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EAD 150036-00-0301

August 2018

European Assessment Document for

Flouraluminate rapid setting cement

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1 SCOPE OF THE EAD

1.1 Description of the construction product

Fluoraluminate rapid setting cement is a special cement, i.e., a hydraulic binder with quick set and strength rise features. Initial setting time is between 1 min 30 s and 15 min. Fluoraluminate rapid setting cement is produced in industrial-scale. Clinker for Fluoraluminate rapid setting cement is made by sintering a precisely specified mixture of several raw materials. These raw materials, finely divided, are intimately mixed and therefore result in a homogeneous mixture.

Short term strength is developed by hydration of calcium fluoroaluminate that, in presence of water and calcium sulphate, forms ettringite, $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$. A stable strength development extending over a long time is due to hydration of alite (C_3S^1) and belite (C_2S), forming calcium silicate hydrates, see [1]². Due to this hydration process, chemical composition and pore structure of the matrix of the Fluoraluminate rapid setting cement, at long term, are the same as of the matrix of common Portland cement.

The Fluoraluminate rapid setting cement is based on a clinker with particular composition:

- It contains calcium fluoroaluminate (C₁₁A₇f)
- The main constituents are tricalcium silicate (C₃S) and bicalcium silicate (C₂S)

The composition of the Fluoraluminate rapid setting cement is:

- Clinker 80 %–94.5 %
- Calcium oxide 0 %–3 %

Optionally, Fluoraluminate rapid setting cement may contain one or several of the following constituents.

CEM I or CEM II Portland cement according to EN 197-1³.....0 %–10 %

The product is not fully covered by the following harmonised technical specifications: EN 197-1 and EAD 150008-00-0301.

Deviations of Fluoraluminate rapid setting cement from common cement according to EN 197-1 are:

- It is a special cement with special composition and not a common Portland cement according to EN 197-1.
- Sulphate content, \leq 12.5 %, is higher than EN 197-1 cements of \leq 4.0 %
- Rapid setting feature implies initial setting time, between 1 min 30 s and 15 min, is considerably shorter than for EN 197-1 cements of ≥ 45 min.
- Rapid setting requires different assessment methods than those reported in EN 197-1.
- Fluoraluminate rapid setting cement is not considered as a sulphate resisting cement.
- Fluoraluminate rapid setting cement is not considered as a low heat cement.
- Fluoraluminate rapid setting cement is not considered as a pozzolanic cement.

Deviations of Fluoraluminate rapid setting cement from EAD 150008-00-0301 cement are:

- Different to EAD 150008-00-0301 the Fluoraluminate rapid setting cement of present EAD is produced in industrial-scale and its raw materials are not "from a single specific homogeneous geological seam".
- Mineralogy of the Fluoraluminate rapid setting cement of present EAD is different to EAD 150008-00-0301. In particular, the Fluoraluminate rapid setting cement contains fluor, essential for the Fluoraluminate rapid setting cement.

¹ C = Calcium oxide, S = Silicon dioxide, A = Aluminium oxide, and f = Calcium fluoride

² Reference documents are listed in Clause 4.

³ All undated references to standards or to EADs in this EAD are to be understood as references to the dated versions listed in Clause 4.

 Fluoraluminate rapid setting cement has peculiar features that need to be reflected in the assessment. Consequently, the assessment methods for Fluoraluminate rapid setting cement and assessment methods in EAD 150008-00-0301 will differ from each other.

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 transport, storage, maintenance, replacement and repair of the product, as he considers necessary.

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

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

1.2 Information on the intended uses of the construction product

1.2.1 Intended uses

Fluoraluminate rapid setting cement is intended to be used to produce concretes, mortars, grouts, and other mixes for construction and for the manufacture of prefabricated construction elements.

The intended use includes Fluoraluminate rapid setting cement in blends with CEM I cements according to EN 197-1 with up to 75 % of CEM I cement.

Due to quick setting and hardening of Fluoraluminate rapid setting cement, concrete with Fluoraluminate rapid setting cement allows for quick demoulding and mortars are typically used for finishing and repairing works.

Heat treatment is not intended for Fluoraluminate rapid setting cement.

1.2.2 Working life/Durability

The assessment methods included or referred to in this EAD have been written based on the manufacturer's request to take into account a working life of concrete, mortar and grout with Fluoraluminate rapid setting cement that is similar to the one of concrete, mortar and grout with ordinary Portland cement, and for the intended use of 100 years when installed in the works, provided that the Fluoraluminate rapid setting 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 defined 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 works⁴.

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

1.3.1 Symbols

1.3.1.1 Expansion

L(0)..... mm...... Initial reading of comparator measurement L(t)..... mm...... Reading of comparator measurement at time t

⁴ 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.

	i	Specimen number i				
	İts	Specimen number i, immersed in test solution				
	iw	Specimen number i, immersed in distilled water				
	tweek	Time of immersion				
	Li(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				
	$\Delta L_i(t) \dots mm \dots$	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				
	εi(t) mm/m	Expansion of specimen i at time t				
	$\epsilon(t)$ mm/m	Mean expansion of specimens immersed in test solution				
1.3.1.2	Porosity					
	rnm	pore radius				
	p MPa	applied absolute pressure				
1.3.1.3	Resistance to chloride penetration	n				
		non-steady-state chloride migration coefficient,				
	z	absolute value of ion valence, for chloride, $z = 1$				
	FJ/(V · mol)	Faraday constant, F = $9.648 \cdot 10^4 \text{ J/(V} \cdot \text{mol})$				
	UV	absolute value of the applied voltage				
	RJ/(K·· mol)	gas constant, R = $8.314 \text{ J/(K} \cdot \text{ mol)}$				
	ТКК.	average value of the initial and final temperatures in the anolyte solution				
	Lmm	thickness of the specimen				
	x dm	average value of the penetration depths				
	tss	test duration, seconds				
	erf ^{_1}	inverse of error function				
	CdNN	chloride concentration at which the colour changes, $c_d \approx 0.07$ N for OPC concrete				
	coN	chloride concentration in the catholyte solution, $c_0 \approx 2 \text{ N}$				
1.3.1.4	Carbonation					
	C _{dcr}	Abbreviation for testing carbonation by carbonation depth determination according to EN 12390-10				
	dĸ mm	Carbonation depth				
	tc days	Duration of carbonation				
	k_{c} $\frac{mm}{\sqrt{days}}$	Rate of carbonation				

1.3.1.5 Freeze-thaw resistance without de-icing agent

FT_{CF} Abbreviation for testing freeze-thaw resistance with CF-procedure, i.e., testing with de-ionized water and without de-icing agent according to CEN/TS 12390-9, section 7

1.3.1.6 Freeze-thaw resistance with de-icing agent

FTS_{CDF} — Abbreviation for testing freeze-thaw resistance with CDF-procedure, i.e., testing with 3 % by mass sodium chloride (NaCI) solution as deicing agent according to CEN/TS 12390-9, section 7

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 Fluoraluminate rapid setting 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

Nº	Essential characteristic	Assessment method	Type of expression of product performance
	Basic Work Requirement 1: Mechanical resistance and stability		
1	Early strength	2.2.1	Level
2	Standard strength	2.2.2	Level
3	Initial setting time	2.2.3	Level
4	Soundness	2.2.4	Level
5	Loss on ignition	2.2.5	Level
6	Sulphate content	2.2.6	Level
7	Chloride content	2.2.7	Level
8	Expansion	2.2.8	Level
9	Mineralogy	2.2.9	Level
10	Porosity	2.2.10	Level, description
11	Effect of high temperature on mortar hardened under standard conditions	2.2.11	Level
12	Effect of high temperature on mortar at early age	2.2.12	Level
13	Setting time of blend with CEM I cement	2.2.13	Level
14	Resistance to chloride penetration	2.2.14	Level
15	Carbonation	2.2.15	Level, description
16	Freeze-thaw resistance without de-icing agent	2.2.16	Level
17	Freeze-thaw resistance with de-icing agent	2.2.17	Level
	Basic Work Requirement 3: Hy	giene, health and the envi	ronment
18	Water-soluble hexavalent chromium content	2.2.18	Level
19	Release of dangerous substances	EN 197-1, Annex ZA.1, Note 1 and Note 2	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 Early strength

Early strength of Fluoraluminate rapid setting cement is the compressive strength determined at 3 hours.

Early strength shall be determined and expressed according to EN 196-1. Due to the rapid setting characteristic, the following deviations to EN 196-1 shall apply for Fluoraluminate rapid setting cement.

Composition of mortar

- Sand......1 350 g
- Cement675 g
- Water270 g

Water to cement ratio shall be equal to 0.4.

Ratio sand to cement shall be equal to 2.

A dry premix of the solid constituents, i.e., sand and cement, may be prepared prior to addition of water. The premix can be made by e.g., agitating by hand the two constituents in a closed polyethylene bag.

After preparing the dry mix, water shall be added and immediately mixed at a speed of 14.7 rad/s for 10 seconds and subsequently at 29.8 rad/s for 15 seconds.

NOTE 1 14.7 rad/s [≙] 140 rpm
In EN 196-1 140 rpm is designated "low speed".
29.8 rad/s [≙] 285 rpm
In EN 196-1 285 rpm is designated "high speed".

NOTE 2 Tolerance of speeds are according to EN 196-1.

NOTE 3 Moulding and conditioning of test specimens shall be according to EN 196-1.

Compression tests shall be carried out after 3 hours \pm 5 min.

Arithmetic mean of compressive strength in MPa shall be stated as early strength in the ETA.

2.2.2 Standard strength

Standard strength of Fluoraluminate rapid setting cement is the compressive strength determined at 28 days. Standard strength shall be expressed according to EN 196-1.

Due to the rapid setting characteristic, standard strength shall be determined as early strength according to Clause 2.2.1, except testing for compressive strength shall be performed after 28 days \pm 8 h.

NOTE Moulding and conditioning of test specimens shall be according to EN 196-1.

Arithmetic mean of compressive strength in MPa shall be stated as standard strength in the ETA.

2.2.3 Initial setting time

Initial setting time of Fluoraluminate rapid setting cement shall be determined according to EN 196-3. Due to the rapid setting characteristic, the following deviations to EN 196-3 shall apply for Fluoraluminate rapid setting cement.

Composition of cement paste

Cement500 g

- Water180 g

Water to cement ratio shall be equal to 0.36 \pm 0.01. Consistence shall be:

- High in viscosity so that no cement paste leaks out when filling the Vicat mould and during testing.
- Low in viscosity so that on the first attempt the needle penetrates through cement paste down to baseplate.

Water shall be added to the Fluoraluminate rapid setting cement and immediately mixed at a speed of 14.7 rad/s for 10 seconds and subsequently at 29.8 rad/s for 15 seconds.

NOTE 1 14.7 rad/s \triangleq 140 rpm In EN 196-1 140 rpm is designated "low speed". 29.8 rad/s \triangleq 285 rpm In EN 196-1 285 rpm is designated "high speed".

NOTE 2 Tolerance of speeds are according to EN 196-1.

Initial setting time in min and water to cement ratio shall be stated in the ETA.

2.2.4 Soundness

Soundness of Fluoraluminate rapid setting cement shall be determined according to EN 196-3. Due to the rapid setting characteristic, the following deviations to EN 196-3 shall apply for Fluoraluminate rapid setting cement.

Composition of cement paste

- Cement500 g
- Water180 g

Water to cement ratio shall be equal to 0.36 ± 0.01 as used in testing initial setting time according to Clause 2.2.3.

Water shall be added to the Fluoraluminate rapid setting cement and immediately mixed at a speed of 14.7 rad/s for 10 seconds and subsequently at 29.8 rad/s for 15 seconds.

 NOTE 1 14.7 rad/s [△] 140 rpm In EN 196-1 140 rpm is designated "low speed".
 29.8 rad/s [△] 285 rpm In EN 196-1 285 rpm is designated "high speed".

NOTE 2 Tolerance of speeds are according to EN 196-1.

Soundness⁵ shall be \leq 10 mm.

For soundness the maximum value in mm and water to cement ratio shall be stated in the ETA.

2.2.5 Loss on ignition

Loss on ignition of Fluoraluminate rapid setting cement shall be determined according to EN 196-2. Loss on ignition⁵ shall be \leq 5.0 % by mass.

Maximum value of loss on ignition in percentage by mass shall be stated in the ETA.

2.2.6 Sulphate content

Sulphate content of Fluoraluminate rapid setting cement shall be determined according to EN 196-2. Sulphate content, expressed as SO₃, in percentage by mass shall be stated in the ETA.

⁵ The required performance originates from EN 197-1.

2.2.7 Chloride content

Chloride content of Fluoraluminate rapid setting cement shall be determined according to EN 196-2. Chloride content⁶ shall be \leq 0.10 % by mass.

Maximum chloride content in percentage by mass shall be stated in the ETA.

2.2.8 Expansion

Expansion of Fluoraluminate rapid setting cement shall be determined according to Annex B.

Arithmetic mean of expansion in mm/m shall be stated in the ETA.

2.2.9 Mineralogy

2.2.9.1 General

The chemical composition of the clinker of Fluoraluminate rapid setting cement shall be determined according to EN 196-2. The quantitative mineralogical composition shall be calculated with the content of the main oxides, determined by chemical analysis, according to the modified Bogue method as described in Clause 2.2.9.2.

The qualitative mineralogy composition shall be also determined in accordance with the test method described in the Clause 2.2.9.3.

The main constituents of Fluoraluminate rapid setting cement are:

- Tricalcium Silicate (C₃S)
- Bicalcium Silicate (C₂S)
- Calcium Fluoroaluminate (C₁₁A₇f)
- Ferrite (C₄AF)
- Calcium Sulphate, mainly as Anhydrite (CaSO₄)
- 2.2.9.2 Mineralogical composition of clinker

Mineralogical composition shall be calculated – called Bogue method – with the chemical composition of the clinker in terms of main oxides, i.e., CaO, SiO₂, Al₂O₃, Fe₂O₃.

Furthermore, content of SO3 and free lime, CaOfree, shall be input values for the calculation.

The free lime content, CaOfree, shall be determined according to EN 451-1.

The principle of the calculation is:

- Assume that the composition of the 4 major phases is C₃S, C₂S, C₄AF, C₁₁A₇f.
- Assume that the Fe_2O_3 occurs as C₄AF.
- Assume that the remaining AI_2O_3 occurs as $C_{11}A_7f$.
- Deduct from the CaO content the amount attributable to C₄AF, C₁₁A₇f, calcium sulphate, and free lime, and solve to simultaneous equations to obtain the contents of C₃S and C₂S.

This leads to Equation 2.2.9.2.1, Equation 2.2.9.2.2, Equation 2.2.9.2.3, and Equation 2.2.9.2.4.

$$\begin{array}{rcl} C_3S & = & 4.0710 \cdot CaO & - & 7.6024 \cdot SiO_2 & - & 3.8335 \cdot Al_2O_3 & - & 3.2613 \cdot Fe_2O_3 & - \\ & & - & 4.0710 \cdot CaO_{free} - & 2.8466 \cdot SO_3 \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{rcl} \mbox{Equation 2.2.9.2.1} \\ $

 $\begin{array}{rcl} C_2S & = & -\ 3.0710 \cdot CaO \ + \ 8.6024 \cdot SiO_2 \ + \ 2.8916 \cdot Al_2O_3 \ + \ 2.4600 \cdot Fe_2O_3 \ + \\ & +\ 3.0710 \cdot CaO_{free} \ + \ 2.1472 \cdot SO_3 \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{rcl} \mbox{Equation } 2.2.9.2.2 \ + \ 2.4600 \cdot Fe_2O_3 \ + \$

⁶ The required performance originates from EN 197-1.

Equation 2.2.9.2.4

$C_4AF = 3.0432 \cdot Fe_2O_3$	Equation 2.2.9.2.3
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 $C_{11}A_7f = 1.9735 \cdot Al_2O_3 - 1.2601 \cdot Fe_2O_3$

- 2.2.9.3 Mineralogy by X-ray diffraction
- 2.2.9.3.1 Introduction

X-ray diffraction is a direct method for qualitative and also quantitative characterisation of fine-grained materials such as clinker [2].

2.2.9.3.2 Preparation of specimen

Careful, consistent preparation is necessary to obtain a homogeneous specimen for analysis. Critical factors are particle and crystallite size, specimen thickness, preferred orientation, strain and surface planarity. To minimize these potential problems, care shall be taken in grinding and specimen preparation.

NOTE See [3] for further information on preparation of specimen.

Grinding with a laboratory mill shall be to a fineness of

- ≤ 10 % on 40 μ m sieve
- Mean grain size 10 μm–15 μm

The ground clinker shall be pressed to pellets for X-ray diffraction measurement.

2.2.9.3.3 X-ray diffractometer

The X-ray diffractometer performs measurement of the X-ray diffraction pattern from which the crystalline phases are identified, and proportion of each phase can be quantitatively determined. X-ray diffractometers comprise the following elements:

- Source of radiation (X-ray tube);
- Detection and counting equipment;
- Specimen holder and goniometer.

An X-ray diffractometer with the following parameters shall be used:

- Peak position, $2\cdot\theta,$ shall be within \pm 0.02 ° of a clearly identifiable peak, e.g., quartz 101 peak as reference
- Peak resolution allows to separate, e.g., 5 peaks around quartz 203 peak as reference
- Peak intensity shall be at least 2 000 counts, e.g., of the quartz 101 peak as reference
- Ratios of peak intensities shall be within 5 % of reference peak intensities
- The X-ray diffractometer performs digital data acquisition

2.2.9.3.4 Measurement

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

2.2.9.3.5 Qualitative analysis

The phases, commonly present in clinkers and cements, are summarised in Table 2.2.9.3.5.1.

Notation	Designation	Chemical Formula	
C₃S	Tricalcium Silicate	Ca ₃ SiO ₅	
C ₂ S	Bicalcium Silicate	Ca ₂ SiO ₄	
C ₁₁ A ₇ f	Calcium Fluoroaluminate	Ca11Al14O32CaF2	
C ₃ A	Tricalcium Aluminate	Ca ₃ Al ₂ O ₆	
C4AF	Calciumaluminateferrit	Ca ₂ (Al, Fe) ₂ O ₅	
Calcium sulphate dihydrate	Gypsum	CaSO ₄ ·2H ₂ O	
Calcium sulphate hemihydrate	Hemihydrate	CaSO ₄ · 1/2H ₂ O	
Calcium sulphate	Anhydrite	CaSO ₄	

Table 2.2.9.3.5.1 Phases commonly present in clinkers and cements

2.2.9.4 Test results

The mineralogy of clinker, as determined according to Clause 2.2.9.2 for C_3S , C_2S , C_4AF , and $C_{11}A_7f$, shall be checked qualitatively as indicated in Clause 2.2.9.3.

Contents of C₃S, C₂S, C₄AF, and C₁₁A₇f, expressed as percentage by mass, shall be stated in the ETA.

2.2.10 Porosity

2.2.10.1 Introduction

Total porosity and porosimetric distribution of Fluoraluminate rapid setting cement shall be determined on mortar specimens.

Total porosity and the porosimetric distribution shall be compared to that of mortars based on Portland cement CEM I or CEM II, strength class 42.5 or higher according to EN 197-1. Total porosity and porosimetric distribution shall be expressed as the results of mercury porosimetry.

Mercury porosimetry allows for determining porosity of cementitious pastes, mortars or concretes. The available procedures are able to analyse pores with diameters from 10⁻⁹ up to 10⁻³ m. The test shall be performed by recording the mercury volume that penetrates into the porous structure as a function of pressure. Total porosity and porosity distribution shall be determined with mercury at increasing controlled pressure up to 1 000 MPa while measuring the amount of mercury introduced. The results shall be analysed by means of a theoretical model, see Clause 2.2.10.3.

2.2.10.2 Preparation of specimen

The microstructure of hardened mortar depends on its composition in terms of cement, admixtures, and water content and it is also a function of curing time and temperature. If the above-mentioned parameters are constant, humidity of the hardened cement paste further is an important parameter affecting the microstructural properties of cementitious pastes. For this reason, composition and curing of the cement paste, specimen preparation including specimen conditioning can considerably affect the results of porosimetric measures and are specified below.

Mortar prisms shall be made with Fluoraluminate rapid setting cement and with Portland cement CEM I or CEM II according to Clause 2.2.1. The following deviations to Clause 2.2.1 shall apply:

- Cement with an amount of (660 ± 5) kg/m³ for mortar prisms:
 - Mortar prism with fluoraluminate rapid setting cement shall be made and
 - Mortar prism with Portland cement CEM I or CEM II, strength class 42.5 or higher according to EN 197-1 shall be made.

- Sand
- Water

Water to cement ratio shall be equal to 0.55.

Curing

The mortar prisms shall be cured under water for 28 days at the temperature of (20 \pm 2) °C.

Extraction of specimen

Extraction of specimen from hardened material shall be performed in a way to avoid local overheating and mechanical shocks. An example for specimen extraction is:

- After curing, the mortar prism shall be removed from water.
- A water-cooled diamond saw shall be employed for cutting to obtain a specimen with dimensions of about 10 mm and thickness of 2 mm from the prism middle.

Conditioning of specimen

The important extraction of free water from capillary pores shall be carried out gradually and with particular care, in order to avoid the collapse of the existing pores and the formation of micro-cracks.

NOTE See [4], [5], [6], [7], and [8] for further information on conditioning of specimens.

In particular, one of the following techniques shall be applied:

- Drying under vacuum by using a humidity trap at low temperature, 77 °C⁷
- Drying under vacuum at high temperature 70 °C-105 °C
- Preconditioning at laboratory temperature with low relative humidity, at about 11 %, and then drying under vacuum at 100 °C

Results obtained with these techniques have been shown to be equivalent.

2.2.10.3 Measurement and data analysis

A porosimeter according to ISO 15901-1 shall be employed. Porosity measurement shall be performed according to ISO 15901-1 at high pressure operation mode.

The measured volume of mercury introduced into the specimen at controlled and measured pressure gives the results of mercury porosimetry. The Washburn relation, based on the hypothesis of cylindrical and straight pores gives, in the case of mercury, Equation 2.2.10.3.1.

$$r = \frac{750}{p}$$

Where

r.....pore radius

p..... MPaapplied absolute pressure

2.2.10.4 Expression of results

The test results shall be expressed as stated below.

- Total porosityValue of detected porosity expressed as volumetric percentage and rounded to the nearest 0.1 %.
- Porosimetric distributionDistribution of the porosity as a function of the pore radius. For each pore radius shall be given the fraction of the total porosity that has been detected in correspondence of that specific value of pore radius.

Total porosity in % for Fluoraluminate rapid setting cement and for Portland cement shall be stated in the ETA.

Equation 2.2.10.3.1

⁷ This temperature corresponds to the temperature of solid CO₂.

2.2.11 Effect of high temperature on mortar hardened under standard conditions

The effect of high temperature of Fluoraluminate rapid setting cement shall be determined as compressive strength of mortar with Fluoraluminate rapid setting cement, hardened under standard conditions, see Clause 2.2.2, and then subjected to high temperatures. The results shall be compared with mortar according to Clause 2.2.2, cured at 20 °C for a corresponding period.

9 series of mortar prisms according to Clause 2.2.2 shall be prepared. The specimens shall be demoulded after 1 day, and then immersed in water at 20 °C \pm 2 °C during further 27 days.

After 28 days, 3 series of mortar prisms shall be immersed in water at 20 °C \pm 2 °C, 3 series of mortar prisms in water at 40 °C \pm 2 °C, and 3 series of mortar prisms in water at 80 °C \pm 2 °C.

After 3, 7, and 28 days one series of each immersion temperature shall be removed from water and tested for compressive strength according to EN 196-1. Compressive strengths determined on specimens stored at 40 °C and 80 °C shall be compared with those of specimens stored at 20 °C.

After 3, 7, and 28 days the differences of mean compressive strength at high temperatures to mean compressive strength at standard temperature, 20 °C, shall be expressed in percent, relative to mean compressive strength at standard temperature. Separately for temperature of 40 °C and for temperature of 80 °C, the maximum differences in percent shall be stated in the ETA.

2.2.12 Effect of high temperature on mortar at early age

Effect of high temperature of Fluoraluminate rapid setting cement shall be determined as compressive strength of mortar with Fluoraluminate rapid setting cement, preliminary hardened for 3 hours under standard conditions, see Clause 2.2.1, and then subjected to high temperatures. The results shall be compared with mortar according to Clause 2.2.1, cured at 20 °C for a corresponding period.

3 series of mortar prisms according to Clause 2.2.1 shall be prepared. The specimens shall be demoulded after 3 hours.

1 series of mortar prisms immersed in water at 20 °C \pm 2 °C, 1 series of mortar prisms in water at 40 °C \pm 2 °C, and 1 series of mortar prisms in water at 80 °C \pm 2 °C.

After 3 days, the series of each immersion temperature shall be removed from water and tested for compressive strength according to EN 196-1. Compressive strengths determined on specimens stored at 40 °C and 80 °C shall be compared with those of specimens stored at 20 °C.

The differences of mean compressive strength at high temperatures to mean compressive strength at standard temperature, 20 °C, shall be expressed in percent, relative to mean compressive strength at standard temperature. Separately for temperature of 40 °C and for temperature of 80 °C, the differences in percent shall be stated in the ETA.

2.2.13 Setting time of blend with CEM I cement

Fluoraluminate rapid setting cement and CEM I cement according to EN 197-1 shall be blended. The blend comprises:

Fluoraluminate rapid setting cement......25 % by mass

CEM I cement according to EN 197-175 % by mass

CEM I cements with following parameters shall be selected:

- CEM I 42.5 R-SR 0, determined according to Clause 2.2.9.3, evaluated with Rietveld quantification
- CEM I 42.5 R with Na_{equ} \leq 0.6 %, determined according to EN 196-2
- CEM I 42.5 R with Na_{equ} > 1 %, determined according to EN 196-2
- CEM I 42.5 R with C₃A content > 8 %, determined according to Clause 2.2.9.3, evaluated with Rietveld quantification
- CEM I 52.5 R with C₃A content > 8 %, determined according to Clause 2.2.9.3, evaluated with Rietveld quantification

Each CEM I cement shall be blended with Fluoraluminate rapid setting cement in a mixer to obtain a homogeneous blend. At least 2 kg of each blend shall be made.

The blends shall be tested for:

- Initial setting time according to EN 196-3
- Final setting time according to EN 196-3

Preparation of paste for testing shall be according to Clause 2.2.3. 0.2 % carboxylic acid, e.g., tartaric acid or citric acid, shall be added to the water for mixing. 0.2 % is related to the total mass of the blend of cements.

Initial setting times in min and final setting times in min of the blends shall be stated in the ETA.

2.2.14 Resistance to chloride penetration

Resistance to chloride penetration of Fluoraluminate rapid setting cement shall be determined in accordance with Annex C, Clause C.1, Non-steady-state chloride migration coefficient – D_{nssm} .

Separately for the Fluoraluminate rapid setting cement and reference concrete, the arithmetic mean of chloride coefficient D_{nssm} after 35 days and the arithmetic mean of chloride coefficient D_{nssm} after 97 days shall be stated in the ETA. In the ETA the quantity in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – and the type of retardant shall be specified once an addition to concrete has been made or it shall be stated, no such addition was made.

2.2.15 Carbonation

Carbonation of Fluoraluminate rapid setting cement shall be determined in accordance with EN 12390-10, procedure when using climate controlled chamber. Preparation of specimens, determination of compressive strength and calculation of rate of carbonation shall be according to Annex D, Clause D.1, Carbonation resistance – C_{dcr} . Mean carbonation depths after 140 days and mean rate of carbonation shall be calculated and included in Figure G.1, Figure G.2, Figure G.3, and Figure G.4:

- Mean carbonation depth shall be plotted in Figure G.2 and Figure G.4. Compressive strength shall be taken as determined according to Annex D, Clause D.1.
- Mean rate of carbonation shall be plotted in Figure G.1 and Figure G.3. Compressive strength shall be taken as determined according to Annex D, Clause D.1.

In the ETA, separately for curing time of 7 days and for curing time of 28 days, shall be given:

- Mean carbonation depths in mm after 140 days
- Mean rates of carbonation in mm \cdot d^{-0.5} after 140 days
- Figure G.1, Figure G.2, Figure G.3, and Figure G.4 with plotted mean carbonation depths and mean rates of carbonation
- Quantity in percentage by mass related to the mass of Fluoraluminate rapid setting cement and the type of retardant, once an addition to concrete has been made or it shall be stated, no such addition was made

2.2.16 Freeze-thaw resistance without de-icing agent

Freeze-thaw resistance without de-icing agent of Fluoraluminate rapid setting cement shall be determined in accordance with CEN/TS 12390-9, CF-Procedure – FT_{CF}, see Annex E, Clause E.1.

Arithmetic mean of cumulative mass in g/m^2 of scaled material after 28 freeze-thaw cycles and arithmetic mean relative dynamic modulus of elasticity after 28 freeze-thaw cycles shall be stated in the ETA. In the ETA, the quantity in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – and the type of retardant shall be specified once an addition to concrete has been made or it shall be stated, no such addition was made.

2.2.17 Freeze-thaw resistance with de-icing agent

Freeze-thaw resistance with de-icing agent of Fluoraluminate rapid setting cement shall be determined in accordance with CEN/TS 12390-9, Freeze-thaw and de-icing resistance, CDF-Procedure – FTS_{CDF}, see further information in Annex F, Clause F.1.

Arithmetic mean of cumulative mass in g/m^2 of scaled material after 28 freeze-thaw cycles and arithmetic mean of relative dynamic modulus of elasticity after 28 freeze-thaw cycles shall be stated in the ETA. In the ETA, the quantity in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – and the type of retardant shall be specified once an addition to concrete has been made or it shall be stated, no such addition was made.

2.2.18 Water-soluble hexavalent chromium content

Water-soluble hexavalent chromium content of Fluoraluminate rapid setting cement shall be determined according to EN 196-10. Water-soluble hexavalent chromium content⁸ shall be \leq 2 ppm or the exceptions of the Regulation of the European Parliament and of the Council (EC) Nº 1907/2006, as amended, apply.

Water-soluble hexavalent chromium content in ppm shall be stated in the ETA.

⁸ The required performance originates from Regulation of the European Parliament and of the Council (EC) № 1907/2006, as amended, in particular amendment by Commission Regulation (EC) № 552/2009 and by Commission Regulation (EU) № 126/2013.

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: Decision 97/555/EC as amended by 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
	een er plan		

Nº	Subject of control	Test or control method	Criteria	Minimum number of samples	Minimum frequency of control
	Factory productior testing of samples taken at the factor	n control (FP) y in accorda	C), including nce with a pr	escribed test	t plan
1	Early strength	2.2.1	1)	1 sample	2/week ²⁾ , 4/week ³⁾
2	Standard strength	2.2.2	1)	1 sample	2/week ²⁾ , 4/week ³⁾
3	Initial setting time	2.2.3	1)	1 sample	2/week ²⁾ , 4/week ³⁾
4	Soundness	2.2.4	≤ 10 mm	1 sample	1/week ²⁾ , 4/week ³⁾
5	Loss on ignition	2.2.5	≤ 5.0 %	1 sample	2/month ²⁾ , 1/week ³⁾
6	Sulphate content	2.2.6	1)	1 sample	2/week ²⁾ , 4/week ³⁾
7	Chloride content	2.2.7	≤ 0.10 %	1 sample	2/month ²⁾ , 1/week ³⁾
8	Clinker composition	2.2.9	1)	1 sample	1/month ²⁾ , 1/week ³⁾
9	Cement composition	4)	4)	1 sample	1/month ²⁾ , 1/week ³⁾

¹⁾ According to control plan of the Fluoraluminate rapid setting cement

²⁾ Routine situation, following the initial period according to EN 197-2

³⁾ Initial period according to EN 197-2

⁴⁾ The composition of the Fluoraluminate rapid setting cement shall be determined by an appropriate verification method, see EN 197-1, clause 9, table 6 footnote i.

The composition of the Fluoraluminate rapid setting cement is checked by the manufacturer on a spot sample, taken at the point of release of the cement. The cement composition shall meet the specification. The specification includes limits of the content of the main constituents as reference values to be met by the average, calculated from the spot samples taken in the control period. For single results, maximum deviations of -2% at the lower and +2% at the higher limits of the specification are observed. Suitable procedures during production and appropriate verification methods to ensure conformity to this requirement are applied and documented.

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 the Fluoraluminate rapid setting cement are laid down in Table 3.3.1.

Table 3.3.1	Control p	lan for the	notified body	v – Cornerstones
	001101 p		nounou sou	,

Nº	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 Fluoraluminate rapid setting 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, asse	essment, and evaluat	ion of factory	production of	control	
2	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	
Auc	Audit-testing of samples taken by the notified product certification body at the manufacturing plant or at the manufacturer's storage facilities					
3	Standard strength	2.2.2	1)	1 sample	6 per year 2)	
4	Early strength	2.2.1	1)	1 sample	6 per year 2)	
5	Initial setting time	2.2.3	1)	1 sample	6 per year 2)	
6	Soundness	2.2.4	≤ 10 mm	1 sample	6 per year 2)	
7	Loss on ignition	2.2.5	≤ 5.0 %	1 sample	6 per year 2)	

Nº	Subject of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
8	Sulphate content	2.2.6	1)	1 sample	6 per year 2)
9	Chloride content	2.2.7	≤ 0.10 %	1 sample	6 per year 2)
10	Clinker composition	2.2.9	1)	1 sample	6 per year 2)
11	Effect of high temperature	2.2.11 2.2.12	1)	1 sample	3 per year 2)
12	Cement composition	3)	3)	1 sample	1 per year 2)
13	Water-soluble hexavalent chromium content	2.2.18	1)	1 sample	1 per year 2)

¹⁾ According to control plan of the Fluoraluminate rapid setting cement

²⁾ In case Fluoraluminate rapid setting cement is continuously dispatched. In case Fluoraluminate rapid setting cement is not continuously dispatched, minimum frequency of sampling is reasonably adapted.

³⁾ According to Table 3.2.1, note ⁴⁾

4 **REFERENCE DOCUMENTS**

EAD 150008-00-0301:05-2017	Rapid setting cement
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 196-3:2016	Methods of testing cement – Part 3: Determination of setting times and soundness
EN 196-10:2016	Methods of testing cement – Part 10: Determination of the water-soluble chromium (VI) content of cement
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 480-11:2005	Admixtures for concrete, mortar and grout – Test methods – Part 11: Determination of air void characteristics in hardened concrete
EN 1015-3:1999 + A1:2004 + A2:2006	Methods of test for mortar for masonry – Part 3: Determination of consistence of fresh mortar (by flow table)
EN 12620:2002 + A1:2008	Aggregates for concrete
EN 12390-1:2021	Testing hardened concrete – Part 1: Shape, dimensions and other requirements for specimens and moulds
EN 12390-2:2019	Testing hardened concrete – Part 2: Making and curing specimens for strength tests
EN 12390-3:2019	Testing hardened concrete - Part 3: Compressive strength of test specimens
EN 12390-10:2018	Testing hardened concrete – Part 10: Determination of the carbonation resistance of concrete at atmospheric levels of carbon dioxide
EN 12617-4:2002	Products and systems for the protection and repair of concrete structures – Test methods – Part 4: Determination of shrinkage and expansion
ISO 15901-1:2016	Evaluation of pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption – Part 1: Mercury porosimetry
CEN/TS 12390-9:2016	Testing hardened concrete – Part 9: Freeze-thaw resistance with de-icing salts – Scaling
CEN/TR 15177:2006	Testing the freeze-thaw resistance of concrete – Internal structural damage

Further information and background for assessment methods are given in the following documents.

- [1] Uchikawa H. and Kohno K., Ultra-rapid hardening cement (Jet cement), in Swamy, New concrete materials, Surrey University Press, 1983
- [2] Paul E. Stutzman, Guide for X-Ray Powder Diffraction Analysis of Portland Cement and Clinker, NISTIR 5755, 1996
- [3] Klug, Harold Philip, Alexander, Leroy E., X-ray diffraction procedures for polycrystalline and amorphous materials, 1974, Wiley
- [4] R.F. Feldman, Density and porosity studies of hydrated Portland cement, Cement Technology, 1972, 3, 1, page 5

- [5] L.J. Parrott, Effect of drying history upon the exchange of pore water with methanol and upon subsequent methanol sorption behaviour in hydrated alite paste, Cement and Concrete Research, 1981, 11, 5, page 651
- [6] L.J. Parrott, Thermogravimetric and sorption studies of methanol exchange in an alite paste, Cement and Concrete Research, 1983, 13, 1, page 18
- [7] Robert L. Day, Bryan K. Marsh, Measurement of porosity in blended cement pastes, Cement and Concrete Research, 1988, 18, 1, page 63
- [8] R.F. Feldman, J.J. Beaudoin, Pretreatment of hardened hydrated cement pastes for mercury intrusion measurements, Cement and Concrete Research, 1991, 21, 2–3, page 297

ANNEX A CONCRETE COMPOSITIONS FOR TESTING

Concrete	Composition	Aggregates size distribution, EN 12620											
Concrete I	Fluoraluminate rapid setting cement $320 \pm 1 \text{ kg/m}^3$ Aggregates $\pm 1 \text{ kg/m}^3$ W/C = 0.5 (or others)	Size in mm 0.25 0.5 1 2 4 8 16 Passing in % by mass 6 14 22 32 46 68 100											
Concrete II	Fluoraluminate rapid setting cement 450 ± 2 g Aggregates 1350 ± 5 g W = 225 ± 1 g (or others)	Size in mm 0.25 0.5 1 2 4 8 Passing in % by mass 8 21.5 36 46.5 67.5 100											
Concrete IIIa	Fluoraluminate rapid setting cement 350 \pm 2 kg/m ³ Aggregates \pm 5 kg/m ³ W/C = 0.5 (or others)	Size in mm 0.125 0.25 0.5 1 2 4 8 16 32											
Concrete IIIb	Cement CEM I 52.5R $350 \pm 2 \text{ kg/m}^3$ Aggregates $\pm 5 \text{ kg/m}^3$ W/C = 0.5 (or others)	Passing in % by 5 9 14 20 30 43 62 89 100 mass											
Concrete IV	Fluoraluminate rapid setting cement $300 \pm 2 \text{ kg/m}^3$ Aggregates $\pm 5 \text{ kg/m}^3$ W/C = 0.6 (or others)	Size in mm 0.125 0.25 0.5 1 2 4 8 16 32 Passing in % by mass 1.5 5 23 35 45 56 70 85 100											
Concrete V	Fluoraluminate rapid setting cement $320 \pm 2 \text{ kg/m}^3$ Aggregates $\pm 5 \text{ kg/m}^3$ W/C = 0.5 (or others)												
Concrete VI	Fluoraluminate rapid setting cement $320 \pm 2 \text{ kg/m}^3$ Aggregates $\pm 5 \text{ kg/m}^3$ Concrete with air entraining agent. The air content of the fresh concrete shall be 4.5 ± 0.5 Vol%. W/C = 0.5 (or others)	Size in mm 0.25 0.5 1 2 4 8 16 Passing in % by mass 6 14 22 32 46 68 100											

Table A.1 Concrete composition for testing

ANNEX B TESTING OF EXPANSION

B.1 Equipment and test solution

B.1.1 Container

The container for immersion in test solution has a capacity to contain the specimens under the conditions of Clause B.2.2. The container shall be fitted with a light-proof lid and made in a material that does not react with its content.

B.1.2 Test solution

The test solution shall be saturated calcium hydroxide, Ca(OH)₂, solution at 20 °C.

B.2 Preparation of specimens

B.2.1 Preparation of specimens

The specimens, 4 prisms made of mortar according to Clause 2.2.1, with dimensions of $25 \text{ mm} \times 25 \text{ mm} \times 25 \text{ mm} \times 25 \text{ mm} \times 25 \text{ mm} \times 160 \text{ mm}$, shall be equipped with stainless steel studs at both ends for comparator measurement. Both lengths are considered to achieve equivalent results.

B.2.2 Curing

The moulded specimens shall be placed in the moist cabinet at a temperature of (20 ± 0.5) °C for 23 hours. Thereafter, the specimens shall be removed from the moulds and immersed in water of 20 °C for 1 hour.

B.3 Test procedure

B.3.1 Measurements and immersion

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

After 1 hour immersed in water, the specimens shall be removed for initial measurement, performed according to EN 12617-4, clause 6.3, L(0).

After initial measurement the specimens shall be placed in a container with test solution, 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,
- On supports, at least 2 mm clear from the container bottom, and
- A water head of at least 5 mm above the specimens.

The container shall be sealed with a lid and kept at (20 ± 2) °C.

Taking readings of comparator measurement according to EN 12617-4, clause 6.5.2, of the specimens, L(t), immersed in test solution.

- After (48 \pm 2) hours, i.e., 2 days immersion
- After (168 \pm 2) hours, i.e., 1 week immersion
- After (336 \pm 2) hours, i.e., 2 weeks immersion

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

Where

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

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

B.4 Test results

The readings of comparator measurements on the specimens shall be corrected with readings on the calibration rod according to Equation B.4.1 and Equation B.4.2.

$$L_{i, corr}(0) = L_i(0) - L_{Cr}(0)$$
Equation B.4.1

$$L_{i, corr}(t) = L_i(t) - L_{Cr}(t)$$

The corrected readings at time t shall be subtracted from the corrected initial readings, i.e., measurements prior to commencing the immersion test, to obtain the change in length of the specimens, see Equation B.4.3.

$$\Delta L_i(t) = L_i, corr(t) - L_i, corr(0)$$

This change in length shall be divided by the gauge length of the specimen to obtain the expansion of the specimen according to Equation B.4.4.

$$\epsilon_{i}(t) = \frac{\Delta L_{i}(t)}{L_{g, i}} \cdot 1000$$
 Equation B.4.4

The expansion of immersion in test solution shall be calculated by Equation B.4.5 as the arithmetic mean of four specimens immersed in test solution.

$$\varepsilon(t) = \frac{1}{4} \cdot \sum_{i=1}^{4} \varepsilon_i(t)$$
 Equation B.4.5

Where

i	Specimen number i
İts	Specimen number i, immersed in test 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
ΔLi(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
ε _i (t) mm/m	Expansion of specimen i at time t
ε(t) mm/m	Mean expansion of specimens immersed in test solution

•

Equation B.4.2

Equation B.4.3

ANNEX C RESISTANCE TO CHLORIDE PENETRATION

C.1 Non-steady-state chloride migration coefficient – D_{nssm}

C.1.1 General

The non-steady-state chloride migration coefficient determined by the method is a measure of the resistance of the tested material to chloride penetration. This non-steady-state chloride 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.

The resistance to chloride penetration of concrete with the Fluoraluminate rapid setting cement and with Portland cement CEM I according to EN 197-1 as reference shall be determined.

C.1.2 Principle

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

C.1.3 Reagents

Distilled or deionised water, Calcium hydroxide Ca(OH)₂, technical quality, Sodium chloride NaCl, chemical quality, Sodium hydroxide NaOH, chemical quality and Silver nitrate AgNO₃, chemical quality.

C.1.4 Apparatus

- Water-cooled diamond saw.
- Migration set-up, one design (see Figure C.1) includes the following parts:
 - Silicone rubber sleeve, inner/outer diameter 100 mm/115 mm, about 150 mm long
 - Clamp, diameter range (105–115) mm, 20 mm wide, stainless steel, see Figure C.4
 - Catholyte reservoir, plastic box, 370 mm × 270 mm × 280 mm (length × width × height)
 - Plastic support, see Figure C.2
 - Cathode, stainless steel plate, see Figure C.2, about 0.5 mm thick
 - Anode, stainless steel mesh or plate with holes, see Figure C.3, 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 °C to 25 °C.

- Power supply, capable of supplying (0–60) V DC regulated voltage with an accuracy of \pm 0.1 V
- Ammeter, capable of displaying current to \pm 1 mA
- Thermometer or thermocouple with readout device capable of reading to \pm 1 °C
- Any suitable device for splitting the specimen
- Spray bottle
- Slide calliper with a precision of ± 0.1 mm
- Ruler with a minimum scale of 1 mm

C.1.5 Preparation of the test specimen

6 cylinders from each **concrete I**, see Annex A, Table A.1, with a diameter of 100 mm and a length of 200 mm (EN 12390-1/4.3.1) shall be made in accordance to EN 12390-2, see Table C.1.5.1.

Concrete age at time to start migration test	Number of cylinders for migration tests of concrete with Fluoraluminate rapid setting cement	Number of for cylinders for migration tests of concrete with CEM I cement				
days	—	—				
35	3	3				
97	3	3				

Table C.1.5.1 Number of specimens for migration tests

Due to the very early setting, the addition of a retarding admixture makes the preparation of the cylinders easier. When used, the nature and the dosage in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – of retarding admixture shall be given in the ETA.

The cylinders shall be stored for 24 hours in the mould at climate (20 °C/95 % relative humidity). After demoulding, the cylinders shall be stored in water at (20 \pm 5) °C until cutting. At an age of 28 days and 90 days, 3 cylinders of each concrete shall be taken out of the water.

In the middle of each cylinder a (50 ± 2) mm thick slice shall be cut out as specimen for migration testing. Measure the thickness of each specimen with a slide calliper and read to 0.1 mm.

NOTE The term 'cut' here means to saw perpendicularly to the axis of a core or cylinder, using a watercooled diamond saw.

Until commencing the test procedure for chloride migration, the specimens shall be stored immersed in water at (20 \pm 5) °C. The non-steady-state chloride migration coefficient of concrete (D_{nssm}) with the Fluoraluminate rapid setting cement shall be compared to the non-steady-state chloride migration coefficient of the reference concrete at an age of 35 and 97 days.

C.1.6 Test procedure

C.1.6.1 Catholyte and anolyte

The catholyte solution shall be 10 % NaCl by mass in tap water (100 g NaCl in 900 g water, about 2 N) and the anolyte solution shall be 0.3 N NaOH in distilled or de-ionised water (approximately 12 g NaOH in 1 litre water). Store the solutions at a temperature of 20 $^{\circ}$ C–25 $^{\circ}$ C.

C.1.6.2 Temperature

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

C.1.6.3 Preparation of the test

- Fill the catholyte reservoir with about 12 litres of 10 % NaCl solution.
- Fit the rubber sleeve on the specimen as shown in Figure C.3 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 C.1.
- Fill the sleeve above the specimen with 300 ml anolyte solution, 0.3 N NaOH.
- Immerse the anode in the anolyte solution.
- Connect the cathode to the negative pole and the anode to the positive pole of the power supply.

C.1.6.4 Migration test

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

- Record the final current and temperature before terminating the test.

C.1.6.5 Measurement of chloride penetration depth

- Disassemble the specimen by following the reverse of the procedure in "Preparation of the test". 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 C.5, to obtain seven depths, see notes 1, 2 and 3. Measure the depth to an accuracy of 0.1 mm.
 - NOTE 1 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.
 - If there is a significant defect in the specimen which results in a penetration front much larger than NOTE 2 the average, ignore this front as indicative of the penetration depth, but note and report the condition.
 - NOTE 3 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 C.5).

C.1.7 Test results

Calculate the non-steady-state chloride migration coefficient with Equation C.1.7.1.

$$D_{nssm} = \frac{R \cdot T}{z \cdot F \cdot E} \cdot \frac{x_d - \alpha \cdot \sqrt{x_d}}{t}$$
Equation C.1.7.1

Where α shall be according to Equation C.1.7.2 and E shall be according to Equation C.1.7.3.

 $\alpha = \sqrt{\frac{\mathbf{R} \cdot \mathbf{T}}{\mathbf{z} \cdot \mathbf{F} \cdot \mathbf{E}}} \cdot \operatorname{erf}^{-1} \left(1 - \frac{2 \cdot \mathbf{c}_{d}}{\mathbf{c}_{0}}\right)$ Equation C.1.7.2

$$E = \frac{U-2}{L}$$
 Equation C.1.7.3

Where

D_{nssm}.....non-steady-state chloride migration coefficient, m²/s zabsolute value of ion valence, for chloride, z = 1 F.....Faraday constant, $F = 9.648 \cdot 10^4 \text{ J/(V} \cdot \text{mol})$ 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 Lthickness of the specimen, m x_d.....average value of the penetration depths, m ttest duration, seconds erf⁻¹inverse of error function

 c_dchloride concentration at which the colour changes, $c_d \approx 0.07$ N for OPC concrete c_0chloride concentration in the catholyte solution, $c_0 \approx 2$ N

Since Equation C.1.7.4 applies, the simplified Equation C.1.7.5 can be used.

$$erf^{-1}\left(1 - \frac{2 \cdot 0.07}{2}\right) = 1.28$$
 Equation C.1.7.4

$$D_{nssm} = \frac{0.0239 \cdot (273 + T) \cdot L}{(U - 2) \cdot t} \cdot \left(x_{d} - 0.0238 \cdot \sqrt{\frac{(273 + T) \cdot L \cdot x_{d}}{U - 2}} \right)$$
Equation C.1.7.5

Where

D_{nssm}.....non-steady-state migration coefficient, 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

Lthickness of the specimen, mm

x_d.....average value of the penetration depths, mm

ttest duration, hour

The non-steady-state chloride migration coefficient of concrete (D_{nssm}) with the Fluoraluminate rapid setting cement shall be compared to the non-steady-state chloride migration coefficient of the reference concrete at an age 35 and 97 days.

Table C.1 Test voltage and duration

Initial current L30V, with 30 V	Applied voltage U After adjustment	Possible new initial current l₀	Test duration t
mA	V	mA	hours
l ₀ < 5	60	l ₀ < 10	96
$5 \leq I_0 < 10$	60	$10 \leq l_0 < 20$	48
10 ≤ l₀ < 15	60	$20 \leq l_0 < 30$	24
15 ≤ I₀ < 20	50	$25 \le I_0 < 35$	24
$20 \leq l_0 < 30$	40	$25 \leq l_0 < 40$	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
$60 \le I_0 < 90$	25	$50 \leq I_0 < 75$	24
$90 \le I_0 < 120$	20	$60 \le I_0 < 80$	24
$120 \le I_0 < 180$	15	$60 \le I_0 < 90$	24
$180 \le I_0 < 360$	10	$60 \leq I_0 < 120$	24
$l_0 \geq 360$	10	$l_0 \geq 120$	6



Figure C.1 Testing for chloride migration



Figure C.2 Plastic support and cathode



Figure C.3 Specimen, assembled with rubber sleeve, clamps, and anode



Figure C.4 Stainless steel clamp



Dimensions in mm

Figure C.5 Measurement of chloride penetration

ANNEX D CARBONATION

D.1 Carbonation resistance – Cdcr

The carbonation depth of concrete made with Fluoraluminate rapid setting cement shall be measured according to EN 12390-10. The resistance of carbonation depth has to be tested on 3 prisms ($40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}$) with aggregates according to EN 12620.

The carbonation resistance shall be tested on **concrete II**, see Annex A, Table A.1. If the consistence of this new fresh concrete/mortar varies significantly with respect to the reference fresh concrete/mortar (EN 1015-3 or other methods) other ratio is allowed to obtain a similar consistence with the reference fresh concrete/mortar).

Due to the very early setting, the addition of a retarding admixture makes the preparation of the specimens easier. When used, the nature and the dosage in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – of retarding admixture shall be given in the ETA.

The specimens shall be prepared according to EN 196-1/4.4, except the respect for the gap of (3 ± 1) mm between the bowl and the blade that cannot be respected by the size grading of the sand.

After demoulding half of the specimens shall be stored immersed in water (20 \pm 2) °C until the age of 7 days and the other half until the age of 28 days.

Afterwards the specimens shall be stored in climate 20 °C, 65 % relative humidity and ambient CO₂ content (commonly 350 to 450 p.p.m.) (EN 12390-10, Annex A).

Measurements of carbonation depth shall be performed after 14, 28, 56, 98, and 140 days.

NOTE It is recommended to continue the tests on the same specimens after 1, 2, and 5 years in order to extend the obtained results after 140 days and improve the knowledge.

Furthermore, compressive strength shall be determined according to EN 196-1 for plotting in Figure G.1, Figure G.2, Figure G.3, and Figure G.4.

- On the set of specimens n°1, at the age of 35 days (after 7 days pre-storing in water and 28 days in climate 20 °C, 65 % relative humidity) and, optionally, at the age of 147 days (after 7 days pre-storing in water and 140 days in climate 20 °C, 65 % relative humidity),
- On the set of specimens n°2, at the age of 35 days (after 28 days pre-storing in water and 7 days in climate 20 °C, 65 % relative humidity) and, optionally, at the age of 168 days (after 28 days pre-storing in water and 140 days in climate 20 °C, 65 % relative humidity).

Carbonation depth and rate of carbonation of the concrete with Fluoraluminate rapid setting cement shall be plotted in Figure G.1, Figure G.2, Figure G.3, and Figure G.4 of Annex G, see Clause 2.2.15.

Rate of carbonation k_c shall be calculated by linear regression from Equation D.1.1.

$$d_k = d_{k,0} + k_c \cdot \sqrt{t_c}$$

Equation D.1.1

Carbonation depth and rate of carbonation of the concrete with Fluoraluminate rapid setting cement shall be plotted in Figure G.1, Figure G.2, Figure G.3, and Figure G.4 of Annex G, see Clause 2.2.15.

Where

 d_k Carbonation depth in mm

t_c Duration of carbonation in days

 k_{c}Rate of carbonation in $\frac{mm}{\sqrt{days}}$

 $d_{k,0}$Carbonation depth at time t = 0. This specific parameter which depends on the storage and will be lower at a later start of testing the carbonation.

ANNEX E FREEZE-THAW RESISTANCE WITHOUT DE-ICING AGENT

E.1 Freeze-thaw resistance (CF-Procedure) – FTCF

The freeze-thaw resistance of concrete with the Fluoraluminate rapid setting cement shall be determined according to CEN/TS 12390-9/7 ("CF-Procedure"). The internal structural damage shall be determined according to CEN/TR 15177/9.

The freeze-thaw resistance ("CF-Procedure") shall be tested on 3 specimens of **concrete V**, see Annex A, Table A.1. If the consistence of this new fresh concrete/mortar varies significantly with respect to the reference fresh concrete (EN 1015-3 or other methods) other ratio is allowed to obtain a similar consistence with the reference fresh concrete.

Due to the very early setting, the addition of a retarding admixture makes the preparation of the specimens easier. When used, the nature and the dosage in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – of retarding admixture shall be given in the ETA.

The relative dynamic modulus of elasticity (RDM) and scaling shall be measured after 0, 4, 10, 16, 22, and 28 freeze-thaw cycles. The scaling and the relative dynamic modulus of elasticity (RDM) after 28 freeze-thaw cycles (CF-Procedure) shall be given in the ETA.

ANNEX F FREEZE-THAW AND DE-ICING SALT RESISTANCE

F.1 Freeze-thaw and de-icing resistance (CDF-Procedure) – FTS_{CDF}

The freeze-thaw and de-icing salt resistance of concrete with the Fluoraluminate rapid setting cement shall be determined according to CEN/TS 12390-9/7 ("CDF-Procedure"). Furthermore, the internal structural damage shall be determined according to CEN/TR 15177/9.

The freeze-thaw resistance with de-icing salt ("CDF-Procedure") shall be tested on 3 specimens of **concrete VI**, see Annex A, Table A.1. If the consistence of this new fresh concrete/mortar varies significantly with respect to the reference fresh concrete/mortar (EN 1015-3 or other methods) other ratio is allowed to obtain a similar consistence with the reference fresh concrete/mortar.

Due to the very early setting, the addition of a retarding admixture makes the preparation of the specimens easier. When used, the nature and the dosage in percentage by mass – related to the mass of Fluoraluminate rapid setting cement – of retarding admixture shall be given in the ETA.

The relative dynamic modulus of elasticity (RDM) and scaling shall be measured after 0, 4, 6, 14, and 28 freeze-thaw cycles. The scaling in g/m^2 after 28 freeze-thaw cycles (CDF-Procedure) shall be given in the ETA.

ANNEX G EVALUATION OF THE CARBONATION RESISTANCE - CDCR

Table G.1 Carbonation on concrete, W/C = 0.50 and pre-storage 7 days

Carbonation test on concrete (W/C = 0.50) - 7 days pre-storage

		fc in MPa				Carbonation depth in mm								Rate of carbonation 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	f _{C, P} -0.5 N ^{-0.5} - I	VC,140d	VC,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	C 80 %	5; LL 2	0 %)												
	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	S-V) (C	<u> 65 %</u>	s; S 15	%; V 2	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
		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
CEMI									·						
	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

Table G.2 Carbonation on concrete, W/C = 0.50 and pre-storage 28 days

								``		,		•	0			
		f _c in MPa			Carbonation depth in mm								° in mm	Rate of carbonation 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	f _{C, P} ^{-0.!} N ^{-0.5} .	VC,140d	VC,5a	
CEM II/B-M (S,	V-LL)								u				•			
	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	0 %)													
	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 %)		1	1								
	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 9	%)				1	1								
	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	
CEMI																
	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	

Carbonation test on concrete (W/C = 0.50) – **28** days pre-storage



NOTE The diagrams are only appropriate for a specific concrete composition. Other compositions are not applicable.