EAD **150035-00-0301**

www.eota.eu

December 2020

European Assessment Document for

Special cements – Carbonation hardening cement requiring a particular curing process

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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).

Contents

1 SCOPE OF THE EAD

1.1 Description of the construction product

The Special cements – Carbonation hardening cements requiring a particular curing process are binders based on calcium and silicon. In the text the product is designated as “Carbonation hardening cement”.

NOTE 1 The product subject to assessment is the Carbonation hardening cement but not the concrete made with the Carbonation hardening cement. However, the assessment methods are performed on mortar specimens or concrete specimens with the Carbonation hardening cement. Testing of mortar specimens or concrete specimens is not a particularity for Carbonation hardening cement, but is common in the cement domain.

The Carbonation hardening cement is manufactured by burning the raw mix in a kiln to a special clinker. This clinker is further processed, including grinding, to obtain a fine-grained inorganic product – the Carbonation hardening cement – that only sets and hardens in a particular curing process by carbonation reactions. In terms of composition, Carbonation hardening cement best matches a cement EN 197-1[1] CEM I. The Carbonation hardening cement does not contain more than 1,0 % by weight or volume – whichever is the more onerous – of homogeneously distributed organic material.

NOTE 2 With the paragraph above the composition of the Carbonation hardening cement is established. The reference to "organic material" is included for reasons related to the fire performance of the Carbonation hardening cement.

After hardening the Carbonation hardening cement retains strength and stability even under water. Concrete is, beside the Carbonation hardening cement, composed of aggregates and water, and possibly admixtures and additions. Fresh concrete retains workability until it sets and hardens in a particular curing process.

NOTE 3 With the paragraph above the behaviour of the Carbonation hardening cement regarding strength when exposed to water is described to be same as for common cement. Moreover, it is expressed that the Carbonation hardening cement is used to make mixtures to obtain concrete with aggregates, water, admixtures, and additions. This is not a particularity for Carbonation hardening cement, but is common in the cement domain.

The particular curing process includes exposure to CO_2 enriched surrounding in a closed chamber. Curing of products and specimens made of the Carbonation hardening cement – The particular curing process – requires:

- A special chamber that allows in its interior for controlled curing conditions, including controlled CO_2 content,

- A predetermined series of curing conditions, including their durations. These series include exposure to CO_2 enriched surrounding in the closed chamber.

Concerning the particular curing process, the description of the product provides data on:

- Ratio sum of surfaces of all specimens to volume of chamber.

- Placement of and clearance between specimens in chamber.

- Gas in chamber.

- CO_2 concentration.

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1 All undated references to standards in this EAD are to be understood as references to the dated versions listed in chapter 4.

– Humidity.

– Temperature.

– Duration of chamber treatment.

– Ratio mass of specimens after to prior chamber treatment.

Chamber and series of curing conditions are proprietary to the individual Carbonation hardening cement.

NOTE 4 All specimens for testing undergo the same particular curing process. Subject of testing are the specimens after the particular curing process. Since the particular curing process is specific to the individual Carbonation hardening cement, it is not part of the EAD.

Compared to common cement, concrete produced with the Carbonation hardening cement attains similar strength and retains similar properties with regard to chemical and mechanical actions. However, the concrete does not provide for corrosion protection of ordinary steel reinforcement.

The product is not fully covered by EN 197-1 as:

– Different to common cement, Carbonation hardening cement sets and hardens in a particular curing process.

– Mainly because of that particular curing process, assessment of the characteristics “early and standard compressive strength”, “setting time”, “soundness by expansion”, and “heat of hydration” of EN 197-1 are not applicable to the Carbonation hardening cement.

  – Early and standard compressive strength are related to setting and hardening of EN 197-1 cement. Since for the Carbonation hardening cement the times of testing specified in EN 197-1 are without relevance due to the particular curing process – significant different to EN 197-1 cement –, these characteristics are omitted. Instead, the specific essential characteristic Compressive strength is included for Carbonation hardening cement in Clause 2.2.3.

  – Setting time is related to gaining hardness of EN 197-1 cement. Since for the Carbonation hardening cement setting and strength development is all within the particular curing process – significant different to EN 197-1 cement –, this characteristic is omitted.

  – Soundness by expansion of EN 197-1 cement is tested by setting and hardening under specified conditions. Since for the Carbonation hardening cement setting and strength development is all within the particular curing process – significant different to EN 197-1 cement –, this characteristic is omitted.

  – Heat of hydration of EN 197-1 cement is tested by setting and hardening under specified conditions. Since for the Carbonation hardening cement setting and strength development is all within the particular curing process – significant different to EN 197-1 cement –, this characteristic is omitted.

– Moreover, “pozzolanicity” is not applicable as the Carbonation hardening cement is not a pozzolanic cement. “C3A in clinker” only relevant for sulfate resisting common cements is replaced by the essential characteristic “Sulphate resistance” in the document.

– In addition to EN 197-1, the following essential characteristics have been added:

  – Sum of active phases, Content of free CaO, and Na_2O equivalent of the Carbonation hardening cement reflects composition of EN 197-1 cement.

  – Resistance against acid attack, Alkali silica reaction, freeze thaw resistance without de-icing agent, internal structural damage, and Freeze thaw resistance with de-icing agent, scaling of the Carbonation hardening cement reflects durability of EN 197-1 cement.

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 in accordance with the manufacturer's instructions or (in absence of such instructions) in accordance with 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 of the construction product

1.2.1 Intended use(s)

The Carbonation hardening cement is intended for preparation of concrete for construction for factory made products and for applications on site. Setting and hardening are by a particular curing process.

Concrete produced with the Carbonation hardening cement is intended to be used for external, internal, and for only non-structural applications. The intended use includes concrete for e.g., roofing, masonry units, pavement, etc.

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 Carbonation hardening cement for the intended use of 100 years when installed in the works, provided that the Carbonation hardening cement is subject to appropriate installation, see Clause 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[2].

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.

[2] 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.

1.3 Specific terms used in this EAD

1.3.1 Symbols

1.3.1.1 Sulphate resistance

Square prism method

$L(0)$ .... mm...................... Initial reading of comparator measurement

$L(t)$ .... mm...................... Reading of comparator measurement at time t

i ....—....................... Specimen number i

i_{ts} ....—....................... Specimen number i, immersed in sulphate solution

i_{w} ....—....................... Specimen number i, immersed in distilled water

t................ week .... Time of immersion

$L_i(0)$........... mm...................... Initial reading of comparator measurement on specimen i, t = 0 weeks

$L_i(t)$............ mm...................... Reading of comparator measurement on specimen i at time t

$L_{Cr}(0)$.......... mm...................... Initial reading of comparator measurement on calibration rod, t = 0 weeks

$L_{Cr}(t)$.......... mm...................... Reading of comparator measurement on calibration rod at time t

$L_{i,\ corr}(0)$ ...... mm...................... Corrected initial reading of comparator measurement on specimen i, t = 0 weeks

$L_{i,\ corr}(t)$ .... mm...................... Corrected reading of comparator measurement on specimen i at time t

$\Delta L_i(t)$.......... mm...................... Change in length of specimen i at time t

$L_{g,\ i}$............. mm...................... Gauge length of specimen, determined according to EN 12617-4, clause 6.6

$\varepsilon_i(t)$ .... mm/m.................... Expansion of specimen i at time t

$\varepsilon(t)$........... mm/m.................... Mean expansion of specimens immersed in sulphate solution

$\varepsilon(32)$......... mm/m.................... Mean expansion of specimens immersed in sulphate solution for 32 weeks, square prism method

Flat prism method

i ....—....................... Specimen number i

$i_{ts,\ 5}$ ....—....................... Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts,\ 5}$ = 1 to 3

$i_{ts,\ 20}$ ....—....................... Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts,\ 20}$ = 1 to 3

$i_{Ca,\ 5}$ ....—....................... Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca,\ 5}$ = 1 to 3

$i_{Ca,\ 20}$ ....—....................... Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca,\ 20}$ = 1 to 3

t................ week .... Time of immersion

$L_i(0)$ .... mm .... Initial reading of comparator measurement on specimen i, t = 0 weeks

$L_i(t)$ .... mm .... Reading of comparator measurement on specimen i at time t

$L_{Cr}(0)$ .... mm .... Initial reading of comparator measurement on calibration rod, t = 0 weeks

$L_{Cr}(t)$ .... mm .... Reading of comparator measurement on calibration rod at time t

$L_{i, corr}(0)$ ...... mm .... Corrected initial reading of comparator measurement on specimen i, t = 0 weeks

$L_{i, corr}(t)$ .... mm .... Corrected reading of comparator measurement on specimen i at time t

$\Delta L_i(t)$ .... mm .... Change in length of specimen i at time t

$L_{g, i}$ .... mm .... Gauge length of specimen i, determined according to EN 12617-4, clause 6.6

$\varepsilon_i(t)$ .... mm/m .... Expansion of specimen i at time t

$\varepsilon_5(t)$ .... mm/m .... Mean expansion of specimens immersed in sulphate solution at 5 °C

$\varepsilon_{20}(t)$ .... mm/m .... Mean expansion of specimens immersed in sulphate solution at 20 °C

$\varepsilon_5(32)$ .... mm/m .... Mean expansion of specimens immersed in sulphate solution at 5 °C for 32 weeks, flat prism method

$\varepsilon_{20}(32)$ ...... mm/m .... Mean expansion of specimens immersed in sulphate solution at 20 °C for 32 weeks, flat prism method

i ....—...................... Specimen number i

$i_{ts, 5}$ ....—...................... Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts, 5}$ = 1 to 3

$i_{ts, 20}$ ....—...................... Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts, 20}$ = 1 to 3

$i_{Ca, 5}$ ....—...................... Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca, 5}$ = 1 to 3

$i_{Ca, 20}$ ....—...................... Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca, 20}$ = 1 to 3

t .... weeks .... Time of immersion

$E_{dyn, i}(t)$ .... GPa .... Dynamic modulus of elasticity of specimen i at time t

$E_{dyn, ts, 5}(t)$ ... GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in sulphate solution at 5 °C

$E_{dyn, ts, 20}(t)$ .. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in sulphate solution at 20 °C

$E_{dyn, Ca, 5}(t)$ .. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in calcium hydroxide solution at 5 °C

$E_{dyn, Ca, 20}(t)$. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in calcium hydroxide solution at 20 °C

$RDM_5(t)$ .... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 5 °C, to specimens immersed in calcium hydroxide solution at 5 °C, at time t

$RDM_{20}(t)$ ...... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 20 °C, to specimens immersed in calcium hydroxide solution at 20 °C, at time t

$RDM_5(0)$ .... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 5 °C, to specimens immersed in calcium hydroxide solution at 5 °C, prior to commencing immersion, t = 0 weeks

$RDM_{20}(0)$ ..... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 20 °C, to specimens immersed in calcium hydroxide solution at 20 °C, prior to commencing immersion, t = 0 weeks

$\Delta RMD_5(t)$ ..... % .... Change in relative dynamic modulus of elasticity for specimens immersed at 5 °C, at time t

$\Delta RMD_{20}(t)$ .... % .... Change in relative dynamic modulus of elasticity for specimens immersed at 20 °C, at time t

$\Delta RMD_5(32)$ .. % .... Change in relative dynamic modulus of elasticity for specimens immersed at 5 °C for 32 weeks, flat prism method

$\Delta RMD_{20}(32)$. % .... Change in relative dynamic modulus of elasticity for specimens immersed at 20 °C for 32 weeks, flat prism method

i .... — .... Specimen number i

$i_{ts,5}$ .... — .... Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts,5}$ = 1 to 3

$i_{ts,20}$ .... — .... Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts,20}$ = 1 to 3

$i_{Ca,5}$ .... — .... Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca,5}$ = 1 to 3

$i_{Ca,20}$ .... — .... Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca,20}$ = 1 to 3

t .... weeks .... Time of immersion

$M_i(0)$ .... g .... Mass of specimen i prior to immersion, at time t = 0 weeks

$M_i(t)$ .... g .... Mass of specimen i at time t

$\Delta M_i(t)$ .... g .... Change in mass of specimen i at time t, i.e., mass at time t minus mass prior to immersion

$m_i(t)$ .... % .... Relative change in mass of specimen i at time t

$m_{ts,5}(t)$ .... % .... Mean relative change in mass of specimens at time t, immersed in sulphate solution at 5 °C

$m_{ts,20}(t)$ .... % .... Mean relative change in mass of specimens at time t, immersed in sulphate solution at 20 °C

$m_{Ca,5}(t)$ .... % .... Mean relative change in mass of specimens at time t, immersed in calcium hydroxide solution at 5 °C

$m_{Ca,20}(t)$ .... % .... Mean relative change in mass of specimens at time t, immersed in calcium hydroxide solution at 20 °C

$\Delta m_5(t)$ .... % .... Difference in relative change in mass at time t between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C

$\Delta m_{20}(t)$ .... % .... Difference in relative change in mass at time t between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C

$\Delta m_5(32)$ .... % .... Difference in relative change in mass between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C for 32 weeks, flat prism method

$\Delta m_{20}(32)$ ...... % .... Difference in relative change in mass between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C for 32 weeks, flat prism method

1.3.1.2 Resistance against acid attack

i .... — .... Specimen number i

$m_{0, i}$ .... g .... Mass of specimen i prior to immersion in acetic acid solution

$m_{21, i}$ .... g .... Mass of specimen i after 21 days immersion in acetic acid solution

$\Delta m_{21, i}$ .... g .... Mass loss of specimen i after 21 days immersion in acetic acid solution

A_{pris} .... m^2 .... Surface area of specimen.
Apply A_{pris} = 0,028 8 m^2 for prism 40 mm × 40 mm × 160 mm

$L_{21, I}$ .... kg/m^2 .... Mass loss per unit area of specimen i

$L_{21, mean}$ ...... kg/m^2 .... Mean mass loss per unit area of 3 specimens

1.3.1.3 Resistance against alkali silica reaction

i .... — .... Specimen number i

t .... days .... Time of immersion

$L_i(0)$ .... mm .... Initial reading of comparator measurement on specimen i, t = 0 days

$L_i(t)$ .... mm .... Reading of comparator measurement on specimen i at time t

$L_{Cr}(0)$ .... mm .... Initial reading of comparator measurement on calibration rod, t = 0 days

$L_{Cr}(t)$ .... mm .... Reading of comparator measurement on calibration rod at time t

$L_{i, corr}(0)$ ...... mm .... Corrected initial reading of comparator measurement on specimen i, t = 0 days

$L_{i, corr}(t)$ .... mm .... Corrected reading of comparator measurement on specimen i at time t

$\Delta L_i(t)$ .... mm .... Change in length of specimen i at time t

$L_{g, i}$ .... mm .... Gauge length of specimen i, determined according to EN 12617-4, clause 6.6

$\varepsilon_i(t)$ .... mm/m .... Expansion of specimen i at time t

$\varepsilon(t)$ .... mm/m .... Mean expansion of specimens immersed in test solution

$\varepsilon(14)$ .... mm/m .... Mean expansion of specimens immersed in test solution for 14 days.

2 ESSENTIAL CHARACTERISTICS AND RELEVANT ASSESSMENT METHODS AND CRITERIA

2.1 Essential characteristics of the product

Table 2.1.1 shows how the performance of the Carbonation hardening cement is assessed in relation to the essential characteristics.

Table 2.1.1 Essential characteristics of the product and methods and criteria for assessing the performance of the product in relation to those essential characteristics

№	Essential characteristic	Assessment method	Type of expression of product performance
Basic Works Requirement 2: Safety in case of fire			
1	Reaction to fire	2.2.1	Class
Basic Works Requirement 3: Hygiene, health and the environment			
2	Content, emission and/or release of dangerous substances	2.2.2	Level
Basic Works Requirement 4: Safety and accessibility in use			
3	Compressive strength	2.2.3	Level and Description
4	Fineness	2.2.4	Level
5	Loss on ignition	EN 196-2, clause 4.4.1	Level
6	Insoluble residue	EN 196-2, clause 4.4.3	Level
7	Sum of active phases	2.2.5	Level
8	Content of free CaO	EN 451-1	Level
9	Chloride content	EN 196-2, clause 4.5.16	Level
10	SO_3 content	EN 196-2, clause 4.4.2	Level
11	Na_2O equivalent	EN 196-2, clause 4.5.19	Level
12	Sulphate resistance	2.2.6	Level and Description
13	Resistance against acid attack	2.2.7	Level and Description
14	Alkali silica reaction	2.2.8	Level and Description
15	Freeze thaw resistance without de-icing agent, internal structural damage	2.2.9	Level and Description
16	Freeze thaw resistance with de-icing agent, scaling	2.2.10	Level and Description

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

This chapter is intended to provide instructions for TABs. Therefore, the use of wordings such as “shall be stated in the ETA” or “it has to be given in the ETA” shall be understood only as such instructions for TABs on how results of assessments shall be presented in the ETA. Such wordings do not impose any obligations for the manufacturer and the TAB shall not carry out the assessment of the performance in relation to a given essential characteristic when the manufacturer does not wish to declare this performance in the Declaration of Performance.

2.2.1 Reaction to fire

The Carbonation hardening cement as described in Clause 1.1 is considered to satisfy the requirements of class A1 of the reaction-to-fire performance in accordance with the Commission Decision 96/603/EC, as amended by Commission Decision 2000/605/EC and Commission Decision 2003/424/EC without the need for testing on the basis of it fulfilling the conditions set out in that Decision and its intended use being covered by that Decision.

Therefore, when the conditions referred to above are fulfilled, the performance of the product is class A1 which shall be given in the ETA.

2.2.2 Content, emission and/or release of dangerous substances

2.2.2.1 General

The performance of the product regarding the emissions and/or release and, where appropriate, the content of dangerous substances will be assessed on the basis of the information provided by the manufacturer[3] after identifying the release scenarios taking into account the intended use(s) of the product and the Member States where the manufacturer intends his product to be made available on the market.

The identified intended release scenarios for this product and intended use with respect to dangerous substances are:

IA1: Product with direct contact to indoor air

IA2: Product with indirect contact to indoor air (e.g. covered products) but possible impact on indoor air

S/W1: Product with direct contact to soil, ground- and surface water.

S/W2: Product with indirect contact to soil, ground- and surface water.

NOTE 1 With regard to Cr VI the Carbonation hardening cement is subject to Commission Regulation (EC) № 552/2009.

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[3] The manufacturer may be asked to provide to the TAB the REACH related information which shall accompany the DoP, c.f. Article 6(5) of Regulation (EU) No 305/2011.

The manufacturer is **not** obliged to:

- Provide the chemical constitution and composition of the product (or of constituents of the product) to the TAB, or
- Provide a written declaration to the TAB stating whether the product (or constituents of the product) contain(s) substances which are classified as dangerous according to Directive 67/548/EEC and Regulation (EC) No 1272/2008 and listed in the "Indicative list on dangerous substances" of the SGDS, taking into account the installation conditions of the construction product and the release scenarios resulting from there.

Any information provided by the manufacturer regarding the chemical composition of the products is not to be distributed to EOTA to other TABs or beyond.

NOTE 2 With regard to ionising radiation the Carbonation hardening cement is subject to Council Directive 2013/59/EURATOM, corrected by Corrigendum OJ L 72 of 17.3.2016, page 69 and Corrigendum OJ L 152 of 11.6.2019, page 128.

2.2.2.2 SVOC, VOC, and PAH

For the intended use covered by the release scenarios IA1 and IA2 the Carbonation hardening cement, as described in Clause 1.1 as an inorganic matter, shall not be tested regarding semi-volatile organic compounds (SVOC), volatile organic compounds (VOC), and polycyclic aromatic hydrocarbons (PAH).

The nature of the Carbonation hardening cement, being an inorganic matter, shall be stated in the ETA.

2.2.2.3 Leachable Substances

For the intended use covered by the release scenario the performance of the product concerning leachable substances shall be assessed. A leaching test with subsequent eluate analysis shall be performed, each in triplicate.

Elution specimens shall be made of concrete with the composition of Table 2.2.2.3.1.

Table 2.2.2.3.1 Concrete composition for elution specimens

Concrete composition for elution specimens	
Item	**Amount**
Binder	Carbonation hardening cement 300 kg/m^3
Water to cement ratio	0,50
Aggregate	Aggregates in accordance with EN 12620 and with grading of Table 2.2.2.3.2.

Table 2.2.2.3.2 Grading of aggregates for elution specimens

Grading of aggregates for elution specimens								
Sieve size	mm	0,25	0,5	1	2	4	8	16
Passing	% by mass	6	14	22	32	46	68	100

Concrete cubes with dimensions of 100 mm × 100 mm × 100 mm shall be moulded in accordance with Clause 2.2.3. Form oil shall not be used. After completion of the particular curing process, keep the specimens covered with a damp cloth for 3 days and subsequently commence elution.

Elution is performed by a tank test in accordance with EN 16637-2. The eluates taken after 6 hours, 24 hours, 54 hours, 4 days, 9 days, 16 days, 36 days, and 64 days shall be analysed for the environmentally relevant parameters of Table 2.2.2.3.3.

Table 2.2.2.3.3 Environmentally relevant parameters

Linie №	Environmentally relevant parameters
1	antimony, arsenic, barium, lead, cadmium, chromium (total), chromate (Cr VI), cobalt, copper, molybdenum, nickel, mercury, thallium, vanadium, zinc
2	chloride (Cl^-), sulphate (SO_4^{2-}), fluoride (F^-)
3	TOC
4	pH-value, electrical conductivity, colour, turbidity

The parameters shall be analysed with standardised test methods in accordance with Table 2.2.2.3.4.

Table 2.2.2.3.4 Analytical test methods

Analytical test methods	
Parameter	**Test method**
antimony (Sb), arsenic (As), barium (Ba), lead (Pb), cadmium (Cd), total chromium (Cr), chromate VI (Cr VI), cobalt (Co), copper (Cu), molybdenum (Mo), nickel (Ni), mercury (Hg), thallium (Ti), vanadium (V), zinc (Zn), chloride (Cl^-), sulphate (SO_4^{2-}), fluoride (F^-)	EN 17195
TOC, pH-value, electrical conductivity	EN 17332
Colour	EN ISO 7887
Turbidity	EN ISO 7027-1

– Measured concentration of the leaching test according to EN 16637-2 of hardened concrete shall be recorded per step for each parameter in line 1 and line 2 of Table 2.2.2.3.3 in µg/L and mg/m². Additionally, the cumulatively released quantities shall be expressed for each parameter in mg/m².

– For the parameters in line 1 and line 2 of Table 2.2.2.3.3 the cumulatively released quantities expressed in mg/m² shall be stated in the ETA.

2.2.3 Compressive strength

Compressive strength shall be tested in accordance with EN 196-1 taking account of the particularities of the Carbonation hardening cement:

Specimens for testing in accordance with Table 2.2.3.2 shall be prisms in accordance with EN 196-1, clause 7, made of mortar in accordance with Table 2.2.3.1.

There are two cures. Firstly, the particular curing process that serves to prepare the specimens for testing. Secondly, once the particular curing process is complete, the specimens are subjected to curing conditions given in Table 2.2.3.2. After the specified curing times have elapsed, strength testing follows. Just after the particular curing process is completed, a sample of the specimens is strength tested. These are the initial test results as further strength testing is once the curing times of Table 2.2.3.2 have elapsed. I.e. these strength tests come later.

Moulding and testing of specimens shall be:

– Laboratory and equipment in accordance with EN 196-1, clause 4,

– Mix of mortar in accordance with EN 196-1, clause 6.2, with composition in accordance with Table 2.2.3.1,

– Determine mass of empty mould,

– Mould specimens on vibrating table,

– Determine prism mass by weighing of filled mould. Adjusting the prism masses by:

  - ✓ Calculate arithmetic mean, standard deviation and coefficient of variation as standard deviation divided by arithmetic mean of the specimen masses.
  - ✓ If coefficient of variation is greater than 0,010, identify those specimens with lowest mass that cause exceeding the coefficient of variation.
  - ✓ Discard all these specimens.
  - ✓ Replace all discarded specimens with new ones.
  - ✓ Repeat the process for all specimens from the beginning until coefficient of variation is ≤ 0,010.

– Store specimens in moist cabinet at (+ 20,0 ± 1,0) °C and at a relative humidity not less than 95 %,

– Demould specimens after (18 ± 2) h,

– Keep demoulded specimens covered by a damp cloth until start of the particular curing process,

– Subject the specimens to the particular curing process,

– After completion of the full particular curing process remove the specimens from the curing chamber,

– Subject the specimens to curing conditions set out in Table 2.2.3.2,

– After removing the specimens from curing, keep specimens covered by a damp cloth until strength testing,

– Continue with strength testing in accordance with EN 196-1, clause 9.2.

Table 2.2.3.1 Mortar composition for testing compressive strength

Mortar composition for testing compressive strength	
Item	**Amount**
Binder	Carbonation hardening cement (450 ± 2) g
Water to cement ratio	0,40
Aggregate	CEN standard sand in accordance with EN 196-1, clause 5.1 (1 350 ± 5) g

Table 2.2.3.2 Time of testing compressive strength

Time in days	0 1)	7	14	28	56	90	6 months	12 months
Curing	**Number of prisms per test date**							
Curing in still air 2)	6	2	2	2	2	2	2	2
Curing in tap water 3)	—	2	2	2	2	2	2	2
Curing in deionised water 4)	—	2	2	2	2	2	2	2
Curing in water and air 5)	—	—	—	2	2	2	2	2

1) Compressive strength, tested after the completed particular curing process, initial test results
2) Temperature 20 °C, relative humidity 55 %
3) Temperature 20 °C, tap water 3,5 mmol $CaCO_3$/l
4) Temperature 20 °C, deionized water ≤ 0,025 mmol $CaCO_3$/l
5) Weekly change between air at temperature 20 °C and relative humidity 55 %, and deionized water at 20°C

Test results:

– Mean values of compressive strength versus time shall be plotted and the trend over time shall be described.

– Test results after 12 months shall be compared to the initial test results. The changes prior and after 12 months immersion shall be calculated in percentage.

The test results, i.e., mean compressive strength in MPa after the completed particular curing process, initial test results, description of the trend over time, and the changes in percentage after 12 months shall be stated in the ETA.

2.2.4 Fineness

Fineness shall be determined by sieving and air permeability:

– Residue after sieving on a 90 µm mesh sieve shall be tested in accordance with EN 196-6, clause 5.

– Specific surface shall be tested as air permeability in accordance with EN 196-6, clause 4.

Residue after sieving in % and specific surface in g/cm^2 shall be stated in the ETA.

2.2.5 Sum of active phases

Sum of crystalline phases shall be determined by X-ray diffraction with Rietveld quantification. X-ray diffraction with Rietveld quantification is a direct method for qualitative and also quantitative characterisation of fine-grained materials such as clinker.

X-ray diffraction with Rietveld quantification is performed with a X-ray diffractometer.

X-ray diffractometer

The X-ray diffractometer performs measurement of the X-ray diffraction pattern from which the crystalline phases shall be identified, and proportion of each phase shall be quantitatively determined.

X-ray diffractometers comprise the following elements:

– Source of radiation (X-ray tube);

- Detection and counting equipment;
- Specimen holder and goniometer.

The suitability of the X-ray diffractometer shall be established with the following parameters:

- Peak position is within $\pm$ 0,02 ° 2 · θ of a clearly identifiable peak, e.g., quartz 101 peak.
- Peak resolution allows to separate, e.g., 5 peaks around quartz 203 peak.
- Peak intensity is at least 2 000 counts, e.g., of the quartz 101 peak.
- Ratios of peak intensities are within 5 % of nominal peak intensities.
- The X-ray diffractometer performs digital data acquisition.

Specimen preparation

Specimens shall be pressed pellets. Prior to pellet pressing, the material shall be ground to obtain a particle size distribution with at least 95 % finer than 45 µm. Respective particle size distribution shall be determined with laser diffraction.

Measurement

The pressed pellet shall be measured with the X-ray diffractometer. Measured diffraction pattern shall be aligned with data from control files, representing diffraction patterns of the phases of the Carbonation hardening cement. I.e., this is called Rietveld quantification to quantitatively determine phase proportions.

Measurements for phase proportions shall be performed with pellets of:

- Carbonation hardening cement,
- Homogenised blend of Carbonation hardening cement and measured quantity of crystalline material.

For Sum of active crystalline phases, wollastonite, pseudo-wollastonite, rankinite, and belite, shall be determined from the measurement. Quantified amorphous components shall be considered in the evaluation.

Test results

Sum of active crystalline phases in %.

The test results shall be stated in the ETA.

2.2.6 Sulphate resistance

Sulphate resistance shall be determined on specimens of mortar with the Carbonation hardening cement in accordance with Table 2.2.6.1.

Table 2.2.6.1 Mortar composition for testing sulphate resistance

Mortar composition for testing sulphate resistance	
Item	**Amount**
Binder	Carbonation hardening cement (450 ± 2) g
Water to cement ratio	0,40
Aggregate	CEN standard sand in accordance with EN 196-1, clause 5.1 (1 350 ± 5) g

Specimens with Carbonation hardening cement shall be moulded in accordance with Clause 2.2.3.

The specimens shall be subjected to two immersion tests in accordance with:

- Annex A, the square prism method and
- Annex B, the flat prism method.

Test results:

- Expansion for square prism method after 32 weeks of immersion,

  $\varepsilon(32)$ in mm/m in accordance with Annex A,

- Expansion for flat prism method after 32 weeks of immersion,

  $\varepsilon_5(32)$ in mm/m in accordance with Annex B,

  $\varepsilon_{20}(32)$ in mm/m in accordance with Annex B,

- Change of dynamic modulus of elasticity for flat prism method after 32 weeks of immersion,

  $\Delta RMD_5(32)$ in % in accordance with Annex B,

  $\Delta RMD_{20}(32)$ in % in accordance with Annex B,

- Difference in relative change in mass between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution,

  $\Delta m_5(32)$ in % in accordance with Annex B,

  $\Delta m_{20}(32)$ in % in accordance with Annex B,

- For square prism method and for flat prism method, description of signs of degradation or the conclusion no degradation is present.

Where

$\varepsilon(32)$......... mm/m.................... Mean expansion of specimens immersed in sulphate solution for 32 weeks, square prism method

$\varepsilon_5(32)$ .... mm/m.................... Mean expansion of specimens immersed in sulphate solution at 5 °C for 32 weeks, flat prism method

$\varepsilon_{20}(32)$ ...... mm/m.................... Mean expansion of specimens immersed in sulphate solution at 20 °C for 32 weeks, flat prism method

$\Delta RMD_5(32)$ .. %....................... Change in relative dynamic modulus of elasticity for specimens immersed at 5 °C for 32 weeks, flat prism method

$\Delta RMD_{20}(32)$. %....................... Change in relative dynamic modulus of elasticity for specimens immersed at 20 °C for 32 weeks, flat prism method

$\Delta m_5(32)$........ %....................... Difference in relative change in mass between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C for 32 weeks, flat prism method

$\Delta m_{20}(32)$ ...... %....................... Difference in relative change in mass between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C for 32 weeks, flat prism method

The test results shall be stated in the ETA.

2.2.7 Resistance against acid attack

Resistance against acid attack shall be determined in comparison on specimens with dimensions of 40 mm × 40 mm × 160 mm of:

- Mortar with the Carbonation hardening cement in accordance with Table 2.2.7.1 and
- Mortar with Portland cement CEM I-SR 0 in accordance with EN 197-1, see Table 2.2.7.1

Table 2.2.7.1 Mortar composition for testing resistance against acid attack

<table>
<tr><th colspan="3">Mortar composition for testing resistance against acid attack</th></tr>
<tr><th>Item</th><th>Amount for CEM I-SR 0</th><th>Amount for Carbonation hardening cement</th></tr>
<tr><td>Binder</td><td rowspan="3">Mortar in accordance with EN 196-1, clause 5 and clause 6.1, with cement CEM I-SR 0 in accordance with EN 197-1</td><td>Carbonation hardening cement (450 ± 2) g</td></tr>
<tr><td>Water to cement ratio</td><td>0,30</td></tr>
<tr><td>Aggregate</td><td>CEN standard sand in accordance with EN 196-1, clause 5.1 (1 350 ± 5) g</td></tr>
</table>

- 3 specimens with Carbonation hardening cement shall be moulded in accordance with Clause 2.2.3. After completion of the particular curing process, keep the specimens for 3 days in water at (+ 20 ± 2) °C. Remove the specimens from water and commence test for resistance against acid attack.
- 3 specimens with Portland cement shall be moulded in accordance with EN 196-1, clause 7, clause 8.1, and clause 8.2. After demoulding the specimens shall be submerged in water for further 27 days. Remove the specimens from water and commence test for resistance against acid attack.
- The specimens shall be tested for resistance against acid attack in accordance with Annex C.

Test results:

– Mean mass loss per unit area of specimens with Carbonation hardening cement after immersion for 21 days in acetic acid solution,

  $L_{21,\,mean}$ in kg/m² in accordance with Annex C,

– Mean mass loss per unit area of specimens with cement CEM I-SR 0 after immersion for 21 days in acetic acid solution,

  $L_{21,\,mean}$ in kg/m² in accordance with Annex C,

Where

  $L_{21,\,mean}$ ...... kg/m² .... Mean mass loss per unit area of 3 specimens

The test results shall be stated in the ETA.

2.2.8 Alkali silica reaction

Resistance against alkali silica reaction shall be determined in comparison on specimens with dimensions of 40 mm × 40 mm × 160 mm of:

– Mortar with the Carbonation hardening cement in accordance with Table 2.2.8.1 and

– Mortar with Portland cement CEM I 42,5 R in accordance with EN 197-1, see Table 2.2.8.1

Table 2.2.8.1 Mortar composition for testing resistance against alkali silica reaction

Mortar composition for testing resistance against alkali silica reaction		
Item	**Amount for CEM I 42,5 R**	**Amount for Carbonation hardening cement**
Binder	Mortar in accordance with EN 196-1, clause 5 and clause 6.1, with cement CEM I 42,5 R in accordance with EN 197-1	Carbonation hardening cement (450 ± 2) g
Water to cement ratio		0,40
Aggregate	Aggregates, size 0/4 mm [1)] in accordance with Clause D.1	Aggregates, size 0/4 mm [1)] in accordance with Clause D.1 (1 350 ± 5) g
[1)] The gap between blade and bowl of the mixer allows for aggregates of 4 mm size.		

– 3 specimens with Carbonation hardening cement shall be moulded in accordance with Clause 2.2.3. After completion of the particular curing process, commence test for alkali silica reaction. The specimens shall be provided with measurement studs to facilitate length variation measurement.

– 3 specimens with Portland cement shall be moulded in accordance with EN 196-1, clause 7, clause 8.1, and clause 8.2. After demoulding the specimens, 24 hours after moulding, commence test for alkali silica reaction. The specimens shall be provided with measurement studs to facilitate length variation measurement.

– The specimens shall be tested for resistance against alkali silica reaction according Annex D.

Test results:

– Mean expansion of specimens with Carbonation hardening cement after 14 days of immersion in sodium hydroxide solution.

  $\varepsilon(14)$ in mm/m in accordance with Annex D, tested with Carbonation hardening cement.

– Mean expansion of specimens with cement CEM I 42,5 R after 14 days of immersion in sodium hydroxide solution.

$\varepsilon(14)$ in mm/m in accordance with Annex D, tested with cement CEM I 42,5 R.

Where

$\varepsilon(14)$......... mm/m.................... Mean expansion of specimens immersed in test solution for 14 days

The test results shall be stated in the ETA.

2.2.9 Freeze thaw resistance without de-icing agent, internal structural damage

Freeze thaw resistance for internal structural damage shall be tested accordance to the beam test of CEN/TR 15177 in comparison on specimens of:

– Concrete with the Carbonation hardening cement in accordance with Table 2.2.9.1,

– Concrete with Portland cement CEM I in accordance with EN 197-1, see Table 2.2.9.1.

The tests shall be performed on prismatic specimens, dimensions

length × width × depth = 400 mm × 100 mm × 40 mm.

Table 2.2.9.1 Concrete composition for testing freeze thaw resistance without de-icing agent, internal structural damage

Concrete composition for testing freeze thaw resistance without de-icing agent, internal structural damage		
Item	**Amount for CEM I**	**Amount for Carbonation hardening cement**
Binder	CEM I in accordance with EN 197-1 495 kg/m^3	Carbonation hardening cement 495 kg/m^3
Water to cement ratio	0,55	0,30
Aggregate	Aggregates for reference concrete as specified in EN 1766 [1)] with maximum aggregate size 4 mm [2)]	Aggregates for reference concrete as specified in EN 1766 [1)] with maximum aggregate size 4 mm [2)]

1) Specification of aggregates in accordance with EN 1766, clause 5.1, with grading in accordance with Figure 2.2.9.1.
2) The gap between blade and bowl of the mixer allows for aggregates of 4 mm size.

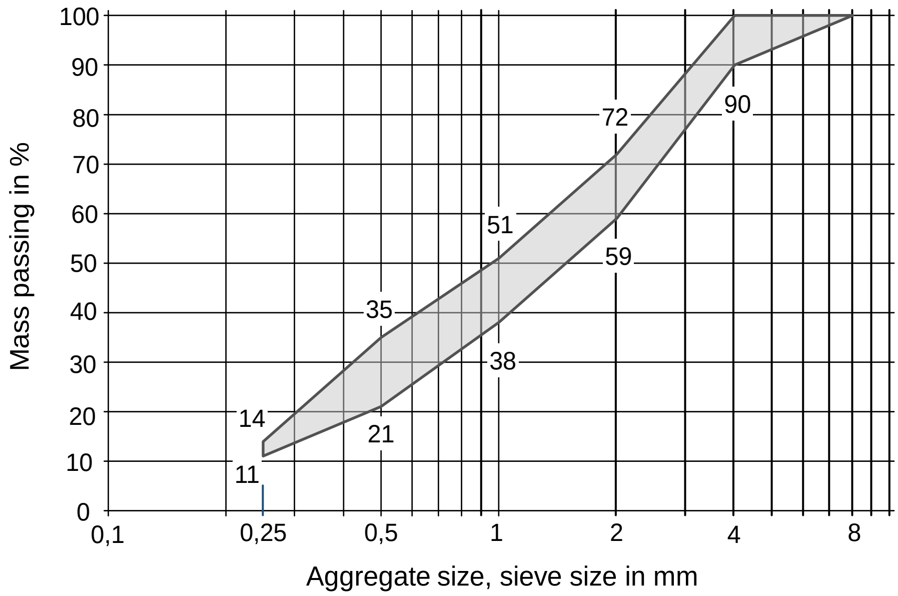

Figure 2.2.9.1 Aggregate grading

– 3 Specimens with Carbonation hardening cement shall be moulded in accordance with Clause 2.2.3. After completion of the particular curing process, submerge the specimens up to an age of 14 days in water at (+ 20 ± 2) °C. Remove the specimens from water and commence testing freeze thaw resistance without de-icing agent.

NOTE Age of specimen in days is counted from the day of manufacturing the specimen. I.e., day of manufacturing is the day of mixing the fresh concrete.

– 3 Specimens with Portland cement shall be moulded following EN 196-1, clause 7, clause 8.1, and clause 8.2.

– Determine air content of fresh concrete in accordance with EN 12350-7, clause 6. Aggregate correction factor, determined in accordance with EN 12350-7, Annex B, shall be considered in accordance with EN 12350-7, clause 7.

– After demoulding, cure the specimens in accordance with CEN/TR 15177, beam test. Commence testing freeze thaw resistance without de-icing agent once curing is completed.

The specimens are submitted to a beam test in accordance with CEN/TR 15177 with ultrasonic pulse transit time measured. Measurement of ultrasonic pulse transit time shall be commenced before subjecting the specimens to freeze-thaw cycles and continued in accordance with CEN/TR 15177.

Test results:

– The relative dynamic modulus of elasticity in % after 56 freeze-thaw cycles,

– Description whether the relative dynamic modulus of elasticity after 56 freeze-thaw cycles of concrete with CEM I cement is higher or lower than concrete with Carbonation hardening cement.

The test results shall be stated in the ETA.

2.2.10 Freeze thaw resistance with de-icing agent, scaling

Freeze thaw resistance with de-icing agent shall be tested for scaling with a slab test in accordance with CEN/TS 12390-9 in comparison on specimens of:

– Concrete with the Carbonation hardening cement in accordance with Table 2.2.10.1.

– Concrete with Portland cement CEM I in accordance with EN 197-1, see Table 2.2.10.1.

The tests shall be performed on slabs, dimensions

length × width × depth = 150 mm × 150 mm × 40 mm.

Instead of saw cut specimens, the slabs shall be moulded in their final dimensions.

Table 2.2.10.1 Concrete composition for testing freeze thaw resistance with de-icing agent, scaling

Concrete composition for testing freeze thaw resistance with de-icing agent, scaling		
Item	**Amount for CEM I**	**Amount for Carbonation hardening cement**
Binder	CEM I in accordance with EN 197-1 495 kg/m³	Carbonation hardening cement 495 kg/m³
Water to cement ratio	0,45	0,30
Aggregate	Aggregates for reference concrete as specified in EN 1766 [1)] with maximum aggregate size 4 mm [2)]	Aggregates for reference concrete as specified in EN 1766 [1)] with maximum aggregate size 4 mm [2)]
[1)] Specification of aggregates in accordance with EN 1766, clause 5.1, with grading in accordance with Figure 2.2.9.1. [2)] The gap between blade and bowl of the mixer allows for aggregates of 4 mm size.		

– 4 specimens with Carbonation hardening cement shall be moulded in accordance with Clause 2.2.3. After completion of the particular curing process, keep the specimens up to an age of 14 days in water at (+ 20 ± 2) °C. Remove the specimens from water and commence testing freeze thaw resistance with de-icing agent.

NOTE Age of specimen in days is counted from the day of manufacturing the specimen. I.e., day of manufacturing is the day of mixing the fresh concrete.

– 4 specimens with Portland cement shall be moulded following EN 196-1, clause 7, clause 8.1, and clause 8.2.

– Determine air content of fresh concrete in accordance with EN 12350-7, clause 6. Aggregate correction factor, determined in accordance with EN 12350-7, Annex B, shall be considered in accordance with EN 12350-7, clause 7.

– After demoulding, cure the specimens in accordance with CEN/TS 12390-9, slab test. Commence testing freeze thaw resistance with de-icing agent once curing is completed.

The specimens shall be submitted to a slab test in accordance with CEN/TS 12390-9.

Test results:

– Cumulative amount of scaled material per unit area in g/m² after 56 freeze-thaw cycles.

– Description whether the cumulative amount of scaled material per unit area after 56 cycles of concrete with CEM I cement is higher or lower that concrete with Carbonation hardening cement.

The test results shall be 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 product covered by the EAD the applicable European legal act is Commission Decision 97/555/EC, amended by Commission Decision 2010/683/EU.

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 3.2.1.

Table 3.2.1 Control plan for the manufacturer – Cornerstones

№	Subject of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
Factory production control (FPC) including testing of samples taken at the factory in accordance with the prescribed test plan					
1	Compressive strength	2.2.3 [1)]	[2)]	1 sample	2 per week [3)], 4 per week [4)]
2	Fineness	2.2.4	[2)]	1 sample	2 per week [3)], 4 per week [4)]
3	Loss on ignition	EN 196-2, clause 4.4.1	[2)]	1 sample	2 per week [3)], 4 per week [4)]
4	Insoluble residue	EN 196-2, clause 4.4.3	[2)]	1 sample	2 per week [3)], 4 per week [4)]
5	Sum of active phases	2.2.5	[2)]	1 sample	2 per week [3)], 4 per week [4)]
6	Content of free CaO	EN 451-1	[2)]	1 sample	2 per week [3)], 4 per week [4)]
7	Chloride content	EN 196-2, clause 4.5.16	[2)]	1 sample	2 per week [3)], 4 per week [4)]
8	SO_3 content	EN 196-2, clause 4.4.2	[2)]	1 sample	2 per week [3)], 4 per week [4)]
9	Na_2O equivalent	EN 196-2, clause 4.5.19 [5)]	[2)]	1 sample	2 per week [3)], 4 per week [4)]
10	Composition	[6)]	[2)]	1 sample	1 per month [3)], 1 per week [4)]

1) Only compressive strength after the particular curing process.
2) In accordance with the Control plan of the Carbonation hardening cement.
3) Routine situation
4) Initial period
5) Equivalent determination method by X-ray fluorescence in accordance with EN 196-2, clause 5, with EN 196-2, clause 4.5.19, as the reference method.
6) Appropriate test method in accordance with the Control plan.

3.3 Tasks of the notified body

The cornerstones of the actions to be undertaken by the notified body in the procedure of assessment and verification of constancy of performance for Carbonation hardening cement are laid down in Table 3.3.1.

Table 3.3.1Control plan for the notified body – Cornerstones

№	Subject of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
Initial inspection of the manufacturing plant and of factory production control					
1	Notified Body will ascertain that the factory production control with the staff and equipment are suitable to ensure a continuous and orderly manufacturing of the Carbonation hardening cement.	Verification of the complete FPC as described in the Control plan agreed between the TAB and the manufacturer Details on the procedure for initial inspection are given in EN 197-2.	According to Control plan	According to Control plan	When starting the production or a new line
Continuing surveillance, assessment, and evaluation of factory production control					
1	The Notified Body will ascertain that the system of factory production control and the specified manufacturing process are maintained taking account of the Control plan.	Verification of the controls carried out by the manufacturer as described in the Control plan agreed between the TAB and the manufacturer with reference to the raw materials, to the process and to the product as indicated in Table 3.2.1 Details on the procedure are given in EN 197-2.	According to Control plan	According to Control plan	Once per year
Audit-testing of samples taken by the notified product certification body at the manufacturing plant or at the manufacturer's storage facilities					
1	Compressive strength	2.2.3 [1]	[2]	1 sample	6 per year
2	Fineness	2.2.4	[2]	1 sample	6 per year
3	Loss on ignition	EN 196-2, clause 4.4.1	[2]	1 sample	6 per year
4	Insoluble residue	EN 196-2, clause 4.4.3	[2]	1 sample	6 per year
5	Sum of active phases	2.2.5	[2]	1 sample	6 per year
6	Content of free CaO	EN 451-1	[2]	1 sample	6 per year
7	Chloride content	EN 196-2, clause 4.5.16	[2]	1 sample	6 per year
8	SO_3 content	EN 196-2, clause 4.4.2	[2]	1 sample	6 per year
9	Na_2O equivalent	EN 196-2, clause 4.5.19	[2]	1 sample	6 per year

1) Only compressive strength after the particular curing process.
2) According to the Control plan of the Carbonation hardening cement.

REFERENCE DOCUMENTS

EN 196-1:2016	Methods of testing cement – Part 1: Determination of strength
EN 196-2:2013	Methods of testing cement – Part 2: Chemical analysis of cement
EN 196-6:2018	Methods of testing cement – Part 6: Determination of fineness
EN 197-1:2011	Cement – Part 1: Composition, specifications and conformity criteria for common cements
EN 197-2:2020	Cement – Part 2: Assessment and verification of constancy of performance
EN 451-1:2017	Method of testing fly ash – Part 1: Determination of free calcium oxide content
EN 1766:2017	Products and systems for the protection and repair of concrete structures – Test methods – Reference concretes for testing
EN 12350-7:2019/AC:2022	Testing fresh concrete – Part 7: Air content – Pressure methods
EN 12617-4:2002	Products and systems for the protection and repair of concrete structures – Test methods – Part 4: Determination of shrinkage and expansion
EN 12620:2002+A1:2008	Aggregates for concrete
EN 16637-2:2023	Construction products: Assessment of release of dangerous substances – Part 2: Horizontal dynamic surface leaching test
EN 17195:2023	Construction products: Assessment of release of dangerous substances – Analysis of inorganic substances in eluates
EN 17332:2023	Construction products: Assessment of release of dangerous substances - Analysis of organic substances in eluates
EN ISO 7027-1:2016	Water quality – Determination of turbidity – Part 1: Quantitative methods
EN ISO 7887:2011	Water quality – Examination and determination of colour
CEN/TR 15177:2006	Testing the freeze-thaw resistance of concrete – Internal structural damage
CEN/TS 12390-9:2016	Testing hardened concrete – Freeze-thaw resistance with de-icing salts – Scaling

ANNEX A: SULPHATE RESISTANCE – SQUARE PRISM METHOD

A.1 Equipment and test solution

A.1.1 Containers

The containers for immersion in distilled water and test solution, see Clause A.1.2, have a capacity of 1,5 litre and 2,5 litre with inner dimensions of at least 180 mm × 80 mm. Each container is suitable for (1,0 ± 0,1) litre of liquid, so that the depth of the liquid is at least 25 mm. All containers shall be fitted with light-proof lids and made in a material that does not react with its content.

A.1.2 Test solution

The test solution is sulphate solution in accordance with Clause A.1.3.

A.1.3 Sulphate solution

The sulphate solution has a concentration of (16 ± 0,5) g SO_4 per litre. It 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. The SO_4-content of the Na_2SO_4 shall be determined before the solution is prepared or the SO_4-content of the solution shall be measured and, if necessary, corrected.

A.2 Preparation of specimens

A.2.1 Preparation of specimens

The specimens, 6 prisms made of mortar in accordance with Clause 2.2.6, with dimensions of 20 mm × 20 mm × 160 mm, shall be equipped with stainless steel studs for comparator measurement at both ends.

A.2.2 Curing

The moulded specimens shall be cured in accordance with Clause 2.2.3. After the particular curing process, the specimens shall be placed in 2 containers, 3 specimens in each container with 1 litre of distilled water.

The specimens shall be placed side by side, with:

- A clear distance of at least 5 mm between them,
- A clear distance of at least 5 mm to the container wall, and
- A water head of at least 5 mm above the specimens.

The specimens shall be placed on supports, at least 2 mm clear from the container bottom.

A.3 Test procedure

A.3.1 Measurements and immersion

The specimens shall be measured with a comparator in accordance with EN 12617-4, clauses 5.3 and 5.4, for change in length.

At the age of 28 days of the specimens, the immersion test in commenced. Prior to initial immersion of the specimens in sulphate solution, initial comparator measurements in accordance with EN 12617-4, clause 6.3, shall be performed, L(0), on specimens to be immersed in distilled water and specimens to be immersed in sulphate solution.

NOTE Age of specimen in days shall be counted from the day of manufacturing the specimen. I.e., day of manufacturing is the day of mixing the fresh concrete.

- Replace the distilled water of one container with fresh distilled water and immerse three specimens into the container as described in A.2.2.
- Replace the distilled water in the second container with 1 litre sulphate solution and immerse the remaining three specimens into the container as described in A.2.2.

All containers shall be sealed with lids and kept at (20 ± 2) °C.

The distilled water shall not be changed during the full testing period but filled up with additional distilled water to maintain the water level.

The sulphate solution shall be replaced every 28 days.

Continue the immersion test by taking readings of comparator measurement in accordance with EN 12617-4, clause 6.5.2, of the specimens, L(t), immersed in distilled water and immersed in sulphate solution:

- After 2 weeks immersion, t = 2,
- After 4 weeks immersion, t = 4,
- After 8 weeks immersion, t = 8,
- After 12 weeks immersion, t = 12,

For longer immersion times, a tolerance of ± 1 day is accepted:

- After 15 weeks + 6 days to 16 weeks + 1 day immersion, t = 16,
- After 19 weeks + 6 days to 20 weeks + 1 day immersion, t = 20,
- After 25 weeks + 6 days to 26 weeks + 1 day immersion, t = 26,
- After 31 weeks + 6 days to 32 weeks + 1 day immersion, t = 32.

Inspect the specimens during comparator measurement for any sign of degradation, like formation of cracks, scaling, etc., and record the findings. Take photos of the specimens.

Where

L(0) .... mm...................... Initial reading of comparator measurement

L(t) .... mm...................... Reading of comparator measurement at time t

A.3.2 Test results

The readings of comparator measurements on the specimens shall be corrected with readings on the calibration rod in accordance with Equation (A.3.2.1) and Equation (A.3.2.2).

$$L_{i,\,corr}(0) = L_i(0) - L_{Cr}(0) \qquad \text{(A.3.2.1)}$$

$$L_{i,\,corr}(t) = L_i(t) - L_{Cr}(t) \qquad \text{(A.3.2.2)}$$

The corrected readings at time t shall be subtracted from the corrected initial readings by Equation (A.3.2.3), i.e., measurements prior to commencing the immersion test, to obtain the change in length of the specimens.

$$\Delta L_i(t) = L_{i,\,corr}(t) - L_{i,\,corr}(0) \qquad \text{(A.3.2.3)}$$

This change in length shall be divided by the gauge length of the specimen in accordance with Equation (A.3.2.4) to obtain the expansion.

$$\varepsilon_i(t) = \frac{\Delta L_i(t)}{L_{g,\,i}} \cdot 1\,000 \qquad \text{(A.3.2.4)}$$

The expansion of immersion in sulphate solution shall be calculated by Equation (A.3.2.5) by the mean value of three specimens immersed in sulphate solution, subtracted with the mean value of three specimens immersed in distilled water.

$$\varepsilon(t) = \frac{1}{3} \cdot \sum_{i = i_{ts} = 1}^{3} \varepsilon_i(t) - \frac{1}{3} \cdot \sum_{i = i_w = 1}^{3} \varepsilon_i(t) \qquad \text{(A.3.2.5)}$$

Where

- i ....—...................... Specimen number i
- i_{ts} ....—...................... Specimen number i, immersed in sulphate solution
- i_w ....—...................... Specimen number i, immersed in distilled water
- t .... week .... Time of immersion
- $L_i(0)$ .... mm...................... Initial reading of comparator measurement on specimen i, t = 0 weeks
- $L_i(t)$ .... mm...................... Reading of comparator measurement on specimen i at time t
- $L_{Cr}(0)$ .... mm...................... Initial reading of comparator measurement on calibration rod, t = 0 weeks
- $L_{Cr}(t)$ .... mm...................... Reading of comparator measurement on calibration rod at time t
- $L_{i,\,corr}(0)$ ...... mm...................... Corrected initial reading of comparator measurement on specimen i, t = 0 weeks
- $L_{i,\,corr}(t)$ .... mm...................... Corrected reading of comparator measurement on specimen i at time t
- $\Delta L_i(t)$ .... mm...................... Change in length of specimen i at time t
- $L_{g,\,i}$ .... mm...................... Gauge length of specimen, determined according to EN 12617-4, clause 6.6
- $\varepsilon_i(t)$ .... mm/m.................... Expansion of specimen i at time t
- $\varepsilon(t)$ .... mm/m.................... Mean expansion of specimens immersed in sulphate solution

ANNEX B: SULFATE RESISTANCE – FLAT PRISM METHOD

B.1 Preparation of specimens

B.1.1 Moulding of specimens

The specimens shall be 24 flat prisms made in accordance with Clause 2.2.6, with dimensions of 10 mm × 40 mm × 160 mm:

– 12 flat prisms with stainless steel studs for comparator measurement at both ends,

– 12 flat prisms without stainless steel studs,

The specimens shall be moulded and compacted in accordance with Clause 2.2.3.

B.1.2 Curing of specimens

After the particular curing process, the 24 flat prisms shall be immersed in saturated calcium hydroxide, $Ca(OH)_2$, solution at 20 °C, standing on edge and on gratings, until the age of 14 days. This describes the particular curing of the specimens.

NOTE Age of specimen in days shall be counted from the day of manufacturing the specimen. I.e., day of manufacturing is the day of mixing the fresh concrete.

B.2 Test procedure

B.2.1 Immersion of specimens

The specimens shall be immersed in sodium sulphate solution and calcium hydroxide solution as below:

– 6 specimens, i.e., 3 with stainless steel studs and 3 without stainless steel studs shall be immersed in a container with 4,4 % sodium sulphate solution at a temperature of (+ 5 ± 1) °C.

– 6 specimens, i.e., 3 with stainless steel studs and 3 without stainless steel studs shall be immersed in a container with 4,4 % sodium sulphate solution at a temperature of (+ 20 ± 1) °C.

– 6 specimens, i.e., 3 with stainless steel studs and 3 without stainless steel studs shall be immersed in a container with saturated calcium hydroxide solution at a temperature of (+ 5 ± 1) °C.

– 6 specimens, i.e., 3 with stainless steel studs and 3 without stainless steel studs shall be immersed in a container with saturated calcium hydroxide solution at a temperature of (+ 20 ± 1) °C.

All specimens shall be immersed, standing on edge and on gratings in the containers.

In all containers, the ratio of $\frac{\text{volume of solution}}{\text{volume of solid}}$ shall be between $\frac{3}{1}$ and $\frac{5}{1}$. The solution of 4,4 % Na_2SO_4 shall be replaced every 14 days with fresh Na_2SO_4 solution, temperature-controlled at 5 °C or 20 °C. The saturation of $Ca(OH)_2$ solution shall be checked every 14 days and, if necessary, corrected.

B.2.2 Measurements

The specimens with stainless steel studs shall be measured with a comparator in accordance with EN 12617-4, clauses 5.3 and 5.4, for change in length.

The specimens without stainless steel studs shall be measured for dynamic modulus of elasticity by ultrasonic pulse transit time measurement in accordance with CEN/TR 15177, clauses 4.2.2 and 7.3.2.

At the age of 14 days and prior to commencing the immersion test:

– 12 specimens, i.e., 6 with stainless steel studs and 6 without stainless steel studs shall be immersed in a container with saturated calcium hydroxide solution at a temperature of (+ 5 ± 1) °C for (24 ± 2) h.

– All 24 specimens shall be weighed after removing excess calcium hydroxide solution with a moist sponge at a resolution of 0,1 g.

– All 12 specimens with stainless steel studs shall be measured for initial comparator measurements in accordance with EN 12617-4, clause 6.3, L(0).

– With all 12 specimens without stainless steel studs the dynamic modulus of elasticity shall be determined by ultrasonic pulse transit time measurement.

After initial measurements immerse the specimens in accordance with Clause B.2.1 and perform measurements on the immersed specimens, i.e., weighing, comparator measurements in accordance with EN 12617-4, clause 6.5.2, L(t), and dynamic modulus of elasticity by ultrasonic pulse transit time measurement:

– After 2 weeks immersion, t = 2,

– After 4 weeks immersion, t = 4,

– After 8 weeks immersion, t = 8,

– After 12 weeks immersion, t = 12,

For longer immersion times, a tolerance of ± 1 day is accepted:

– After 15 weeks + 6 days to 16 weeks + 1 day immersion, t = 16,

– After 19 weeks + 6 days to 20 weeks + 1 day immersion, t = 20,

– After 25 weeks + 6 days to 26 weeks + 1 day immersion, t = 26,

– After 31 weeks + 6 days to 32 weeks + 1 day immersion, t = 32.

Inspect the specimens during the measurements for any sign of degradation, like formation of cracks, scaling, etc., and record the findings. Take photos of the specimens.

B.2.3 Test results

The expansion of the specimens with stainless steel studs shall be determined as mean of 3 specimens immersed in sulphate solution subtracted by the mean of 3 specimens immersed in calcium hydroxide solution.

The readings of comparator measurements on the specimens shall be corrected with readings on the calibration rod in accordance with Equation (B.2.3.1) and Equation (B.2.3.2).

$$L_{i,\,corr}(0) = L_i(0) - L_{Cr}(0) \qquad \text{(B.2.3.1)}$$

$$L_{i,\,corr}(t) = L_i(t) - L_{Cr}(t) \tag{B.2.3.2}$$

The corrected readings shall be subtracted from the corrected initial readings by Equation (B.2.3.3), i.e., measurements prior to commencing the immersion test, to obtain the change in length of the specimens.

$$\Delta L_i(t) = L_{i,\,corr}(t) - L_{i,\,corr}(0) \tag{B.2.3.3}$$

This change in length shall be divided by the gauge length of the specimen by Equation (B.2.3.4) to obtain the expansion.

$$\varepsilon_i(t) = \frac{\Delta L_i(t)}{L_{g,\,i}} \cdot 1\,000 \tag{B.2.3.4}$$

The expansion of immersion in sulphate solution shall be calculated by Equation (B.2.3.5) and Equation (B.2.3.6) as the mean value of three specimens immersed in sulphate solution, subtracted with the mean value of three specimens immersed in calcium hydroxide solution.

$$\varepsilon_5(t) = \frac{1}{3} \cdot \sum_{i = i_{ts,\,5} = 1}^{3} \varepsilon_i(t) - \frac{1}{3} \cdot \sum_{i = i_{Ca,\,5} = 1}^{3} \varepsilon_i(t) \tag{B.2.3.5}$$

$$\varepsilon_{20}(t) = \frac{1}{3} \cdot \sum_{i = i_{ts,\,20} = 1}^{3} \varepsilon_i(t) - \frac{1}{3} \cdot \sum_{i = i_{Ca,\,20} = 1}^{3} \varepsilon_i(t) \tag{B.2.3.6}$$

Where

i ....—...................... Specimen number i

$i_{ts,\,5}$ ....—...................... Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts,\,5}$ = 1 to 3

$i_{ts,\,20}$ ....—...................... Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts,\,20}$ = 1 to 3

$i_{Ca,\,5}$ ....—...................... Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca,\,5}$ = 1 to 3

$i_{Ca,\,20}$ ....—...................... Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca,\,20}$ = 1 to 3

t .... week .... Time of immersion

$L_i(0)$ .... mm...................... Initial reading of comparator measurement on specimen i, t = 0 weeks

$L_i(t)$ .... mm...................... Reading of comparator measurement on specimen i at time t

$L_{Cr}(0)$ .... mm...................... Initial reading of comparator measurement on calibration rod, t = 0 weeks

$L_{Cr}(t)$ .... mm...................... Reading of comparator measurement on calibration rod at time t

$L_{i,\,corr}(0)$ ...... mm...................... Corrected initial reading of comparator measurement on specimen i, t = 0 weeks

$L_{i,\,corr}(t)$ .... mm...................... Corrected reading of comparator measurement on specimen i at time t

$\Delta L_i(t)$ .... mm...................... Change in length of specimen i at time t

$L_{g,\,i}$ .... mm...................... Gauge length of specimen i, determined according to EN 12617-4, clause 6.6

$\varepsilon_i(t)$ .... mm/m.................... Expansion of specimen i at time t

$\varepsilon_5(t)$ .... mm/m.................... Mean expansion of specimens immersed in sulphate solution at 5 °C

$\varepsilon_{20}(t)$ .... mm/m.................... Mean expansion of specimens immersed in sulphate solution at 20 °C

The dynamic modulus of elasticity shall be determined in accordance with Equation (B.2.3.7), Equation (B.2.3.8), Equation (B.2.3.9), and Equation (B.2.3.10) as mean value of 3 specimens immersed in sulphate solution and the mean of 3 specimens immersed in calcium hydroxide solution.

$$E_{dyn,\,ts,\,5}(t) = \frac{1}{3} \cdot \sum_{i = i_{ts,\,5} = 1}^{3} E_{dyn,\,i}(t) \qquad \text{(B.2.3.7)}$$

$$E_{dyn,\,ts,\,20}(t) = \frac{1}{3} \cdot \sum_{i = i_{ts,\,20} = 1}^{3} E_{dyn,\,i}(t) \qquad \text{(B.2.3.8)}$$

$$E_{dyn,\,Ca,\,5}(t) = \frac{1}{3} \cdot \sum_{i = i_{Ca,\,5} = 1}^{3} E_{dyn,\,i}(t) \qquad \text{(B.2.3.9)}$$

$$E_{dyn,\,Ca,\,20}(t) = \frac{1}{3} \cdot \sum_{i = i_{Ca,\,20} = 1}^{3} E_{dyn,\,i}(t) \qquad \text{(B.2.3.10)}$$

The change in dynamic modulus of elasticity relative to the specimens immersed in calcium hydroxide solution shall be calculated by Equation (B.2.3.11), Equation (B.2.3.12), Equation (B.2.3.13), and Equation (B.2.3.14).

$$RDM_5(t) = \frac{E_{dyn,\,ts,\,5}(t)}{E_{dyn,\,Ca,\,5}(t)} \cdot 100 \qquad \text{(B.2.3.11)}$$

$$RDM_{20}(t) = \frac{E_{dyn,\,ts,\,20}(t)}{E_{dyn,\,Ca,\,20}(t)} \cdot 100 \qquad \text{(B.2.3.12)}$$

$$\Delta RMD_5(t) = RDM_5(t) - RDM_5(0) \qquad \text{(B.2.3.13)}$$

$$\Delta RMD_{20}(t) = RDM_{20}(t) - RDM_{20}(0) \qquad \text{(B.2.3.14)}$$

Where

i ....—...................... Specimen number i

$i_{ts,\,5}$ ....—...................... Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts,\,5}$ = 1 to 3

$i_{ts,\,20}$ ....—...................... Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts,\,20}$ = 1 to 3

$i_{Ca,\,5}$ ....—...................... Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca,\,5}$ = 1 to 3

$i_{Ca, 20}$ ....—........................ Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca, 20}$ = 1 to 3

t................ weeks .... Time of immersion

$E_{dyn, i}(t)$ .... GPa .... Dynamic modulus of elasticity of specimen i at time t

$E_{dyn, ts, 5}(t)$ ... GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in sulphate solution at 5 °C

$E_{dyn, ts, 20}(t)$ .. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in sulphate solution at 20 °C

$E_{dyn, Ca, 5}(t)$ .. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in calcium hydroxide solution at 5 °C

$E_{dyn, Ca, 20}(t)$. GPa .... Mean dynamic modulus of elasticity of 3 specimens at time t, immersed in calcium hydroxide solution at 20 °C

$RDM_5(t)$ .... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 5 °C, to specimens immersed in calcium hydroxide solution at 5 °C, at time t

$RDM_{20}(t)$ ...... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 20 °C, to specimens immersed in calcium hydroxide solution at 20 °C, at time t

$RDM_5(0)$ .... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 5 °C, to specimens immersed in calcium hydroxide solution at 5 °C, prior to commencing immersion, t = 0 weeks

$RDM_{20}(0)$ ..... % .... Relative dynamic modulus of elasticity from specimens immersed in sulphate solution at 20 °C, to specimens immersed in calcium hydroxide solution at 20 °C, prior to commencing immersion, t = 0 weeks

$\Delta RMD_5(t)$ ..... % .... Change in relative dynamic modulus of elasticity for specimens immersed at 5 °C, at time t

$\Delta RMD_{20}(t)$ .... % .... Change in relative dynamic modulus of elasticity for specimens immersed at 20 °C, at time t

Change in mass of specimens to mass prior to commencing immersion, t = 0 weeks, shall be calculated in accordance with Equation (B.2.3.15).

$$\Delta M_i(t) = M_i(t) - M_i(0) \qquad \text{(B.2.3.15)}$$

By Equation (B.2.3.16) change in mass relative to mass prior to commencing immersion, t = 0 weeks, shall be calculated.

$$m_i(t) = \frac{\Delta M_i(t)}{M_i(0)} \cdot 100 \qquad \text{(B.2.3.16)}$$

Calculation of mean relative mass of specimens for the respective immersion shall be calculated in accordance with Equation (B.2.3.17), Equation (B.2.3.18, Equation (B.2.3.19), Equation (B.2.3.20), Equation (B.2.3.21), and Equation (B.2.3.22).

$$m_{ts, 5}(t) = \frac{1}{3} \cdot \sum_{i = i_{ts, 5} = 1}^{3} m_i(t) \qquad \text{(B.2.3.17)}$$

$$m_{ts,\,20}(t) = \frac{1}{3} \cdot \sum_{i = i_{ts,\,20} = 1}^{3} m_i(t) \qquad \text{(B.2.3.18)}$$

$$m_{Ca,\,5}(t) = \frac{1}{3} \cdot \sum_{i = i_{Ca,\,5} = 1}^{3} m_i(t) \qquad \text{(B.2.3.19)}$$

$$m_{Ca,\,20}(t) = \frac{1}{3} \cdot \sum_{i = i_{Ca,\,20} = 1}^{3} m_i(t) \qquad \text{(B.2.3.20)}$$

$$\Delta m_5(t) = m_{ts,\,5}(t) - m_{Ca,\,5}(t) \qquad \text{(B.2.3.21)}$$

$$\Delta m_{20}(t) = m_{ts,\,20}(t) - m_{Ca,\,20}(t) \qquad \text{(B.2.3.22)}$$

Where

i	—	Specimen number i
$i_{ts,\,5}$	—	Specimen number i, immersed in sulphate solution at 5 °C, $i_{ts,\,5}$ = 1 to 3
$i_{ts,\,20}$	—	Specimen number i, immersed in sulphate solution at 20 °C, $i_{ts,\,20}$ = 1 to 3
$i_{Ca,\,5}$	—	Specimen number i, immersed in calcium hydroxide solution at 5 °C, $i_{Ca,\,5}$ = 1 to 3
$i_{Ca,\,20}$	—	Specimen number i, immersed in calcium hydroxide solution at 20 °C, $i_{Ca,\,20}$ = 1 to 3
t	weeks	Time of immersion
$M_i(0)$	g	Mass of specimen i prior to immersion, at time t = 0 weeks
$M_i(t)$	g	Mass of specimen i at time t
$\Delta M_i(t)$	g	Change in mass of specimen i at time t, i.e., mass at time t minus mass prior to immersion
$m_i(t)$	%	Relative change in mass of specimen i at time t
$m_{ts,\,5}(t)$	%	Mean relative change in mass of specimens at time t, immersed in sulphate solution at 5 °C
$m_{ts,\,20}(t)$	%	Mean relative change in mass of specimens at time t, immersed in sulphate solution at 20 °C
$m_{Ca,\,5}(t)$	%	Mean relative change in mass of specimens at time t, immersed in calcium hydroxide solution at 5 °C
$m_{Ca,\,20}(t)$	%	Mean relative change in mass of specimens at time t, immersed in calcium hydroxide solution at 20 °C
$\Delta m_5(t)$	%	Difference in relative change in mass at time t between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C
$\Delta m_{20}(t)$	%	Difference in relative change in mass at time t between specimens immersed in sulphate solution and specimens immersed in calcium hydroxide solution, at 5 °C

Description of signs of degradation or the conclusion no degradation is present.

ANNEX C: DETERMINATION OF RESISTANCE TO ATTACK BY ACID

C.1 Test solutions and reagents

Test solution shall be acetic acid solution, composed of:

- 20 l of deionised water and
- 2,75 l acetic acid with 80 % concentration.

The reagent for pH adjustment shall be acetic acid with 80 % concentration.

C.2 Test procedure

C.2.1 Measurements and immersion

After taking the specimens out from water, remove excess water with a moist sponge, and weigh the specimens at a resolution of 0,1 g before immersion.

A plastic container with dimensions of about 600 mm × 300 mm shall be filled with about 20 l of acetic acid solution. The specimens shall be immersed in the acetic acid solution with clearance of about 15 mm to bottom and wall of the container as well as between the individual specimens. The container shall be tightly closed with a lid and kept at (+ 20 ± 2) °C.

After 1 day, 4 days, 7 days, and 14 days immersion the solution in the container shall be stirred and the pH-value measured. A pH-value of 4,0 and above shall be compensated by adding acetic acid until pH-value of about 3,5 is attained.

The immersion is completed after 21 days.

The specimens shall be removed from the container:

- Rinsed off with tap water,
- Manually brushed to remove loose parts of the specimens with a wire brush, bristles 0,5 mm diameter and 20 mm long.
- Weighed at a resolution of 0,1 g.

C.2.2 Test results

The mass loss of each specimen shall be calculated by Equation (C.2.2.1) as the difference of mass before immersion and mass after 21 days immersion in acetic acid solution.

$$\Delta m_{21,\,i} = m_{0,\,i} - m_{21,i\,i} \qquad \text{(C.2.2.1)}$$

Divide by Equation (C.2.2.2) mass loss by surface area of specimen.

$$L_{21,\,i} = \frac{\Delta m_{21,\,i}}{1\,000 \cdot A_{pris}} \qquad \text{(C.2.2.2)}$$

Calculate mean value of mass loss per unit area of all specimens in accordance with Equation (C.2.2.3).

$$L_{21,\,mean} = \frac{1}{3} \cdot \sum_{i=1}^{3} L_{21,\,i} \qquad \text{(C.2.2.3)}$$

Where

i ....—....................... Specimen number i

$m_{0,\,i}$ .... g........................ Mass of specimen i prior to immersion in acetic acid solution

$m_{21,\,i}$ .... g........................ Mass of specimen i after 21 days immersion in acetic acid solution

$\Delta m_{21,\,i}$ .... g........................ Mass loss of specimen i after 21 days immersion in acetic acid solution

A_{pris} .... m^2 .... Surface area of specimen.
Apply A_{pris} = 0,028 8 m^2 for prism 40 mm × 40 mm × 160 mm

NOTE In 3-dimensional Euclidean space, the surface of a prism comprises 6 sub-surfaces. The surface area of this prism is the algebraic sum of the areas of its 6 sub-surfaces. Each sub-surface is a rectangle, and the area of the rectangle is the algebraic product of its side lengths. Using the nominal dimensions, the surface area of a test specimen is calculated as:

$4 \cdot 40\ mm \cdot 160\ mm + 2 \cdot 40\ mm \cdot 40\ mm = 4 \cdot 6\,400\ mm^2 + 2 \cdot 1\,600\ mm^2 = 25\,600\ mm^2 + 3\,200\ mm^2 = 28\,800\ mm^2 = 0{,}028\,8\ m^2$

$L_{21,\,i}$ .... kg/m^2.................... Mass loss per unit area of specimen i

$L_{21,\,mean}$ ...... kg/m^2.................... Mean mass loss per unit area of 3 specimens

ANNEX D: DETERMINATION OF RESISTANCE AGAINST ALKALI SILICA REACTION

D.1 Aggregates

The aggregates for testing obtain the following performances:

- Size 0/4 mm,
- Composed of fractions obtained by sieving in accordance with Table D.1.1.
- Elongation in immersion test in accordance with Clause D.2, Clause D.3, and Clause D.3.2 with Portland cement CEM I 42,5 R is 1,2 ‰ to 1,5 ‰.

Table D.1.1 Composition of test aggregates

Size	Fraction
mm	g
0,125/0,25	202
0,25/0,5	338
0,5/1	338
1/2	337
2/4	135
Sum	1 350

D.2 Test solution

3 000 ml of sodium hydroxide solution, composed of:

- 120 sodium hydroxide diluted in and
- 3 000 ml tap water.

D.3 Test procedure

D.3.1 Measurements and immersion

The specimens shall be measured with a comparator in accordance with EN 12617-4, clauses 5.3 and 5.4, for change in length.

The test procedure:

- The specimens shall be immersed in tap water in a closed container, supported on triangular battens at a temperature of (+ 80 ± 2) °C for (24 ± 2) h.

– The warm specimens shall be measured for initial comparator measurements in accordance with EN 12617-4, clause 6.3, L(0), observing not to substantially drop the temperature of the specimens.

– The specimens shall be immersed in sodium hydroxide solution in a closed container, inner dimensions 200 mm × 200 mm × 200 mm, supported on triangular battens at a temperature of (+ 80 ± 2) °C. Clearance between specimens and container wall shall be at least 10 mm.

– Comparator measurements in accordance with EN 12617-4, clause 6.5.2, L(t), shall be performed after 2 days, 7 days, 9 days, and 14 days after the initial comparator measurement. At each measurement, the temperature of the specimens shall not substantially drop.

D.3.2 Test results

The expansions of the specimens shall be determined as comparator measurement results after 14 days subtracted by initial comparator measurement results and the mean value of expansions of all specimens shall be calculated.

The readings of comparator measurements on the specimens shall be corrected with readings on the calibration rod in accordance with Equation (D.3.2.1) and Equation (D.3.2.2).

$$L_{i,\,corr}(0) = L_i(0) - L_{Cr}(0) \qquad \text{(D.3.2.1)}$$

$$L_{i,\,corr}(t) = L_i(t) - L_{Cr}(t) \qquad \text{(D.3.2.2)}$$

The corrected readings shall be subtracted from the corrected initial readings in accordance with Equation (D.3.2.3), i.e., measurements prior to commencing the immersion test, to obtain the change in length of the specimens.

$$\Delta L_i(t) = L_{i,\,corr}(t) - L_{i,\,corr}(0) \qquad \text{(D.3.2.3)}$$

By Equation (D.3.2.4) this change in length shall be divided by the gauge length of the specimen to obtain the expansion.

$$\varepsilon_i(t) = \frac{\Delta L_i(t)}{L_{g,\,i}} \cdot 1\,000 \qquad \text{(D.3.2.4)}$$

The expansion of immersion in test solution shall be calculated by Equation (D.3.2.5) as the mean value of three specimens immersed in test solution.

$$\varepsilon(t) = \frac{1}{3} \cdot \sum_{i=1}^{3} \varepsilon_i(t) \qquad \text{(D.3.2.5)}$$

Where

i ....—..................... Specimen number i

t ....days.................... Time of immersion

$L_i(0)$ .... mm..................... Initial reading of comparator measurement on specimen i, t = 0 days

$L_i(t)$ .... mm..................... Reading of comparator measurement on specimen i at time t

$L_{Cr}(0)$ .... mm..................... Initial reading of comparator measurement on calibration rod, t = 0 days

$L_{Cr}(t)$ .... mm..................... Reading of comparator measurement on calibration rod at time t

$L_{i, corr}(0)$ ...... mm...................... Corrected initial reading of comparator measurement on specimen i, t = 0 days

$L_{i, corr}(t)$ .... mm...................... Corrected reading of comparator measurement on specimen i at time t

$\Delta L_i(t)$........... mm...................... Change in length of specimen i at time t

$L_{g, i}$.............. mm...................... Gauge length of specimen i, determined according to EN 12617-4, clause 6.6

$\varepsilon_i(t)$ .... mm/m.................... Expansion of specimen i at time t

$\varepsilon(t)$............ mm/m.................... Mean expansion of specimens immersed in test solution