

## EUROPEAN ASSESSMENT DOCUMENT

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# NATURAL CALCINED POZZOLANA AS TYPE II ADDITION FOR CONCRETE

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This European Assessment Document (EAD) has been developed taking into account up-to-date technical and scientific knowledge at the time of issue and is published in accordance with the relevant provisions of Regulation No (EU) 305/2011 as a basis for the preparation and issuing of European Technical Assessments (ETA).

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## 1 SCOPE OF THE EAD

### 1.1 Description of the construction product

The product is a natural calcined pozzolana of volcanic origin (defined origin), that is finely divided and activated by thermal treatment (450°C). It consists essentially of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The content of reactive SiO<sub>2</sub>, as defined and described in EN 197-1, amounts to at least 25 % by mass (acc. to EN 450-1).

Mineralogically the pozzolana consists mainly of the minerals zeolite, alkali-feldspar, aegirine augite and wollastonite. Other components are calcite, götzenite, melanite, apatite, titanite.

The product is not covered by a harmonised European standard (hEN).

Concerning product packaging, transport, storage, maintenance, replacement and repair it is the responsibility of the manufacturer to undertake the appropriate measures and to advise his clients on the transport, storage, maintenance, replacement and repair of the product as he considers necessary.

It is assumed that the product will be installed according to the manufacturer's instructions or (in absence of such instructions) according to the usual practice of the building professionals.

Relevant manufacturer's stipulations having influence on the performance of the product covered by this European Assessment Document shall be considered for the determination of the performance and detailed in the ETA.

### 1.2 Information on the intended use(s) of the construction product

#### 1.2.1 Intended uses

The product is a type II addition (pozzolanic) for production of concrete, including in particular cast-in-situ or prefabricated structural concrete conforming to European standard EN 206.

"Natural calcined pozzolana as type II addition" is also intended to be used in mortars and grouts.

The recommended maximum replacement of cement by "natural calcined pozzolana as type II addition" is 25 % by mass. The maximum content of addition to be taken into account is 33 % by mass of the cement content.

The product is intended to be used in concrete with all types of common cement acc. to EN 197-1. It is also intended to be used with verified k-value concept in concrete made of Portland cement CEM I, and, if requested, further types of common cement acc. to EN 197-1.

Therefore the types of cements for which the suitability of the k-value concept is verified are CEM I, and, if requested, further types of common cement acc. to EN 197-1. The k-value concept can be used for all exposure classes acc. to EN 206 except XF2 and XF4.

#### 1.2.2 Working life/Durability

The assessment methods included or referred to in this EAD have been written based on the manufacturer's request to take into account a working life of concrete incorporating the "natural calcined pozzolana as type II addition" for the intended use of 50 years when installed in the works provided that the concrete incorporating the "natural calcined pozzolana as type II addition" is subject to appropriate installation (see 1.1)). These provisions are based upon the current state of the art and the available knowledge and experience.

When assessing the product, the intended use as foreseen by the manufacturer shall be taken into account. The real working life may be, in normal use conditions, considerably longer without major degradation affecting the basic requirements for works<sup>1</sup>.

The indications given as to the working life of the construction product cannot be interpreted as a guarantee neither given by the product manufacturer or his representative nor by EOTA when drafting this EAD nor by the Technical Assessment Body issuing an ETA based on this EAD, but are regarded only as a means for expressing the expected economically reasonable working life of the product.

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<sup>1</sup> The real working life of a product incorporated in a specific works depends on the environmental conditions to which that works is subject, as well as on the particular conditions of the design, execution, use and maintenance of that works. Therefore, it cannot be excluded that in certain cases the real working life of the product may also be shorter than referred to above.

## 2 ESSENTIAL CHARACTERISTICS AND RELEVANT ASSESSMENT METHODS AND CRITERIA

### 2.1 Essential characteristics of the product

Table 1 shows how the performance of "natural calcined pozzolana as type II addition" is assessed in relation to the essential characteristics.

Assessment acc. to clauses 2.2.1 to 2.2.11 is done on 5 samples taken from a current production of the product.

The "natural calcined pozzolana as type II addition"-sample with the lowest relative compressive strength (see clause 2.2.8) is used for the assessment acc. to clauses 2.2.12 to 2.2.15.

**Table 1 Essential characteristics of the product and methods and criteria for assessing the performance of the product in relation to those essential characteristics**

No	Essential characteristic	Assessment method	Type of expression of product performance
<b>Basic Works Requirement 1: Mechanical resistance and stability</b>			
1	Loss on ignition	2.2.1	Level
2	Chloride content	2.2.2	Level
3	Sulfate content	2.2.3	Level
4	Total content of alkalis	2.2.4	Level
5	Content of soluble alkalis	2.2.5	Level
6	Fineness	2.2.6	Level
7	Specific surface	2.2.7	Level
8	Activity index	2.2.8	Level
9	Initial setting time	2.2.9	acc. to EN 450-1: ≤ twice as long as the initial setting time of a 100 % (by mass) test cement paste
10	Soundness	2.2.10	acc. to EN 450-1: expansion ≤ 10 mm
11	Pozzolanic reactivity (Content of reactive silicon dioxide)	2.2.11	acc. to EN 450-1: content of reactive SiO <sub>2</sub> ≥ 25 % by mass
12	Carbonation of concrete	2.2.12	Description
13	Freeze thaw resistance of concrete	2.2.13	Method FT <sub>cube</sub> : Level Method FT <sub>CF</sub> : Level

No	Essential characteristic	Assessment method	Type of expression of product performance
14	Suitability of the k-value concept	2.2.14	Description
15	Resistance against chloride penetration	2.2.15	Description and level ( $D_{mig}$ )

## 2.2 Methods and criteria for assessing the performance of the product in relation to essential characteristics of the product

Regarding the test methods according to EN 196-2 the reference test methods shall be used for the different characteristics according to clause 2.2.1 to 2.2.5 and 2.2.11 unless stated otherwise.

### 2.2.1 Loss on ignition

The loss on ignition is determined by the method described in EN 196-2 but using an ignition time of 1 hour.

The loss on ignition shall be stated.

### 2.2.2 Chloride content

The chloride content, expressed as  $Cl^-$ , is determined by the method described in EN 196-2.

The chloride content is not higher than 0,10 % by mass acc. to EN 15167-1.

If the  $Cl^-$  content is higher than 0,10 % by mass, the upper limit value shall be stated.

### 2.2.3 Sulfate content

The sulfate content, expressed as  $SO_3$ , is determined by the method described in EN 196-2.

The sulfate content shall be stated.

### 2.2.4 Total content of alkalis

The total content of alkalis is determined by the method described in EN 196-2 and calculated as  $Na_2O$  equivalent.

The total content of alkalis, calculated as  $Na_2O$  equivalent, shall be stated.

Different national provisions adopt different principles but in general only a very small proportion of alkalis in "natural calcined pozzolana as type II addition" are considered to contribute to alkali silica reaction, CEN Report CR 1901:1995.

For the content of soluble alkalis see clause 2.2.5.

### 2.2.5 Content of soluble alkalis

The content of soluble alkalis is determined by the method described in EN 196-2 except that the leaching is done in accordance with EN 1744-1, clause 7. The content of soluble alkalis is calculated as  $Na_2O$  equivalent.

The content of soluble alkalis, calculated as Na<sub>2</sub>O equivalent, shall be stated.

### **2.2.6 Fineness**

The fineness of the addition is expressed as the mass proportion of the retained addition in percent when wet sieved on a 0,045 mm sieve and determined in accordance with EN 451-2.

The fineness of the addition shall be stated.

### **2.2.7 Specific surface**

The specific surface is determined in accordance with the air permeability method specified in EN 196-6.

Other methods than the one indicated may be used provided they give results correlated and equivalent to those obtained with the reference method.

The specific surface shall be stated.

### **2.2.8 Activity index**

The activity index is the ratio (in percent) of the compressive strength of standard mortar bars, prepared with 75 % test cement plus 25 % addition by mass, to the compressive strength of standard mortar bars prepared with 100 % test cement, when tested at the same age.

Preparation of standard mortar bars and determination of the compressive strength shall be carried out in accordance with EN 196-1.

NOTE: For test cement see EN 450-1 clause 3.3

The activity index at 7 days and at 28 days shall be stated.

NOTE: The result of the activity index tests gives no direct information on the strength contribution of the addition in concrete, nor is the use of the addition limited to the mixing ratio used in these tests.



### **2.2.9 Initial setting time**

The initial setting time is determined on a cement paste consisting of 25 % addition plus 75 % test cement (by mass) in accordance with EN 196-3.

NOTE: For test cement see EN 450-1 clause 3.3

The initial setting time is less than twice as long as the initial setting time of a 100 % by mass test cement paste acc. to EN 450-1. Both initial setting times shall be stated.

### **2.2.10 Soundness**

The soundness is determined by testing in accordance with EN 196-3. The expansion is determined on a cement paste consisting of 30 % addition plus 70 % test cement (by mass).

NOTE: For test cement see EN 450-1 clause 3.3

The expansion shall not be higher than 10 mm acc. to EN 450-1 and shall be stated.

NOTE: Because of the low temperature of the thermal treatment (450°C), free CaO is not existent, even in case of calcined pozzolana.

### **2.2.11 Pozzolanic reactivity (Content of reactive silicon dioxide)**

The content of reactive silicon dioxide is defined and described in 3.2 of EN 197-1. The content of reactive SiO<sub>2</sub> is determined by the method described in EN 196-2.

The content of reactive silicon dioxide is at least 25 % by mass acc. to EN 450-1 and shall be stated.

### **2.2.12 Carbonation of concrete**

The carbonation depth of concrete made with addition and without addition is measured according to RILEM CPC 18.

EN 12390-10 will replace RILEM CPC 18 after publication (cp. prEN 12390-10:2017).

The test of carbonation depth is made on prism (40 mm x 40 mm x 160 mm) with aggregates according to EN 12620.

The concrete compositions are given below:

	Concrete mixtures for 3 specimens															
<b>concrete Ia</b> without "natural calcined pozzolana as type II addition"	c = 450 g CEM I 32,5 R <sup>1</sup> g = 1350 g aggregates <sup>2</sup> w = 225 g water $\left(\frac{w}{c} = 0,50\right)$															
<b>concrete Ib</b> with "natural calcined pozzolana as type II addition"	c = 337,5 g CEM I 32,5 R <sup>1</sup> p = 112,5 g addition <sup>4</sup> g = 1350 g aggregates <sup>2</sup> m = .... ml admixture <sup>3</sup> w = (225 – m) g water $\left(\frac{w + m}{c + p} = 0,50\right)$															
<p>1 The reference cement (CEM I 32,5 R) shall contain less than 0,6 % by mass alkalis, calculated as Na<sub>2</sub>O equivalent.</p> <p>2 Aggregates according to EN 12620 with the following grading curve shall be used:</p> <table border="1" data-bbox="258 1025 1214 1160"> <thead> <tr> <th>Size [mm]</th> <th>0,25</th> <th>0,5</th> <th>1</th> <th>2</th> <th>4</th> <th>8</th> </tr> </thead> <tbody> <tr> <td>Passing [% by mass]</td> <td>8</td> <td>21,5</td> <td>36</td> <td>46,5</td> <td>67,5</td> <td>100</td> </tr> </tbody> </table> <p>3 Liquid admixture of type "water reducing admixtures" or "plasticizing admixtures" according to EN 934-2 shall be used. The amount of admixture shall be conformed to the flow table test (<math>\pm 5</math> mm) according to EN 1015-3 of the concrete without addition. The amount of admixture shall not exceed the compliance dosage.</p> <p>4 The "natural calcined pozzolana as type II addition"-sample with the lowest relative compressive strength shall be taken, see clause 2.2.8.</p>			Size [mm]	0,25	0,5	1	2	4	8	Passing [% by mass]	8	21,5	36	46,5	67,5	100
Size [mm]	0,25	0,5	1	2	4	8										
Passing [% by mass]	8	21,5	36	46,5	67,5	100										

NOTE: The recommended maximum replacement of cement by "natural calcined pozzolana as type II addition" is 25 % by mass.

The types of cement that are tested depend on the application, e.g.

- if the applicant applies for CEM I, then CEM I shall be tested
- if the applicant applies for CEM I, CEM II/A-LL and CEM II/B-M (T-LL), then CEM II/A-LL and CEM II/B-M (T-LL) shall be tested.

The specimens are prepared according to EN 196-1, except the respect for the gap of ( $3 \pm 1$ mm) between the bowl and the blade that cannot be respected by the size grading of the sand.

After demoulding half of the specimens are stored immersed in water ( $20 \pm 2$ ) °C until the age of 7 days (set of specimens n°1) and the other half until the age of 28 days (set of specimens n°2).

Afterwards the specimens are stored in climate 20/65 and ambient CO<sub>2</sub> content (commonly 350 to 450 p.p.m.).

Measurements of carbonation depth shall be performed after 14, 28, 56, 98 and 140 days for the delivery of the ETA. It is recommended to continue the tests on the same samples after 1, 2 and 5 years in order to verify the obtained results after 140 days and improve the knowledge.

Furthermore the compressive strength is determined according to EN 196-1:

- on the set of specimens n°1, at the age of 35 days (after 7 days pre-storing in water and 28 days in climate 20/65) and at the age of 147 days (after 7 days pre-storing in water and 140 days in climate 20/65),
- on the set of specimens n°2, at the age of 35 days (after 28 days pre-storing in water and 7 days in climate 20/65) and at the age of 168 days (after 28 days pre-storing in water and 140 days in climate 20/65).

The carbonation depth resp. the carbonation speed of the concrete with and without addition is compared to data according to **Annex B**. The carbonation depth and the carbonation speed of the concrete with and without addition shall be located below or around the limit curve.

Note 1: The carbonation speed  $v_c$  is calculated by linear regression with:

$$d_c = d_0 + v_c \cdot \sqrt{t_c} \text{ expressed in mm / } \sqrt{d}$$

with:

$d_c$  = carbonation depth (mm)

$t_c$  = duration of carbonation (days)

$v_c$  = carbonation speed (in  $mm / \sqrt{\text{day}}$ )

$d_0$  = carbonation depth at time  $t = 0$ ; this specific parameter which depends on the storage and will be lower at a later start of testing the carbonation.

Alternatively the concrete compositions 2a (reference) and 2b (see 2.2.14) may be tested.

The carbonation depth resp. the carbonation speed shall be plotted in the diagrams according to Annex B (diagrams in Annex B1 for concrete composition 1a and 1b, diagrams in Annex B2 for concrete composition 2a and 2b). The respective diagrams are given in the ETA.

### 2.2.13 Freeze-thaw resistance

#### Method 1: Freeze-thaw resistance (Cube-Procedure) - FT<sub>cube</sub>

The freeze-thaw resistance of concrete with addition is determined according to CEN/TS 12390-9 ("cube procedure").

The freeze-thaw resistance ("cube procedure") is tested on the concrete composition 2a (reference) and 2b (see 2.2.14).

NOTE: The recommended maximum replacement of cement by "natural calcined pozzolana as type II addition" is 25 % by mass.

The scaling shall be measured after 10, 25, 50, 75 and 100 freeze-thaw cycles.

Furthermore the compressive strength of concrete 2a and 2b is determined according to EN 12390-3 at 28 days. The concrete specimens are immersed in water after demoulding until the age of 7 days. Afterwards the specimens are stored in normal climate 20/65.

The scaling after 100 freeze-thaw-cycles shall be stated.

#### Method 2: Freeze-thaw resistance (CF-Procedure) - FT<sub>CF</sub> (Reference method)

The freeze-thaw resistance of concrete with and without addition is determined according to CEN/TS 12390-9 ("CF-Procedure"). The internal structural damage is determined according to CEN/TR 15177.

The freeze-thaw resistance ("CF-Procedure") is tested on the following concrete compositions:

	Composition per m <sup>3</sup> fresh concrete																
<b>concrete IIa</b> without "natural calcined pozzolana as type II addition"	c = 320 kg CEM I 32,5 R <sup>1</sup> g = .... kg aggregates <sup>2</sup> w = 160 kg water $\frac{w}{c} = 0,50$																
<b>concrete IIb</b> with "natural calcined pozzolana as type II addition"	c = 240 kg CEM I 32,5 R <sup>1</sup> p = 80 kg addition <sup>3</sup> g = ..... kg aggregates <sup>2</sup> m = .... ml admixture <sup>4</sup> w = (160 – m) g water $\left( \frac{w + m}{c + p} = 0,50 \right)$																
1 The reference cement (CEM I 32,5 R) shall contain less than 0,6 % by mass alkalis, calculated as Na <sub>2</sub> O equivalent. 2 Aggregates according to EN 12620 with the following grading curve shall be used: <table border="1" data-bbox="258 981 1310 1115"> <thead> <tr> <th>Size [mm]</th> <th>0,25</th> <th>0,5</th> <th>1</th> <th>2</th> <th>4</th> <th>8</th> <th>16</th> </tr> </thead> <tbody> <tr> <td>Passing [% by mass]</td> <td>6</td> <td>14</td> <td>22</td> <td>32</td> <td>46</td> <td>68</td> <td>100</td> </tr> </tbody> </table> 2 The "natural calcined pozzolana as type II addition"-sample with the lowest relative compressive strength shall be taken, see clause 2.2.8. 3 Liquid admixture of type "water reducing admixtures" according to EN 934-2 shall be used. The fresh concrete shall be adjusted to flow class F3 according to EN 206-1.		Size [mm]	0,25	0,5	1	2	4	8	16	Passing [% by mass]	6	14	22	32	46	68	100
Size [mm]	0,25	0,5	1	2	4	8	16										
Passing [% by mass]	6	14	22	32	46	68	100										

NOTE: The recommended maximum replacement of cement by "natural calcined pozzolana as type II addition" is 25 % by mass.

Furthermore the compressive strength of concrete IIa and IIb is determined according to EN 12390-3 at 28 days. The specimens are immersed in water after demoulding until the age of 7 days. Afterwards the specimens are stored in normal climate 20/65.

The relative dynamic modulus of elasticity (RDM) and scaling is measured after 0, 4, 10, 16, 22 and 28 freeze-thaw cycles.

The scaling after 28 freeze-thaw cycles (CF-procedure) and the relative dynamic modulus of elasticity (RDM) shall be stated.

### 2.2.14 Suitability of the k-value concept

The compressive strength of concretes with and without "natural calcined pozzolana as type II addition" is determined over a range of water/binder-ratio.

The recommended replacement of cement by "natural calcined pozzolana as type II addition" is 25 % by mass maximum.

The types of cement for which the suitability of the k-value concept shall be verified are Portland cement CEM I and, if requested, further types of common cement acc. to EN 197-1.

The concrete compositions are given below:

	Concrete No					
	1a	1b	2a	2b	3a	3b
Cement	340 kg/m <sup>3</sup>	255 kg/m <sup>3</sup>	320 kg/m <sup>3</sup>	240 kg/m <sup>3</sup>	290 kg/m <sup>3</sup>	217,5 kg/m <sup>3</sup>
Addition	-	85 kg/m <sup>3</sup>	-	80 kg/m <sup>3</sup>	-	72,5 kg/m <sup>3</sup>
Water	170 kg/m <sup>3</sup>		192 kg/m <sup>3</sup>		217,5 kg/m <sup>3</sup>	
w/c-value	0,50	0,67	0,60	0,80	0,75	1,00
w/b-value		0,50		0,60		0,75

The following cements will be used:

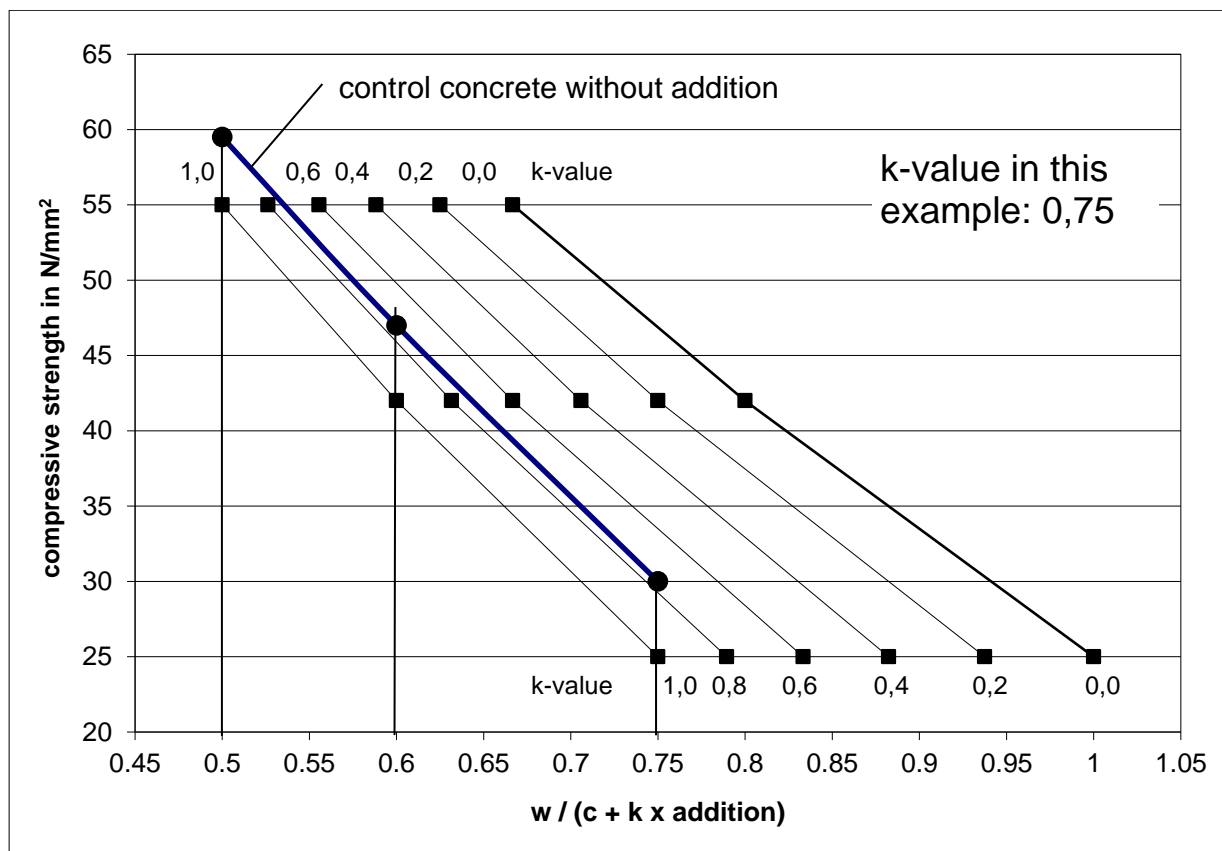
- Portland cement CEM I 32,5 R with Na<sub>2</sub>O equivalent ≤ 0,6 % by mass
- Portland cement CEM I 32,5 R with Na<sub>2</sub>O equivalent 0,8 to 0,9 % by mass
- Portland cement CEM I 32,5 R with Na<sub>2</sub>O equivalent ≥ 1,2 % by mass

Additional common cement types requested by the applicant:

- Portland limestone cement CEM II/A-LL 32,5 R
- Portland composite cement CEM II/B-M (T-LL) 42,5 N
- ...

The concrete compressive strength is determined acc. to EN 12390-3 after 2, 7, 28 and 90 days.

The k-value is derived from the relationship between compressive strength and water/cement-ratio w/c resp. w/(c + k x addition). The following figure illustrates the principle of such a derivation with exemplary data.



The k-value is derived from the compressive strength of concretes with and without "natural calcined pozzolana as type II addition". The compressive strengths and the k-values are stated.

NOTE: The validity of the k-value with respect to durability is verified in 2.2.12 and 2.2.13, where carbonation and freeze thaw resistance of concrete are tested for k = 1,0.

### 2.2.15 Resistance against chloride penetration

The resistance against chloride penetration of concrete with "natural calcined pozzolana as type II addition" is determined in accordance with the test method given in **Annex A**.

The resistance against chloride penetration is tested on the concrete composition IIa and IIb (see clause 2.2.13).

The chloride migration coefficient of concrete ( $D_{\text{mig}}$ ) with "natural calcined pozzolana as type II addition" is compared to the chloride migration coefficient of the reference concrete at the age of 35 and 97 days.

The chloride migration coefficient  $D_{\text{mig}}$  is stated in the ETA.

### 3 ASSESSMENT AND VERIFICATION OF CONSTANCY OF PERFORMANCE

#### 3.1 System of assessment and verification of constancy of performance to be applied

For the products covered by this EAD the applicable European legal act is: Decision 1999/469/EC.

The system is: 1+

#### 3.2 Tasks of the manufacturer

The cornerstones of the actions to be undertaken by the manufacturer of the product in the procedure of assessment and verification of constancy of performance are laid down in Table 2.

**Table 2 Control plan for the manufacturer; cornerstones**

No	Subject/type of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
<b>Factory production control (FPC)</b> [including testing of samples taken at the factory in accordance with a prescribed test plan]					
1	Loss on ignition	2.2.1	See control plan	1	1/day
2	Chloride	2.2.2		1	1/month
3	Sulfate	2.2.3		1	1/month
4	Fineness	2.2.6		1	1/day
5	Specific surface	2.2.7		1	1/day
6	Activity index	2.2.8		1	2/month

### 3.3 Tasks of the notified body

The cornerstones of the actions to be undertaken by the notified body of the product in the procedure of assessment and verification of constancy of performance are laid down in Table 3.

**Table 3 Control plan for the notified body; cornerstones**

No	Subject/type of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
<b>Initial inspection of the manufacturing plant and of factory production control</b>					
1	acc. to EN 450-2				
<b>Continuous surveillance, assessment and evaluation of factory production control</b>					
2	acc. to EN 450-2				1/year
<b>Audit-testing of samples taken by the notified product certification body at the manufacturing plant or at the manufacturer's storage facilities</b>					
3	Loss on ignition	2.2.1	See control plan	1	6/year
4	Chloride	2.2.2	See control plan	1	6/year
5	Sulfate	2.2.3	See control plan	1	6/year
6	Fineness	2.2.6	See control plan	1	6/year
7	Specific surface	2.2.7	See control plan	1	6/year
8	Activity index	2.2.8	See control plan	1	6/year



## 4 REFERENCE DOCUMENTS

As far as no edition date is given in the list of standards thereafter, the standard in its current version at the time of issuing the European Technical Assessment is of relevance.

EN 196-1	Methods of testing cement — Determination of strength
EN 196-2	Methods of testing cement — Chemical analysis of cement
EN 196-3	Methods of testing cement — Determination of setting time and soundness
EN 196-6	Methods of testing cement — Determination of fineness
EN 197-1	Cement — Part 1: Composition, specification and conformity criteria for common cements
EN 206	Concrete — Specification, performance, production and conformity
EN 450-1	Fly ash for concrete - Part 1: Definition, specifications and conformity criteria
EN 450-2	Fly ash for concrete - Part 2: Conformity evaluation
EN 451-2	Methods of testing fly ash — Part 2: Determination of fineness by wet sieving
EN 934-2	Admixtures for concrete, mortar and grout - Part 2: Concrete admixtures - Definitions, requirements, conformity, marking and labelling
EN 1015-3	Methods of test for mortar for masonry - Part 3: Determination of consistence of fresh mortar (by flow table)
EN 1744-1	Tests for chemical properties of aggregates — Part 1 Chemical analysis
EN 12390-2	Testing hardened concrete – Part 2: Making and curing specimens for strength tests
EN 12390-3	Testing hardened concrete — Part 3: Compressive strength of test specimens
prEN 12390-10:2017	Testing hardened concrete — Part 10: Determination of the carbonation resistance of concrete at atmospheric levels of carbon dioxide
EN 12620	Aggregates for concrete
EN 15167-1	Ground granulated blast furnace slag for use in concrete, mortar and grout - Part 1: Definitions, specifications and conformity criteria
CEN/TR 15177	Testing the freeze-thaw resistance of concrete - Internal structural damage
CEN/TS 12390-9	Testing hardened concrete - Part 9: Freeze-thaw resistance, Scaling
CEN CR 1901:1995	Regional specifications and recommendations for the avoidance of damaging alkali-silica reactions in concrete
RILEM CPC 18	Measurement of hardened concrete carbonation depth, Materials and structures, Vol. 21, December 1988

## **Annex A Testing the resistance against chloride penetration by the non-steady-state-migration experiments**

### **A.1 References**

EN 12390-2            Testing hardened concrete – Part 2: Making and curing specimens for strength tests

### **A.2 Scope**

This procedure is for determination of the chloride migration coefficient in concrete, mortar or cement-based repair materials from non-steady-state migration experiments.

### **A.3 Field of Application**

The method is applicable to hardened specimens cast in the laboratory or drilled from field structures. The chloride migration coefficient determined by the method is a measure of the resistance of the tested material to chloride penetration. This non-steady-state migration coefficient cannot be directly compared with chloride diffusion coefficients obtained from other test methods, such as the non-steady-state immersion test or the steady-state migration test.

### **A.4 Test Method**

#### **A.4.1 Principle**

An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration, the specimen is axially split and a silver nitrate solution is sprayed on to one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth.

#### **A.4.2 Reagents and apparatus**

##### **A.4.2.1 Reagents**

- Distilled or deionised water.
- Calcium hydroxide:  $\text{Ca(OH)}_2$ , technical quality.
- Sodium chloride:  $\text{NaCl}$ , chemical quality.
- Sodium hydroxide:  $\text{NaOH}$ , chemical quality.
- Silver nitrate:  $\text{AgNO}_3$ , chemical quality.

##### **A.4.2.2 Apparatus**

- Water-cooled diamond saw.
- Migration set-up: One design (see A.1) includes the following parts:
  - Silicone rubber sleeve: inner/outer diameter 100/115 mm, about 150 mm long.
  - Clamp: diameter range 105 ~ 115, 20 mm wide, stainless steel (see Figure A.2).
  - Catholyte reservoir: plastic box, 370 × 270 × 280 mm (length × width × height).
  - Plastic support: (see Figure A.3).
  - Cathode: stainless steel plate (see Figure A.3), about 0,5 mm thick.
  - Anode: stainless steel mesh or plate with holes (see Figure A.4), about 0,5 mm thick.

Other designs are acceptable, provided that temperatures of the specimen and solutions during the test can be maintained in the range of 20 to 25 °C.

- Power supply: capable of supplying 0 ~ 60 V DC regulated voltage with an accuracy of  $\pm 0,1$  V.
- Ammeter: capable of displaying current to  $\pm 1$  mA.
- Thermometer or thermocouple with readout device capable of reading to  $\pm 1$  °C.
- Any suitable device for splitting the specimen.
- Spray bottle.
- Slide calliper with a precision of  $\pm 0,1$  mm.
- Ruler with a minimum scale of 1 mm.

### A.4.3 Preparation of the test specimen

Six cylinders from each concrete with a diameter of 100 mm and a length of 200 mm shall be made in accordance to EN 12390-2.

The specimens shall be stored for 24 hours in the mould at 20 °C and a relative air humidity of > 95 % r.H. After demoulding the specimens shall be stored immersed in water at  $20 \pm 5$  °C until testing. At an age of 28 days respectively 90 days 3 specimens of each concrete are taken out of the water.

In the middle of each cylinder a  $50 \pm 2$  mm thick slice is cut out. Measure the thickness of each slice with a slide calliper and read to 0,1 mm.

Note 1: The term 'cut' here means to saw perpendicularly to the axis of a core or cylinder, using a water-cooled diamond saw.

Until the test procedure the slices are stored immersed in water. The test procedure is started at an age of 35 days and 97 days.

### A.4.4 Test procedure

#### A.4.4.1 Catholyte and anolyte

The catholyte solution is 10 % NaCl by mass in tap water (100 g NaCl in 900 g water, about 2 N) and the anolyte solution is 0,3 N NaOH in distilled or de-ionised water (approximately 12 g NaOH in 1 litre water). Store the solutions at a temperature of 20 – 25 °C.

#### A.4.4.2 Temperature

Maintain the temperatures of the specimen and solutions in the range of 20 – 25 °C during the test.

#### A.4.4.3 Preparation of the test

- Fill the catholyte reservoir with about 12 litres of 10 % NaCl solution.
- Fit the rubber sleeve on the specimen as shown in Figure A.4 and secure it with two clamps. If the curved surface of the specimen is not smooth, or there are defects on the curved surface which could result in significant leakage, apply a line of silicone sealant to improve the tightness.
- Place the specimen on the plastic support in the catholyte reservoir (see Figure A.1).
- Fill the sleeve above the specimen with 300 ml anolyte solution (0,3 N NaOH).
- Immerse the anode in the anolyte solution.
- Connect the cathode to the negative pole and the anode to the positive pole of the power supply.

#### A.4.4.4 Migration test

- Turn on the power, with the voltage preset at 30 V, and record the initial current through each specimen.
- Adjust the voltage if necessary (as shown in Table A.1). After adjustment, note the value of the initial current again.
- Record the initial temperature in each anolyte solution, as shown by the thermometer or thermocouple.
- Choose appropriate test duration according to the initial current (see Table A.1).
- Record the final current and temperature before terminating the test.

#### A.4.4.5 Measurement of chloride penetration depth

- Disassemble the specimen by following the reverse of the procedure in A.4.4.3. A wooden rod is often helpful in removing the rubber sleeve from the specimen.
- Rinse the specimen with tap water.
- Wipe off excess water from the surfaces of the specimen.
- Split the specimen axially into two pieces. Choose the piece having the split section more nearly perpendicular to the end surfaces for the penetration depth measurement, and keep the other piece for chloride content analysis (optional).
- Spray 0.1 M silver nitrate solution on to the freshly split section.

- When the white silver chloride precipitation on the split surface is clearly visible (after about 15 minutes), measure the penetration depth, with the help of the slide calliper and a suitable ruler, from the centre to both edges at intervals of 10 mm (see Figure A.5) to obtain seven depths (notes 2, 3 and 4). Measure the depth to an accuracy of 0,1 mm.

Note 2: If the penetration front to be measured is obviously blocked by the aggregate, move the measurement to the nearest front where there is no significant blocking by aggregate or, alternatively, ignore this depth if there are more than five valid depths.

Note 3: If there is a significant defect in the specimen which results in a penetration front much larger than the average, ignore this front as indicative of the penetration depth, but note and report the condition.

Note 4: To obviate the edge effect due to a non-homogeneous degree of saturation or possible leakage, do not make any depth measurements in the zone within about 10 mm from the edge (see Figure A.5).

## A.5 Expression of results

### A.5.1 Test results

Calculate the non-steady-state migration coefficient from Equation (1):

$$D_{\text{nssm}} = \frac{RT}{zFE} \cdot \frac{x_d - \alpha \sqrt{x_d}}{t} \quad (1)$$

Where:

$$E = \frac{U-2}{L} \quad (2)$$

$$\alpha = 2 \sqrt{\frac{RT}{zFE}} \cdot \text{erf}^{-1} \left( 1 - \frac{2c_d}{c_0} \right) \quad (3)$$

$D_{\text{nssm}}$ : non-steady-state migration coefficient, m<sup>2</sup>/s;

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

$F$ : Faraday constant,  $F = 9,648 \times 10^4$  J/(V·mol);

$U$ : absolute value of the applied voltage, V;

$R$ : gas constant,  $R = 8,314$  J/(K·mol);

$T$ : average value of the initial and final temperatures in the anolyte solution, K;

$L$ : thickness of the specimen, m;

$x_d$ : average value of the penetration depths, m;

$t$ : test duration, seconds;

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

$c_d$ : chloride concentration at which the colour changes,  $c_d \approx 0,07$  N for OPC concrete;

$c_0$ : chloride concentration in the catholyte solution,  $c_0 \approx 2$  N.

Since  $\text{erf}^{-1} \left( 1 - \frac{2 \cdot 0,07}{2} \right) = 1,28$  the following simplified equation can be used:

$$D_{\text{nssm}} = \frac{0,0239(273+T)L}{(U-2)t} \left( x_d - 0,0238 \sqrt{\frac{(273+T)L x_d}{U-2}} \right) \quad (4)$$

Where:

- $D_{nssm}$ : non-steady-state migration coefficient,  $\times 10^{-12}$  m<sup>2</sup>/s;  
 U: absolute value of the applied voltage, V;  
 T: average value of the initial and final temperatures in the analyte solution, °C;  
 L: thickness of the specimen, mm;  
 $x_d$ : average value of the penetration depths, mm;  
 t: test duration, hour.

## A.6 Appendix

Table A.1: Test voltage and duration for concrete specimen with normal binder content

Initial current L30V (with 30 V)	Applied voltage U (After adjustment)	Possible new initial current $I_0$	Test duration t
mA	V	mA	hour
$I_0 < 5$	60	$I_0 < 10$	96
$5 \leq I_0 < 10$	60	$10 \leq I_0 < 20$	48
$10 \leq I_0 < 15$	60	$20 \leq I_0 < 30$	24
$15 \leq I_0 < 20$	50	$25 \leq I_0 < 35$	24
$20 \leq I_0 < 30$	40	$25 \leq I_0 < 40$	24
$30 \leq I_0 < 40$	35	$35 \leq I_0 < 50$	24
$40 \leq I_0 < 60$	30	$40 \leq I_0 < 60$	24
$60 \leq I_0 < 90$	25	$50 \leq I_0 < 75$	24
$90 \leq I_0 < 120$	20	$60 \leq I_0 < 80$	24
$120 \leq I_0 < 180$	15	$60 \leq I_0 < 90$	24
$180 \leq I_0 < 360$	10	$60 \leq I_0 < 120$	24
$I_0 \geq 360$	10	$I_0 \geq 120$	6

Note: For specimens with a special binder content, such as repair mortars or grouts, correct the measured current by multiplying by a factor (approximately equal to the ratio of normal binder content to actual binder content) in order to be able to use the above table.

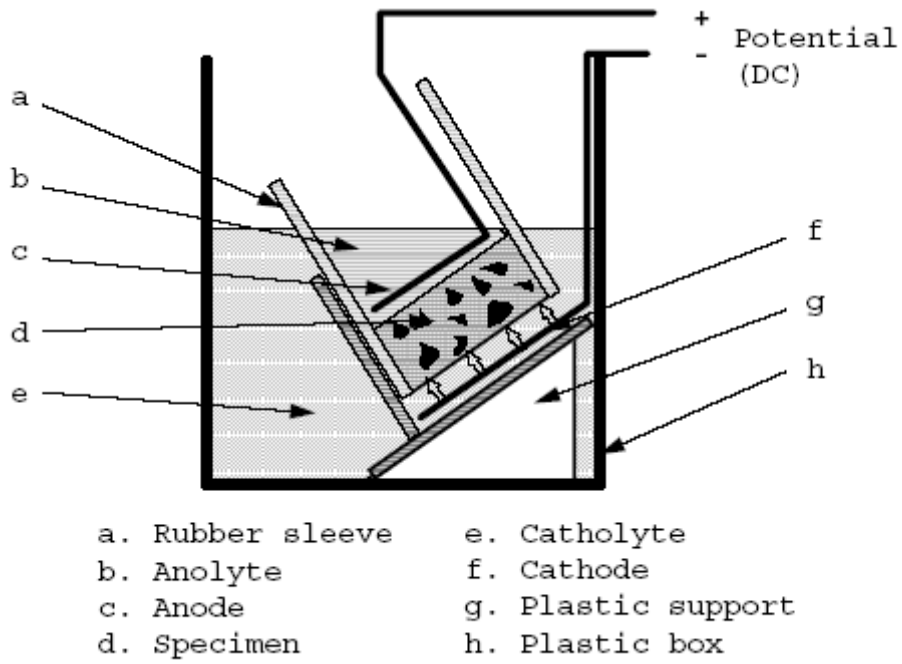


Figure A.1: Arrangement of the migration set-up



Figure A.2: Stainless steel

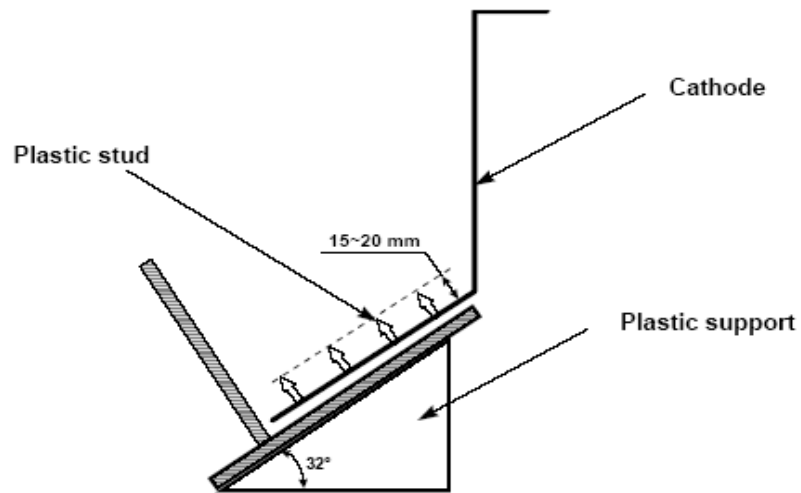


Figure A.3: Plastic support and cathode

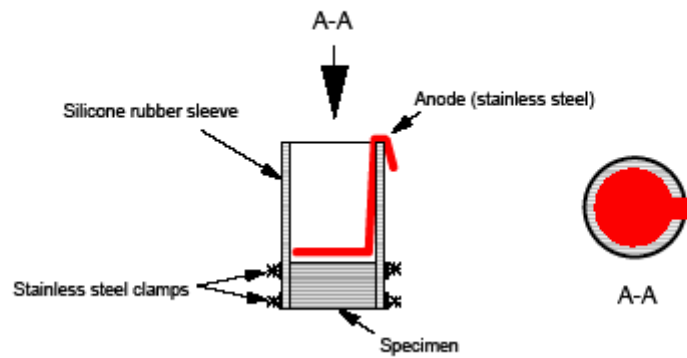


Figure A.4: Rubber sleeve assembled with specimen, clamps and anode

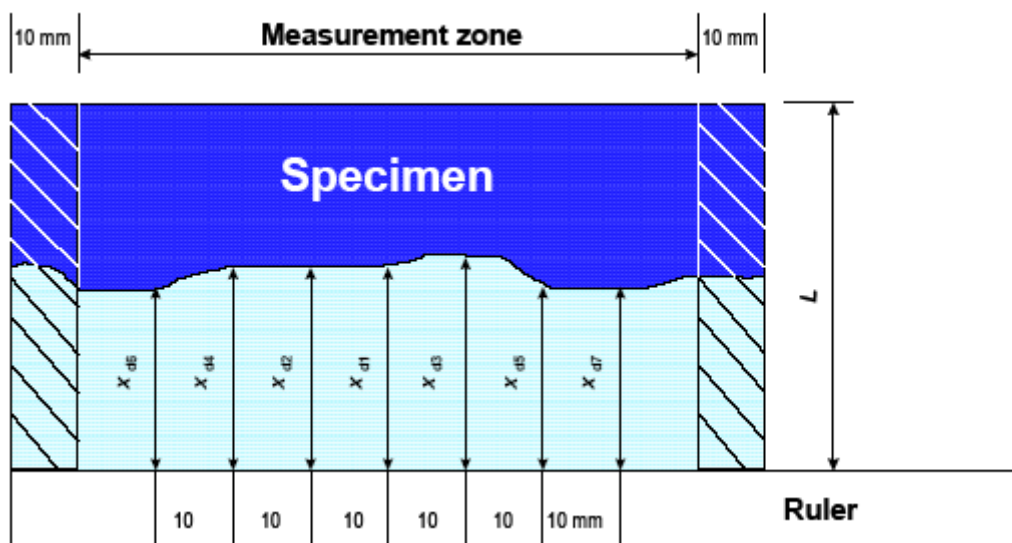


Figure A.5: Illustration of measurement for chloride penetration depths

**ANNEX B EVALUATION OF THE CARBONATION RESISTANCE - C<sub>DCR</sub>****B1 Data for concrete composition Ia and Ib**

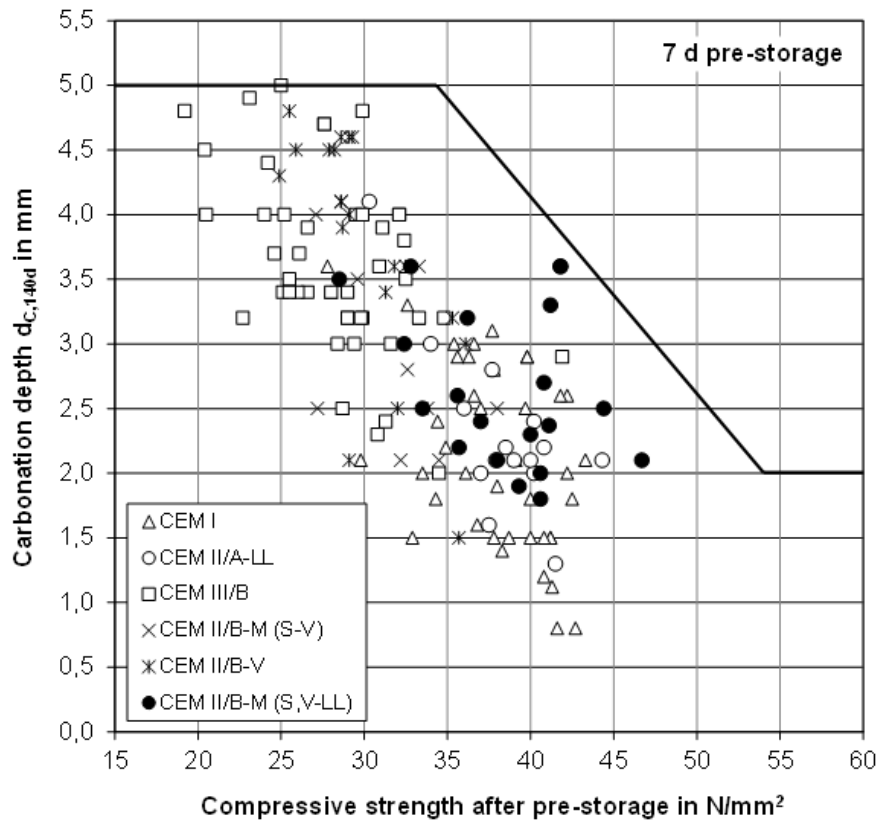
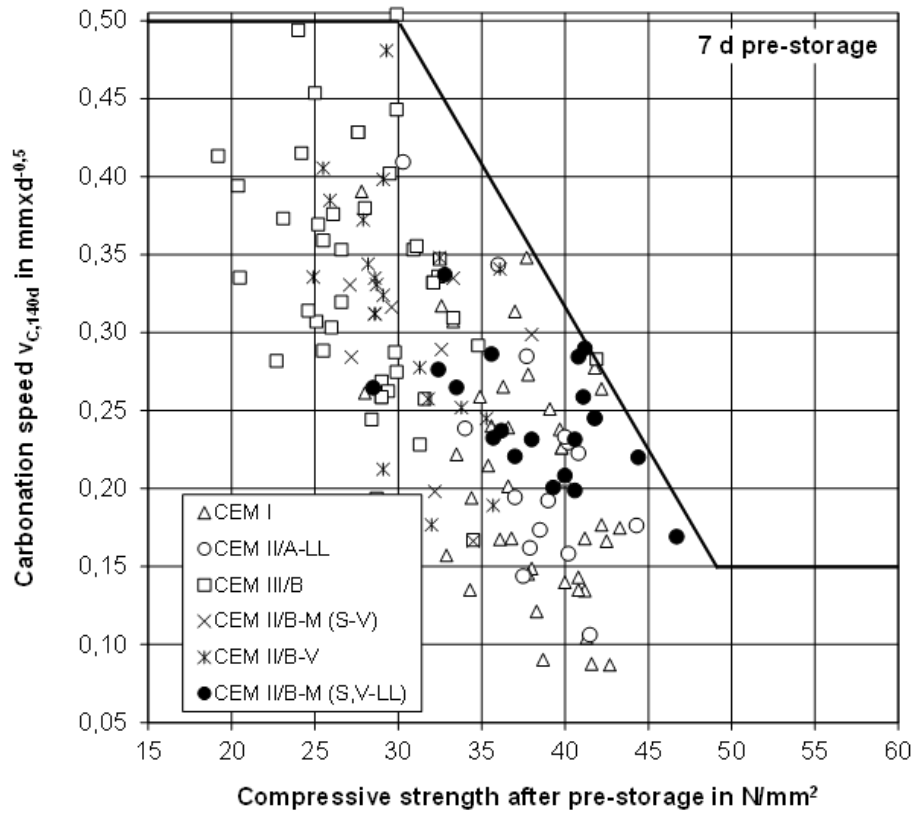
Carbonation test on concrete (w/c = 0,50) - 7 days pre-storage

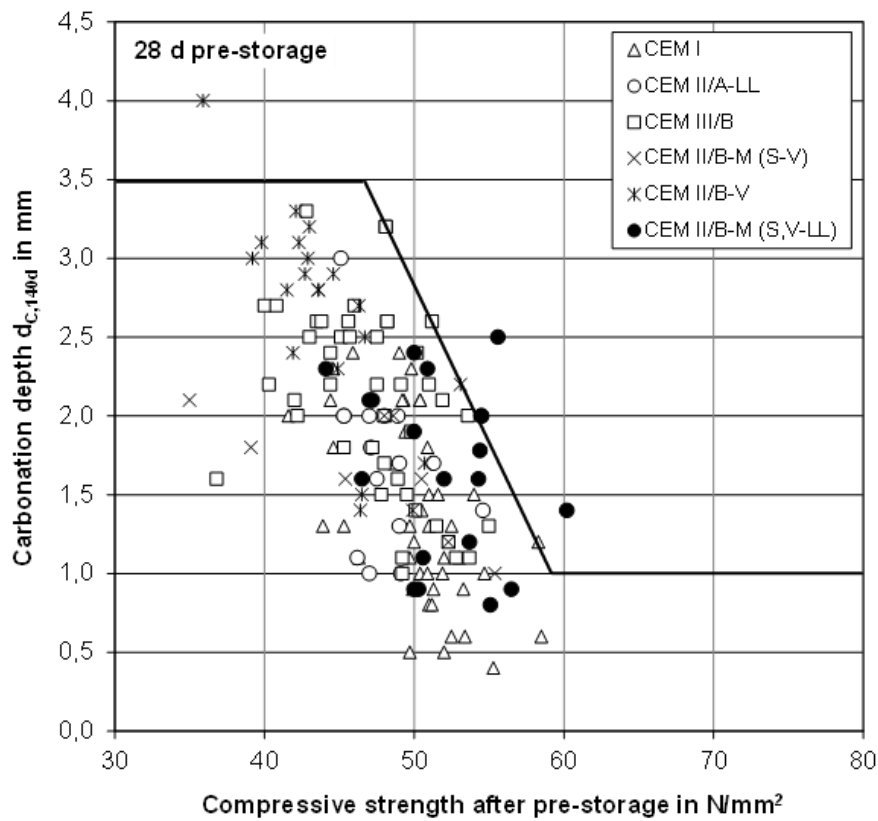
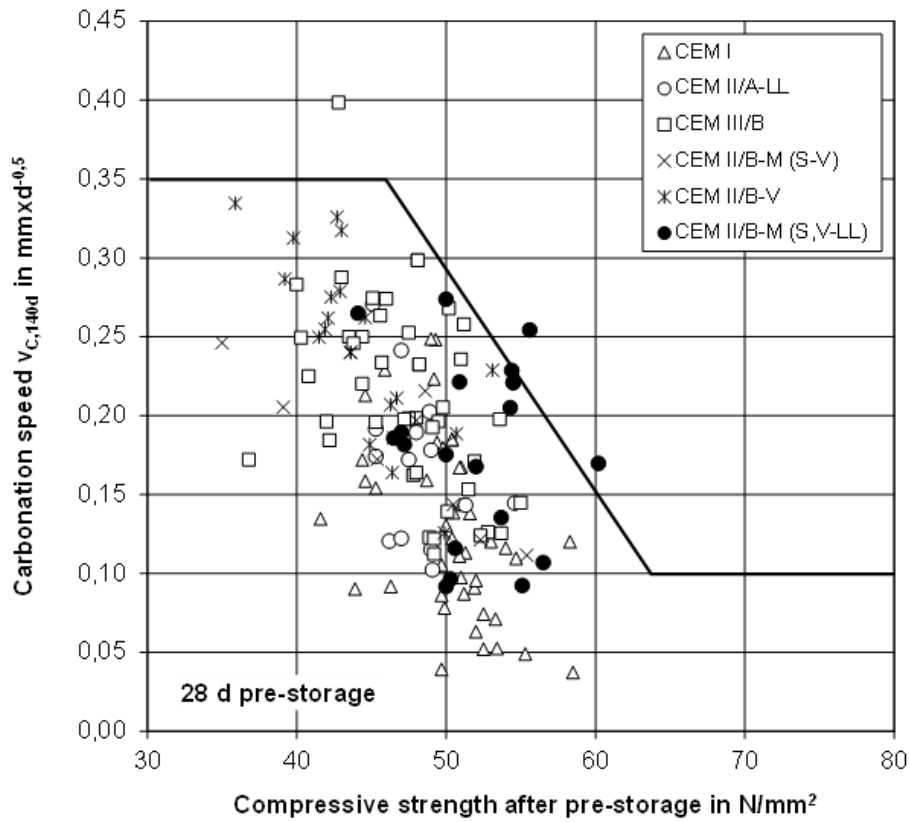
	f <sub>c</sub> in N/mm <sup>2</sup>			Carbonation depth in mm									F <sub>C,P</sub> <sup>0,5</sup> in N <sup>0,5</sup> x mm	Carbo. speed in mm / d <sup>0,5</sup>	
	Pre-st. 7 d	35 d	140 d main-st.	14 d	28 d	56 d	98 d	140 d	1 a	2 a	5 a	V <sub>C,140d</sub>		V <sub>C,5a</sub>	
<b>CEM II/B-M (S, V-LL)</b>															
min	28,5	51,2	50,8	0,2	0,4	1,0	1,5	1,8	3,1	3,7	6,8	0,146	0,169	0,147	
max	46,7	66,2	71,0	1,6	1,8	2,7	3,2	3,6	4,7	6,0	10,1	0,187	0,337	0,243	
AVG	38,1	56,6	60,2	0,7	1,0	1,6	2,1	2,7	4,0	5,2	8,6	0,163	0,246	0,203	
s	4,3	4,0	6,0	0,5	0,5	0,5	0,5	0,6	0,6	1,3	1,1	0,010	0,039	0,032	
<b>CEM II/A-LL (C 80 %; LL 20 %)</b>															
min	30,3	36,1	31,6	0,0	0,2	0,6	1,0	1,3	2,3	4,2	7,0	0,150	0,106	0,173	
max	44,3	64,3	63,7	1,0	1,7	2,6	3,5	4,1	6,0	7,8	12,9	0,182	0,409	0,290	
AVG	38,3	54,0	55,0	0,5	0,9	1,5	2,0	2,3	3,8	5,8	9,0	0,162	0,218	0,217	
s	3,3	6,2	7,4	0,3	0,4	0,5	0,6	0,6	1,0	1,0	1,5	0,007	0,079	0,031	
<b>CEM II/B-M (S-V) (C 65 %; S 15 %; V 20 %)</b>															
min	27,1	45,6	45,8	0,0	0,2	1,0	1,4	2,1	3,7	4,9	7,2	0,162	0,166	0,178	
max	38,0	58,8	64,7	1,3	1,8	2,7	3,2	4,0	6,5	8,3	14,3	0,192	0,335	0,327	
AVG	31,8	50,9	55,3	0,6	1,1	1,8	2,3	2,9	4,7	6,3	9,5	0,178	0,277	0,226	
s	3,7	4,7	6,0	0,4	0,5	0,6	0,6	0,7	1,1	1,4	2,3	0,010	0,062	0,049	
<b>CEM II/B-V (C 70 %; V 30 %)</b>															
min	24,9	40,7	43,3	0,0	0,1	0,5	1,0	1,5	3,5	5,3	8,0	0,166	0,177	0,179	
max	36,1	60,9	64,5	1,7	2,4	3,2	4,5	4,8	8,6	9,6	14,3	0,200	0,481	0,318	
AVG	30,1	48,4	51,9	1,1	1,7	2,5	3,1	3,7	5,4	7,2	10,6	0,183	0,316	0,240	
s	3,2	5,0	5,5	0,5	0,6	0,7	0,8	0,9	1,3	1,3	1,7	0,010	0,075	0,036	
<b>CEM III/B</b>															
min	19,2	35,3	36,6	0,1	0,9	1,5	1,5	2,0	3,1	5,5	7,5	0,154	0,167	0,178	
max	41,9	62,0	67,6	1,8	2,6	3,5	4,2	5,0	8,0	10,5	17,1	0,228	0,504	0,394	
AVG	28,3	49,1	52,4	0,9	1,5	2,3	3,0	3,6	5,5	7,6	11,5	0,190	0,330	0,269	
s	4,3	5,3	5,8	0,4	0,4	0,5	0,7	0,7	1,1	1,4	2,4	0,015	0,079	0,055	
<b>CEM I</b>															
min	27,8	45,5	46,6	0,0	0,1	0,1	0,3	0,8	2,0	3,2	5,0	0,152	0,087	0,121	
max	43,3	63,0	64,0	1,4	1,8	2,2	3,2	3,6	6,2	7,8	9,9	0,190	0,391	0,247	
AVG	37,2	56,1	58,1	0,5	0,8	1,3	1,7	2,2	3,4	4,7	6,9	0,165	0,202	0,164	
s	4,2	3,7	3,9	0,3	0,4	0,5	0,7	0,7	0,9	1,0	1,4	0,010	0,072	0,030	



## Carbonation test on concrete (w/c = 0,50) - 28 days pre-storage

	f <sub>c</sub> in N/mm <sup>2</sup>			Carbonation depth in mm									F <sub>C,p-0,5</sub> in N <sup>0,5</sup> x mm	Carbo. speed in mm / d <sup>0,5</sup>	
	Prest. 28 d	35 d	140 d main-st.	14 d	28 d	56 d	98 d	140 d	1 a	2 a	5 a	VC,140d		VC,5a	
<b>CEM II/B-M (S, V-LL)</b>															
min	44,1	50,6	61,2	0,0	0,2	0,3	0,5	0,8	1,8	4,0	4,9	0,129	0,092	0,130	
max	60,2	67,6	76,4	0,7	1,0	1,5	2,2	2,5	3,6	5,1	9,9	0,151	0,274	0,247	
AVG	51,7	58,9	67,2	0,3	0,5	0,9	1,4	1,7	2,5	4,4	7,6	0,139	0,182	0,187	
s	4,0	27,0	30,6	0,2	0,3	0,4	0,5	0,6	0,6	0,6	1,4	0,005	0,062	0,031	
<b>CEM II/A-LL (C 80 %; LL 20 %)</b>															
min	45,1	52,5	60,0	0,0	0,0	0,0	0,4	1,0	2,0	3,0	6,0	0,135	0,102	0,157	
max	54,6	67,8	67,3	0,8	1,2	1,5	2,4	3,0	4,2	6,1	9,6	0,149	0,271	0,221	
AVG	48,0	58,2	62,9	0,3	0,6	0,9	1,3	1,7	3,2	4,7	7,7	0,144	0,170	0,192	
s	2,5	3,7	2,4	0,2	0,3	0,4	0,5	0,5	0,6	0,7	0,9	0,004	0,047	0,017	
<b>CEM II/B-M (S-V) (C 65 %; S 15 %; V 20 %)</b>															
min	35,0	48,3	59,8	0,0	0,0	0,1	0,3	1,0	2,0	2,5	4,4	0,134	0,112	0,109	
max	55,4	65,3	73,1	0,4	0,7	1,4	1,8	2,1	3,3	4,5	8,6	0,169	0,246	0,204	
AVG	46,8	58,7	65,8	0,2	0,5	0,9	1,3	1,7	2,7	3,6	6,4	0,147	0,177	0,154	
s	6,8	5,5	4,2	0,2	0,2	0,4	0,5	0,4	0,5	0,7	1,4	0,012	0,048	0,032	
<b>CEM II/B-V (C 70 %; V 30 %)</b>															
min	35,9	45,6	53,2	0,0	0,1	0,5	0,7	1,4	2,4	3,7	5,9	0,137	0,126	0,144	
max	53,1	62,5	69,9	1,1	1,9	2,4	3,1	4,0	5,2	6,9	11,3	0,167	0,335	0,253	
AVG	44,2	55,4	61,0	0,6	1,0	1,6	2,2	2,6	4,0	5,5	8,3	0,151	0,244	0,195	
s	4,0	4,4	4,6	0,3	0,5	0,5	0,6	0,7	0,8	0,9	1,4	0,007	0,056	0,028	
<b>CEM III/B</b>															
min	36,8	43,6	56,9	0,0	0,0	0,0	0,5	1,0	2,0	2,9	5,0	0,135	0,112	0,122	
max	55,0	63,6	73,0	0,8	1,3	1,9	3,0	3,3	5,4	7,8	11,5	0,165	0,399	0,279	
AVG	47,3	55,2	64,7	0,4	0,7	1,2	1,7	2,1	3,4	5,1	7,9	0,146	0,212	0,193	
s	4,1	4,6	4,2	0,2	0,3	0,4	0,5	0,6	0,9	1,1	1,6	0,007	0,061	0,038	
<b>CEM I</b>															
min	41,6	51,9	59,2	0,0	0,0	0,0	0,2	0,4	1,0	2,2	3,6	0,131	0,037	0,090	
max	58,5	71,3	72,6	1,0	1,1	1,6	2,2	2,4	3,5	4,7	7,8	0,155	0,249	0,177	
AVG	50,3	60,3	66,0	0,3	0,5	0,8	1,1	1,4	2,3	3,3	5,2	0,141	0,128	0,126	
s	3,5	4,1	3,3	0,2	0,3	0,4	0,5	0,6	0,7	0,8	1,0	0,005	0,056	0,022	





**B2 Data for concrete composition 2a and 2b**

