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RAPID HARDENING SULFATE RESISTANT CALCIUM SULPHOALUMINATE BASED CEMENT

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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 (EU) 305/2011 as a basis for the preparation and issuing of European Technical Assessments (ETA). This EAD not yet published in the Official Journal of the European Union (OJEU).

Contents

1 SCOPE OF THE EAD

1.1 Description of the construction product

The construction product referred to in this document as “Rapid hardening sulfate resistant calcium sulphoaluminate based cement” is a hydraulic binder with rapid hardening features and sulfate resistance property.
The manufacturing composition of the product is the following;

Calcium sulphoaluminate clinker	50 – 90 %
Cement CEM I acc. hEN 197-1	0 – 50 %
Calcium sulfate (as defined in hEN 197-1, clause 5.4)	0 – 30 %
Minor additional constituents	< 5 %
Additives (as defined in hEN 197-1, clause 5.5)	< 2 %
of which organic additives	<0.2%

The main constituent of the product is calcium sulphoaluminate clinker (CSAK) which is made by sintering a precisely specified mixture of raw materials (raw meal, paste or slurry) containing elements, usually expressed as oxides (e.g. CaO, Al_2O_3, SiO_2, Fe_2O_3, SO_3) and small quantities of other materials.

The calcium sulphoaluminate clinker is a hydraulic material which is composed mainly by $C_4A_3SO_3$ or $C_4A_3\$$ (Yeelimite); the content of $C_4A_3SO_3$ is not less than 50% by mass, while the remaining is formed of calcium silicates ($2CaO{\cdot}SiO_2$) and other compounds. Common cement CEM I (as defined by hEN 197-1) with additional requirement for tricalcium aluminate content (C_3A) can be the other main addition. Some minor additional constituents may be added and consist mainly by the inorganic mineral materials derived from the production processes of CSAK and/or Portland cement clinker, or inorganic natural mineral materials or constituents as specified in hEN 197-1, clause 5.3.

Hydraulic hardening of “Rapid hardening sulfate resistant calcium sulphoaluminate based cement” is primarily due to $C_4A_3SO_3$ but also to Portland cement clinker content in CEM I, if present. The CSA and CSA-CEM I blend hydration products are variable depending on the proportions and characteristics of the two types of cement, as well as the calcium sulfate content.
The "Rapid hardening sulfate resistance calcium sulphoaluminate based cement" has a high sulfate resistance. It should be stated that low C_3A content of the product is not the only reason of the sulfate resistance of the product and that even an appropriate proportioning of the cement constituents is essential.

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 EAD 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 use(s)

The product is a rapid hardening special cement for the preparation of concrete, mortar, grouts and other mixes for construction and for the manufacture of construction products, including in particular cast-in-situ and prefabricated structural concrete in accordance with EN 206.

In addition the product is suitable for the preparation of concrete, mortar, grouts exposed to chemical attack from natural soils and ground water (e.g. sulfate attack).

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 the product for the intended use of 50 years when incorporated in concrete, mortars and/or grouts. The assumed working life of the product stored in appropriate conditions is 1 year. 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[1].
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.

1.3 Specific terms used in this EAD

CEM I = portland cement CEM I acc. to EN 197-1
CSA = calcium sulphoaluminate based cement
CSAK = calcium sulphoaluminate clinker
CS = calcium sulfate acc. to EN 197-1

Abbreviations:

S_{FPM} = sulfate resistance - flat prism method
S_{SPM} = sulfate resistance - square prism method
C_{dcr} = direct carbonation resistance
C_{rcr} = relative carbonation resistance
D_{nssm} = chloride non-steady state migration coefficient
D_{nss} = chloride non-steady state diffusion coefficient
FT_{cube} = Freeze – thaw resistance – cube procedure
FT_{CF} = Freeze – thaw resistance – CF procedure or CIF procedure
RDM = Relative Dynamic Modulus of elasticity
FT_{beam} = Freeze – thaw resistance – beam procedure
FTS_{CDF} = Freeze thaw resistance with de-icing salt – CDF procedure
FTS_{slab} = Freeze thaw resistance with de-icing salt – slab procedure

1 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 the "Rapid hardening sulfate resistant calcium sulphoaluminate based cement" is assessed in relation to the essential characteristics. The assessed composition and the nature of raw materials shall be stated in the ETA.

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

N°	Essential characteristic	Assessment method	Type of expression of product performance *(level, class, description)*
Basic Works Requirement 1: Mechanical resistance and stability			
1	Cement composition *CSAK content* *CEM I content* *Calcium sulfate content*	See 2.2.1	Level [% by mass] Level [% by mass] Level [% by mass]
2	Calcium sulphoaluminate ($C_4A_3\$$) content in the product	See 2.2.2	Level [% by mass]
3	C_3A content of the clinker	See 2.2.3	Level [% by mass]
4	Early strength ($3 \leq t \leq 8$ h)	See 2.2.4	Level [MPa]
5	Standard strength (28 days)	See 2.2.5	Class [MPa] acc. to hEN 197-1 table 3
6	Initial setting time	See 2.2.6	Level [min]
7	Soundness	See 2.2.7	Level [mm]
8	Sulfate content	See 2.2.8	Level [% by mass]
9	Chloride content	See 2.2.9	Level [% by mass]
10	Shrinkage	See 2.2.10	Level [mm/m]
11	Density	See 2.2.11	Level [g/cm^3]
12	Fineness	See 2.2.12	Level [cm^2/g]
13	Effect of high temperature on mortar hardened under standard conditions	See 2.2.13	Description [graph]
14	Effect of high temperature on mortar at early age	See 2.2.14	Description [graph]
15	Sulfate Resistance	See 2.2.15	Level [%]
16	Carbonation of concrete	See 2.2.16	Level [mm]; [$mm/d^{0.5}$] Description [graph]

N°	Essential characteristic	Assessment method	Type of expression of product performance *(level, class, description)*
17	Resistance to chloride penetration	See 2.2.17	Level [m^2/s]
18	Freeze-thaw resistance (without de-icing agent)	See 2.2.18	Level [%]
19	Freeze-thaw and de-icing salt resistance	See 2.2.19	Level [kg/m^2] Level [%]

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

General

The reference for taking and preparing samples of cement needed for the assessments described in the following clauses is EN 196-7.

2.2.1 Cement composition

The composition of the cement shall be checked using XRD analysis with Rietveld refinement; a spot sample taken at the point of release of the cement may be used.
The cement composition shall meet the requirements specified in Table 2.

Table 2 - Cement composition

Calcium sulphoaluminate clinker	50-90 (% by mass)
Cement CEM I acc. hEN 197-1	0-50 (% by mass)
Calcium sulfate (as defined in hEN 197-1, clause 5.4)	0-30 (% by mass)

Note. The values in the table refer to the sum of the main and minor additional constituents; also the note at clause 6.1 of the hEN 197-1 shall apply. In the case of the product, the calcium sulfate is intended as main constituent.

The values shall be stated in the ETA.

2.2.2 Calcium sulphoaluminate ($C_4A_3\$$) content of the product

The calcium sulphoaluminate content in the cement, expressed as $C_4A_3\$$, shall be determined with XRD analysis with Rietveld refinement. The total amount of $C_4A_3\$$ is obtained by adding the content of the two polymorphs ($C_4A_3\$$ cubic and $C_4A_3\$$ orthorhombic) coming directly from the XRD analysis of the calcium sulphoaluminate based cement sample.
The calcium sulphoaluminate content in the cement, expressed as $C_4A_3\$$, shall be stated in the ETA.

2.2.3 C_3A content of the clinker

The determination shall be carried out on the clinkers used in the product utilizing EN 196-2 test methods (clauses 4.5.10 and 4.5.11 for the determination of Aluminium and Iron (III) oxides) applying Equation (1) of clause 5.2.1 of hEN 197-1 (relatively to CEM I); only in the case of sulphoaluminate clinker, the

determination of the C_3A content - according hEN 197-1 clause 5.2.1 (relatively to CEM I) as above- shall be made by XRD analysis with Rietveld refinement.
The value of C_3A content of the clinker shall be stated in the ETA.

2.2.4 Early strength ($3 \leq t \leq 8$ h)

The early compressive strength of the product shall be tested according to the standard EN 196-1 amended as follows: the determination shall be carried out on halves of plastic mortar prisms, demoulded after ($3 \leq t \leq 8$) hours (± 10'), broken as in clause 9.1 and immediately subjected to the compression test.
Early compressive strength and time of demoulding shall be stated in the ETA.

Remark. Mortars based on CSA cement are sometimes characterised by quick loss of workability; in these cases this property can make difficult the correct filling of the moulds and may be necessary to apply some precautions, for example cooling of the constituents of the mortar or by addition of suitable retarding agents. When used, the cooling and/or the nature and the dosage of retarding admixture shall be stated in the ETA.

2.2.5 Standard strength

The standard compressive strength of the product shall be determined according to the standard EN 196-1 at 28 days. The compressive strength class shall be stated in the ETA.

Remark. Mortars based on CSA cement are sometimes characterised by quick loss of workability; in these cases this property can make difficult the correct filling of the moulds and may be necessary to apply some precautions, for example cooling of the constituents or by addition of suitable retarding agents. When used, the cooling and/or the nature and the dosage of retarding admixture shall be stated in the ETA.

2.2.6 Initial setting time

The determination of the initial setting time shall be carried out according to the standard EN 196-3. The value of the initial setting time shall be stated in the ETA.
In case that the manufacturer needs to fix the value of water-cement ratio, this value shall be stated in the ETA.

2.2.7 Soundness (expansion)

The determination of the soundness shall be carried out according to the standard EN 196-3.
The soundness of the product shall be ≤ 10 mm according to hEN 197-1 table 3. No test result shall exceed the above value taking into account EN 196-3 clause 7.4.
In case that the manufacturer needs to fix the value of water-cement ratio, this value shall be stated in the ETA.

2.2.8 Sulfate content

The determination of the sulfate content of the product, expressed as SO_3, shall be carried out according to the standard EN 196-2. The sulfate content of the product, expressed as SO_3, shall be stated in the ETA.

2.2.9 Chloride content

The determination of the chloride content of the product shall be carried out according to the standard EN 196-2.
The chloride content of the product shall not be greater than 0.10% by mass (acc. hEN 197-1 table 4). If the chloride content is greater than 0.10 % by mass, the upper limit value shall be stated in the ETA. For pre-stressing applications see note [f] in Table 4 of hEN 197-1; in this case the lower value shall be stated in the ETA.

2.2.10 Shrinkage (concrete method - Shr_C)

The shrinkage of concrete with the product shall be determined according to ISO 1920-8 on specimens with the dimension 75 mm x 75 mm x 280 mm.
The shrinkage shall be tested on concrete I, see below.

Table 3 - Composition of concrete I for the determination of shrinkage

concrete I	(Composition per m³ of fresh concrete) c = 320 kg g = **…..** kg aggregates according to EN 12620 with the following grading curve w/c = 0.60 (with w = effective water)						
Size [mm]	0.2	0.5	1	2	4	8	16
Passing [% by mass]	6	14	22	32	46	68	100

The specimens are to be stored for (24 ± 2) hours in the mould, covered for protection against drying, at (20 ± 2) °C and > 95 % relative humidity.
After demoulding the concrete prisms shall be cured in water maintained at (20 ± 2) °C. At an age of 7 days the specimens shall be removed from water and the surfaces shall be wiped dry with a damp cloth. The specimens shall then be stored in climate 20/65 ((20 ± 2) °C and (65 ± 5) % relative humidity) in order to determine the drying shrinkage.

The weight measurement and the length measurement shall be determined immediately after demoulding and during the water curing at 24 hours, 3 and 7 days of age. After curing in water and during conditioning in air, the weight and the length measurements shall be performed at the ages (starting from moulding) of 14, 21, 28, 35, 60, 90, 120 and 180 days. During each measurement step shall be checked the presence of cracks or any other faults of the surfaces and record them in the test report.

The shrinkage at 180 d of concrete (concrete method – **Shr_C**) prepared with the product shall be stated in the ETA, together with any cracks or faults detected during the checks as above.

2.2.11 Density

The determination of the density of the product shall be carried out according to the standard EN 1097-7; different or automatic equipment may be used provided that they can be shown to give the same results as the specified apparatus of the reference test method. The density of the product shall be stated in the ETA.

2.2.12 Fineness

The determination of the fineness of the product shall be carried out according to the standard EN 196-6. The fineness of the product shall be stated in the ETA.

2.2.13 Effect of high temperature on mortar hardened under standard conditions

The effect of aging in hot water of the mortar prepared with the product and cured under standard conditions shall be checked by comparing the compressive strength results shown by mortar prisms prepared - according to EN 196-1 - with the product and with a cement type CEM I as reference; after the curing the prisms are kept in water at the temperature of 20, 40 and 60 °C for some specified number of days, as below. The obtained results are inserted into a table; it is useful also to use a graph that can better show and compare the developments of compressive strength at different aging and water temperatures.

This test method requires the preparation of prisms of mortar, according to EN 196-1, with the product and with the reference cement CEM I cured under standard conditions for 28 days. A first set of specimens (of the product and of the reference cement CEM I) is subjected to a compression test without further aging

and their compressive strength values represent the basic value of the product and of the reference cement; the other prisms are placed in separate tanks already containing water at a temperature of (20 ± 2) °C, (40 ± 2) °C and (60 ± 2) °C where they shall be stored for further 7, 28 and 62 days. At the end of each step and at a the total age of the prisms of 35, 56 and 90 days, part of them are extracted and subjected to a compression test; the results for each temperature and aging are compared among them for the product and for the reference CEM I.

It is recommended to use non-demineralized water for storage, coming - for example - from the same containers of the first aging, in order to oppose the leakage of minerals from specimens; it is also useful to maintain other sets of prisms under water to achieve the total age of 180 and 365 days, to improve the knowledge of the product performance.
The evaluation and the assessment is carried out by comparing the strenght resistance of the product and of the reference cement CEM I for each aging and water temperature. The obtained values and diagrams shall be attached to the ETA.

2.2.14 Effect of high temperature on mortar at early age

The effect of aging in hot water of the mortar prepared with the product at early age shall be checked by comparing the compressive strength results shown by mortar prisms prepared according EN 196-1 with the product and with a cement type CEM I as reference; the prisms are demoulded after ($3 \leq x \leq 8$) hours, then cured in water at a temperature of 20, 40 and 80 °C for some specified number of days, as below. The obtained results are inserted into a table; it is useful also to use a graph that can better show and compare the developments of compressive strength at different aging and water temperatures.

This test method require the preparation of prisms of mortar with the product and with the reference cement CEM I, according to EN 196-1, cured in a moist air cabinet maintained at a temperature of (20.0 ± 1.0) °C and a relative humidity of not less than 90% for ($3 \leq x \leq 8$) hours. A first set of specimens (of the product and of the reference cement CEM I) is subjected to a compression test without further aging and represents the compressive strength value at early age; the other prisms are placed in separate tanks already containing water at a temperature of (20 ± 2) °C, (40 ± 2) °C and (80 ± 2) °C where they shall be stored for further time, in order to reach a total age of the prisms of 24 hours, 2 and 3 days. At the end of each age part of them are extracted and subjected to a compression test; the results for each temperature and aging are compared among them.

It is recommended to use non-demineralised water for storage, coming - for example - from the same containers of the first aging, in order to oppose the leakage of minerals from specimens.
The evaluation and the assessment is carried out by comparing the strength resistance of the product and of the reference cement CEM I for each aging and water temperature. The obtained values and diagrams shall be attached to the ETA.

2.2.15 Sulfate resistance

Sulfate resistance of the product shall be determined using Flat Prism Method ($\mathbf{S_{FPM)}}$ and Square Prism Method ($\mathbf{S_{SPM}}$), as reported in Annexes A and B. Some further reference sulfate resistant cements acc. hEN 197-1 - for example CEM I-SR and/or CEM III / B-SR and/or CEM IV/ -SR - shall be tested at the same moment and in the same conditions in order to compare the sulfate resistant performances of the product with the reference cements.

Method 1 - Flat prism method - S_{FPM}

Sulfate resistance $\mathbf{S_{FPM}}$ of the product shall be tested according the test method described in Annex A on mortar specimens aged up to 26 weeks in a solution of Na_2SO_4, and in a solution of saturated Calcium hydroxide. Differences of expansion and of Relative Dynamic Modulus of elasticity (RDM) of the product specimens aged in the two solutions at various ages shall be calculated.
That result and the same of the reference cements at 180 days, both at 20 and 5°C, together with diagrams and a description of the failure or cracks of the specimens – if any - shall be given in the ETA.

Method 2 - Square prism method - S_{SPM}

Sulfate resistance of the product shall be tested following the test method described in Annex B on mortar specimens aged up to 26 or 52 weeks in a solution of Na_2SO_4, and in distilled water. Differences of expansion of the specimens aged in the two solutions shall be compared. After 26 weeks the difference of expansion in percent of the product specimens stored in water shall be subtracted from the correspondent stored in sulfate solution. The result shall be compared with the same of the reference cements; the difference of expansion of the product after 26 weeks shall be lower than or equal to the same values of the reference cements at the same age.
If at that time this requirement is not fulfilled, the evaluation shall be repeated after 52 weeks.
The comparative evaluation will be considered positive if the value of the expansion of the prisms of the product after 52 weeks will be lower than or equal to the expansion value of the reference cements at the same age.
Difference of expansion in percent of all cements after 26 or 52 weeks as specified above with a description of the failure or cracks of the specimens – if any - shall be stated in the ETA.

2.2.16 Carbonation of concrete

The carbonation resistance of concrete prepared with the product shall be determined through the "direct carbonation resistance" – **C_{dcr}** – (Method 1) and by the "Relative carbonation resistance" – **C_{rcr}** – (Method 2).

Method 1: direct carbonation resistance – C_{dcr}

The direct carbonation resistance of mortar made with the product shall be measured according to prEN 12390-10:2017 and determined as follows.
The direct carbonation resistance shall be tested on concrete II as below, on prisms (40 x 40 x 160) mm prepared acc. to EN 196-1 using a modified mixer with a blade-bowl distance of (9.0 ± 1.0) mm due to the different grain size of the aggregates.

Table 4 - Composition of concrete II for the determination of carbonation depth

concrete II	(concrete mixtures for 3 specimens) c = 450 g g = 1350 g aggregates acc. to EN 12620 with the following grading curve w = 225 g water (with w = effective water)					
Size [mm]	0.25	0.5	1	2	4	8
Passing [% by mass]	8	21.5	36	46.5	67.5	100

After demoulding half of the specimens shall be cured in water (20.0 ± 1.0) °C for 6 days, until the age of 7 days (first group) and the other half (second group) for 27 days, until the age of 28 days. Then the specimens shall be stored in climate 20/65 and roughly 0.03% CO_2 concentration; measurements of carbonation depth shall be performed after the first storage in water (7 and 28 days of age) and then after 14, 28, 56, 98 and 140 days of climate 20/65 plus CO_2 storage for the delivery of the ETA. It is recommended to continue the tests on the same samples until 1, 2 and 5 years, in order to verify the first results and to improve the knowledge of the properties of the product.
In addition the compressive strength shall be determined according to EN 196-1 on the specimens as follow; the compressive strength at the end of the pre-storing and after 140 days in climate 20/65 shall be compared with the depth of carbonation at the same ages. These results shall be included in the figures X and X in Annex C.

- on specimens of the first group, 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 specimens of the second group, 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 compressive strength values in N/mm² is f_c in tables of Annex C – see below;

Record the depth of carbonation after aging in water and after each aging step in air at 20/65 and CO_2; calculate the speed of carbonation as follows.
The carbonation speed $\mathbf{v}_c$ is calculated by linear regression with:

$$d_C = d_0 + v_C \cdot \sqrt{t_C} \quad \text{and then:} \quad v_C = (d_C - d_0) / \sqrt{t_C} \quad mm / d^{0.5}$$

where:

d_0 = carbonation depth (mm) at time t = 0
d_c = carbonation depth (mm) at time t_c
t_c = duration of carbonation exposure (days)
v_c = carbonation speed (in mm / $d^{0.5}$)

The determined carbonation depth together with the strength resistance and the calculated carbonation speed of prisms shall be inserted in the diagrams and figures given in Annex C. Values of d_c, v_c, figures and diagrams shall be stated in the ETA.

Method 2: relative carbonation resistance – C_{rcr}

The relative carbonation resistance of concrete made with the product and of reference cement CEM I 52,5 R acc. hEN 197-1 shall be tested according to CEN/TS 12390-10; the carbonation resistance of the concretes made with the product and with the reference cement shall be tested on concrete III_a (concrete made with the product) and concrete III_b (reference concrete), see the following table.

Table 5 - Composition of concrete for the determination of relative carbonation resistance

Composition per m³ of fresh concrete	
concrete III_a	c = 350 kg of product g = **…** kg aggregates according to EN 12620 – see*) – shall be used w = 175 l water (w=effective water)
concrete III_b	c = 350 kg of CEM I 52.5 R g = … kg aggregates according to EN 12620– see*) – shall be used w = 175 l water (w=effective water)

*) Aggregates according to EN 12620 with the following grading curve

Size [mm]	0.125	0.25	0.5	1	2	4	8	16	32
Passing [% by mass]	5	9	14	20	30	43	62	89	100

Due to the very early setting time of some formula, the addition of a retarding admixture can facilitate the preparation of test specimens. When used, the nature and the dosage of the retarder shall be reported in the ETA.
Concrete prisms shall be stored in a climate controlled chamber with 20/65 conditions and a CO_2 content of (0.035±0.005)%.

The assessment of the relative carbonation resistance of the concretes shall be performed on prisms aged for 182 days, when the carbonation depth of the concretes shall be measured. Measurements of carbonation depth shall be performed after 182 days in order to achieve the ETA, but it is recommended to

continue the tests on the same samples after 273, 365, 547 and 730 days in order to verify the first results and to improve the knowledge of the properties of the product. The determined carbonation depth of the concretes after 182 days shall be stated in the ETA.

2.2.17 Resistance to chloride penetration

The resistance to chloride penetration in the concrete prepared with the product shall be determined through the "non-steady-state migration experiments" (Chloride migration coefficient - **D_{nssm}** - Method 1) and/or by the non-steady-state diffusion coefficient of chlorides (Chloride diffusion coefficient – **D_{nss}** - Method 2). The former is the reference test method, the latter is optional.

Method 1: Chloride migration coefficient - D_{nssm}

The chloride migration coefficient of concrete made with the product and with Portland cement CEM I as reference shall be determined in accordance with the test method given in Annex D, on concrete IV_a and IV_b as in the following table.

Table 6 - Composition of concrete for the determination to chloride penetration

Composition per m³ of fresh concrete	
concrete IV_a	c = 320 kg of product g = **…** kg aggregates according to EN 12620 – see*) – shall be used $\frac{w}{c} = 0{,}50$ (w=effective water)
concrete IV_b	c = 320 kg of CEM I 52.5 R g = **…** kg aggregates according to EN 12620 – see*) – shall be used $\frac{w}{c} = 0{,}50$ (w=effective water)

*) Aggregates according to EN 12620 with the following grading curve

Size [mm]	0.25	0.5	1	2	4	8	16
Passing [% by mass]	6	14	22	32	46	68	100

The determination of the chloride migration coefficient (**D_{nssm}**) of concrete made with the product and of the reference concrete shall be performed on specimens aged for 35 and 97 days applying the test method given in Annex D. The comparison of the concrete of the product with the concrete made with CEM I 52.5 R shall be used to verify the correct execution of the test and for a further evaluation of the properties of the product.
The chloride "non-steady-state migration coefficient" of concrete (**D_{nssm}**) made with the product at an age of 97 days shall be stated in the ETA.

Method 2: Chloride diffusion coefficient - D_{nss}

The resistance to chloride penetration of concrete made with the product and with CEM I as reference shall be determined in accordance to the standard EN 12390-11 by the non-steady-state chloride diffusion coefficient (D_{nss}). The resistance to chloride penetration shall be tested on cylindrical specimens of concrete IVa (concrete made with the product) and concrete IVb (reference concrete), see above; after division, the sub-specimens shall be sealed as in clause 6.2.2 a) of the standard and shall be exposed by immersion (clause 7.2.2 of the standard).
The evaluation of the resistance to penetration of chlorides by method **D_{nss}** shall be performed through the comparison of the results obtained on the concretes prepared with the product and with the CEM I.
The value of **D_{nss}** of concretes made with the product and with the reference cement shall be stated in the ETA.

2.2.18 Freeze-thaw resistance (without de-icing agent)

In order to verify the freeze - thaw resistance of the concrete prepared with the product, three different test methods are reported below; none of the results is comparable with the others but they can provide specific and peculiar information. Test method 3 (Beam-Procedure) shall be used as reference method, while the others are optional.

Method 1: Freeze-thaw resistance - scaling (Cube-Procedure) - FT_{cube}

The "freeze-thaw resistance – scaling – (Cube procedure) - **FT_{cube}**" of concrete made with the product shall be determined according to CEN/TS 12390-9 clause 6 on concrete type V, see below; the compressive strength of concrete V made with the product shall be determined according to EN 12390-3 after 28 days and reported in the ETA. The freezing medium (clause 6.2.4 of the standard) shall be de-ionised water.

Table 7 - Composition of concrete for the determination of the freeze-thaw resistance without de-icing agent

Composition per m³ of fresh concrete	
concrete V	c = 300 kg of product g = **…** kg aggregates according to EN 12620 – see*) – shall be used $\frac{w}{c} = 0{,}60$ (w=effective water)

*) Aggregates according to EN 12620 with the following grading curve

Size [mm]	0.125	0.25	0.5	1	2	4	8	16	32
Passing [% by mass]	1.5 **)	5	23	35	45	56	70	85	100

**) recommended value

In order to assess the scaling resistance of the concrete type V prepared with the product, the cumulative mass of the dried scaled material at 56 days/56 cycles shall be reported in the ETA, together with the results of visual inspections on samples for the presence of cracks or major defects, if any, and the compressive strength of the concrete type V. The TAB can also choose to continue the measurement of the scaling and the visual inspection until to 75 and 100 freeze-thaw cycles in order to verify the first results.

Method 2: Freeze-thaw resistance (CF-Procedure) - FT_{CF}

The freeze-thaw resistance of concrete made with the product shall be tested according to CEN/TS 12390-9 clause 7 - "CF-test" - to determine the loss of material (scaling) and according to CEN/TR 15177 clause 9 – CIF-test to determine the internal structural damage (Relative Dynamic Modulus of elasticity - RDM). Both test methods shall be applied on concrete IV_a (see 2.2.17 – Method 1) and de-mineralized water shall be used as freezing medium. Furthermore the compressive strength of concrete IVa made with the product shall be determined according to EN 12390-3 after 28 days and reported in the ETA. 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 scaling and the Relative Dynamic Modulus of elasticity (RDM) shall be measured after 7, 14, 28, 42 and 56 freeze-thaw cycles. The capillary suction of water of any specimen should be measured and reported for further information.

The evaluation of the results of the two tests allow to give an overall information about the internal and external damage of the specimen subjected to freeze-thaw cycles as above. The results of the surface scaling and the Relative Dynamic Modulus of elasticity after 28 and 56 freeze-thaw cycles shall be stated in the ETA, together with the compressive strength as above and any information for visual defects of the specimens.

Method 3: Freeze-thaw resistance (Beam-Procedure) - FT_{beam}

The freeze-thaw resistance - internal structural damage (Beam-Procedure) - **FT_{beam}** of concrete made with the product shall be determined according to CEN/TR 15177 clause 7 on concrete IIIa (see 2.2.16 – Method 2). Record the values of the Relative Dynamic Modulus of elasticity – RDM - after 7, 14, 28, 42 and 56 freeze-thaw cycles; the test may be continued until 100, 150, 200, 250 and 300 freeze-thaw cycles in order to verify the first results. Report the RDM values after 56 cycles and, when appropriate, also after 300 freeze-thaw cycles in the ETA.

2.2.19 Freeze-thaw and de-icing salt resistance

In order to verify the freeze - thaw and de-icing resistance of the concrete prepared with the product, two different test methods are reported below; in any case, test method 2 is the reference one, according to CEN/TS 12390-9. Even each result is not comparable with the other, together they can provide an overall evaluation of the product characteristics, especially referred to the internal and external damage of the specimen subjected to freeze-thaw cycles.

Method 1: Freeze-thaw and de-icing resistance (CDF-Procedure) - FTS_{CDF}

The freeze-thaw and de-icing resistance - **FTS_{CDF}** - of concrete made with the product shall be tested according to CEN/TS 12390-9 clause 7 - "CDF-test" - to determine the loss of material (scaling) after 28 freeze-thaw cycles and according to CEN/TR 15177 clause 9 – "CIF-test" to determine the internal structural damage (Relative Dynamic Modulus of elasticity - RDM). Both test methods shall be applied on concrete VI (see below) and shall use a de-icing solution as freezing medium. The scaling shall be measured after 4, 6, 14 and 28 freeze-thaw cycles, while the Relative Dynamic Modulus of elasticity (RDM) shall be measured after 7, 14, 28, 42 and 56 freeze-thaw cycles. The concrete shall contain (4.5 ± 0.5)% of air voids when determined according to EN 480-11.

Table 8 - Composition of concrete for freeze-thaw resistance with de-icing agent determination

Composition per m³ of fresh concrete	
concrete VI	c = 320 kg of product g = **…** kg aggregates according to EN 12620 – see*) – shall be used $\frac{w}{c} = 0{,}50$ (w=effective water) Concrete with air entraining agent. (The air content of the fresh concrete shall be 4.5 ± 0.5 Vol.-%.)

*)Aggregates according to EN 12620 with the following grading curve

Size [mm]	0.25	0.5	1	2	4	8	16
Passing [% by mass]	6	14	22	32	46	68	100

The compressive strength of concrete VI made with the product shall be determined according to EN 12390-3 after 28 days and reported in the ETA. 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 shall be measured after 0, 4, 6, 14 and 28 freeze-thaw cycles. The air void content shall be determined according to EN 480-11 on concrete VI.
The values of the surface scaling of the concrete prepared with the product and tested according to CEN/TS 12390-9 clause 7 – "CDF-Test" - after 28 freeze-thaw cycles shall be stated in the ETA.
The Relative Dynamic Modulus of elasticity of the concrete prepared with the product and tested according to CEN/TR 15177 clause 9 – "CIF-test" - shall be stated in the ETA, together with any information about visual defects of the specimens.

Method 2: Freeze-thaw resistance (Slab-Procedure) - FT_{slab}

The freeze-thaw and de-icing resistance (Slab-procedure) - **FT_{Slab}** - of concrete made with the product shall be tested according to CEN/TS 12390-9 clause 5 to determine the loss of material (scaling) and according to CEN/TR 15177 clause 8 to determine the internal structural damage as Relative Dynamic Modulus of elasticity - RDM. Both test methods shall be applied on concrete IIIa, (see 2.2.16 – method 2) and shall use a de-icing solution as freezing medium.
The loss of material - scaling – of the concrete type IIIa tested according to CEN/TS 12390-9 clause 5 – (Slab Test) - after 28 freeze-thaw cycles shall be reported in the ETA.
The Relative Dynamic Modulus of elasticity - RDM - of the concrete type IIIa prepared with the product and tested according to CEN/TR 15177 clause 8 – **FT_{Slab}** - shall be measured after 7, 14 and 28 freeze-thaw cycles; the results of the RDM after 28 freeze-thaw cycles shall be stated in the ETA, together with any information about visual defects of the specimens.

3 ASSESSMENT AND VERIFICATION OF CONSTANCY OF PERFORMANCE

3.1 System(s) of Assessment and Verification of Constancy of Performance to be applied

Decision 1997/555/EC as amended by Decision 2010/683/EU is the European legal act applicable to the products covered by this EAD.
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 9.

Table 9 - Control plan for the manufacturer: cornerstones

N°	Subject/ type of control *(product, raw/constituent material, component - indicating characteristic concerned)*	Test or control method	Criteria, if any	Minimum number of samples [g]	Minimum frequency of control	Statistical assessment procedure [a]	
						Variables [b]	Attributes
Factory Production Control (FPC) [including testing of samples taken at the manufacturing plant by the manufacturer in accordance with the prescribed test plan]							
1	Cement	Cement composition: CSAK content; CEM I content Calcium sulfate content	Control plan	1	1/month [d] 1/week [e]		
2		Calcium sulphoaluminate ($C_4A_3\$$) content	Control plan	1	1/month [d] 1/week [e]		x
3	Constituent clinker	C_3A content in clinker	Control plan	1	Every 2 weeks [d] 1/week [e]		x [f]
4	Cement	Early strength ($3 \leq t \leq 8$ h)	Control plan	1	2/week [d] 4/week [e]	x	
5		Standard strength (28 days)	Control plan	1	2/week [d] 4/week [e]	x	
6		Initial setting time	Control plan	1	2/week [d] 4/week [e]		x [f]
7		Soundness	Control plan	1	2/week [d] 4/week [e]		x

N°	Subject/ type of control *(product, raw/constituent material, component - indicating characteristic concerned)*	Test or control method	Criteria, if any	Minimum number of samples g)	Minimum frequency of control	Statistical assessment procedure a)	
						Variables b)	Attributes
8	Cement	Sulfate content	Control plan c	1	2/week d) 4/week e)		x f)
9		Chloride content	EN 196-2 c)	1	Every 2 weeks d) 1/week e)		x f)
10		Density	Control plan c)	1	1/month d) 1/week e)		x f)
11		Fineness	EN 196-6 c)	1	2/week d) 4/week e)		x f)

a) The statistical assessment procedure is carried out according to hEN 197-1, clause 9.2.

b) If the data are not normally distributed then the method of assessment may be decided on a case-by-case method.

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

d) Routine situation.

e) Initial period (3 months).

f) If the number of samples is at least one per week during the control period, the assessment may be made by variables.

g) The methods used to take and prepare samples shall be in accordance with EN 196-7.

The evaluation of the results of the FPC shall be done in accordance with Clause 9.2 of the standard hEN 197-1 and Clause 5.3 of the standard EN 197-2.

3.3 Tasks of the notified product certification body

The cornerstones of the actions to be undertaken by the notified product certification body in the process of assessment and verification of constancy of performance for the product shall be based on the standard EN 197-2 and on the following table 10.

Table 10 - Control plan for the notified product certification body: cornerstones

N°	Subject/type of control *(product, raw/constituent material, component - indicating characteristic concerned)*		Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
Initial inspection of the manufacturing plant and of the Factory Production Control						
1	The initial inspection of the factory and of the factory production control shall comply with clause 5.5.1 and/or clause 5.5.2 of the Standard EN 197-2. In addition, the inspection shall assess the suitability of the production equipment in relation to the Works' quality manual, according to clause 5.5.3 of the Standard EN 197-2.				At the beginning of the contract between notified product certification body and manufacturer or in the case of a new type of product (acc. clause 5.5.2 of the Standard EN 197-2)	
Continuous surveillance, assessment and evaluation of the Factory Production Control						
2	Initial period (first three months of dispatching)	Audit sampling (three samples)	See table 9		1	1/month
		Evaluation of the first and of the following three samples results together with the corresponding autocontrol test results	According to clause 5.6 of EN 197-2			At the end of the tests
3	Routine situation	Audit sampling of the product	See table 9		1	6/year
		Assessment of the factory production control and evaluation of the autocontrol test results according to clause 6.2.1 of EN 197-2. Evaluation of the audit test results according to clause 6.2.2 of EN 197-2. Issuing a report of surveillance.				1/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 – Part 1: Determination of strength
EN 196-2	Methods of testing cement - Part 2: Chemical analysis of cement
EN 196-3	Methods of testing cement - Part 3: Determination of setting times and soundness
EN 196-6	Methods of testing cement - Part 6: Determination of fineness
EN 196-7	Methods of testing cement - Part 7: Methods of taking and preparing samples of cement
EN 197-1	Cement - Part 1: Composition, specification and conformity criteria for common cements
EN 197-2	Cement – Part 2: Conformity evaluation
EN 206	Concrete - Specification, production, performance and conformity
EN 480-11	Admixtures for concrete, mortar and grout - Test methods – Part 11: Determination of air void characteristics in hardened concrete; German version
EN 1097-7	Tests for mechanical and physical properties of aggregates. Determination of the particle density of filler. Pyknometer method
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
EN 12620	Aggregates for concrete
CEN/TS 12390-9	Testing hardened concrete - Part 9: Freeze-thaw resistance. Scaling
CEN/TS 12390-10	Testing hardened concrete - Part 10: Determination of the relative carbonation resistance of concrete
prEN 12390-10:2017	Testing hardened concrete – Part 10: Determination of the carbonation resistance of concrete at atmospheric levels of carbon dioxide
prCEN/TS 12390-11	Testing hardened concrete - Determination of the chloride resistance of concrete, unidirectional diffusion
CEN/TR 15177	Testing the freeze-thaw resistance of concrete - Internal structural damage
ISO 1920-8	Testing of concrete – Part 8: Determination of drying shrinkage of concrete for samples prepared in the field or in the laboratory

ANNEX A – SULFATE RESISTANCE – FLAT PRISM METHOD

Testing the Sulfate Resistance of Special Cements – Flat prism method S_{FPM}

A.1 References

EN 196-1 Methods of testing cement - Part 1: Determination of strength.

A.2 General description of the test method

The sulfate resistance of the hydraulic binder according to the flat prism method (**S_{FPM}**) shall be tested on specimens made of mortar according to EN 196-1; some specimens will be fitted with pins for measurement of the length, the others will be used for the control of the Relative Dynamic Modulus of elasticity (RDM).
The curing shall be made up to 2 weeks of age in a saturated solution of calcium hydroxide; Then, half of the prisms will be transferred into two containers with a sodium sulphate solution at 4.4%, one of them maintained at the temperature of 5°C and the other at 20°C. The other prisms will be kept still in saturated calcium hydroxide solution, half of these at 5 and half at 20 °C. All of them will be weighed before the measurement to check for any weight changes; all measures will be carried out at every maturities up to 180 days.
All the values shall be reported in the ETA together with any annotations on the presence of cracks or swelling or otherwise.

A.3 Preparation of test specimens

24 flat prisms with dimensions (10 x 40 x 160) mm (12 with and 12 without measuring pin) from each mortar shall be prepared in accordance with and/or following EN 196-1 and compacted by means of a vibrating table.

A.4 Storage of test specimens

The 24 flat prisms shall stored for 2 days in the mould at (20.0±1.0)°C and a relative air humidity percentage not less than 95 % RH.
After demoulding the 24 flat prisms shall be pre-stored until the age of 14 days, on edge, standing on gratings in saturated Ca(OH)2 solution at 20 °C.
At the age of 14 days, a set of 3 flat prisms with measuring pin and 3 flat prisms without measuring pin shall be stored on edge, standing on gratings in a 4.4% Na2SO4 solution at (5.0±1.0)°C; another set of 3+3 flat prisms shall be maintained in an identical solutions maintained at (20.0±1.0)°C (sulfate storage at 5 and 20°C).
One set of 3 flat prisms with measuring pin and 3 flat prisms without measuring pin shall be stored on edge, standing on gratings in a saturated Ca(OH)2 solution at (5.0±1.0)°C; another set of 3+3 flat prisms shall remain stored in saturated Ca(OH)2 solution at (20.0±1.0) °C (reference storage at 5 and at 20°C).
In all storages the ratio of volumes of solution/solid must be 3:1 to 5:1. The solution of Na2SO4 is to be replaced every 14 days with a new Na2SO4 solution, temperature-controlled at 5 °C respectively 20 °C. The saturation of the Ca(OH)2 solution is to be checked every 14 days; if necessary it has to be concentrated.

A.5 Test

The length of the flat prisms and their RDM will be measured after a period of storage of 0, 14, 28, 56, 90, and 180 days into the solutions of sodium sulfate and calcium hydroxide. In addition the mass of the flat prisms is to be determined.
Photos of the specimens should be taken after every testing to illustrate the formations of the cracks.

A.6 Analysis

The elongation of the flat prisms is to be determined as mean value of the measured values from 3 specimens and the difference in elongation between the sulfate storage and the reference storage is to be assessed.
The Relative Dynamic Modulus of elasticity is to be determined as mean value of the group of three specimens.
The elongation difference and the Relative Dynamic Modulus of elasticity are to be stated in the test report for all test steps.

The expansion (in percent) of every prism at time (t) shall be determined by the equation:

$$\Delta_L\ (\%) = L_t - L_0 * 100/160$$

where: Δ_L is the length variation between time (t) and time (0), as a percentage of the length of the prism (160 mm) excluding pins.

L_0 is the prism length at time (0)

L_t is the prism length at time (t)

The difference between the average of Δ_L (%) of the group of prisms cured in sulfate solution and the corresponding cured in calcium hydroxide solution shows the length variation Δ_{SS} (%) due to the sulfate storage at time (t); the value of Δ_{SS} may be included in a diagram with the aim of showing more clearly the trend of the samples length in time.

ANNEX B – SULFATE RESISTANCE – SQUARE PRISM METHOD

Testing the Sulfate Resistance –Square prism method S_{SPM}

This test method is adapted from the CUR – NL —Civieltechnisch Centrum Uitvoering Research en Regelgeving (Centre for Civil Engineering Research and Codes) - Recommendation 48 – suitability test for new cements for application in concrete

B.1 References

EN 196-1 Methods of testing cement - Part 1: Determination of strength.

B.2 Apparatus and solution

B.2.1 Containers

The containers for storage of distilled water and sulfate solution must have a capacity of 1.5 and 2.5 liter and measure at least 180 mm x 80 mm. Each container must be capable of containing 1.0 ± 0.1 liter of liquid, so that the depth of the liquid reaches at least 25 mm. All the containers must be fitted with light-proof lids and must be manufactured in a material that does not react with its content.
It is allowed to put specimens of different cement types in a single container provided that the chemical composition of the cement is equivalent. In this case 1.0 ± 0.1 liter of liquid are used per three specimens.

B.2.2 Sulfate Solution

The sulfate solution must have a concentration of 16 ± 0.5 g SO_4 per liter, and is prepared by adding Na_2SO_4 or $Na_2SO_4 \cdot 10\ H_2O$ of analytical purity to distilled water, or to water of the same purity.

Note: The SO_4-content of the Na_2SO_4 must be measured before the solution is prepared, or the SO_4-content of the solution must be measured and corrected, if necessary.

B.3 Preparation of test specimens

The mortar shall be prepared in accordance with paragraph 6 of EN 196-1 Standard using CEN Standard sand, distilled water or water of equal purity, 6 prisms from each mortar with the dimensions (20 x 20 x 160) mm with two stainless steel studs shall be made and then demoulded in accordance with clauses 7 and 8 of EN 196-1.

B.4 Conditioning

The specimens shall be cured according to clause 8.3 of EN 196-1; immediately after demoulding the specimens shall be placed in 2 containers (3 specimens in every container) each one containing 1 liter of distilled water.
The specimens must be placed along each other with at least 5 mm of space between them, at least 5 mm water above and at least 5 mm of distance from the sides of the containers. The specimens must be placed on supports at least 2 mm clear from the bottom of the containers.

B.5 Testing procedure

At the age of 28 days the length of each specimen shall be measured. Before carrying out the measurements, the apparatus must be calibrated using the reference bar. Note the result or adjust the measuring apparatus to the standard value. Remove one specimen at a time and clean the measuring points with a damp cloth. Note the measured value L(0).

Immediately after the measurement, enter the prisms in the containers: replace the first three in the container with distilled water, and the second three in a new container with 1 liter of sulfate solution for the next storage period. All the containers shall be stored with a sealed lid at (20 ± 2)°C.
The distilled water shall not be changed during the whole storage period but fill up the water level with additional water if necessary.
The sulfate solution shall be replaced every 28 days.

B.6 Test

Measure the length of the prisms L(t) in the same way after 4, 8, 12, 16, 20, 26, 29, 40 and 52 weeks in the container.
Photos of the specimens should be taken after every testing to illustrate the eventual formations of some cracks. Record any visible degradation of the specimens.

B.7 Analysis

For each storage period (t), measure the changes in the length of each specimen in relation to the length L_0 as a percentage of the standard length of 160 mm, rounded to an accuracy of 0.005 %.

The expansion (in percent) of every prism at time (t) shall be determined by the equation:

$$\Delta_L\ (\%) = L_t - L_0 * 100/160$$

where: Δ_L is the length variation between time (t) and time (0), as a percentage of the length of the prism (160 mm) excluding pins.

L_0 is the prism length at time (0)

L_t is the prism length at time (t)

The difference between the average of Δ_L (%) of the group of prisms cured in sulfate solution and the corresponding cured in water shows the length variation Δ_{SS} (%) due to the sulfate storage at time (t); the values of Δ_{SS} may be included in a diagram with the aim of showing more clearly the trend of the samples length in time.

ANNEX C – CARBONATION RESISTANCE

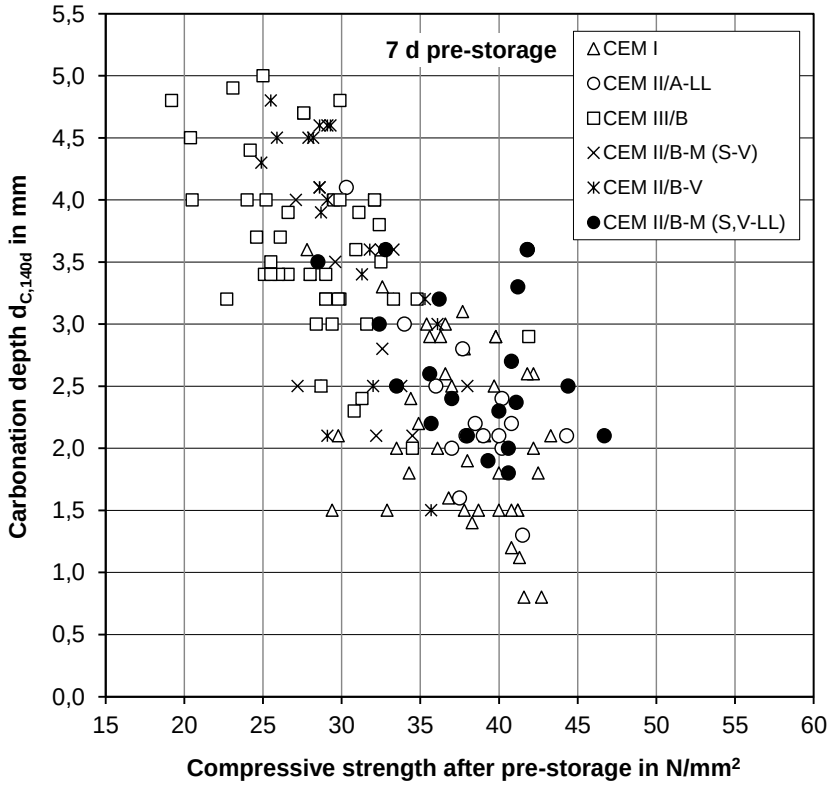
Evaluation of the carbonation resistance - $\mathbf{C_{dcr}}$

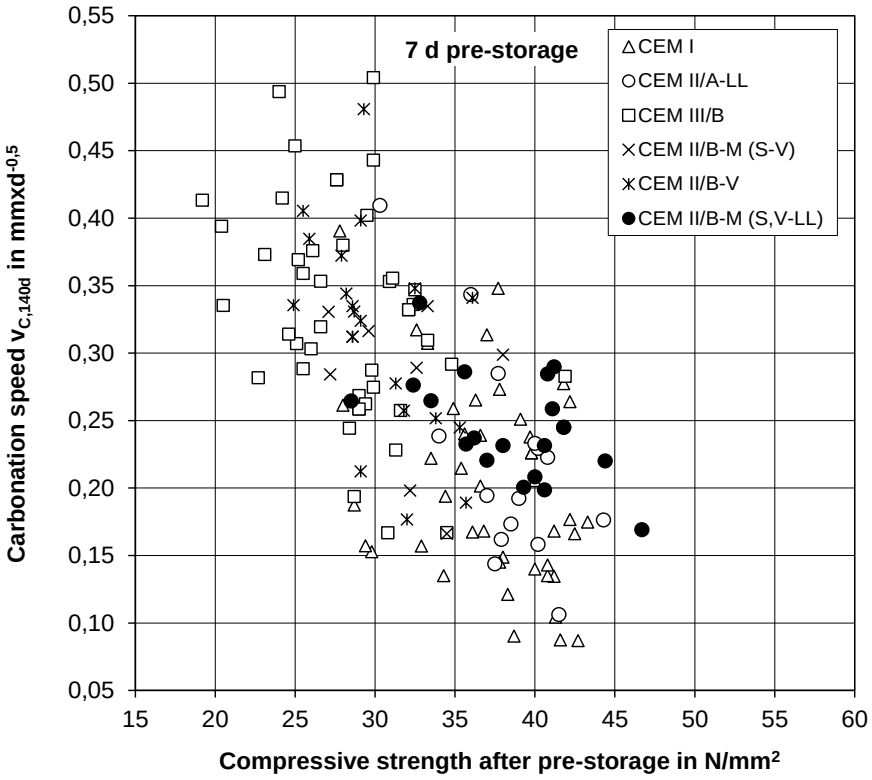
Carbonation test on concrete (w/c = 0.50) - **7** days pre-storage

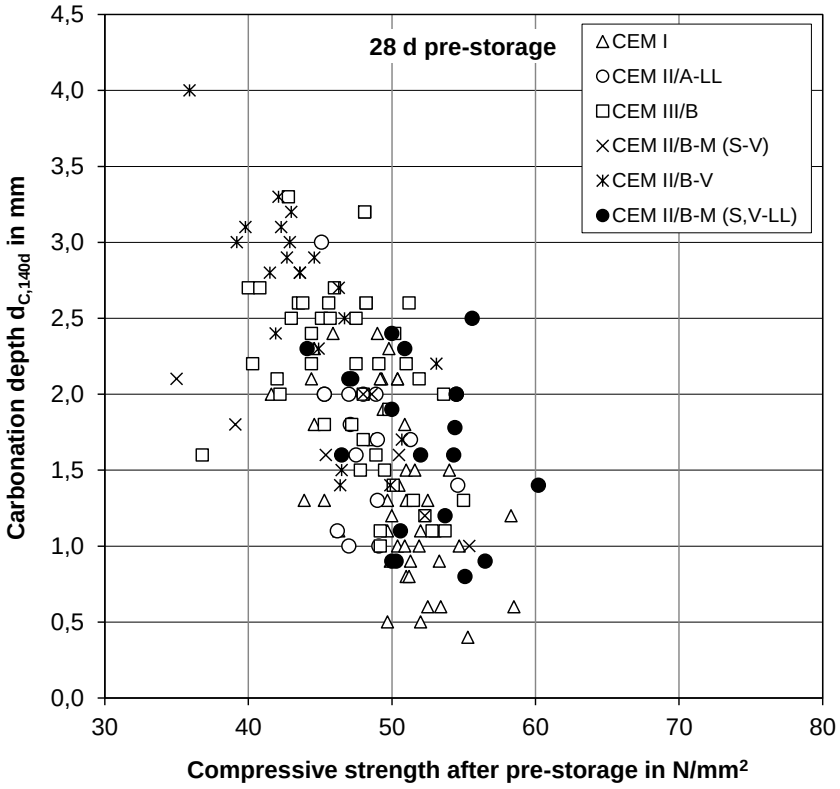
	f_C in N/mm²			Carbonation depth in mm								$F_{C,P}^{-0.5}$ in $N^{-0.5}$ x mm	Carbo. speed in mm / $d^{0.5}$	
	Pre-st. 7 d	35 d	140 d main-st.	14 d	28 d	56 d	98 d	140 d	1 a	2 a	5 a		$v_{C.140d}$	$v_{C.5a}$
CEM II/B-M (S. V-LL)														
min	28.5	51.2	50.8	0.2	0.4	1.0	1.5	1.8	3.1	3.7	6.8	0.146	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
CEM II/A-LL (C 80 %; LL 20 %)														
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
CEM II/B-M (S-V) (C 65 %; S 15 %; V 20 %)														
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
CEM II/B-V (C 70 %; V 30 %)														
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
CEM III/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
CEM I														
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

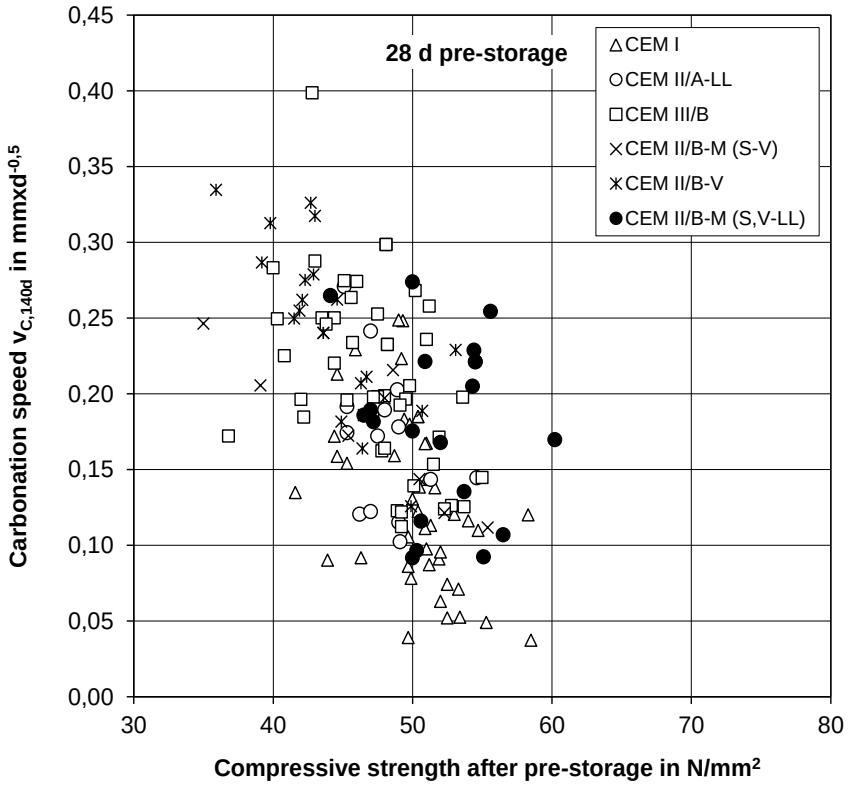
Carbonation test on concrete (w/c = 0,50) - **28** days pre-storage

	f_C in N/mm²			Carbonation depth in mm								$F_{C,P}^{-0,5}$ in $N^{-0,5}$ x mm	Carbo. speed in mm / $d^{0,5}$	
	Pre-st. 28 d	35 d	140 d main-st.	14 d	28 d	56 d	98 d	140 d	1 a	2 a	5 a		$v_{C,140d}$	$v_{C,5a}$
CEM II/B-M (S, V-LL)														
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
CEM II/A-LL (C 80 %; LL 20 %)														
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
CEM II/B-M (S-V) (C 65 %; S 15 %; V 20 %)														
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
CEM II/B-V (C 70 %; V 30 %)														
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
CEM III/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
CEM I														
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


7 d pre-storage
CEM I
CEM II/A-LL
CEM III/B
CEM II/B-M (S-V)
CEM II/B-V
CEM II/B-M (S,V-LL)
Carbonation depth $d_{C,140d}$ in mm
Compressive strength after pre-storage in N/mm²
5,5
5,0
4,5
4,0
3,5
3,0
2,5
2,0
1,5
1,0
0,5
0,0
15
20
25
30
35
40
45
50
55
60


7 d pre-storage
CEM I
CEM II/A-LL
CEM III/B
CEM II/B-M (S-V)
CEM II/B-V
CEM II/B-M (S,V-LL)
Carbonation speed $v_{C,140d}$ in mmxd$^{-0,5}$
Compressive strength after pre-storage in N/mm²
0,55
0,50
0,45
0,40
0,35
0,30
0,25
0,20
0,15
0,10
0,05
15
20
25
30
35
40
45
50
55
60


28 d pre-storage
CEM I
CEM II/A-LL
CEM III/B
CEM II/B-M (S-V)
CEM II/B-V
CEM II/B-M (S,V-LL)
Carbonation depth $d_{C,140d}$ in mm
Compressive strength after pre-storage in N/mm²
4,5
4,0
3,5
3,0
2,5
2,0
1,5
1,0
0,5
0,0
30
40
50
60
70
80


28 d pre-storage
CEM I
CEM II/A-LL
CEM III/B
CEM II/B-M (S-V)
CEM II/B-V
CEM II/B-M (S,V-LL)
Carbonation speed $v_{C,140d}$ in mmxd$^{-0,5}$
Compressive strength after pre-storage in N/mm²
0,45
0,40
0,35
0,30
0,25
0,20
0,15
0,10
0,05
0,00
30
40
50
60
70
80

ANNEX D – RESISTANCE TO CHLORIDE PENETRATION

Testing the Resistance to Chloride Penetration by the Non-Steady-State Migration Experiments – Chloride migration coefficient D_{mig}

This test method is adapted from NT Build 492 - Chloride migration coefficient from non-steady-state migration experiments

D.1 Scope

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

D.2 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.

D.3 References

EN 12390-1 Testing hardened concrete – Part 1: Shape, dimensions and other requirements for specimens and moulds
EN 12390-2 Testing hardened concrete – Part 2: Making and curing specimens for strength tests

D.4 Test method

D.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 be then measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth.

D.4.2 Reagents and apparatus

D.4.2.1 Reagents

Distilled or deionised water.
Calcium hydroxide: $Ca(OH)_2$, technical quality.
Sodium chloride: NaCl, chemical quality.
Sodium hydroxide: NaOH, chemical quality.
Silver nitrate: $AgNO_3$, chemical quality.

D.4.2.2 Apparatus

Water-cooled diamond saw.
Catholyte reservoir.
Migration set-up (Figure D 1 in Appendix D 1) includes the following parts:
Silicone rubber sleeve: inner/outer diameter 100 ÷ 115 mm, about 150 mm long.
Stainless steel clamp: diameter range 105 ÷ 115 mm, about 20 mm wide (see Figure D 2 in Appendix D 1).
Plastic support: (see Figure D 3 in Appendix D 1).
Cathode: stainless steel plate (see Figure D 3 in Appendix D 1), about 0.5 mm thick.

Anode: stainless steel mesh or plate with holes (see Figure D 4 in Appendix D 1), 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 ±0.1 V.
Ammeter: capable of displaying current to ±1 mA.
Thermometer or thermocouple with readout device capable of reading to ±1°C.
Any suitable device for splitting the specimen.
Spray bottle.
Slide calliper with a precision of ±0.1 mm.
Ruler with a minimum scale of 1 mm.

D.4.3 Preparation of the test specimen

6 cylinders from each concrete with a diameter of 100 mm and a length of 200 mm shall be made in accordance to EN 12390-1 and EN 12390-2.
The specimens shall be stored for 24 hours in the mould at climate (20/95). After demoulding the specimens shall be stored in water at 20 ± 5°C until testing. At an age of 28 days respectively 90 days 3 specimens of each concrete are taken out of the water.

Prepare the test specimen by first cutting each cylinder into two halves (i.e. into ø 100 x 100 mm cylinders), and then cutting a 50 ± 2 mm thick slice from one half. The end surface that was nearer to the first cut (the middle surface) is the one to be exposed to the chloride solution (catholyte). Measure the thickness of each slice with a slide calliper and read to 0.1 mm.

Note D 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.

D.4.4 Test procedure

D.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 liter of water). Store the solutions at a temperature of 20÷25 °C.

D.4.4.2 Temperature

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

D.4.4.3 Preparation of the test (for any migration set-up)

Fill the catholyte reservoir with 10% NaCl solution.
Fit the rubber sleeve on the specimen as shown in Figure D 4 in Appendix D 1 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 D 1 in Appendix D 1).
Fill the sleeve above the specimen with 300 ml anolyte solution (0.3 N NaOH). On no account must the two solutions be allowed to mix.
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.

D.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 D 1 in Appendix D 2). 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 D 1 – Appendix D 2).
Record the final current and temperature before terminating the test.

D.4.4.5 Measurement of chloride penetration depth

Disassemble the specimen by following the reverse of the procedure in D.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
Spray 0.1 M silver nitrate solution on to the freshly split sections.
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 D 5 in Appendix D 1) to obtain seven depths (notes 2, 3 and 4). Measure the depth to an accuracy of 0.1 mm. The arithmetic mean x_d is calculated to 0.5 mm from the individual values of the penetration depth of the test specimens (x_1, x_2,.. x_n). The maximum penetration depth x_{max} should also be reported.

Note D 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 D 3: if there is a significant defect in the specimen which results in a penetration front much greater than the average, ignore this front as indicative of the penetration depth, but note and report the condition.
Note D 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 D 5 in Appendix 1).

D.5 Expression of results

D.5.1 Test results

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

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

Where:

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

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

D_{nssm}: non-steady-state migration coefficient, m²/s;
z: absolute value of ion valence, for chloride, z = 1;
F: Faraday constant, F = 9.648 ×10⁴ 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;

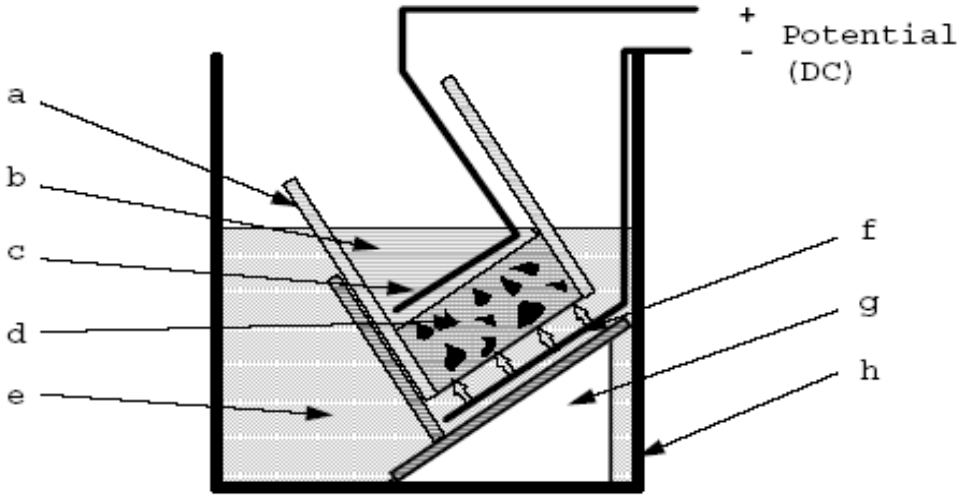
x_d: average value of the penetration depths, m;
t: test duration, seconds;
erf^{-1}: inverse of error function;
c_d: chloride concentration at which the colour changes, $c_d \approx 0.07$ N for OPC concrete;
c_0: chloride concentration in the catholyte solution, $c_0 \approx 2$ N.

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

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

Where:
D_{nssm}: non-steady-state migration coefficient. $\times 10^{-12}$ m²/s;
U: absolute value of the applied voltage, V;
T: average value of the initial and final temperatures in the anolyte solution, °C;
L: thickness of the specimen, mm;
x_d: average value of the penetration depths, mm;
t: test duration, hour.

Appendix D 1



a. Rubber sleeve
b. Anolyte
c. Anode
d. Specimen
e. Catholyte
f. Cathode
g. Plastic support
h. Plastic box

Figure D.1 - Arrangement of the migration set-up.

Figure D.2: Stainless steel clamp

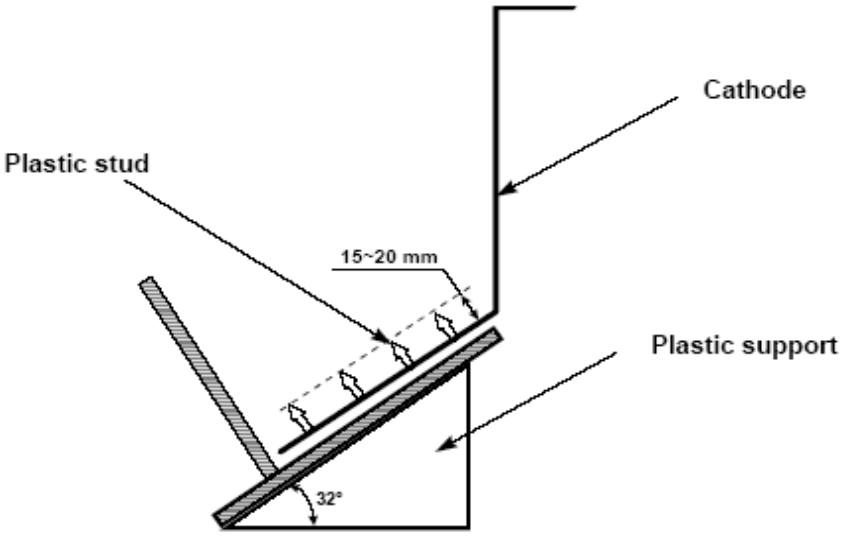


Figure D 3: Plastic support and cathode

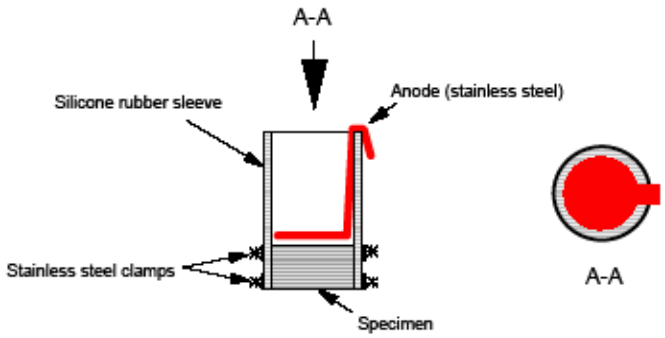


Figure D 4: rubber sleeve assembled with specimen, clamps and anode

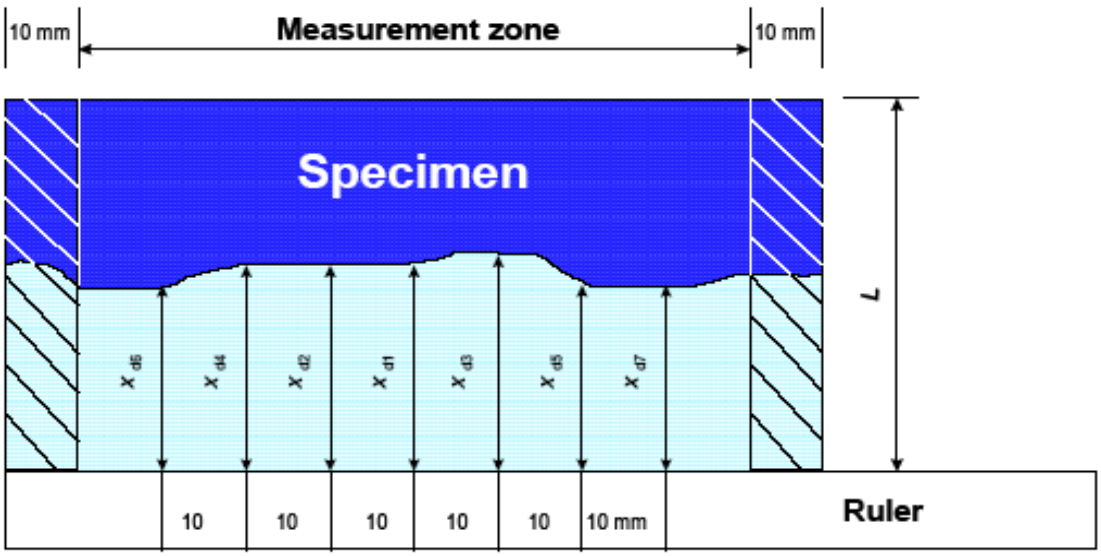


Figure D 5: Illustration of measurement for chloride penetration depths

Appendix D 2

Table D 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 1: 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.

Note 2: it is sometimes useful to carry out a preliminary test using a sample of sacrifice in order to verify the application time of the electric current required to obtain a front of penetration of chlorides placed at about half of the specimen section.