, up to 28 days, dependent on the type of binder [24].

Also the "ASR exposure conditions" (i.e. moisture conditions, type of container, use of any wrapping, exposure temperature, length of the storage period, addition of any external alkalis) and prism size varies between various performance test methods used in the different countries.

In the PhD study, an extensive laboratory program has been performed. The test series cover the variations in test conditions in the most commonly used ASR test methods. Additionally, some test

series include measures intended to reduce the extent of alkali leaching. This paper presents the technical background for the choice of test procedures and gives an overview of the laboratory programme (see Section 2), that in total has included 58 ASR test series (see Section 2.3) and comprehensive complementary testing for documentation (see Section 2.4). Furthermore, the paper presents and evaluates the results from measurements of concrete porosity, internal moisture state and transport properties. As a basis for this evaluation, some important findings in the literature review [23] regarding concrete internal moisture state and concrete transport properties, in particular with respect to influence on development of ASR, are briefly discussed in sections 1.4 and 1.5, respectively.

In a separate paper [25], results from the periodic measurements of alkali leaching from the concrete prisms, concrete prism expansion and dynamic E-modulus are presented and evaluated, together with main findings from some of the supplementary tests (see Section 2.4). Additionally, consequences for ASR test procedures are discussed.

## **1.4 Importance of the internal moisture state**

### *1.4.1. Description of the moisture state in concrete*

The moisture conditions in concrete can be described in two different ways ([26], [27]):

1. Relative humidity (RH) at a certain temperature.
2. The pore water content expressed either as the percentage of mass or volume, or as the degree of saturation. The degree of capillary saturation (DCS) expresses the % filling of the pores in concrete that are able to draw in water by capillary action (i.e. gel and capillary pores; not air entrained pores or voids).

It is important to note that the RH is a measure of the thermodynamic state of the pore water, and is not a direct measure of the amount of water [28]. At a fixed moisture content, the RH is a function of the pore structure, the temperature, the chemical composition of the pore water and the moisture history of the concrete. Hedenblad [29] reported that the chemical effect on the pore water can be as much as 4 % reduction of the RH, i.e. a fully saturated concrete may only exert 96 % RH.

In general, the relation between the RH in concrete and the DCS varies a lot depending on several factors, where the w/cm is the most important one [27]. High w/cm concretes have a steeper desorption isotherm than low w/cm concretes [26]. In other words, for a given loss of moisture, RH decreases less in open concretes compared with denser concretes. Note also that the RH-DCS relation depends on whether a given moisture state is obtained during adsorption or desorption.

RH is easy to measure, also in situ and over time. However, such measurements require much care and experience to be meaningful. Many sources of errors exist, where temperature difference between the sensors and the concrete often is the cause of unreliable results. RH measurements in the field are notoriously uncertain.

Measurement of DCS on samples cut from structures is done by weighing the samples immediately after unwrapping in the laboratory, after immersion 7 days in water and after drying [30] (see Section 2.4). DCS measurements, in contradiction to RH measurements, are easy to perform accurately, but they are destructive and more cumbersome to do, involving cutting samples from the structure and taking them to a laboratory in an “undisturbed” condition. Thus, much care must be taken during sampling.

#### *1.4.2. The role of water in the alkali–silica reaction*

Moisture is generally accepted to be one of the main factors affecting ASR. Water is important as a transport medium for ions. The role of water is also important in the expansion stage. The overall expansion and cracking of concrete is basically caused by sorption of water by the alkali–silica gel, which in turn swells and thereby causes damage. In a review by Pedersen [31], very few discussions of the fundamental aspects of moisture state in concrete in connection with ASR were found in the literature, and most scientists seem to use relative humidity as the only measure of moisture. The critical limit for developing ASR is reported to lie in the range of 80–90 % RH depending on several factors, as discussed by Larive et al. [32]. In general, the 80 % RH limit has been most frequently used in the literature.

DCS is, however, a more relevant parameter to describe the in situ moisture state if it is the amount of water that is controlling the expansion. Based on a survey of a large number of Norwegian concrete bridges, Lindgård et al. [27] showed a rather good correlation between the presence of ASR and the DCS. With only a few exceptions DCS of the concrete structures with pronounced ASR was higher than 90 %. The extent of damages generally increased with increasing water content above this level.

The concrete moisture state might also influence the expansion properties of the alkali-silica gel ("ASR-gel"). Bokern [33] showed that the viscosity of the ASR gel decreased at higher RH levels and thus decreased the expansive pressure exerted.

#### *1.4.3. Consequences of self-desiccation*

The hydration process of cement gives a reduction in the volume of the reacted cement and water. This phenomenon is referred to as chemical shrinkage, and has some major effects:

- 1) In the hardening phase, the chemical shrinkage results in partly empty pores within the concrete. These pores will remain partly empty if no water is supplied from the surroundings. This leads to a lowering of the RH in the concrete, a phenomenon called self-desiccation. Generally, the extent of self-desiccation increases with decreasing w/cm. The type of binder, in particular the type and amount of any SCM used, will also influence the extent of self-desiccation.
- 2) The reduced RH implies (capillary) tension in the pore water. This tension produces autogenous shrinkage, which is a bulk volume contraction of the paste and consequently of the entire concrete body.

For practical purposes the effects of self-desiccation becomes more important as w/cm is reduced. For example at w/cm 0.40, RH may be less than 90 % after a few weeks. At lower w/cm the effect is larger and may reduce the RH to below 80 % over a period of time, provided there is no water supplied from the surroundings. Thus, for massive concrete structures the residual concrete mix water, depending on the w/cm, rather than ambient wetting and drying, determines the prevailing interior moisture content ([26], [30], [34], [35], [36]). The depth of the surface layer that is influenced

by external moisture fluctuations will normally be in the range of about 2-10 mm ([35], [37]). This depth will be less with decreasing w/cm as the result of reduced permeability [38].

Even during laboratory testing, a moisture profile through the prism cross-section often develops, with lowest RH in the mid part, in particular if the size of the concrete specimens is relatively large ( $\geq 100$  mm cross-section) combined with a rather low w/cm. Consequently, a minimum limit should be considered for the w/cm for ASR testing. If such limitations are not introduced, the internal RH in laboratory test prisms might be lower than in slim structures exposed to water in service. This could lead to incorrect test conclusions, i.e. some potentially alkali-reactive mixes could be classified as non-reactive based on performance testing because of the lack of water. For this reason, Lindgård et al. [23] suggested that the net influence of a reduced w/cm ( $< 0.40$ ) should be investigated further.

Additionally, the aggregate porosity and the aggregate moisture state at the time of mixing might significantly influence the RH within the concrete. For example might use of pre-wetted aggregates with relatively high porosity supply additional water to the cement paste during the curing period and thereby reducing the self-desiccation ([39], [40]).

### **1.5 Factors effecting transport properties**

Increasing w/cm will result in a higher and more continuous (capillary) porosity, consequently internal transport processes will be accelerated, the rate of alkali leaching will increase and water loss/uptake will take place more easily ([41], [42]).

Furthermore, addition of any SCMs will influence the permeability of the concrete and thus the permeability-related ASR mechanisms, i.e. internal transport processes, alkali leaching, water uptake and sensitivity to drying during exposure and measuring in the laboratory. Additionally, increased permeability in aggregates may enhance the alkali reactivity due to easier access to concrete pore fluids [43].

## 2 THE TEST PROGRAMME

### 2.1 General

The PhD laboratory test programme has included four concrete mixtures (see Section 2.2) and in total 58 ASR test series, most of them using modified versions of the draft RILEM aggregate concrete prism tests; AAR-3, 2000 (38°C, wrapped prisms) [3] and AAR-4.1, 2006 (60°C, unwrapped and wrapped procedure) [5] - see details in Section 2.3. For comparison, six test series with slightly modified versions of the Norwegian 38°C CPT [18] and 12 test series with the ASTM C1293-08b CPT [9] (38°C, unmodified version) were included. The main reason for incorporating the latter method was to establish a link to the comprehensive experience in North America with this method and to document any batch to batch variation; 8 concrete batches were needed to cast all the concrete prisms with the "basis" binder and two batches were prepared with the "open" binder (see Section 2.2).

In addition to the ASR testing, a comprehensive complementary testing program for documentation of concrete properties of importance for development of ASR has been performed. The main parameters include internal moisture state, transport properties (of water and ions) and alkali leaching (see Section 2.4). Additionally, fresh concrete properties (slump, density and air content), 28 days compressive strength of 100 mm cubes and porosity/density properties were measured.

Before the laboratory testing started, a "pilot testing" program was carried out in order to develop detailed laboratory procedures to improve the reliability of the measurements.

The selected components and the concrete mix design used, including the technical background for the choices made, are described in Section 2.2.

### 2.2 Materials and mixture proportions

Two CEM I Portland cements (EN-197-1), one high alkali (1.24 % Na<sub>2</sub>O<sub>eq</sub>) and one low alkali (0.60 % Na<sub>2</sub>O<sub>eq</sub>), and a CEM II/A-V cement containing 21.6 % of a siliceous fly ash (class F, co-grinded

with the clinker) were used in the study, see Table 1 (*comment: The CEM II/A-V cement normally contains 17-20 % fly ash*). This type of blended cement has been widely used for years in Norway, partly in order to avoid ASR in combination with alkali-silica reactive aggregates.

The aggregates used are defined in the Norwegian ASR regulations ([18], [21]) as “reference Norwegian aggregates” and consist of a non-reactive natural sand (mainly containing granites and gneisses, saturated surface-dry density 2660 kg/m<sup>3</sup>, water absorption 0.1 %) and an alkali-silica reactive crushed coarse aggregate (cataclasite with crypto- to microcrystalline quartz, saturated surface-dry density 2760 kg/m<sup>3</sup>, water absorption 0.4 %). In all the test series, the aggregates were blended to produce a 60:40 coarse:fine ratio (by mass). This is in agreement with the grading prescribed in the RILEM CPTs ([3], [5]).

Details of the concrete mixtures are given in Table 2. Based on a series of considerations, the bulk of the testing was produced using a mixture containing 400 kg/m<sup>3</sup> of Portland cement and water-to-cement ratio (w/c) of 0.45 (denoted "basis" binder). The two CEM I cements were blended to produce an alkali content of 3.7 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>eq</sub>. The alkali content was chosen (based on previous testing of the aggregates at SINTEF [20]) with aim to reach a final expansion of the reference test series lying on the steep part of the “expansion versus alkali level (S-shaped) curve”, so that a small loss of alkalis due to alkali leaching would be detectable in terms of reduced expansion. If a high alkali level had been chosen, most of the test series would probably show a rather high expansion (i.e. lie on the plateau of the “expansion versus alkali level curve”), even if significant quantities of alkalis were leached out during the ASR exposure. Then only minor differences in expansion would have been expected between the different test series.

To examine the impact of w/c, two additional concrete mixtures were cast with CEM I cement and w/c of 0.30 and 0.60 (denoted "dense" and "open" binder, respectively). The cement contents of these mixtures were modified to achieve the desired workability, but the alkali content of the mixtures was maintained at 3.7 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>eq</sub> by appropriate blending of the CEM I cements, see Table 2.

Additionally, one mixture was produced with w/cm of 0.45 using the blended cement containing about 20 % fly ash (denoted "fly ash" binder, see Table 2). The alkali content of this mixture was raised from 5.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>eq</sub> (alkalis originating from the blended cement) to 9.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>eq</sub> by adding NaOH to obtain a final expansion of the "fly ash" concrete mixture on the steep part of the "expansion versus alkali level curve".

To achieve an appropriate consistency, a minor quantity of a low-alkali superplasticizer was added to some of the concrete mixes. To avoid any influence on the ASR expansion of varying air content in the different test series, a minor quantity of a de-foaming agent was added to the concrete mix if the air content was measured to be higher than 3.0 %. The concrete samples were compacted manually.

## 2.3 ASR test procedures – test series

### 2.3.1. ASR concrete prism tests

All the CPTs included in the study (see Table 3) are designated for testing alkali-silica reactivity of concrete aggregates. Additionally, the ASTM C-1293 CPT [9] and the Norwegian CPT [18] are frequently used for ASR performance testing. The main differences between the CPTs are *exposure temperature* (38°C or 60°C; only RILEM AAR-4.1, 2006 [5] uses 60°C), *prism cross section* (100x100 mm (only the Norwegian CPT [18]) or 70-75x70-75 mm) and *use of any wrapping* (damp cotton cloth and polyethylene; only RILEM AAR-3, 2000 [3] and RILEM AAR-4.1 "Alternative", 2006 [5]). Except for the "Standard" RILEM AAR-4.1 CPT, 2006 (unwrapped prisms) [5] where small containers are stored inside a larger container ("reactor") over water, the other storage containers are stored in a hot, dry room or an oven (thus being more vulnerable for evaporation of the water in the bottom if the seal of the container is compromised).

*(Comment: Based on results from this study, the RILEM AAR-4.1 "Alternative" CPT (2006) [5] (wrapped prisms) is no longer considered suitable within the RILEM TC 219-ACS. Furthermore, the RILEM AAR-3 CPT 2000 [3] (wrapped prisms) was significantly revised during 2010. In the 2011 draft version of the AAR-3 CPT, the exposure conditions are similar to ASTM C-1293 [9], i.e. no wrapping is applied. The reason for the revisions made*

*was the disturbingly high extent of alkali leaching from the wrapped concrete prisms at early age - further discussed in [25] and [44]).*

### *2.3.2. Modification of the test procedures*

The standard versions of the concrete prism tests, except the ASTM C-1293 CPT [9], have been slightly modified in order to investigate the effects of these modifications. The motivation for the modifications is given in the recently published literature review [23].

During all the testing only deionised water has been used as mixing water, in the moist cotton cloth wrapping (if any) and in the storage containers.

For all test series, the moulds were stored at ambient temperature in the laboratory under plastic foil from casting until de-moulding the following day.

For all test series, each prism was always stored vertically in the storage container with the same prism end pointing upwards (marked with an arrow). This is in contrast to the description for the three RILEM CPTs ([3], [5]) and the ASTM C-1293 CPT [9] that prescribe that each prism should be turned at every reading. The reason for this modification was to be able to document any variation in internal moisture state, extent of alkali leaching and extent of internal cracking over the prism height.

For all standard versions of the CPTs the mass and length were taken after cooling the prisms for about 16 hours inside their storage container in a room kept at  $\sim 20^{\circ}\text{C}$  (see Table 3). However, during cooling some water will evaporate from the prisms and the extent of alkali leaching might increase [23]. As an example, "pilot tests" showed that each prism in the RILEM AAR-4.1 Standard CPT (unwrapped prisms) [5] lost 8-11 g water during cooling from 60 to  $20^{\circ}\text{C}$  inside the container. This mass loss constitutes about 5-6 % reduction in degree of capillary saturation for this concrete quality (CEM I, w/c 0.45). For this reason, all measurements in the modified versions of the various concrete prism tests were taken without pre-cooling the prisms (i.e. similar to what is the normal procedure in the accelerated mortar bar tests RILEM AAR-2 [3] and ASTM C-1260 [8]). To secure accurate

measurements, i.e. reduce the influence of any mass loss and temperature variations from reading to reading, a detailed measuring procedure was developed. The reference readings of the prisms measured without pre-cooling were taken the day after the prisms were exposed to their ASR exposure temperature (see Figure 1).

The following specimen "pre-treatment" and/or ASR exposure conditions have been varied when modifying the RILEM AAR-3 CPT (2000, [3]) and the RILEM AAR-4.1 CPT (2006, [5]) – see Figure 1 and Table 4 for details and motivation:

- The wrapping procedure (if any) was modified, either by adding only half of the water content prescribed or by removing the prescribed polyethylene bag.
- The length of the "pre-storage" period was varied. The prisms were kept at ambient temperature until 1, 7 or 28 days after casting before being exposed to the ASR exposure temperature. However, for all test series, the prisms were prepared for final exposure (e.g. wrapped) and put into the storage container immediately after de-moulding (and any 0.5 h submersion) and the initial measurements of mass and length.
- Some prisms were pre-cured 24h at elevated temperature (60°C) to simulate the curing temperature in a massive concrete structure.
- Some prisms were sealed to avoid any exchange of water with the environment.
- Some prisms were stored submerged in deionised water (to maximize the alkali leaching conditions).
- Some prisms were wrapped with cotton cloth saturated with a basic solution with pH 14.2 (1.5 M OH<sup>-</sup>) or 13.2 (0.15 M OH<sup>-</sup>), respectively (instead of the usual deionised water), in order to try to reduce the extent of alkali leaching. The lowest pH level corresponds to the typical pH found in water filtered from fresh cement paste with a high alkali Norwegian CEM I after half an hour. The highest pH level corresponds to the calculated pH level in the pore water in the concrete with the "basis" binder after about one month of curing when most of the water has been consumed by hydration and concentrated up the salt solution about ten times. *(Comment: Some may find it strange with a pH above 14, but the pH scale from 0 to 14 is just the common range corresponding to 1M H<sup>+</sup> and 1M OH<sup>-</sup>, respectively, and is not "limits").*

To be able to document the effect of raising the exposure temperature from 38°C to 60°C, similar pre-treatment variants were applied for both ASR exposure temperatures as far as possible.

Also the Norwegian CPT was slightly modified by increasing the length of the "pre-storage" period from 1 to 7 days.

During the "pre-storage" period and the ASR exposure all the prisms were stored in containers prepared as prescribed in the "standard" test procedures (see Table 3), but with the following exceptions: For the "Alternative" version of RILEM AAR-4.1 CPT (wrapped prisms) [5] the three prisms from each test series were stored in the same metal container as prescribed for the "Standard" version of RILEM AAR-4.1 (unwrapped prisms) [5], instead of being stored in separate containers.

### *2.3.3. Overview of test series*

Figure 2 shows the notations used to label the various test series. The full notations give a complete description of the pre-treatment and ASR exposure of the prisms. However, to simplify, short names are used in most figures and tables when presenting the results.

Tables 5-8 give an overview of all 58 test series included in the test programme.

## **2.4 Complementary tests**

### *2.4.1. Overview*

In order to document properties of importance for initiation and progress of ASR, comprehensive complementary testing has been an important part of the study. Figure 3 gives an overview of the following tests performed on concrete prisms during and after the ASR exposure (the abbreviations used in Figure 3 are quoted in the brackets below):

- Concrete porosities ("PF-method" - Section 2.4.2)
- Moisture state ("in-situ" (evaporable) water content, "DCS" and "RH" - Section 2.4.3)
- Relative diffusion coefficient ("Relative D" - Section 2.4.4)

- Electrical resistivity (Section 2.4.5)
- Visual inspection (photo) – see [44])
- Microstructural analysis (on polished sections ("PS") and thin sections ("TS"), in addition to use of scanning electron microscope (SEM) – see [25] and [44])

The tests were performed at two ages: four weeks after starting the ASR exposure (in order to document concrete properties in the early stage of the ASR test) and after ending the ASR exposure, i.e. after 39 weeks (all 60°C test series), 52 or 112 weeks (38°C test series).

The tests initiated after four weeks of ASR exposure were performed on an "extra prism" exposed to identical pre-treatment and ASR exposure conditions as the three parallel prisms in the same test series. The tests performed after ending the ASR exposure were executed on one of the three parallel prisms in each test series.

In both cases, the prisms were removed from their container and immediately sealed in polyethylene foil to avoid loss of moisture. After being cooled to ambient laboratory temperature the next day, each prism was unwrapped before the test samples (six samples after four weeks of the ASR exposure and four samples after ending the ASR exposure) with height about 40 mm were split immediately (to avoid loss of moisture) from the prism by use of a rock splitter. Each of the split samples represents a given height from the bottom of the prism.

This paper presents results from the measurements described in Section 2.4.2-2.4.5. Results from the visual inspections and the microstructural analyses are presented in the PhD thesis [44] together with measurements of alkali release from the aggregates and the dynamic E-modulus (measured for all unwrapped prisms at the same point in time as the expansion measurements).

Additionally, the rate and amount of alkali leaching from concrete prisms (measured for all test series at the same point in time as the expansion measurements) are properly discussed in [25].

#### 2.4.2. Concrete porosity

After splitting, each sample was brushed with a wire brush to remove loose particles. The measurements were performed according to the "PF-method" (named "PF" in Figure 3) as described by Sellevold and Farstad [30] by weighing each sample at the following moisture stages:

- Immediately after splitting and brushing the sample.
- After submersion of the sample in water for seven days assumed to saturate the gel and capillary porosity (also submerged mass to obtain volume).
- After drying the samples at 105°C in an oven for seven days.
- After submersion of the sample in water for seven days.
- After submersion of the sample in water in a pressure tank with 50 bar for two days.

The following parameters were measured:

- "In-situ" (evaporable) water content (see Section 2.4.3).
- Degree of capillary saturation,  $DCS = \text{"in situ" water content} / \text{water content after saturation}$  (see Section 2.4.3).
- Gel + capillary porosity (suction porosity [45]) and macro porosity (taken directly as the air content in the hardened concrete) (volume-%).
- Dry -, saturated surface dry (SSD) - and solid densities.

The "PF-method" is frequently used at SINTEF and NTNU as a quality control of a concrete, including estimating the w/cm based on Powers model [30]. Some experiences with the method are summarised in [30]. For equal cement paste contents, a higher suction porosity reflects a more porous concrete with a higher content of capillary pores. The concrete pore system and the content of capillary pores strongly depend on the mix design (in particular on cement type and w/cm), on the degree of hydration and also on the pre-storage conditions (in particular temperature and access to moisture) and the ASR exposure conditions (temperature and access to moisture) [23].

### 2.4.3. Moisture state

The moisture state of the samples split from each prism was documented by the following three parameters (see further description in Section 1.4.1 and 2.4.2):

- "In-situ" (evaporable) water content (mass-% of dry concrete).
- Degree of capillary saturation, DCS (%).
- Relative humidity, RH (%).

As discussed in Section 1.4.1, measurement of RH is connected with fairly large uncertainties. To improve the accuracy of the measurements, efforts were put into selecting humidity sensors and measuring procedures that had proven to be reliable. Based on "pilot testing" with two RH sensors that had been frequently used at SINTEF and discussions with experienced colleagues at the University of Lund in Sweden, the following measuring procedure was used:

- Use of Vaisala sensors "HMP44" with reported accuracy  $\pm 2$  % in the range of 0-90 % RH and accuracy  $\pm 3$  % in the range of 90-100 % RH [46].
- Each sensor was carefully calibrated before and after each measurement.
- One of the concrete samples split from each prism were split into smaller pieces by use of the splitter, before these pieces were crushed with a hammer. The pieces of crushed cement mortar (some being stuck to small aggregate particles) with diameter from about 5-10 mm were collected and put into slim glass tubes (inner diameter 18 mm) until about  $\frac{3}{4}$  of the glass tube was filled. The top of the glass tube was then immediately sealed with putty. Cement mortar particles from the outer about 15-20 mm of the concrete samples were not used. The collected crushed particles thus represent the "inner part" of each prism at the following levels from the bottom of the prisms: 100-160 mm (prisms of length 280 mm) and 120-240 mm (prisms of length 450 mm). Throughout the crushing and sampling procedure care was taken to avoid loss of moisture.
- For some of the large concrete prisms (100x100 mm) used in the Norwegian CPT, cement mortar particles were also collected from the outer 0-25 mm in order to check if there were any moisture gradients.
- A Vaisala sensor was installed in each glass tube before placing the glass tubes in a conditioning room with temperature 20°C and 50 % RH.

- For 4-5 days readings of RH were taken early every morning, before any other activity took place in the room (in order to secure a stable temperature in the samples and thus improve the accuracy of the measurements).
- The readings taken after 2-4 days (when they had stabilised) are presented in Section 3.3.

#### 2.4.4. Relative diffusion coefficient

The rate of drying of concrete samples with identical geometries may be used to calculate relative diffusion coefficients, as done by Relling [26]. This parameter is used to characterize the transport properties of the various ASR test series in the early period of the ASR exposure. Since the diffusion coefficient strongly depends on the internal moisture content, the calculated relative diffusion coefficients (named "Relative D" in Figure 3) are really average values over a certain drying time at isothermal conditions [26].

After 4 weeks of ASR exposure, one of the six samples split from each "extra prism" (as described in Section 2.4.1) were given special treatment during the "PF test procedure" (see Section 2.4.2). After the initial submersion (i.e. before drying the samples at 105°C), this sample was placed in a conditioning room at 20°C and 50 % RH in order to dry out slowly during the following 6 months.

The relative diffusion coefficient ("RelD") was calculated by comparing the time ( $t_{i/2}$ ) various concrete samples need to dry out to a moisture state representing half of the amount of water lost ( $\psi$ ) from saturated state to a moisture state in equilibrium with the surrounding environment relative to a reference sample. The mean calculated drying time for the ASTM samples (7 parallel test series) with the "basis" binder was chosen as a universal reference sample ( $t_{ref,i/2}$ ). The relative diffusion coefficient ( $RelD$ ) could then be calculated according to Eq.1.

$$RelD = t_{ref,i/2} / t_{i/2} \quad (\text{Eq. 1})$$

After six months of drying in the conditioning room, most samples were still drying slowly, in particular the concretes with lowest w/cm. Thus, an estimation of the total water loss at equilibrium

(*c*) was made by simply assuming that the samples had reached a moisture state in equilibrium with the surrounding environment by extrapolation to 400 days.

#### 2.4.5. Electrical resistivity

The electrical resistivity of concrete depends on the porosity, the continuity of the pore system and the degree of water filling of pores (i.e. "physics"). Additionally, it depends on type and amount of ions dissolved in the pore water (i.e. "chemistry") [47]. Several authors have reported a reasonable correlation between electrical resistivity and chloride diffusion for saturated concrete [48]. Hopefully, electrical resistivity might also be used as an indirect measure for concrete transport properties in the present laboratory testing, i.e. as a measure for the ability for water and ions to move to the "reaction site" during the ASR exposure – at least for concrete with the same cement.

After 4 weeks of ASR exposure, supplementary "pilot" measurements of electrical resistivity were performed on five parallel "PF-samples" split from each prism from ten test series. Due to the good correlation obtained between relative diffusion coefficient and electrical resistivity for CEM I cement with similar chemistry (see section 3.4.1), this simpler and less time consuming test method was chosen to describe the transport properties of the concrete prisms from all test series after ending the ASR exposure (instead of measuring relative diffusion coefficients). The measurements were performed on samples with "in-situ" moisture state by applying current at 1000 Hz at 20°C [49]. Electrical resistivity is sensitive to DCS ([35], [50]), but the procedure chosen was later justified by the fact that DCS maximally varied 5 % between various test series. The following test procedure was established (results from measurements according to two additional procedures are included in the coming PhD thesis [44]): After unpacking the prism from the tight polyethylene foil, but before splitting the "PF-samples" (see Section 2.4.1), the electrical resistance ( $R$ , in  $\text{ohm}$ ) across the prism cross section was measured by placing two 100x100 mm metal plates on two opposite sides (not the casting surface) of the mid part of the prism. A conductive gel was evenly distributed on the two plates to ensure good contact, before the readings of the electrical resistance ( $R$ , in  $\text{ohm}$ ) were taken.

When calculating the electrical resistivity ( $\rho$ ) according to Eq. 2 ([47, 49]), the width of the metal plates (100 mm) was as a simplification used as the cross section widths ( $t$ ).

$$\rho = A \cdot R / L = L \cdot t \cdot R / L = t \cdot R \text{ (ohm}\cdot\text{m)} \quad (\text{Eq. 2})$$

where  $A = L \cdot t =$  area of each of the two opposite sides of the sample ( $\text{m}^2$ )

$L =$  length between the metal plates = length of the prism side (m)

Even though the results presented in Section 3.4.3 are somewhat lower than the "true" electrical resistivity (since some current can be transported through the concrete prisms outside the 100 mm zone used in the calculations), the internal ranking between the various test series is believed to be correct.

### 3 RESULTS AND DISCUSSION

#### 3.1 Overall concrete properties – control of mix design and concrete mixes

In order to cast the prisms for all the 58 test series, 12 concrete mixes of about 50 liters were prepared; eight of the "basis" binder, two of the "open" binder and one of each of the two remaining binders (see Table 9). All the 12 concrete mixes showed good workability properties. The slump varied from 120 to 150 mm.

The air (macro) pores might act like evacuation chamber for the ASR gel produced, but the influence of increased air content on the expansion is not fully agreed in the literature [23]. However, the fresh air content was rather low and in the target range ( $< 3.0\%$ ) for all concrete mixes, except one (3.1%). Consequently, the expansions can be compared without consideration of the slightly varying air contents.

Table 9 shows overall hardened concrete properties for the 12 concrete mixes. All the 8 batches with the "basis" binder (CEM I, w/c of 0.45) attain nearly identical hardened concrete properties. The 8 parallel ASTM test series with this binder (see Table 8) also obtain very similar prism expansions

(mean 52 weeks expansion of 0.266 %, c.o.v. of 3.2 %). The variation between the mean prism expansions for these 8 parallel test series is in fact lower than the internal variation between the three parallel prisms within each of the test series (typical c.o.v. is in the range of 4-10 %). Furthermore, the suction porosities (section 2.4.2) measured for the 8 parallel ASTM test series with the "basis" binder after 4 weeks of exposure are comparable (mean value of 11.9 %, c.o.v. of 3.2 %). Based on these 8 parallel suction porosities and by assuming a realistic degree of hydration ( $\alpha = 0.85$  [51]), w/c is estimated to be in the range from 0.44 to 0.47 applying Powers model [30], with a mean w/c of 0.45, agreeing with the mix design.

The two batches with the "open" binder (CEM I, w/c of 0.60) also obtain nearly identical hardened concrete properties, with internal variations for the various parameters tested in the same range as for the "basis" binder (Table 9). The mean prism expansions are also comparable (mean 52 weeks expansion of 0.225 %, c.o.v. of 8.5 %). Furthermore, based on the suction porosities after 4 weeks of exposure (mean of 13.2 vol-%) and by assuming a realistic degree of hydration ( $\alpha = 0.95$  [51]), w/c is estimated to be 0.55 and 0.60, with a mean w/c of 0.58, i.e. a fairly good agreement with the mix design.

Based on the suction porosity after 4 weeks of exposure (10.4 vol-%) and by assuming a realistic degree of hydration ( $\alpha = 0.65$  [51]), w/c for the ASTM test series with the "dense" CEM I binder (w/c of 0.30) is estimated to be 0.30, i.e. agreeing with the mix design.

Based on the suction porosity after 4 weeks of exposure (13.7 vol-%) and by assuming a realistic degree of hydration ( $\alpha = 0.60$  [51]), w/c for the ASTM test series with the "fly ash" binder (CEM II/A-V, w/cm of 0.45) is estimated to be 0.44, i.e. agreeing with the mix design.

Since most of the fly ash still has not reacted after 4 weeks of exposure, the suction porosity for the "fly ash" binder is significantly higher than for the "basis" binder even though w/cm is equal. Note that the differences in concrete suction porosity do not directly reflect the variation in "binder porosity", since the cement paste content varies somewhat from binder to binder (see Table 2).

Based on the discussion above, we conclude that the produced concretes are of designed quality and that test series cast from different batches can be compared.

### **3.2 Change in porosity and mass during the ASR exposure**

#### *3.2.1. Porosity*

Tables 10-13 show the suction and macro porosities for all the test series after 4 weeks of exposure based on suction and submersion after drying. Additionally, the change in porosities from 4 weeks to the end of the exposure period is given.

As shown in Figure 4, the suction porosity (before drying) for most test series are significantly altered when ASR develops during the exposure period from 4 weeks to 39 weeks (60°C test series) or 52 weeks (38°C test series) of exposure, as are the "in-situ" water contents (see Section 3.2.3). It appears that the suction porosity increases (up to 2.2 vol-% = 22 l/m<sup>3</sup>) for most test series with the CEM I binders with w/c of 0.45 and 0.60, in particular for those revealing high expansion, a natural consequence of the expansion producing cracks that hold ASR-gel and water. However, alteration of the suction porosity also depends on the type of binder (in addition to the magnitude of expansion). For four of the five "fly ash" test series, the suction porosity is reduced in the range of 0.4 to 0.9 vol-%, while being almost unchanged for the remaining "fly ash" test series. Also the test series with the "dense" CEM I binder reveal rather small alterations of the suction porosity (from 0.3 vol-% reduction to 0.6 vol-% increase). One reason for the observed reduction in suction porosity is assumed to be further hydration, in particular of the fly ash, making the paste denser. Additionally, the final expansions are less for these two binders compared with the two CEM I binders with higher w/c (discussed in [25]).

The average changes in macro porosity (air content) during the ASR exposure from 4 to either 39 or 52 weeks of exposure are negligible, in the range of -0.3 to 0.1 vol-%. This is somewhat surprising, since ASR also leads to formation of cracks that are too large to build up capillary forces [52]. One should thus expect an increase in the macro porosity corresponding to the formation of such "coarse" cracks. However, the microstructural analysis showed that ASR gel was present in several air voids and

cracks at the end of the ASR exposure period. In the "PF-analysis", the macro porosity filled with ASR gel (that probably will suck even more water when the concrete samples are submerged), will be part of the measured increase in suction porosity. Since the measured macro porosity did not change significantly, the reduced air void content due to filling with ASR gel seems to be of the same magnitude as the increase in macro porosity due to formation of "coarse" cracks. However, one should not exclude another possible explanation that might have some influence: Some of the cracks formed may be so large that water is running into the cracks when the concrete samples are submerged (i.e. without any capillary forces present). If this water is not released again before the samples are weighed, the water will contribute to an increase in the measured suction porosity. One observation supporting this theory is that the mass increased a little for the (few) samples being submerged longer than the prescribed one week in the "PF-procedure"; 14 days of prolonged submersion raised the suction porosity with about 0.3 vol-% (CEM I, w/c of 0.45) and 0.5 vol-% (CEM I, w/c of 0.60), respectively.

### *3.2.2. Mass change of whole prisms*

Extensive ASR testing has shown that the concrete will absorb water during the exposure period, see for example study by Lindgård and co-workers [20]. The main mechanisms for the water uptake are: 1) Water is absorbed because of self-desiccation; 2) Any cracks developed during the ASR exposure that are able to suck water and any ASR-gel in cracks and pores will absorb water if available.

In addition to document internal moisture state of one prism from each test series (section 2.4.3 and 3.2.3), the net mass change of the whole prisms from de-moulding has been recorded. To avoid loss of moisture, these prisms were cooled inside polyethylene foil before being split. Thus, the net increases in prism mass are assumed to represent the "true" water absorptions at these ages (neglecting the minor influence on the prism mass of alkali leaching). Based on this assumption, the increase in mass might be recalculated from mass-% to volume-% (vol-%) water absorbed, as done in several figures (1 vol-% water corresponds to 10 l/m<sup>3</sup> of concrete). *(Comment: The mass of the three parallel prisms used for expansion readings has additionally been recorded at every measuring point in time, but these*

*results are not presented in this paper. For the wrapped prisms, these periodic mass readings include any water absorbed by the cotton wrapping during the ASR exposure.)*

The fact that concrete will absorb water when ASR develops is evident from Figure 5. A fair correlation exists between the water uptake and the expansion in the period beyond 4 weeks of exposure. When adding trend lines,  $R^2$  is calculated to be about 0.90 for the wrapped prisms and about 0.83 for the unwrapped prisms. At high expansions, the "mass versus expansion trend line" seems to flatten out more for unwrapped prisms compared with wrapped prisms, indicating that wrapped prisms absorb slightly more water during the ASR exposure compared with unwrapped prisms for comparable expansions. Some of the wrapped prisms absorb up to 0.80-0.85 mass-% of water in the period beyond 4 weeks of exposure, corresponding to about 20 litres of water per  $m^3$  of concrete.

The dotted line in Figure 5 represents the linear expansion of the prisms recalculated to volume expansion, assuming a uniform expansion in all directions and that the increased volume is filled with water. Thus, this line represents the new volume generated due to the ASR expansion. As seen, all the test series absorb more water in the period beyond 4 weeks of exposure than the amount representing the new volume generated. A part of this absorption is water entering partly empty pores (i.e. increased DCS – see later). Additionally, this indicates that during the ASR exposure, water is able to enter space that was not available before the ASR started; for instance ASR gel in air voids.

The correlation between the total water absorption from de-moulding and the total prism expansion from the reference readings is less good than the correlation beyond 4 weeks (Figure 5). A possible reason could be that during the first 4 weeks the water uptake is mainly caused by hydration / self-desiccation, while beyond 4 weeks the ASR creates cracks and ASR-gel absorbs water.

*(Comment: Figure 5 clearly indicates that wrapped prisms absorb more water compared with unwrapped prisms for comparable expansions. In the summary of findings in the EU-PARTNER project [7], it was stated that: "The weight measurements showed in general a lower weight increase, and sometimes also a weight loss, for the aggregates tested according to the AAR-4 Alt. method (i.e. wrapped prisms) compared to the reactor version (i.e. unwrapped prisms)".*

*Apparently, the conclusion from the PARTNER tests with respect to effect of wrapping on the mass change of the 60°C prisms was contradictory to the findings discussed above. However, in the PARTNER project the weight results discussed were not net mass of the prisms, rather total mass including any wrapping. Additionally, the prisms were cooled over night to ambient temperature before the readings were taken, and thus some water had evaporated, and then significantly more for the unwrapped prisms - documented in pilot testing in this study).*

Figures 6 and 7 show the average net mass increase of prisms depending on binder type, prism size, test method and time interval. The results for all test series with similar exposure conditions are averaged, irrespective of the length of the pre-storage period at 20°C (see Tables 5-8). However, one exception is made for the "fly ash" binder, where the RILEM AAR-3 and AAR-4.1 results are shown for both 1 and 28 days of pre-storage at 20°C. Figure 8 provides details of the progression of water uptake from de-moulding to the end of the exposure period for the "fly ash" concrete prisms. Similar figures for the other CEM I binders are included in Lindgård's PhD thesis [44]. *(Comments to Figure 8: For the wrapped prisms, the net mass of the prisms is not known at the time of the reference readings. For the unwrapped prisms measured without being pre-cooled to 20°C (i.e. all unwrapped test series except the ASTM test series), the measured reference mass have been corrected (based on results from pilot testing) for the mass loss from the prisms were removed from their containers until the mass measurements 60 seconds later.)*

The following main trends are observed with respect to average water absorption from de-moulding to 4 weeks of exposure (Figure 6):

- The water absorption varies with binder type, prism size, pre-treatment and exposure conditions. The prisms absorb from 8 to 23 litres of water per m<sup>3</sup> of concrete, corresponding to about 0.35 to 0.95 mass-%.
- In general, prisms with the "fly ash" binder absorb significantly less water compared with prisms with the CEM I binders. One reason could be that the hydration products incorporating fly ash bind less water than hydration products of pure OPC, [53] and [54]. For similar pre-storage period at 20°C, the mass gain of the prisms with the "fly ash" binder seems to be independent of test method and exposure temperature.
- The two "fly ash" test series pre-stored in the containers at 20°C for 28 days absorb significantly more water compared with the corresponding test series exposed to elevated temperature after 1 day.

- In general for CEM I binders, exposure to 60°C leads to considerably higher water absorption compared with corresponding test series exposed to 38°C (one exception is unwrapped prisms with w/c of 0.60). Two obvious reasons can explain this higher mass increase when the temperature raises; 1) Some of the 60°C test series have already started to expand somewhat during the first 4 weeks of exposure (up to 0.03 %), leading to suction of water; 2) The relative diffusion coefficient in general increases with increasing exposure temperature (Section 3.4.2), making ingress of water easier.
- In general for the CEM I binders, wrapped prisms absorb significantly more water than corresponding unwrapped prisms (documented by weighing the prisms after unwrapping). It is expected that wrapping the prisms in saturated cloth will increase water availability.
- In general for CEM I binders, the larger Norwegian prisms absorb less water per m<sup>3</sup> of concrete compared with the smaller ASTM prisms, indicating that the interior of the concrete prisms have less access to water when the prism cross section is increased from 70 to 100 mm. An internal moisture gradient is also observed in the larger Norwegian prisms (section 3.3).
- After 4 weeks of exposure, no significant differences in total water absorption was observed between prisms submerged in water for 0.5 h after de-moulding compared with prisms not submerged.

The following additional main trends with respect to average water absorption from 4 weeks to 39/52 weeks of exposure can be drawn from Figure 7:

- The extent of water absorption during the ASR exposure varies with binder type, prism size, pre-treatment and exposure conditions, and is closely connected to extent of ASR expansion; higher expansion in general leads to increased water uptake (see also Figure 5).
- Prisms with the "fly ash" binder exposed to 60°C absorb considerably more water than corresponding prisms exposed to 38°C, primarily due to significantly higher prism expansion. The 38°C "fly ash" test series reveal equal mass increase, independent of prism size and use of any wrapping.
- At 38°C exposure, prisms with the "dense" CEM I binder (w/c of 0.30) absorb significantly less water compared with the prisms with higher w/c. The main reason for this is assumed to be the lower ASR expansion for these "dense" test series, combined with a lower relative diffusion coefficient (Section 3.4.2). Even less water absorption is observed for the "fly ash" test series.

- As expected, the extreme environments "sealed prisms" and "submerged prisms" (Table 4) significantly influence the water uptake at all ages by respectively reducing it (elimination it for the 38°C prisms) and increasing it.

### 3.2.3. Mass change of split samples

After 4 weeks of exposure, the evaporable "in-situ" water content varies from approximately 90-135 l/m<sup>3</sup> (3.8-5.8 mass-%), primarily depending on binder type and exposure conditions (Tables 10-13). As expected, the "dense" binder test series contain least evaporable water, while the test series with the "fly ash" binder and the "open" CEM I binder reveal the highest "in-situ" water contents.

In the period beyond 4 weeks, the change in "in-situ" water content varies from 14 l/m<sup>3</sup> reduction (sealed 38°C test series) to 26 l/m<sup>3</sup> increase (Figure 4). A fairly good correlation exists between the change in suction porosity and the corresponding change in evaporable "in-situ" water content, with a linear correlation ( $R^2$ ) of 0.85 (excluding the sealed samples). The interpretation of a point lying on the 1:1 line in the figure is that the increased suction porosity due to ASR is filled up with a corresponding amount of water (and as discussed earlier also some ASR gel that absorbs water). Most data points, except for the "fly ash" binder and the sealed 38°C prism (lowest data point), lie slightly above the 1:1 line, indicating that DCS increase during the ASR exposure (Section 3.3).

For all test series with the "fly ash" binder and the "dense" CEM I binder, the change in evaporable water content beyond 4 weeks is generally small. In fact, most "fly ash" test series reduce the "in-situ" water content. However, whole prisms with these binders absorb significantly more water in the same period (Figures 6 and 7), indicating that some of the absorbed water has been bound in the cement hydrates during further hydration and is not released when the "PF-samples" are dried at 105°C.

For CEM I test series with higher w/c, the change in "in-situ" water content primarily depends on the expansion. With increasing expansion, the suction porosity and the evaporable water content increase due to formation of internal cracks and growth of ASR gel in cracks and macro pores. At the end of the exposure period, the evaporable water content is still lowest for the "dense" binder (95-110 l/m<sup>3</sup>),

in the range 120-145 l/m<sup>3</sup> for the "fly ash" binder and the "basis" binder, while the test series with the "open" binder contain the most evaporable water (140-155 l/m<sup>3</sup>).

### 3.3 DCS and RH of split samples

#### 3.3.1. Samples split from ASR test prisms

The relation between DCS and internal RH is given in the Figures 9 and 10. Detailed results are shown in the Tables 10-13, including changes of DCS and RH during the ASR exposure.

DCS is relatively easy to measure accurately and is regarded as a reliable measure for the percentage of water filling the gel and capillary pores [30]. A low spread is also documented, both between parallel test series (mean c.o.v. is about 1 % for 8 parallel ASTM test series) and over the prism height (mean c.o.v. for the three parallel "PF-samples" split from various heights of each prism (58 test series) after 4 weeks of exposure is 0.5-0.8 %). An implication of the latter is that no distinct variation in DCS can be observed over the prism height, even though all the prisms have been stored vertically with the same end facing upwards during the whole ASR exposure period.

Due to the test setup and the actions taken to try to minimize the known sources of errors (Section 2.4.3), the RH measurements are generally regarded to be reliable. Parallel measurements generally showed comparable results, being well within the reported accuracy of the sensors. However, for five of the more than 120 RH measurements the sensors drifted, hence the points are uncertain.

At all ages, DCS varies far less than the corresponding RH. After 4 weeks (Figure 9), DCS varies in the range of 91.5-96.5 vol-% (except for the two sealed test series), while the RH varies in the range of 82-97 %. At the end of the exposure period (Figure 10), DCS ranges from about 93-98.5 vol-%. The corresponding RH varies in the range of 83-99 % (except for the sealed 38°C test series).

RH seems to depend strongly on the nature of the binder (w/cm and use of any additions), and for the "dense" binder also on the exposure temperature. In contrast, no significant differences in DCS are observed for the different binders, but the amount of evaporable water is binder dependent.

Primarily due to increased self-desiccation and finer pore structure, and maybe partly due to higher ion concentration in the pore water, the "dense" CEM I binder (w/c of 0.30) reveals the lowest RH values after 4 weeks of exposure (Figure 9), ranging from 82-85.5 % for the 38°C test series (lowest for the larger Norwegian prisms). For corresponding test series exposed to 60°C, RH is higher; in the range of 90-92 %. This RH increase is probably related to the coarsening of the pore structure produced by exposure to elevated temperature (60°C) as shown for cement pastes by Bray and Sellevold [55].

The "fly ash" test series obtain RH in the range of 84.5-90 % after 4 weeks (Figure 9), i.e. almost as low as the "dense" binder. The lowest RH is obtained for the prisms pre-stored at 20°C for 28 days, in particular those later exposed to 60°C. This latter test series also attain the lowest DCS of the "fly ash" test series. Note that these test series are 4 weeks older than the other "fly ash" test series, and consequently more fly ash has reacted and the degree of self-desiccation is thus higher.

At both exposure temperatures, the internal RH in the test series with the CEM I binders with higher w/c (0.45 or 0.60) is always higher than 90 % after 4 weeks of exposure, the majority in the range of 93.5-96 %. During the whole ASR exposure, test series with the most "open" binder always reveal slightly higher or as high RH as the test series with the "basis" binder with highest RH.

The presented values for RH were measured in a climate-controlled room at 20°C after cooling the prisms inside plastic foil (to avoid loss of moisture). As discussed by Lindgård et al. ([22], [23]), a general increase in the temperature will give rise to a small increase in the internal RH provided the moisture content within the concrete is kept constant. For example, Sellevold and Bjøntegaard [28] and Nilsson [56] reported that the RH within a concrete will increase in the range of 0.2-0.3 % RH per °C with a starting RH around 70-90 %. The effect decreases with increasing w/cm ratio and is RH dependent. The effect is most marked in the middle RH-range (around 50–60%), and decreases to zero in very dry and in saturated concrete [28]. For the present test series with RH in the range of 82-97 % RH (measured after cooling to 20°C), the internal RH will increase during the ASR exposure compared with the results presented in Figures 9 and 10 by up to a maximum of 5% when stored at 38°C and up to a maximum of 10% when stored at 60°C. In other words, when the measured RH (at

20°C) for the "dense" binder is significantly lower for the 38°C test series compared with the corresponding 60°C test series, the difference will be even greater during the ASR exposure when the temperature is elevated. The practical importance of this temperature effect for the ASR reaction is not known, but it seems to be reasonable to assume that the higher RH at elevated temperature will contribute to accelerate the ASR expansion.

Knowing that the critical RH limit for developing ASR is in the range of 80-90 %, depending on several factors as discussed by Larive et al. [32], it is likely that the rather low RH revealed for the "dense" binder after 4 weeks of exposure and maybe also for the "fly ash" binder contributes to reduce the rate and extent of ASR, in particular for the test series exposed to 38°C (further discussed in [25]). For all test series with the CEM I binders with higher w/c (0.45 or 0.60), except for the sealed test series, RH is regarded to be sufficient for ASR to develop. Thus, it appears that for these concretes all the test procedures provide sufficient moisture contents for ASR to proceed.

In contrast to the findings by Lindgård et al. [27], no good correlation has been found in the present tests between the rate and extent of ASR and the DCS. However, DCS is higher than 91.5 % for all the test series, while the "critical DCS" for the field concretes was found to be about 90 % DCS [27]. On the other hand, not only the degree of pore filling but also the total amount of evaporable water in the concrete might influence the extent of ASR. One can thus not rule out that the less amount of available (evaporable) water in the "dense" concrete (w/c of 0.30) might contribute to lower extent of ASR compared with the CEM I concretes with higher w/c.

For all binders and all test methods, DCS generally increases during the ASR exposure as water is taken in (only a few exceptions exist). However, the observed changes in RH during the exposure period depend on type of binder, pre-treatment and exposure temperature.

In general, RH increases beyond 4 weeks of exposure for the test series with the CEM I binders, irrespective of w/c (with only a few exceptions). The main trend is that most test series with the "dense" binder (that reveal the lowest RH after 4 weeks of exposure) and those test series with the

"basis" binder that obtain "relative low" RH values after 4 weeks of exposure (except the sealed test series) show the highest increase in RH (+5-7 %).

On the contrary, the internal RH decreases (up to 4.5 %) for most of the "fly ash" test series, despite a small increase in DCS.

At the end of the exposure (Figure 10), the CEM I test series with w/c 0.45 or 0.60 obtain highest RH, the majority lying in the range of 95.5-98 %. The 38°C "dense" binder test series attain RH in the range of 84.5-90.5 %. For corresponding 60°C test series, RH is higher; in the range of 92-95 %. For the "fly ash" binder, RH varies in the range of 83-88.5 % for test series exposed to 38°C, significantly lowest for the larger Norwegian prisms, and in the range of 88-89 % for test series exposed to 60°C.

In general at 38°C exposure, wrapped prisms obtain slightly higher DCS and internal RH compared with corresponding unwrapped prisms at all ages (Figures 9 and 10, Tables 10 and 13). This is valid for all binder types and for all test series (with only a few minor exceptions).

After 4 weeks of exposure to 60°C (Figure 9 and Table 11), wrapped prisms with the CEM I binders obtain slightly higher DCS compared with corresponding unwrapped prisms. This corresponds to the observed higher water uptake for wrapped prisms (Figure 6). Regarding RH, wrapped prisms with the "dense" binder attain slightly higher values than corresponding unwrapped prisms. For higher w/c (0.45 and 0.60), no significant difference in RH is observed between wrapped and unwrapped prisms.

After 39 weeks of exposure to 60°C (Figure 10), no systematic differences in DCS or RH between wrapped and unwrapped prisms are observed. However, a tendency is that RH is slightly less for some of the wrapped test series that reveal significantly lower expansion compared with corresponding unwrapped test series. The assumed reason for this is that more ASR gel is produced for test series that reveal higher expansion, and thus the internal RH is increased.

Except use of any wrapping (or sealed or submerged samples), no systematic differences in DCS or RH are detected between test series with the "basis" binder where other pre-treatments are varied.

During the entire exposure period, the test series submerged in de-ionised water obtain DCS and RH values on the same level as corresponding wrapped test series. (*Comment: no results are available for submerged samples after 4 weeks of exposure to 60°C*).

After 4 weeks (Figure 9), the sealed prisms stored in tight, dry containers placed in the dry 38°C room obtain DCS of about 86.5 vol-% and RH of 87.5 %. After 52 weeks (Figure 10), DCS is reduced to 78 vol-%, while RH is reduced to about 75 % (some uncertainty is related to this latter RH measurement). However, some water is assumed to have evaporated from the prisms during the one year exposure in the dry 38°C room.

After 4 weeks, sealed prisms stored in airtight, dry containers placed inside the humid 60°C reactor obtain DCS of about 79 vol-% and RH of 85 % of exposure (Figure 9; some uncertainty is related to this RH measurement). At 60°C, the sealing with epoxy and aluminium foil is not completely watertight, and some water is taken up beyond age 4 weeks. Thus, no reliable "sealed humidity data" is available for the 60°C test series at the end of the exposure period.

In general for all binders and at all ages, the larger Norwegian prisms (cross section 100x100 mm) obtain slightly lower (or equal) DCS and internal RH compared with the smaller ASTM prisms (70x70 mm) (when excluding the few uncertain RH measurement). This agrees with the observed lower water uptake for the larger prisms in the early period of exposure (Figure 6). After 4 weeks of exposure of the Norwegian prisms, DCS and in particular the corresponding RH are always higher in the outer 25 mm compared with the interior of the prism (Figure 11). The RH gradient is most pronounced for the "dense" binder, where DCS and RH in the interior of the large Norwegian prism are equal to DCS and RH of sealed samples stored in airtight plastic bottles at 20°C until age 11-16 weeks (Figure 11). After 52 weeks of exposure, a substantial RH gradient is observed for the "dense" CEM I binder and the "fly ash" binder (Figure 12). For both binders, the RH gradient is higher than the corresponding gradients after 4 weeks of exposure.

### 3.3.2. Pilot tests with more porous aggregates

Any supply of water from aggregate pores to the cement paste during the curing period will reduce the self-desiccation ([39], [40]) and thus significantly increase the RH compared with use of relatively dry aggregates, in particular for low w/cm binders (see Section 1.4.3). If rather porous ( $\geq 0.8\%$ ) normal density pre-wetted aggregates are used, they may theoretically totally counteract the effect of self-desiccation [39]. As a consequence, it might be conservative to use pre-wetted aggregates in laboratory performance testing instead of dry aggregates that is the practice in some laboratories.

To verify this theory, pilot tests have been performed with two additional aggregates with different porosity and moisture state at the time of mixing. The water absorption of the coarse aggregate fractions was about 1 % (in the upper range for Norwegian aggregates) and 6.5 % (Icelandic basalt), somewhat lower for the fines. Two mixtures were prepared with each of these aggregates, one with dry aggregates and one with moist aggregates (pre-wetted overnight). The same binder with cement CEM I and effective w/c of 0.30 was used in all the mixtures (the amount of extra water added to the mixtures with the dry aggregates was calculated based on the 1 hour water absorption of the aggregates). The four concretes were stored sealed in tight plastic bottles at three temperatures (20, 38 and 60°C) for 2, 3 and 6 weeks (shorter storage when the temperature is increased). Two independent RH measurements were performed on crushed samples (Section 2.4.3) from each of the 12 bottles. The results clearly show the impact on RH of aggregate porosity and aggregate moisture state:

- RH in the concretes containing pre-wetted highly porous Icelandic aggregates was 8-14 % higher compared with the corresponding concrete with dry aggregates. Corresponding differences for the Norwegian less porous aggregate were in the range 5-10 %. *(Comment: Even though the spread in RH measured on parallel crushed samples from the same bottle was rather high, the tendency was clear).*

## 3.4 Transport properties

### 3.4.1. Assessment of methods used for estimation of transport properties

In general, the internal spread for the relative diffusion coefficient between parallel test series is much higher compared with the other parameters measured (Section 3.1-3.3). For example, the c.o.v. of RelD for parallel test series (Table 14) is in the range of 0-24 %. The internal spread for the electrical

resistivity is low (c.o.v. for 5 parallel "PF-samples" split from each prism during the pilot testing was about 4.5 %).

Figure 13 shows mean results from this "pilot" testing of electrical resistivity (Section 2.4.5) plotted against the calculated relative diffusion coefficient measured on the same samples. The figure only includes CEM I cement with w/c in the range of 0.30-0.60, i.e. binders with comparable pore water composition. As seen, a good correlation is obtained between relative diffusion coefficient and electrical resistivity.  $R^2$  for the linear trend line is 0.96 when removing the outlier with a relatively high RelD compared with the other samples. This indicates that both methods can be used for assessment of transport properties of the various test series, provided the binders have equal chemistry, i.e. as long as that the electrical resistivity is not too much influenced by varying chemistry of the pore water. Note that changes in pore water chemistry, DCS or internal cracking (see later) will significantly influence the electrical resistivity.

Results from measurements of RelD after 4 weeks of exposure and electrical resistivity at the end of the exposure period are presented in Section 3.4.2 and 3.4.3, respectively.

#### *3.4.2. Relative diffusion coefficient*

Calculated relative diffusion coefficients (RelD) after 4 weeks of exposure are presented in Table 14.

The following main trends can be drawn from the results:

- The relative diffusion coefficient varies significantly with binder type, exposure conditions, prism size and pre-treatments. RelD for the different test series varies in the range of 0.1-4.1 (RelD = 1.0 for the reference ASTM samples with the "basis" binder).
- RelD for the "fly ash" test series varies far less (in the range of 0.4 to 0.6) compared with the values for the CEM I "basis" binder with equal w/cm (in the range of 0.5-4.1). In other words, the "fly ash" binder is more "robust" against any changes in pre-treatments and exposure conditions compared with the CEM I "basis" binder.

- RelD for various binders exposed to 38°C: "dense" binder < "fly ash" binder < "basis" binder < "open" binder. For all the three CPTs the differences between the binders are significant, and the internal ranking is in accordance with our expectations.
- RelD for various binders exposed to 60°C: "fly ash" binder  $\approx$  "dense" binder  $\ll$  "basis" binder  $\approx$  "open" binder. In general, the internal differences between the two densest binders and the two most open binders, respectively, are less pronounced compared with exposure to 38°C.
- Test series with CEM I binders (w/c of 0.30-0.60) exposed to 60°C for 4 weeks have significantly higher RelD compared with corresponding 38°C test series. One reason is the coarser pore structure when "cured" at a higher temperature [55]. The influence of "curing temperature" (higher permeability) is however, as demonstrated in [55], expected to be even more pronounced when exposed to elevated temperatures directly after casting compared with exposure to elevated temperatures after some time (as in this case, where all test series were cured at 20°C the first day). Still, there is a tendency that exposure to 60°C after 7 or 28 days leads to a somewhat lower RelD compared with exposure to elevated temperature directly after de-moulding. Another factor that might have higher influence on the observed differences in RelDs between the two exposure temperatures is the fact that some of the test series exposed to 60°C already have expanded significantly during the first 4 weeks of exposure (up to 0.03 %). Thus, expected incipient internal micro-cracking have most likely contributed to an increase of RelD already after 4 weeks of exposure to 60°C. Since no test series exposed to 38°C have started to expand significantly after 4 weeks, the influence on any micro-cracking on the revealed RelDs for these test series are assumed to be negligible.
- RelD for test series with CEM I binders (w/c of 0.30-0.60) tested according to various 38°C CPTs (W=Wrapped; U=Unwrapped): Norwegian CPT (U) < RILEM AAR-3 (W) < ASTM C-1293 (U). The main reason for the lowest RelDs for the Norwegian CPT test series is assumed to be that the larger Norwegian concrete prisms dry out more slowly than the smaller prisms (similarly as a dense binder dry out more slowly than a more open binder). The fact that the ASTM test series in general reveal slightly higher RelDs compared with the AAR-3 test series might be due to several reasons, among them earlier exposure to elevated temperature (1 day versus 7 days).

### 3.4.3. Electrical resistivity

Experiences show that 1 % increase in DCS will give approximately 3 % reduction in electrical resistivity [50]. However, DCS values are comparable for all test series, and should thus not influence the measured electrical resistivity too much. In Figure 14, the "in-situ" electrical resistivity measured on whole prisms at the end of the exposure period is plotted against the corresponding expansion. The following main trends can be drawn from the figure:

- The electrical resistivity is influenced by binder type, exposure conditions, pre-treatments and prism expansion. The calculated values vary in the range 40-635 ohm·m.
- As expected [48], the test series with the "fly ash" binder had much higher values (ranging from 330-635 ohm·m) compared with the CEM I binders (ranging from 40-190 ohm·m, except one test series with the "dense" binder with electrical resistivity of 335 ohm·m). Furthermore, the "fly ash" test series exposed to 60°C reveal higher values (540-635 ohm·m) compared with the 38°C test series (330-490 ohm·m), even though the latter test series reveal lower expansions (see later discussion). A similar influence of curing temperature is also observed for the CEM I test series. Possible reasons for this might be higher degree of hydration of the clinker (for the "dense" binder) and reaction of more fly ash at the highest temperature, and thus a denser concrete (the ion concentration in the pore water could also have been slightly reduced).
- There is a tendency that the electrical resistivity decreases when the ASR expansion increases. The reason might be that cracks generated during the ASR exposure are partly filled with ASR gel and water, making the transport of current easier than through concrete with less internal cracking. In particular it can be observed that the wrapped 60°C test series that reveal significantly less expansion than corresponding unwrapped test series, obtain significantly higher electrical resistivity than the unwrapped test series (despite that more alkalis are leached out from the unwrapped prisms at the end of the ASR exposure compared with the wrapped prisms, and consequently less ions are present in the concrete pore water).
- As expected, the test series with the "dense" CEM I binder reveals higher electrical resistivity than the "basis" binder, while test series with the "open" binder reveal the lowest values for comparable expansions.
- For the CEM I binders, a good correlation is revealed after 4 weeks of exposure between the relative diffusion coefficient ( $RelD_{4weeks}$ ) and the electrical resistivity (Figure 13). However, no correlation

exists between  $RelD_{4weeks}$  and the electrical resistivity at the end of the exposure period (except that similar internal ranking is obtained between the CEM I binders). The main reason is assumed to be that the internal cracking due to ASR influence the continuity of the pores (i.e. "opens up" the pore structure), and thus dominates the measured electrical resistivity (for similar binder qualities) late in the exposure period.

## 5 CONCLUSIONS

Based on the comprehensive laboratory study, including 58 test series with modified versions of five concrete prisms tests (CPTs), the following conclusions can be drawn:

- The internal moisture state and the transport properties of a given concrete may be significantly influenced by the specimen "pre-treatment", "ASR exposure conditions" and prism cross-section. The influence depends on the concrete composition, i.e. w/cm and cement type. Consequently, the results of applying a performance test to a given concrete might differ depending on the details of the test method.
- During the ASR exposure, the concrete properties are altered. The general tendency is increased suction porosity and increased internal moisture state with increased ASR expansion, but the alteration depends on the binder composition. The change in macro porosity is negligible. Moreover, a relation exists between water uptake and expansion, i.e. concrete expansion leads to increased porosity that takes up water. However, more water is taken up than the volume corresponding to the increased porosity.
- With respect to the specimen "pre-treatment", the main findings are:
  - ✓ From a "moisture point of view", the 0.5 h submersion after de-moulding described in several CPTs seems unnecessary (has little effect beyond the first weeks of exposure).
  - ✓ The length of pre-storage at 20°C before exposure to elevated temperature might marginally influence the internal moisture state in the first period of exposure, but not at later ages. However, exposure to 60°C directly after de-moulding significantly increase the relative diffusion coefficient ( $RelD$ ) for CEM I binders, making the internal transport of water and ions easier.

- ✓ Prisms wrapped in moist cotton cloth and plastic sheets absorb significantly more water than unwrapped prisms displaying similar expansions in ASR tests. During the first weeks of exposure, this behaviour is particularly pronounced at 60°C. For prisms exposed to 38°C, the wrapping also leads to slightly higher DCS and internal RH after 4 weeks of exposure.
- With respect to the "exposure conditions" and prism cross-section, the main findings are:
  - ✓ The "exposure parameter" confirmed to have the highest impact on the internal moisture state and the transport properties of concrete is the temperature. The prism cross-section can also significantly influence the internal moisture state. For both parameters, the influence depends on the binder composition.
  - ✓ Generally for CEM I binders, exposure to 60°C leads to considerably higher water absorption during the first weeks compared with corresponding test series exposed at 38°C. The internal RH is also significantly higher for the test series exposed to 60°C, both at early age and later. This RH-increase is probably primarily related to the coarsening of the pore structure produced by the elevated temperature and, of course, more water.
  - ✓ At the end of the ASR exposure, the total prism water uptake and the internal moisture state is to a high extent influenced by the extent of ASR and thus the extent of internal cracking and amount of ASR-gel produced. Also the electrical resistivity is influenced – increased ASR expansion tends to decrease the electrical resistivity.
  - ✓ In general for CEM I binders exposed to 38°C, increased prism cross-section leads to less absorbed water in the interior of the prisms, in particular during the first period of the exposure. A pronounced RH gradient is present for test series with the "dense" binder (w/c of 0.30) during the entire exposure period. A similar, but less distinct, gradient is observed in prisms with the "fly ash" binder (w/c of 0.45).
- With respect to influence of binder composition (i.e. w/cm and cement type), the main findings are:
  - ✓ No systematic difference in the degree of capillary saturation (DCS) between binders of different composition can be found, neither at 4 weeks nor at the end of the exposure.
  - ✓ For the CEM I test series, one of the most important properties of the "dense" binder (w/c of 0.30) compared with higher w/c is the much lower internal RH, in particular when

exposed to 38°C (RH in the range of 82-90 %). The reason is primarily the higher extent of self-desiccation. Furthermore, the relative diffusion coefficient is substantially lower also due to a finer pore structure so that the water uptake is slower. Additionally, an assumed higher concentration of ions in the pore water might contribute to reduce the RH.

- ✓ Several concrete properties of the test series with the "fly ash" binder deviate from the CEM I series. Firstly, most concrete properties for the "fly ash" binder seem to be more or less independent of both the specimen pre-treatment and the storage conditions during testing. Secondly, concrete prisms with the "fly ash" binder generally absorb less water compared with prisms with the CEM I binders at the same w/cm, presumably due to the lower "permeability" (measured as a low RelD and a higher electrical resistivity) and less water bound by the hydration products incorporating fly ash. Thirdly, the internal RH is considerably lower in the "fly ash" test series compared with CEM I series with equal w/cm. Fourthly, the general increase in RH seen for the CEM I binders during the ASR exposure is not observed for the "fly ash" binder. Note that all test series are subjected to elevated temperature which is very favourable for the pozzolanic reaction.
- It is likely that the comparatively low RH found for the "dense" binder and probably also for the "fly ash" binder after 4 weeks of exposure contributes together with the measured lower relative diffusion coefficients to reduce the rate and extent of ASR. This is particularly true for the test series exposed to 38°C. Additionally, less amount of available (evaporable) water in the "dense" CEM I concrete (w/c of 0.30) might contribute to the lower extent of ASR observed compared with the CEM I concretes with higher w/c.
- For all test series with the CEM I binders with higher w/c (0.45 or 0.60), RH is regarded to be sufficient for ASR to initiate (well over 90 % RH after 4 weeks). Thus, it appears that for these binders all the specimen pre-treatment and test procedures provide sufficient moisture contents for ASR to proceed.

The consequences of the present results for alkali leaching and prisms expansion are treated in a separate paper [25].

A number of practical details and recommendations on the test procedures are given in the appendix to the thesis [44].

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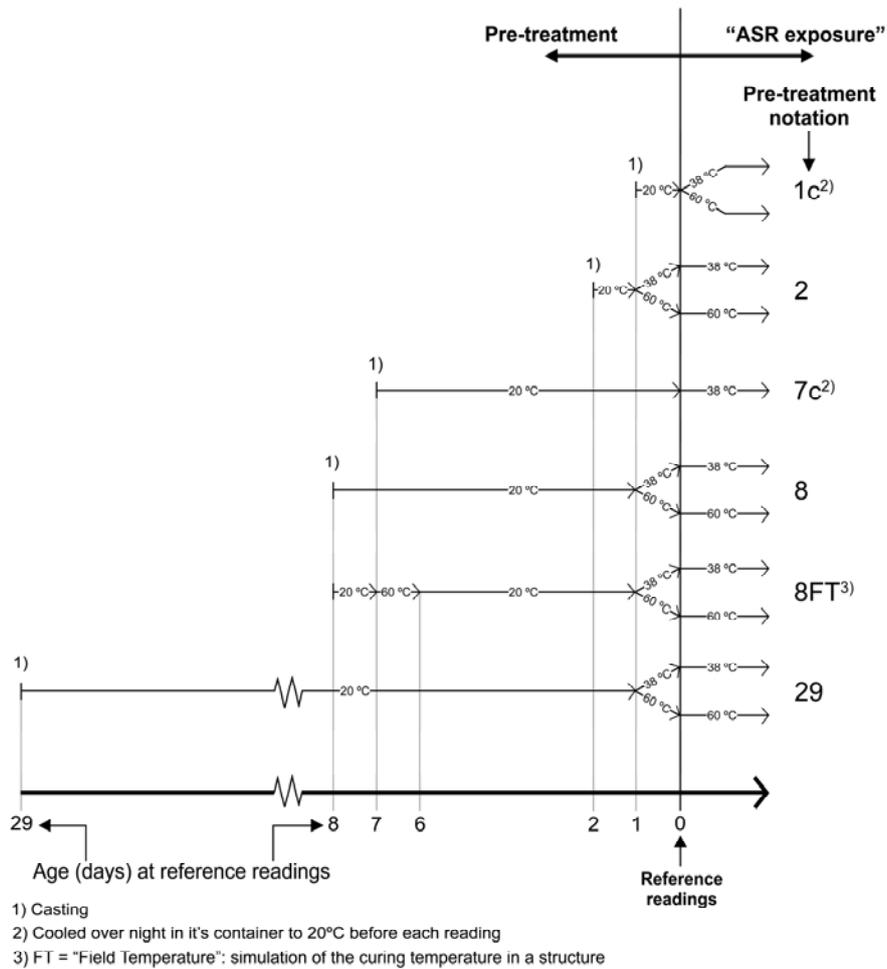


Figure 1 Pre-treatment of the various test series, included notations. For all test series, the prisms were prepared for final storage and put into their storage container immediately after de-moulding (and after the 0.5 h submersion period where used – see Figure 2 and Tables 5-8).

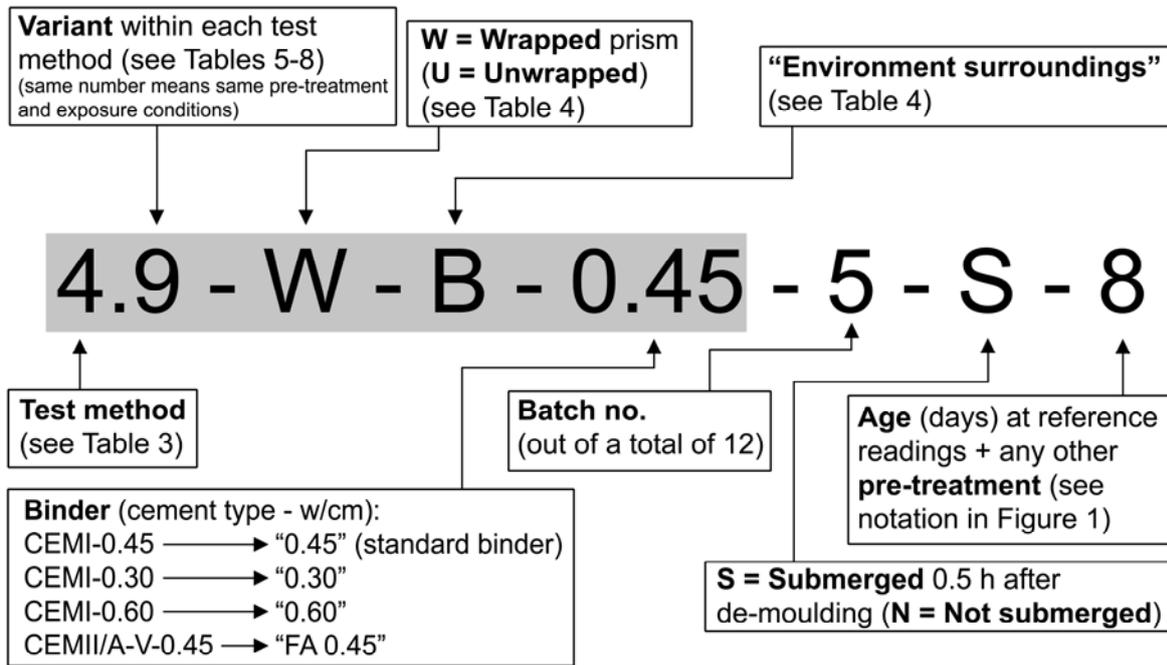


Figure 2 Notations used to name the various test series. The short names (see Tables 5-8) used in figures and tables when presenting results are marked with grey shadow. For repeated test series, the batch no. is additionally included in the short name.

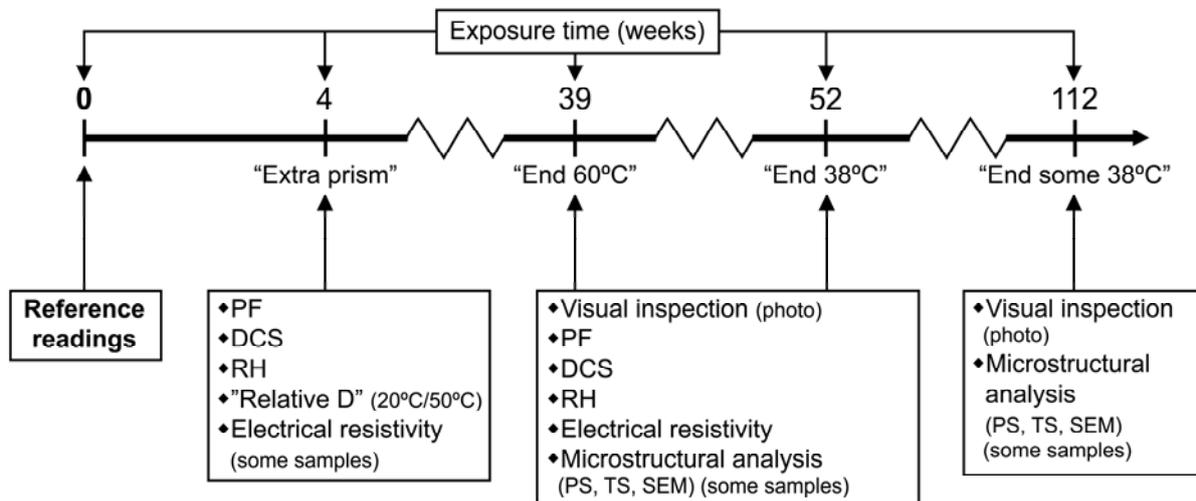


Figure 3 Tests performed on the concrete prisms after start ASR exposure.

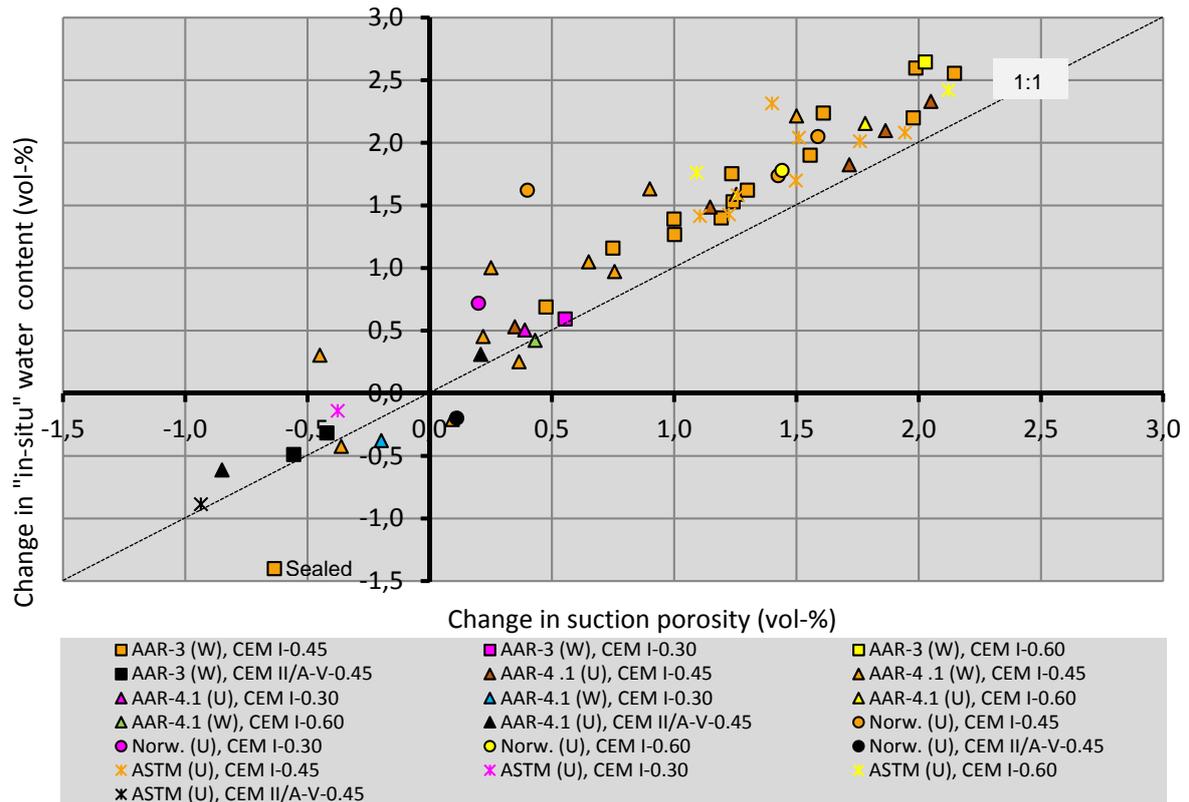


Figure 4. Change in evaporable "in-situ" water content in cut samples versus change in suction porosity from 4 weeks to 39 weeks (60°C) or 52 weeks (38°C) of exposure (W = Wrapped prisms; U = Unwrapped prisms).

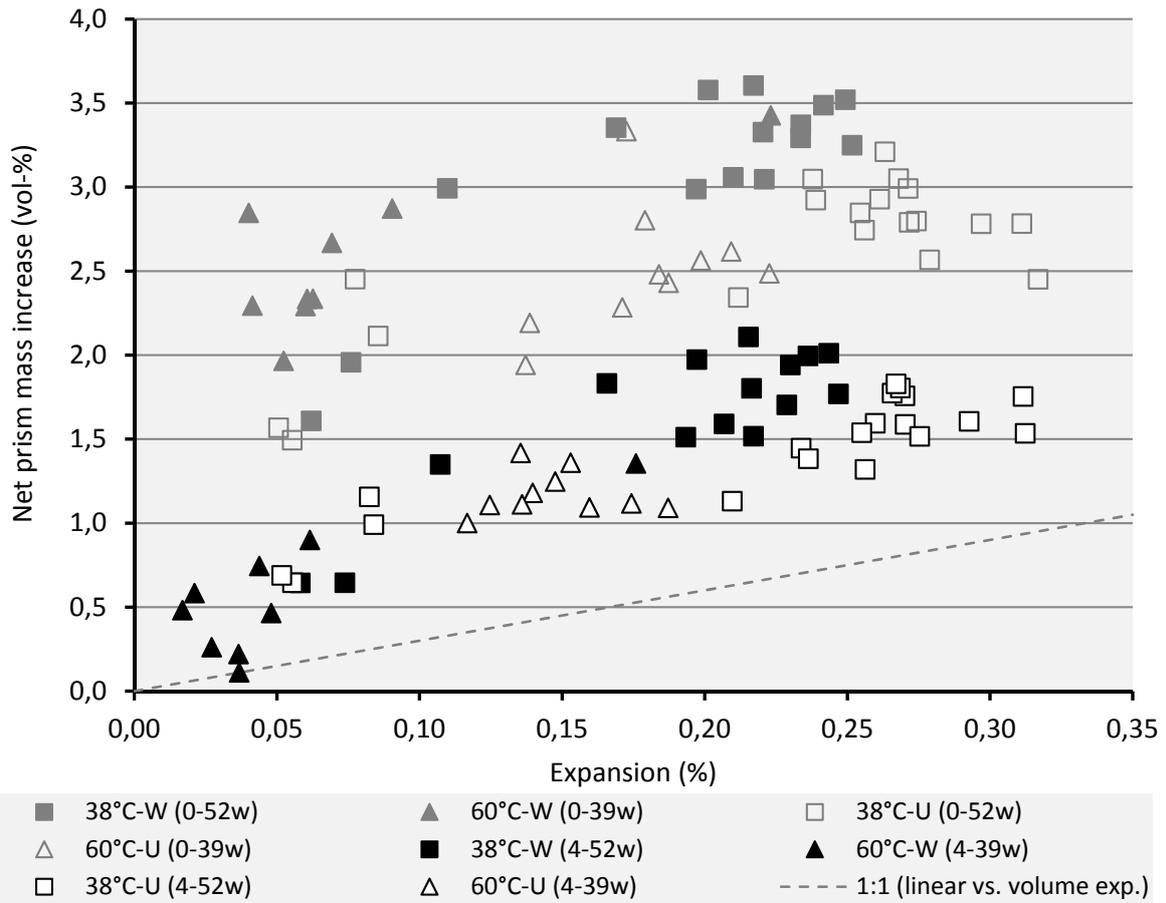


Figure 5. Increase in net mass of whole prisms (in vol-%) versus corresponding prism expansion in various time intervals; upper points (grey symbols) represent mass increase from de-moulding and prism expansion from reference readings until 39/52 weeks of exposure; lower points (black symbols) represent the time interval 4 weeks to 39/52 weeks of exposure. The sealed and submerged test series (see Table 5 and 6) are not included. (1 vol-% constitutes about 0.40 mass-%; W=Wrapped prisms; U=Unwrapped prisms; w=weeks).

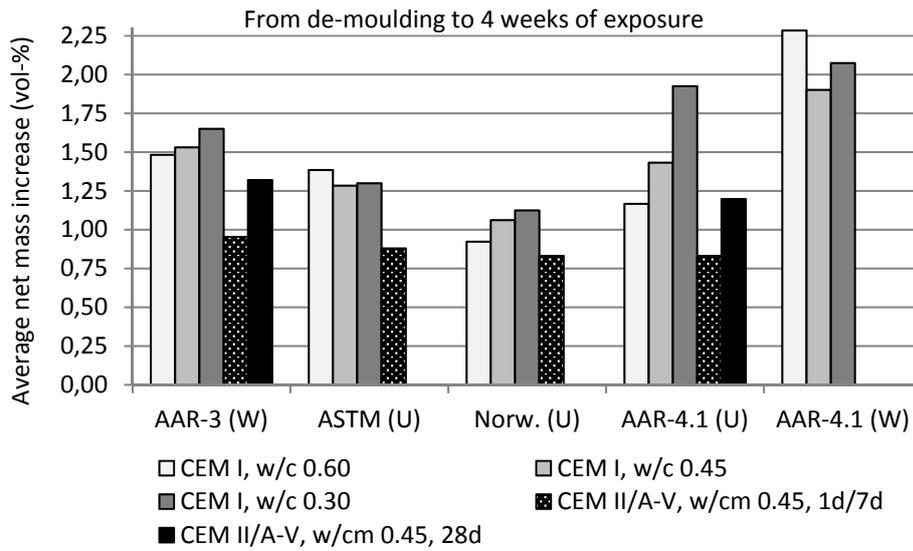


Figure 6. Increase in net mass of whole prisms (re-calculated to volume-% water absorption) from de-moulding to 4 weeks of exposure (W = Wrapped prisms; U = Unwrapped prisms; d = age at exposure to elevated temperature).

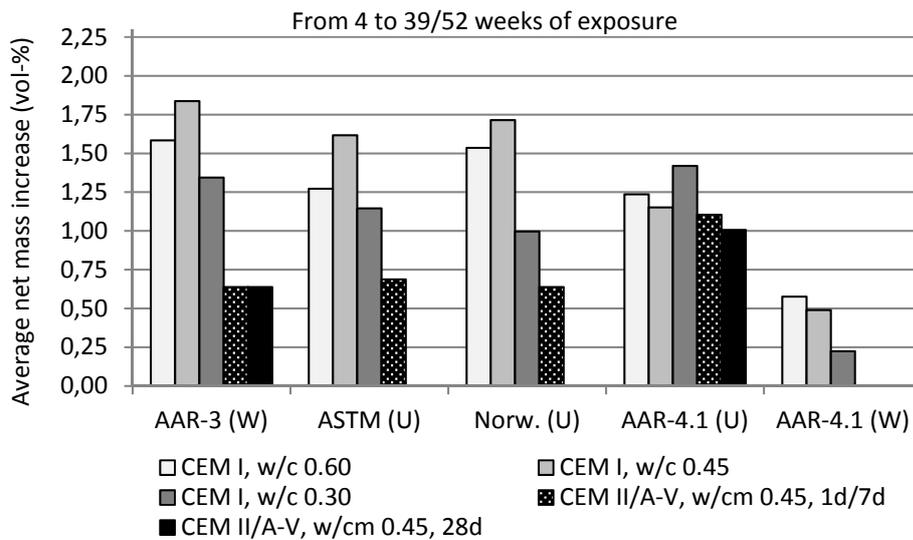


Figure 7. Increase in net mass of whole prisms (re-calculated to volume-% water absorption) from 4 weeks to 39 weeks (60°C) or 52 weeks (38°C) of exposure (W = Wrapped prisms; U = Unwrapped prisms; d = age at exposure to elevated temperature).

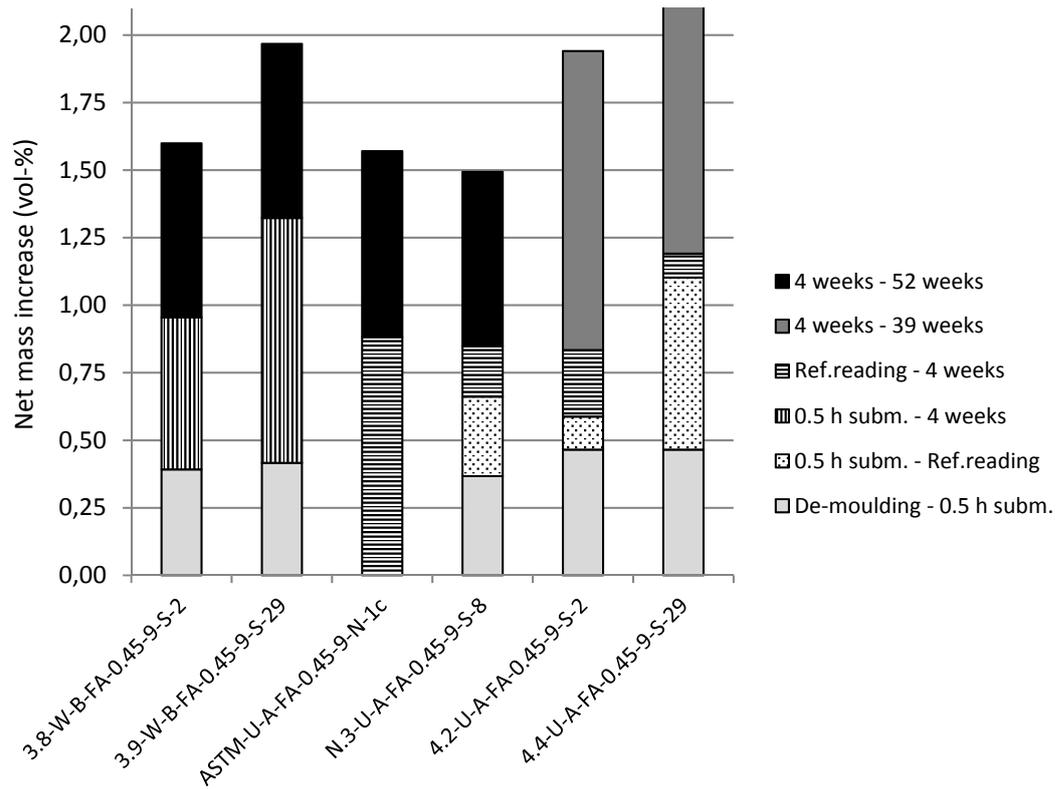


Figure 8. Increase in net mass of whole prisms (re-calculated to volume-% water absorption) from de-moulding to 39 weeks (60°C) or 52 weeks (38°C) of exposure for the six test series with the binder CEM II/A-V, w/cm of 0.45 (W = Wrapped prisms; U = Unwrapped prisms; see Figure 2 for explanations of notations for various test series).

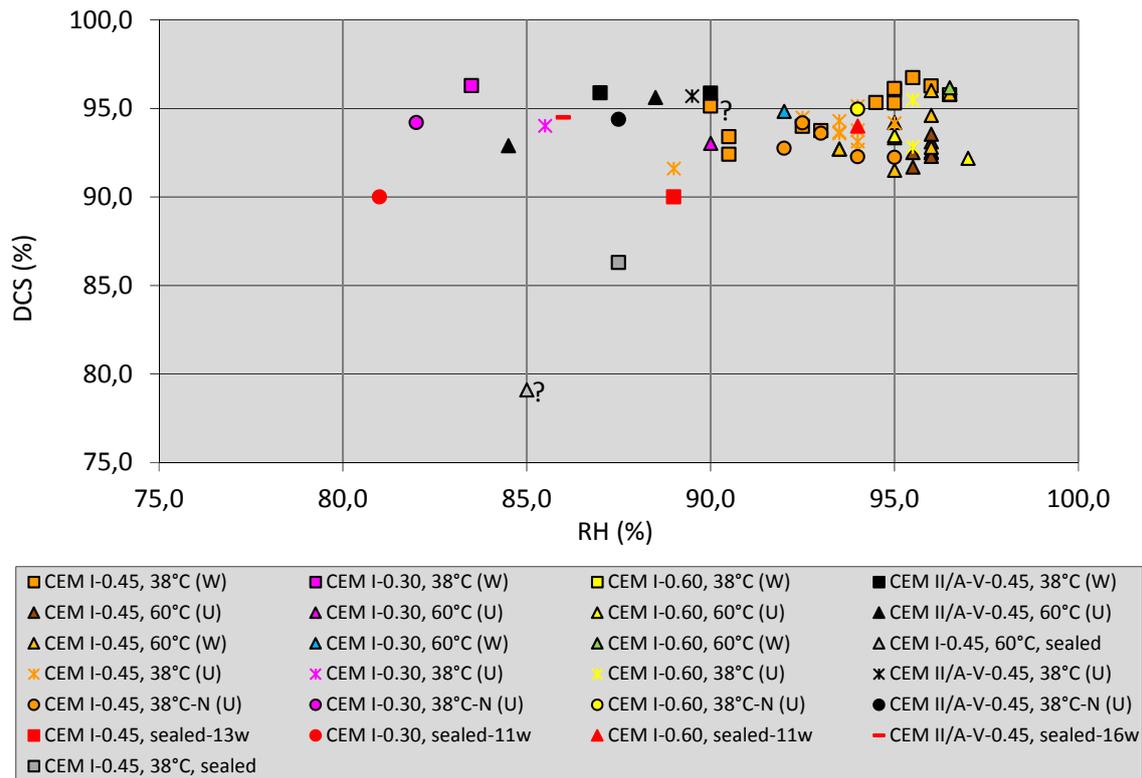


Figure 9. Degree of capillary saturation (DCS) and relative humidity (RH) after 4 weeks of exposure (W = Wrapped prisms; U = Unwrapped prisms; N = larger Norwegian prisms; red points = sealed cured at 20°C in plastic bottles; w = weeks; ? = some uncertainty related to the RH measurement).

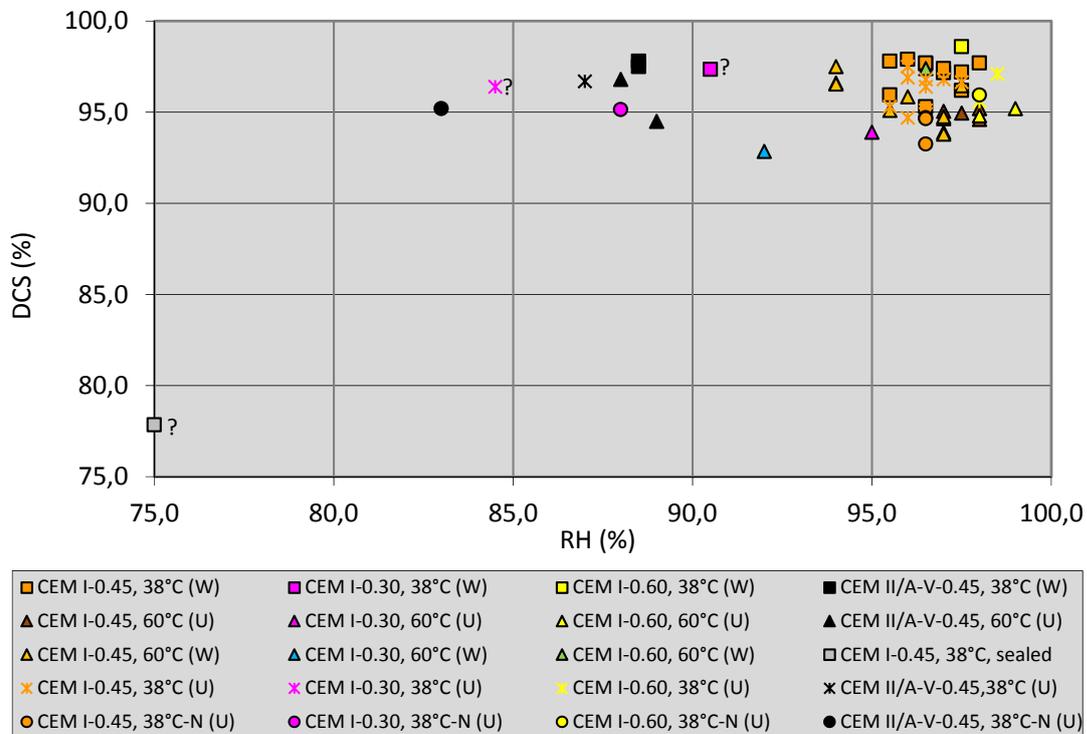


Figure 10. Degree of capillary saturation (DCS) and relative humidity (RH) after 39 weeks (60°C) or 52 weeks (38°C) of exposure (W = Wrapped prisms; U = Unwrapped prisms; N = larger Norwegian prisms; ? = some uncertainty related to the RH measurement).

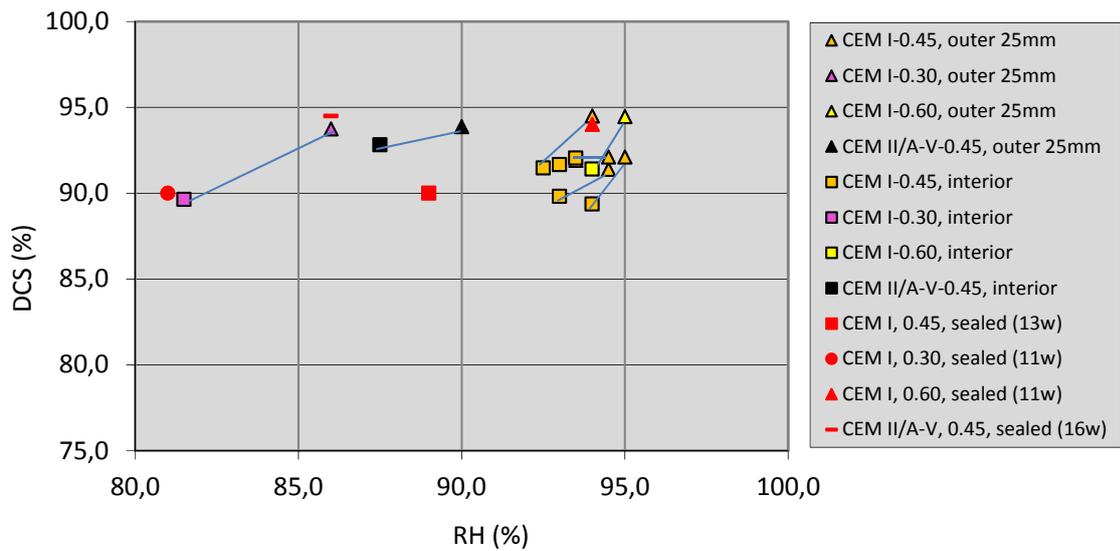


Figure 11. Degree of capillary saturation (DCS) and relative humidity (RH) in the outer 25 mm versus the interior of Norwegian concrete prisms after 4 weeks of exposure (red points = sealed cured at 20° C in plastic bottles; w = weeks).

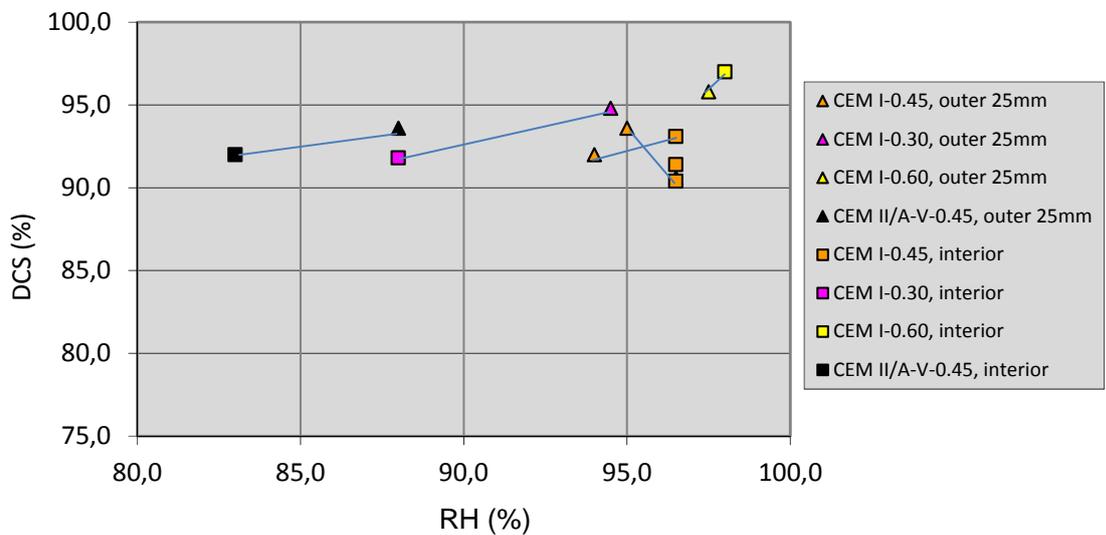


Figure 12. Degree of capillary saturation (DCS) and relative humidity (RH) in the outer 25 mm versus the interior of Norwegian concrete prisms after 52 weeks of exposure.

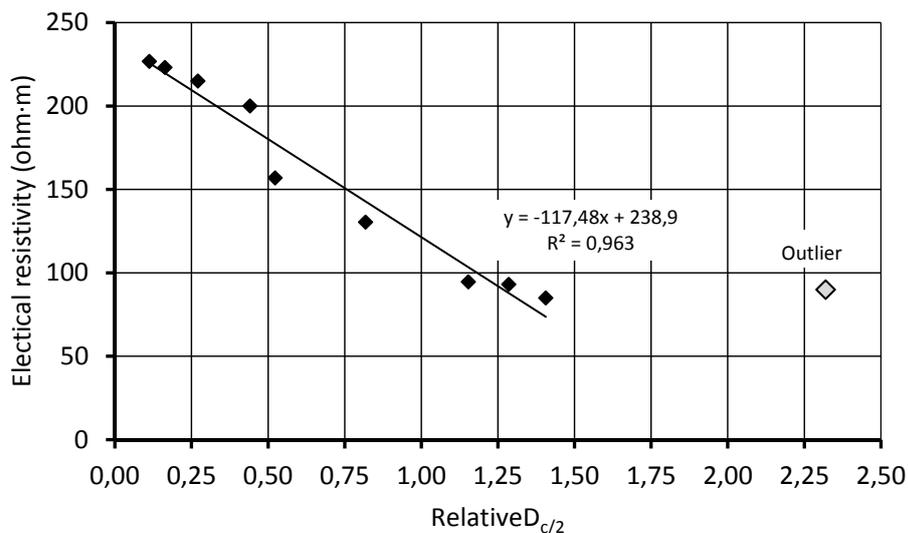


Figure 13. Results from "pilot" testing of electrical resistivity (measured on split "PF-samples" - procedure given in [48]) plotted against the corresponding relative diffusion coefficients after 4 weeks of exposure to 38 or 60°C. One outlier is removed when calculating the linear correlation ( $R^2$ ). The figure only includes CEM I cement with w/c in the range 0.30-0.60.

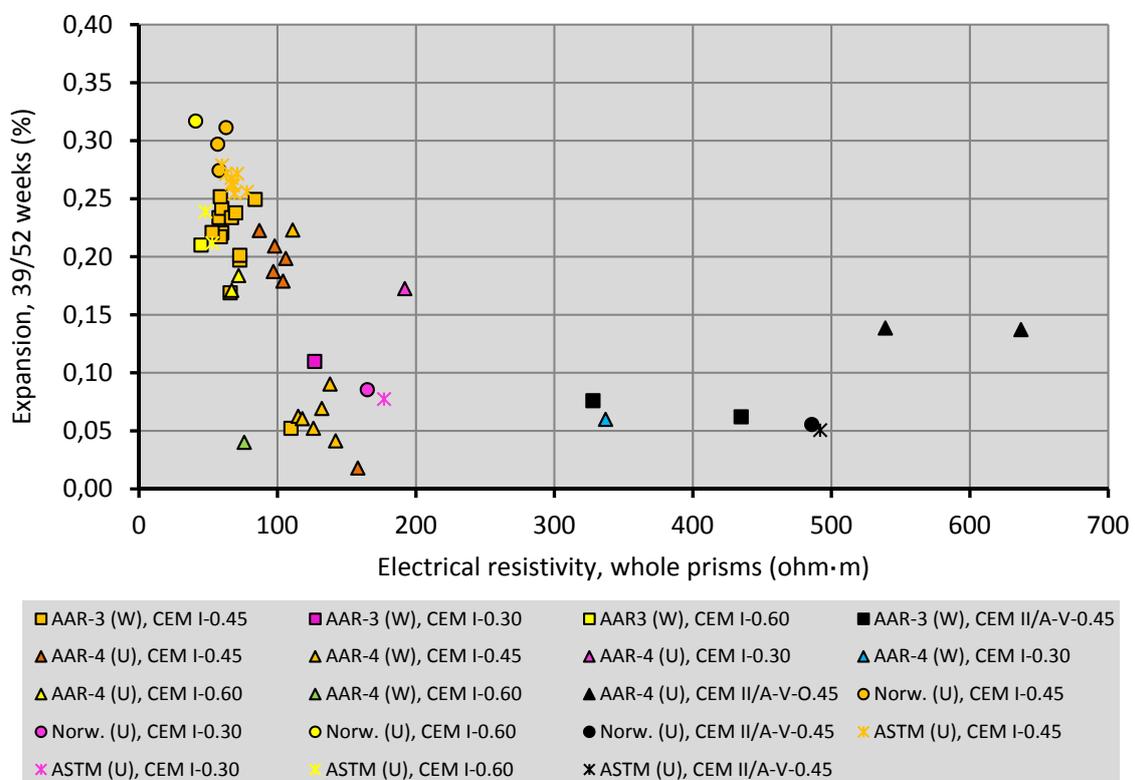


Figure 14. Relation between electrical resistivity and expansion after 39/52 weeks of exposure. Sealed test series are excluded.

Table 1. Chemical composition (EN 196-2) of the three cements used in the study.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	Na <sub>2</sub> O <sub>e</sub>	P <sub>2</sub> O <sub>5</sub>	LOI <sup>1</sup>
High alkali CEM I	19.61	4.87	3.48	61.03	2.83	3.81	1.11	0.51	1.24	0.15	2.44
Low alkali CEM I	20.06	4.67	3.31	63.06	2.01	3.40	0.39	0.34	0.60	0.16	2.24
CEM II/A-V <sup>2</sup>	26.61	8.73	4.24	50.34	2.37	3.28	1.04	0.56	1.25	0.33	1.20

<sup>1</sup> Loss-Of-Ignition.

<sup>2</sup> Blended cement with a class F fly ash content of 21.6 wt%. Manufactured by co-grinding clinker and fly ash. Normally, the content of fly ash is in the range of 17-20 %.

Table 2. Composition of the four concrete mixtures included in the study.

Materials (kg/m <sup>3</sup> )		Binder composition			
		CEM I, 0.45 "basis" binder	CEM I, 0.30 "dense" binder	CEM I, 0.60 "open" binder	CEM II/A-V, 0.45 "fly ash" binder
Cement	High alkali CEM I	200	60	285	---
	Low alkali CEM I	200	490	30	---
	CEM II/A-V	---	---	---	400
Aggregates (SSD <sup>1</sup> )	Årdal (gneiss/granite)	0/4	735	700	755
	Ottersbo (cataclasite)	4/8	185	175	190
		8/11	365	350	375
		11/16	550	525	565
Deionised water (free) (excl. any water in the superplasticizer)		180	165	189	180
NaOH (solids)		---	---	---	5.2
Alkali content (kg Na <sub>2</sub> O <sub>eq</sub> per m <sup>3</sup> )		3.7	3.7	3.7	9.0
Superplasticizer (SIKA SSP 2000)		If necessary, add until workable and stable concrete (aimed slump 120 mm)			
De-foaming agent (SIKA)		If measured air content is > 3.0 %, add until air content is reduced to < 3.0 %			

<sup>1</sup> Saturated surface dry condition

Table 3. Overview of the concrete prism tests (CPTs) included in the laboratory test program.

Method <sup>1</sup>	Notation (Figure 2)	Pre-treatment <sup>2</sup>	Storage conditions			Testing time (weeks)	Acceptance criteria	Binder composition (for aggregate testing)
			Temp. (°C)	Humidity <sup>3,4</sup>	Readings <sup>5</sup> (weeks)			
RILEM AAR-3	3	No submersion after de-moulding, but directly wrapping <sup>6</sup> . Ref. readings ~48h after casting	38±2 (from 7 days)	Each prism wrapped <sup>6</sup> in damp cotton cloth and polyethylene (prism ends not covered)	2,4,13,26,52	52	Single aggregate: <0.05% (52 weeks). (Same limit for alkali threshold with CEM I)	
RILEM AAR-4.1	4	Ref. readings directly after de-moulding, then 0.5h submersion in water	60±2 (from ~24 h)	Unwrapped prisms stored in a metal container (no lining). Several small containers stored in a larger container (“reactor”) on grids over water	5,10,15,20	20	Single aggregate: <0.03% (15 weeks). (Not allowed for measurement of alkali threshold for CEM I)	440kg/m <sup>3</sup> CEM I (0.9-1.2% Na <sub>2</sub> O <sub>eq</sub> ), w/c-ratio 0.50, 5.5 <sup>9</sup> kg/m <sup>3</sup> Na <sub>2</sub> O <sub>eq</sub> alkalis
RILEM AAR-4.1 Alt.	4	No submersion after de-moulding, but directly wrapping <sup>6</sup> before ref. readings are taken		Each prism wrapped <sup>6</sup> in damp cotton cloth and polyethylene (prism ends not covered). RILEM AAR-4.1 Alternative is similar to AAR-3, except the temp.				
ASTM C-1293	ASTM	No submersion after de-moulding, but directly ref. readings	38±2 (from ~24 h)	Unwrapped prisms stored in a plastic container – inside wall covered with a lining with lower part immersed in the deionised water in the bottom	1,4,8,13,26, 39,52 (78,104) <sup>7</sup>	52 (104) <sup>7</sup>	Single aggregate: <0.04% (52 weeks) Pozzolans/slag: <sup>7</sup> <0.04% (104 weeks)	420±10kg/m <sup>3</sup> CEM I (0.9±0.1% Na <sub>2</sub> O <sub>eq</sub> ), w/c-ratio 0.42-0.45, 5.25 <sup>9</sup> kg/m <sup>3</sup> Na <sub>2</sub> O <sub>eq</sub> alkalis
Norwegian CPT	N	Prisms submerged 0.5h in water after de-moulding, before ref. readings are taken		Unwrapped prisms stored in a plastic container – inside wall covered with a lining with lower part immersed in the deionised water in the bottom	1,8,16,26,52 (104) <sup>8</sup>	52 (104) <sup>8</sup>	Single aggregate: <0.040% (52 weeks) Pozzolans/slag: <sup>8</sup> <0.030% (52 weeks)	400±10kg/m <sup>3</sup> CEM I (1.15±0.10% Na <sub>2</sub> O <sub>eq</sub> ), w/c-ratio 0.48±0.02, 5.0 <sup>9</sup> kg/m <sup>3</sup> Na <sub>2</sub> O <sub>eq</sub> alkalis

<sup>1</sup> Three concrete prisms cast for each test series. Prism size ~75x75x250mm<sup>3</sup> (RILEM CPTs and ASTM C-1293) and 100x100x450mm<sup>3</sup> (Norwegian CPT), respectively.

<sup>2</sup> All prisms stored at ~ 20°C under a plastic sheet the first 24 hours, before de-moulding. Subsequent pre-treatment before exposed to elevated temp. is described below.

<sup>3</sup> In all CPTs, the prisms are stored vertically on grids above water, without being in direct contact with the water. A humid environment close to 100% RH is aimed.

<sup>4</sup> Except the RILEM AAR-4.1 CPT (“reactor”), the storage containers are stored in a hot, dry room or an oven.

<sup>5</sup> For all methods the readings are taken after cooling the prisms inside their storage container in a room kept at ~ 20°C. Any wrapping is not removed.

<sup>6</sup> ~80g deionised water added to each cotton cloth before wrapping. Each wrapped prism sealed inside a separate polyethylene bag. 5ml deionised water poured on top surface before sealing the bag and after each reading. Each bag is placed<sup>3</sup> in a separate container (plastic tube with inside lining in contact with the bottom water).

<sup>7</sup> For testing of combinations of aggregates and pozzolans or slag, a 104 weeks testing time is required. Not allowed for measurement of alkali threshold for CEM I.

<sup>8</sup> Other binders than CEM I (alkali threshold), pozzolans or slag must be tested for two years. An extra criterion must then be fulfilled: Expansion <0.060% (104 weeks).

<sup>9</sup> For all test methods, NaOH may be added to reach the aimed alkali content of the binder: 1.25% Na<sub>2</sub>O<sub>eq</sub>.

Table 4. Specimen environment during testing.

Notation	Specimen environment <sup>1</sup>	Comments
A	Three <u>unwrapped</u> prisms stored inside each container	Standard procedure for RILEM AAR-4.1, ASTM C-1293 and Norwegian CPT (see Table 3)
B <sup>3</sup>	Each prism was <u>wrapped</u> <sup>2</sup> in damp cotton cloth and polyethylene (prism ends not covered). Before wrapping, each cotton cloth was submerged for minimum 10 minutes in <u>80g</u> deionised water. Excess water not absorbed by the cotton cloth during submersion (~35-45g) was poured on the top surface before sealing the bag. This lead to a “water reservoir” in the bottom of each polyethylene bag, that after four weeks of exposure was measured to be in the range 4-25g for 38°C exposure and 0-2.5g for 60°C exposure	Standard procedure for RILEM AAR-3 and RILEM AAR-4.1 Alternative, including addition of 5ml deionised water on the top surface after every reading (see Table 3)
C <sup>3</sup>	Equal to “B”, except that each cotton cloth was submerged in half the amount of deionised water (i.e. <u>40g</u> ). All the water was absorbed by the cotton cloth	Motivation: Investigate the importance of the amount of water added to the wrapping
D	Equal to “B”, except that each wrapped prism was <u>not</u> sealed inside a separate <u>polyethylene bag</u> , but was placed on a grid inside the “AAR-3 container”. Neither was 5ml deionised water poured on the top surface at any time	Motivation: Investigate the importance of storing each wrapped prism in a separate polyethylene bag
E	After de-moulding, each prism was coated with <u>epoxy</u> . The next day the prisms were further <u>sealed</u> by packing them in <u>aluminium foil</u> . Further storage in dry containers	Motivation: Try to totally hinder any moisture exchange with the surroundings
F	After de-moulding, the unwrapped prisms were totally <u>submerged</u> in deionised water. After every reading, the water was exchanged with new deionised water	Motivation: Give the prisms the maximum alkali leaching conditions
G <sup>3</sup>	Equal to “B”, except each cotton cloth was submerged for minimum 10 minutes in a basic solution with <u>pH 14.2</u> (Na/K-ratio≈1/3) simulating the pH in the pore water of the “standard CEM I binder” after ~28 days of curing. No extra solution was poured on the top surface, beyond the ~60g absorbed by the cotton cloth when it was submerged	Motivation: Investigate if application of a similar pH in the cotton cloth as in the concrete pore water is able to hinder alkali leaching from the concrete prisms
H <sup>3</sup>	Equal to “G”, except each cotton cloth was submerged in a basic solution with <u>pH 13.2</u> (Na/K-ratio≈1/3) simulating a less basic pore solution. No extra solution was poured on the top surface, beyond the ~50g absorbed by the cotton cloth when it was submerged	Motivation: Investigate if application of a somewhat lower pH in the cotton cloth compared to the concrete pore water is able to reduce the extent of alkali leaching

<sup>1</sup> In all CPTs, the prisms are stored vertically on grids above water, without being in direct contact with the water.

A humid environment close to 100% RH is aimed. See more details about the storage containers in Table 3.

<sup>2</sup> Each wrapped prism was sealed inside a separate polyethylene bag. 5ml deionised water was poured on the top surface before sealing the bag and after each reading. Each bag was placed in a separate “AAR-3 container” with a tight lid.

<sup>3</sup> For 60°C storage, three wrapped prisms were stored in an “AAR-4.1 metal container” instead of single “AAR-3 containers”.

Table 5. Overview of the 18 test series with modified versions of the RILEM AAR-3 38°C CPT (2000) [3].

Test series			Comments
Short name <sup>1</sup>	Full notation <sup>2</sup>		
3.1-W-B-0.45	3.1-W-B-0.45-3-	N-7c	Standard RILEM AAR-3 test procedure ( <u>wrapped</u> prisms, 7 days pre-storage at 20°C, prisms cooled before every reading) <sup>3</sup>
3.2-W-B-0.45	3.2-W-B-0.45-4-	N-8	As 3.1-W-B-0.45, but readings taken without pre-cooling
3.3-W-C-0.45	3.3-W-C-0.45-4-	N-8	As 3.2-W-B-0.45, but less water in wrapping
3.4-W-B-0.45	3.4-W-B-0.45-4-	S-8	As 3.2-W-B-0.45, but prisms 0.5h submerged after de-moulding
3.5- -E-0.45	3.5- -E-0.45-5-	N-8	Sealed storage (epoxy and aluminium foil) after de-moulding (no water in the bottom of the storage containers)
3.6-W-D-0.45	3.6-W-D-0.45-6-	S-8	As 3.4-W-B-0.45, but no polyethylene bag
3.7-U- A-0.45	3.7-U -A-0.45-6-	S-8	As 3.4-W-B-0.45, but no wrapping (one prism in each container)
3.8-W-B-0.45	3.8-W-B-0.45-3-	S-2	As 3.4-W-B-0.45, but 1 day pre-storage at 20°C
3.9-W-B-0.45	3.9-W-B-0.45-2-	S-29	As 3.4-W-B-0.45, but 28 days pre-storage at 20°C
3.10-W-B-0.45-2	3.10-W-B-0.45- 2-	S-8FT	As 3.4-W-B-0.45, but simulating “field curing temperature” (see Fig. 1)
3.10-W-B-0.45-10	3.10-W-B-0.45-10-	S-8FT	As 3.10-W-B-0.45-2, but repeated test series
3.11-U- F-0.45	3.11-U -F-0.45-5-	S-8	Stored submerged in deionised water after de-moulding
3.12-W-G-0.45	3.12-W-G-0.45-12-	S-8	As 3.4-W-B-0.45, but pH 14.2 in wrapping at start
3.13-W-H-0.45	3.13-W-H-0.45-12-	S-8	As 3.4-W-B-0.45, but pH 13.2 in wrapping at start
3.4-W-B-0.30	3.4-W-B-0.30-7-	S-8	As 3.4-W-B-0.45, but lower w/c ratio
3.4-W-B-0.60	3.4-W-B-0.60-8-	S-8	As 3.4-W-B-0.45, but higher w/c ratio
3.8-W-B-FA-0.45	3.8-W-B-FA-0.45-9-S-2	S-2	As 3.8-W-B-0.45, but fly ash binder with boosted alkali level
3.9-W-B-FA-0.45	3.9-W-B-FA-0.45-9-S-29	S-29	As 3.9-W-B-0.45, but fly ash binder with boosted alkali level

<sup>1</sup> Used in figures and tables when presenting results.

<sup>2</sup> See details in Figure 2.

<sup>3</sup> See details in Table 3.

Table 6. Overview of the 22 test series with modified versions of the RILEM AAR-4.1 60°C CPT (2006) [5].

Test series			Comments
Short name <sup>1</sup>	Full notation <sup>2</sup>		
4.1-U-A-0.45	4.1-U -A-0.45-1-	S-1c	Standard RILEM AAR-4.1 test procedure ( <u>unwrapped</u> prisms, “reactor”, 1 day pre-storage at 20°C, prisms 0.5h submerged after de-moulding and cooled before every reading) <sup>3</sup>
4.2-U-A-0.45	4.2-U -A-0.45-1-	S-2	As 4.1-U-A-0.45, but readings taken without pre-cooling
4.3-U-A-0.45	4.3-U -A-0.45-6-	S-8	As 4.2-U-A-0.45, but 7 days pre-storage at 20°C
4.4-U-A-0.45	4.4-U -A-0.45-2-	S-29	As 4.3-U-A-0.45, but 28 days pre-storage at 20°C
4.5-U-A-0.45	4.5-U -A-0.45-2-	S-8FT	As 4.3-U-A-0.45, but simulating “field curing temperature” (see Fig. 1)
4.6-U-F-0.45	4.6-U -F-0.45-5-	S-8	Stored submerged in deionised water after de-moulding
4.7- -E-0.45	4.7- -E-0.45-5-	N-8	Sealed storage (epoxy and aluminium foil) after de-moulding (no water in the bottom of the storage container)
4.8-W-B-0.45-1	4.8-W-B-0.45-1-	S-2	Standard RILEM AAR-4.1 Alt. test procedure ( <u>wrapped</u> prisms <sup>3</sup> , 1 day pre-storage at 20°C), except prisms 0.5h submerged after de-moulding and readings taken without pre-cooling
4.8-W-B-0.45-10	4.8-W-B-0.45-10-	S-2	As 4.8-W-B-0.45-1, but repeated test series
4.9-W-B-0.45	4.9-W-B-0.45-5-	S-8	As 4.8-W-B-0.45-1, but 7 days pre-storage at 20°C
4.10-W-C-0.45-6	4.10-W-C-0.45-6-	N-8	As 4.9-W-B-0.45, but less water in wrapping
4.10-W-C-0.45-12	4.10-W-C-0.45-12-	N-8	As 4.10-W-B-0.45-6, but repeated test series
4.11-W-D-0.45	4.11-W-D-0.45-3-	S-8	As 4.9-W-B-0.45, but no polyethylene bag
4.12-W-G-0.45	4.12-W-G-0.45-12-	S-8	As 4.9-W-B-0.45, but pH 14.2 in wrapping at start
4.13-W-H-0.45	4.13-W-H-0.45-12-	S-8	As 4.9-W-B-0.45, but pH 13.2 in wrapping at start
4.3-U-A-0.30	4.3-U -A-0.30-7-	S-8	As 4.3-U-A-0.45, but lower w/c ratio
4.9-W-B-0.30	4.9-W-B-0.30-7-	S-8	As 4.9-W-B-0.45, but lower w/c ratio
4.3-U-A-0.60-I	4.3-U -A-0.60-8-I	S-8	As 4.3-U-A-0.45, but higher w/c ratio
4.3-U-A-0.60-II	4.3-U -A-0.60-8-II	S-8	As 4.3-U-A-0.60-I, but repeated test series
4.9-W-B-0.60	4.9-W-B-0.60-11-	S-8	As 4.9-W-B-0.45, but higher w/c ratio
4.2-U-A-FA-0.45	4.2-U -A-FA-0.45-9-S-2	S-2	As 4.2-U-A-0.45, but fly ash binder with boosted alkali level
4.4-U-A-FA-0.45	4.4-U -A-FA-0.45-9-S-29	S-29	As 4.4-U-A-0.45, but fly ash binder with boosted alkali level

<sup>1</sup> Used in figures and tables when presenting results.

<sup>2</sup> See details in Figure 2.

<sup>3</sup> See details in Table 3.

Table 7. Overview of the 6 test series with modified versions of the Norwegian 38°C CPT [18].

Test series			Comments
Short name <sup>1</sup>	Full notation <sup>2</sup>		
N.1-U-0.45	N.1-U- A-0.45-3-	S-1c	<u>Standard Norwegian CPT</u> procedure (1 day pre-storage at 20°C, prisms 0.5h submerged after de-moulding and cooled before every reading) <sup>3</sup>
N.2-U-0.45	N.2-U- A-0.45-1-	S-2	As N.1-U-0.45, but readings taken without pre-cooling
N.3-U-0.45	N.3-U- A-0.45-4-	S-8	As N.2-U-0.45, but 7 days pre-storage at 20°C
N.3-U-0.30	N.3-U- A-0.30-7-	S-8	As N.3-U-0.45, but lower w/c ratio
N.3-U-0.60	N.3-U- A-0.60-8-	S-8	As N.3-U-0.45, but higher w/c ratio
N.3-U-FA-0.45	N.3-U- A-FA-0.45-9-S-8		As N.3-U-0.45, but fly ash binder with boosted alkali level

<sup>1</sup> Used in figures and tables when presenting results.

<sup>2</sup> See details in Figure 2.

<sup>3</sup> See details in Table 3.

Table 8. Overview of the 12 test series with the ASTM C-1293 38°C CPT [9].

Test series			Comments
Short name <sup>1</sup>	Full notation <sup>2</sup>		
ASTM-U-0.45-1	ASTM-U-A-0.45-1-	N-1c	<u>Standard ASTM C-1293</u> test procedure (1 day pre-storage at 20°C, prisms cooled before every reading) <sup>3</sup>
ASTM-U-0.45-2	ASTM-U-A-0.45-2-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-3	ASTM-U-A-0.45-3-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-4	ASTM-U-A-0.45-4-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-5	ASTM-U-A-0.45-5-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-6	ASTM-U-A-0.45-6-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-10	ASTM-U-A-0.45-10-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-12	ASTM-U-A-0.45-12-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.30-7	ASTM-U-A-0.30-7-	N-1c	As ASTM-U-0.45-1, but lower w/c ratio
ASTM-U-0.60-8	ASTM-U-A-0.60-8-	N-1c	As ASTM-U-0.45-1, but higher w/c ratio
ASTM-U-0.60-11	ASTM-U-A-0.60-11-	N-1c	As ASTM-U-0.60-8, but repeated test series
ASTM-U-FA-0.45-9	ASTM-U-A-FA-0.45-9-	N-1c	As ASTM-U-0.45-1, but fly ash binder with boosted alkali level

<sup>1</sup> Used in figures and tables when presenting results.

<sup>2</sup> See details in Figure 2.

<sup>3</sup> See details in Table 3.

Table 9. Quality control of the 12 concrete mixes.

Binder composition	Density <sup>3</sup>	Compr. strength, cubes 28 days		Suction porosity <sup>4</sup> , 4 weeks of exposure		Macro porosity <sup>4</sup> , 4 weeks of exposure		Solid density <sup>4</sup> , 4 weeks of exposure	
	$Q_{\text{mean}}$ (kg/m <sup>3</sup> )	$f_{\text{c-mean}}$ (MPa)	C.O.V. (%)	$\epsilon_{\text{suc-mean}}$ (vol-%)	C.O.V. (%)	$\epsilon_{\text{air-mean}}$ (vol-%)	C.O.V. (%)	$Q_{\text{mean}}$ (kg/m <sup>3</sup> )	C.O.V. (%)
CEM I, w/c 0.60 <sup>1</sup>	2450	49.7	1.6	13.3	4.8	1.9	15	2715	0.3
CEM I, w/c 0.45 <sup>2</sup>	2450	70.0	2.1	12.0	3.2	1.8	22	2720	0.2
CEM I, w/c 0.30	2520	103.4	--	10.4	--	1.9	--	2725	--
CEM II/A-V, w/cm 0.45	2450	44.4	--	14.0	--	1.4	--	2735	--

<sup>1</sup> Two batches prepared

<sup>2</sup> Eight batches prepared

<sup>3</sup> Saturated surface dry condition, measured on the compressive strength cubes

<sup>4</sup> Measured on "PF-samples" cut from the ASTM test series (see section 2.4.2)

Table 10. Porosity and internal moisture state for the 18 RILEM AAR-3 38°C CPT series (numbers in brackets are somewhat uncertain).

Test series		Suction porosity (%)		Macro porosity (%)		Water content ("in-situ") (mass-%)			DCS (%)			RH (%)		
		4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$
3.1-W-B-0.45-3-	N-7c	11.9	1.3	1.9	0.5	4.9	6.0	1.1	93.7	97.2	3.4	93.0	97.0	4.0
3.2-W-B-0.45-4-	N-8	11.5	2.1	1.4	0.1	4.9	6.0	1.1	96.3	97.7	1.4	96.0	96.5	0.5
3.3-W-C-0.45-4-	N-8	11.9	1.1	1.5	0.1	4.9	5.6	0.6	95.1	95.3	0.2	(90.0)	96.5	(6.5)
3.4-W-B-0.45-4-	S-8	11.7	1.4	1.5	0.0	5.0	5.8	0.8	96.0	97.7	1.7	95.0	96.5	1.5
3.5- -E-0.45-5-	N-8	12.0	-0.3	2.1	-0.1	4.5	3.9	-0.6	86.3	77.9	-8.4	87.5	(75.0)	(-12.5)
3.6-W-D-0.45-6-	S-8	12.1	0.8	2.3	-0.4	5.1	5.8	0.7	95.3	97.4	2.1	94.5	97.0	2.5
3.7-U -A-0.45-6-	S-8	12.2	0.6	2.1	0.3	4.9	5.7	0.8	93.7	96.7	3.0	93.5	97.5	4.0
3.8-W-B-0.45-3-	S-2	12.2	1.3	1.8	0.3	5.1	6.0	0.9	96.7	96.2	-0.5	95.5	97.5	2.0
3.9-W-B-0.45-2-	S-29	11.7	1.3	1.1	0.6	4.9	5.9	1.0	94.0	97.2	3.2	92.5	97.5	5.0
3.10-W-B-0.45- 2-	S-8FT	11.8	0.9	1.2	0.2	4.9	5.6	0.7	95.3	97.7	2.4	95.0	98.0	3.0
3.10-W-B-0.45-10-	S-8FT	12.4	0.1	1.8	-0.5	5.3	5.6	0.3	96.1	97.9	1.8	95.0	96.0	1.0
3.11-U -F-0.45-5-	S-8	12.2	1.0	1.7	0.1	5.1	5.6	0.5	95.1	96.8	1.7	94.0	97.0	3.0
3.12-W-G-0.45-12-	S-8	11.6	0.6	1.5	-0.2	4.7	5.3	0.6	92.4	96.0	3.6	90.5	95.5	5.0
3.13-W-H-0.45-12-	S-8	11.8	0.3	1.5	0.1	4.9	5.4	0.5	93.4	97.8	4.4	90.5	95.5	5.0
3.4-W-B-0.30-7-	S-8	10.2	-0.3	1.8	0.0	4.1	4.4	0.2	96.3	97.4	1.1	83.5	(90.5)	(7.0)
3.4-W-B-0.60-8-	S-8	12.5	2.5	1.7	0.4	5.4	6.6	1.2	95.8	98.6	2.8	96.5	97.5	1.0
3.8-W-B-FA-0.45-9-S-2		13.9	-0.9	1.4	-0.4	5.6	5.4	-0.2	95.9	97.5	1.6	90.0	88.5	-1.5
3.9-W-B-FA-0.45-9-S-29		14.0	-0.4	1.2	-0.4	5.8	5.7	-0.1	95.9	97.8	1.9	87.0	88.5	1.5

Table 11. Porosity and internal moisture state for the 22 RILEM AAR-4.1 60°C CPT series (numbers in brackets are somewhat uncertain).

Test series	Suction porosity (%)		Macro porosity (%)		Water content ("in-situ") (mass-%)			DCS (%)			RH (%)		
	4 weeks	$\Delta_{4-39}$ weeks	4 weeks	$\Delta_{4-39}$ weeks	4 weeks	39 weeks	$\Delta_{4-39}$ weeks	4 weeks	39 weeks	$\Delta_{4-39}$ weeks	4 weeks	39 weeks	$\Delta_{4-39}$ weeks
4.1-U -A-0.45-1- S-1c	11.7	1.2	2.1	-0.5	4.6	5.6	1.0	92.7	95.2	2.5	95.5	98.0	2.5
4.2-U -A-0.45-1- S-2	11.9	0.9	2.0	-0.3	4.8	5.6	0.8	93.5	94.7	1.1	96.0	97.0	1.0
4.3-U -A-0.45-6- S-8	12.5	-0.3	2.1	-0.2	5.1	5.3	0.2	92.6	95.1	2.5	96.0	97.0	1.0
4.4-U -A-0.45-2- S-29	11.7	0.8	1.2	0.2	4.8	5.4	0.6	92.4	95.0	2.5	96.0	97.5	1.5
4.5-U -A-0.45-2- S-8FT	11.6	1.2	1.1	0.2	4.6	5.5	0.9	92.5	94.6	2.1	95.5	98.0	2.5
4.6-U -F-0.45-5- S-8	12.0 <sup>1</sup>	--	2.3 <sup>1</sup>	--	--	4.9	--	--	93.9	--	--	97.0	--
4.7- -E-0.45-5- N-8	11.7	0.0	2.0	0.3	4.1	4.3	0.2	79.1	84.8	5.7	(85.0)	--	--
4.8-W-B-0.45-1- S-2	12.0	-0.4	1.9	-0.2	5.0	5.1	0.1	95.8	95.9	0.0	96.5	96.0	-0.5
4.8-W-B-0.45-10- S-2	12.1	-0.2	1.5	-0.2	5.2	5.1	-0.1	96.0	95.1	-0.9	96.0	95.5	-0.5
4.9-W-B-0.45-5- S-8	12.0	-0.2	1.6	0.1	5.0	4.8	-0.2	94.2	93.8	-0.4	95.0	97.0	2.0
4.10-W-C-0.45-6- N-8	11.9	0.3	2.1	0.0	4.9	5.3	0.4	92.8	94.8	2.0	96.0	97.0	1.0
4.10-W-C-0.45-12- N-8	10.8	1.3	1.5	-0.3	4.4	5.1	0.7	91.5	96.6	5.1	95.0	94.0	-1.0
4.11-W-D-0.45-3- S-8	11.5	-0.1	1.8	0.6	4.8	5.0	0.2	94.6	96.5	1.8	96.0	97.5	1.5
4.12-W-G-0.45-12- S-8	11.1	1.8	1.4	-0.2	4.6	5.6	1.0	92.7	97.5	4.8	93.5	94.0	0.5
4.13-W-H-0.45-12- S-8	10.5	1.1	1.4	-0.2	4.4	4.9	0.4	92.7	96.6	3.8	93.5	94.0	0.5
4.3-U -A-0.30-7- S-8	10.2	-0.2	1.8	-0.1	4.1	4.4	0.2	93.0	93.9	0.9	90.0	95.0	5.0
4.9-W-B-0.30-7- S-8	9.7	-0.4	1.7	0.0	4.0	3.8	-0.2	94.8	92.9	-2.0	92.0	92.0	0.0
4.3-U -A-0.60-8-I S-8	13.2	1.6	1.6	0.2	5.4	6.3	0.9	93.5	95.2	1.7	95.0	99.0	4.0
4.3-U -A-0.60-8-II S-8	12.9	1.4	1.8	-0.2	5.2	5.9	0.7	92.2	94.8	2.6	97.0	98.0	1.0
4.9-W-B-0.60-11- S-8	13.4	-0.1	2.3	-0.7	5.7	5.9	0.2	96.2	97.4	1.2	96.5	96.5	0.0
4.2-U -A-FA-0.45-9-S-2	14.0	-0.5	1.3	-0.3	5.6	5.8	0.1	95.6	96.8	1.2	88.5	88.0	-0.5
4.4-U -A-FA-0.45-9-S-29	12.6	-0.5	1.1	0.0	5.1	4.9	-0.3	92.9	94.5	1.6	84.5	89.0	4.5

<sup>1</sup> 39 weeks of exposure

Table 12. Porosity and internal moisture state for the 6 Norwegian 38°C CPT series.

Test series	Suction porosity (%)		Macro porosity (%)		Water content ("in-situ") (mass-%)			DCS (%)			RH (%)		
	4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$
	N.1-U- A-0.45-3- S-1c	12.2	0.7	2.1	-0.1	4.8	5.5	0.7	92.3	93.3	1.0	94.0	96.5
N.2-U- A-0.45-1- S-2	11.9	1.0	2.1	-0.1	4.8	5.6	0.8	93.9	94.7	0.8	93.0	96.5	3.5
N.3-U- A-0.45-4- S-8	12.1	1.3	1.7	-0.2	4.9	5.8	0.9	92.7	94.7	1.9	93.0	96.5	3.5
N.3-U- A-0.30-7- S-8	10.1	-0.3	2.0	0.1	3.8	4.1	0.3	94.2	95.2	1.0	82.0	88.0	6.0
N.3-U- A-0.60-8- S-8	13.6	2.0	2.1	0.4	5.8	6.6	0.8	95.0	95.9	1.0	94.0	98.0	4.0
N.3-U- A-FA-0.45-9-S-8	13.9	-0.6	1.4	-0.2	5.5	5.5	-0.1	94.4	95.2	0.8	87.5	83.0	-4.5

Table 13. Porosity and internal moisture state for the 12 ASTM C-1293 38°C CPT series (numbers in brackets are somewhat uncertain).

Test series	Suction porosity (%)		Macro porosity (%)		Water content ("in-situ") (mass-%)			DCS (%)			RH (%)		
	4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$	4 weeks	52 weeks	$\Delta_{4-52 \text{ weeks}}$
	ASTM-U-A-0.45-1- N-1c	12.3	0.6	2.2	-0.2	5.0	5.6	0.6	92.7	95.3	2.6	94.0	95.5
ASTM-U-A-0.45-2- N-1c	12.1	0.5	1.4	-0.1	4.8	5.5	0.7	94.3	96.9	2.6	(93.5)	96.0	(2.5)
ASTM-U-A-0.45-3- N-1c	11.9	1.1	1.9	0.3	4.7	5.6	0.9	94.2	94.7	0.5	95.0	96.0	1.0
ASTM-U-A-0.45-4- N-1c	11.6	1.1	1.4	0.1	4.6	5.5	0.9	93.1	96.4	3.3	94.0	96.5	2.5
ASTM-U-A-0.45-5- N-1c	12.6	0.6	1.9	0.3	5.2	5.8	0.6	93.6	95.1	1.5	93.5	96.5	3.0
ASTM-U-A-0.45-6- N-1c	12.3	1.2	2.5	-0.4	5.1	5.8	0.7	93.8	94.9	1.1	94.0	96.5	2.5
ASTM-U-A-0.45-10- N-1c	11.5	1.3	1.5	-0.1	4.7	5.6	0.9	94.5	96.8	2.3	92.5	96.5	4.0
ASTM-U-A-0.45-12- N-1c	11.8	0.8	1.6	0.0	4.7	5.7	1.0	91.6	97.5	5.9	89.0	96.0	7.0
ASTM-U-A-0.30-7- N-1c	10.4	-0.9	1.9	-0.2	4.2	4.1	-0.1	94.0	96.4	2.4	85.5	(84.5)	(-1.0)
ASTM-U-A-0.60-8- N-1c	12.8	1.7	1.7	0.7	5.3	6.4	1.1	95.5	95.6	0.1	95.5	98.0	2.5
ASTM-U-A-0.60-11- N-1c	13.7	0.6	2.1	-0.1	5.6	6.4	0.8	92.8	97.1	4.3	95.5	98.5	3.0
ASTM-U-A-FA-0.45-9- N-1c	14.0	-1.3	1.4	-0.3	5.7	5.3	-0.4	95.7	96.7	1.0	89.5	87.0	-2.5