



## EUROPEAN ASSESSMENT DOCUMENT

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# PROCESSED BOTTOM ASH FROM MUNICIPAL SOLID WASTE INCINERATORS AS TYPE II ADDITION FOR PRODUCTION OF CONCRETE, MORTAR AND GROUT

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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) No 305/2011 as a basis for the preparation and issuing of European Technical Assessments (ETA).

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## 1 SCOPE OF THE EAD

### 1.1 Description of the construction product

Concrete addition obtained by physical and chemical treatment of municipal solid waste incinerators bottom ashes (MIBA) deriving from municipal solid waste incinerators (waste from households as well as commercial, industrial and institutional waste, which because of its nature and composition is similar to waste from households, excluding hazardous waste).

The treatment process is characterized by wet micronization of the mineral particles, by the high level reached of separation of metal scraps initially present in MIBA and, particularly, by the fact that the final product is metallic aluminium free and therefore does no longer induce hydrogen formation<sup>1</sup>.

The final product (which, after wet micronization in form of slurry, undergoes a partial dehydration phase) is a humid aggregate with sand appearance and a grain size about 0 mm to 6 mm, constituted of elemental fine particles aggregated in clusters. The water present in the product should be considered as hydration water in concrete design.

Maximum grain size is kept below 0.15 mm by sieving of the final product.

The final product has high pozzolanic properties and can be used to produce concrete with improved properties, both in the fresh and hardened states.

The product is normally used in combination with a plasticizer and/or superplasticizer and, in particular conditions, depending on the other components of concrete, the mix design, etc., it may require an addition of a defoamer<sup>2</sup> in order to avoid the entrapment of excess of air.

The product is registered under REACH and its EC number is 939-997-0.

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

Concerning product packaging, transport, and storage, it is the responsibility of the manufacturer to undertake the appropriate measures and to advise his clients on the transport and storage, 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 Intended use(s) of the construction product

Its intended use is as type II addition for concrete, including cast-in-situ or prefabricated structural concrete conforming to European standard EN-206. It may also be used in mortars and grouts.

The granulate form, may require an additional mixing time to disperse the single particles.

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<sup>1</sup> Hydrogen formation may cause problems of expansion on fresh concrete, this is a well known phenomenon of MIBA.

<sup>2</sup> The product is normally added with a certain quantity of defoamer at the production site. However, in some concretes, depending mostly on superplasticizer and also on mix design and the other constituents (cement, aggregates, etc.), it is necessary to add additional quantities of defoamer.

## 2 ESSENTIAL CHARACTERISTICS AND RELEVANT ASSESSMENT METHODS AND CRITERIA

### 2.1 Essential characteristics of the product

Table 1 shows how the performance of the type II addition is assessed in relation to the essential characteristics.

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

No	Essential characteristic	Assessment method	Type of expression of product performance
<b>Basic Works Requirement 1: Mechanical resistance and stability</b>			
1	Composition	2.2.1	Level / Description
2	Activity index	2.2.2	Description
3	Fineness	2.2.3	Description
4	Soundness	2.2.4	Description
5	Moisture content	2.2.5	Description
6	Loss on ignition	2.2.6	Level
7	Particle density	2.2.7	Level
8	Initial setting time	2.2.8	Level
9	Compressive strength	2.2.9	Level
10	Depth of penetration of water under pressure	2.2.10	Level
11	Relative carbonation resistance		Level
12	Chloride diffusion resistance		Level
13	Sulphate resistance		Level

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

### 2.2.1 Composition

Chemical composition shall be assessed according to table 2 and shall be expressed as proportions by mass of dry product:

**Table 2:** Chemical composition tests.

Composition	Assessment method	Performance
SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub>	EN 196-2	Level
Total sulphates (SO <sub>3</sub> )	EN 196-2	≤ 3%, according to EN 450-1
Total chlorides (Cl <sup>-</sup> )	EN 196-2	Level
Water soluble chlorides (Cl <sup>-</sup> )	EN 1744-1	Level
Total phosphates (P <sub>2</sub> O <sub>5</sub> )	EN 196-2	≤ 5%, according to EN 450-1
Notes:		
<ul style="list-style-type: none"> <li>• Before testing, all test samples shall be dried following the procedure described in EN 450-1, clause 7.</li> </ul>		

### 2.2.2 Activity index

Tests according to EN 196-1, as stated in EN 450-1, chapter 5.3.2. A cement type CEM I with a strength class 42,5 R or higher and in which the fineness (Blaine), content of tricalcium aluminate and content of alkali (Na<sub>2</sub>O<sub>eq</sub>) are according to clause 5.3.2 of EN 450-1 is to be used.

The adjustments in preparation of samples of clauses 5.3.3 of EN 13263-1 shall be taken into account due to the high fineness of the product, as it is done for products with similar fineness, and due to the water content of the product:

- Water present in the product should be considered as hydration water.
- The assessed mortar (the one made of ash, reference cement, sand, etc.) should be prepared using a superplasticizer to obtain a consistence equivalent to the reference mortar (the one made of reference cement only, sand, etc.). The consistence is to be measured according to EN 1015-3.  
An equivalent consistence is achieved when the difference between the measured values of consistence of the assessed and reference mortars is lesser than 10 % of their mean value.
- A defoamer (\*) should be used when preparing the assessed mortar in order to obtain a content of air equivalent to the reference mortar. The content of air is to be measured according to EN 1015-7.  
An equivalent content of air is achieved when the difference between the measured values of content of air of the assessed and reference mortars is lesser than 10 % of their mean value.
- Actions to ensure that the granulate product is completely dispersed during mixing of the mortar should be taken.  
(\* ) The product can contain a certain quantity of defoamer that is generally sufficient for concrete, but not for mortars that have different fines content; therefore mortars should be prepared with defoamer to be preferably previously dispersed into water.

The activity index shall meet the criteria of clause 5.3.2 of EN 450-1 regarding the activity index at 28 days and 90 days.

The activity index of the product shall be expressed in the ETA by means of a description.

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

### 2.2.3 Fineness

Fineness shall be determined according to the following two methods:

1. Fineness by wet sieving shall be determined according to EN 451-2 (according to EN 450-1).

Fineness shall be Class S ( $\leq 12\%$ , by mass), according to EN 450-1.

2. Fineness by laser diffraction shall be determined according to ISO 13320.

Because of the granulate/aggregate form of the product, it should be completely dispersed by mechanical or ultrasound agitation prior to testing.

The fineness of the product shall be expressed in the ETA by means of a description.

### 2.2.4 Soundness

Soundness in plastic period shall be determined according to ASTM C827.

The soundness of the product shall be expressed in the ETA by means of a description.

### 2.2.5 Moisture content

Test according to EN 15167-1, Annex A.

The moisture content of the product shall be expressed in the ETA by means of a description.

### 2.2.6 Loss on ignition

Test according to EN 1744-7.

The loss on ignition of the product shall be expressed in the ETA by means of a level.

### 2.2.7 Particle density

Tests according to EN 1097-7.

According to EN 450-1, particle density shall fall within  $\pm 200 \text{ kg/m}^3$  from the value to be stated in the ETA.

The particle density of the product shall be expressed in the ETA by means of a level.

### 2.2.8 Initial setting time

Tests according to EN 196-3.

According to EN 450-1, the initial setting time of a grout (mixture of water and cement) in which the 25 % of cement is replaced by ash, hereinafter called assessment grout, shall not be more than two times the initial setting time of a reference grout made of 100 % of cement. The reference grout shall fulfil the requirements for initial setting time of EN 197-1.

A cement type CEM I with a strength class 42,5 R or higher is to be used.

Because of the granulate/aggregate form of the product, it should be completely dispersed by mechanical or ultrasound agitation prior to testing.

The initial setting time of the product shall be expressed in the ETA by means of a level.

### 2.2.9 Compressive strength and k value

The reference and the assessed concretes are to be specified based on the following characteristics:

- Main characteristics of reference concrete:
  - Type of cement and resistance
  - Type of aggregates and admixtures and relative amount
  - Cement amount ( $c_r$ )
  - Water/cement ratio ( $w_0$ ), including aggregate moisture
- Main characteristics of assessment concrete:
  - Same type of cement as the reference concrete
  - Same type and relative amount of aggregates and admixtures
  - Cement amount for the assessment concrete ( $c_a$ )
  - The maximum addition/cement ratio ( $r_{a/c}$ )
  - Type II addition amount ( $a = r_{a/c} \cdot c_a$ )
  - Type II addition fineness
  - Water content ( $w_a$ ), including type II addition aggregate moisture content

**Table 3:** Concretes information for the assessment of k value.

Reference concrete	Assessment concrete
CEM XX – YY R (Type and resistance)	
Aggregates and admixtures (Type and amount)	
Cement amount: $c_r = \text{__ kg/m}^3$	Cement amount: $c_a = \text{__ kg/m}^3$ Type II addition amount: $a = r_{a/c} \cdot c_a = \text{__ kg/m}^3$ (maximum allowed acc. to manufacturer)
Water / cement ratio: $w_0 = \text{__}$	$w_a / (c_a + a) = \text{__}$

Recommendations indicated in chapter 2.3.4 of CEN TR 16639 shall be taken into account. If necessary, a defoamer can be also used to control porosity.

Compressive strength tests shall be carried out according to EN 12390-3 after 7, 28 and 90 days.

The number of specimens to be tested for each concrete combination shall be in accordance with EN 12390-3.

For each couple of reference and assessment concretes, tests shall be carried out with three water/cement ratios, as indicated in the following scheme:



**Table 4:** Testing scheme for the k value assessment.

<b>CEM XX – YY R (Type and resistance)</b>						
Sample age	7 days		28 days		90 days	
Concrete	Reference	Assessed	Reference	Assessed	Reference	Assessed
$\omega_1$	$\sigma_{r17}$	$\sigma_{a17}$	$\sigma_{r128}$	$\sigma_{a128}$	$\sigma_{r190}$	$\sigma_{a190}$
$\omega_2$	$\sigma_{r27}$	$\sigma_{a27}$	$\sigma_{r228}$	$\sigma_{a228}$	$\sigma_{r290}$	$\sigma_{a290}$
$\omega_3$	$\sigma_{r37}$	$\sigma_{a37}$	$\sigma_{r328}$	$\sigma_{a328}$	$\sigma_{r390}$	$\sigma_{a390}$
Notes: water/cement ratios can be defined as follows: <ul style="list-style-type: none"> <li><math>\omega_1 = \omega_0 \cdot 0,70</math></li> <li><math>\omega_2 = \omega_0</math></li> <li><math>\omega_3 = \omega_0 \cdot 1,20</math></li> </ul> Other different ratios could be used if more adequate.						

This scheme shall be repeated for each cement type to be included in the ETA. This scheme is also only valid for a defined type II addition fineness, and thus a change of fineness shall imply the repetition of the whole set of tests.

The k value shall be calculated according to the method indicated in chapter 2.3.2 of CEN/TR 16639. In this calculation, k will not be a single value but will be a parameter in functional dependence on the water/cement ratio of the reference concretes ( $\omega_0$ ). The minimum k value, within the water content limits used in tests ( $w_1$ ,  $w_2$ ,  $w_3$ ) for each concrete type and sample age shall be the results of the assessment.

The compressive strength and k value shall be expressed in the ETA by means of a level.

### 2.2.10 Equivalent durability

Durability assessment shall be based on the equivalent durability procedure described in CEN/TR 16563. Table 5 shows the durability performances to be assessed according to this procedure.

**Table 5:** Durability assessment test methods.

<b>Concretes durability performance</b>	<b>Assessment method</b>	<b>Exposure assessed classes</b>
Depth of penetration of water under pressure	EN 12390-8	---
Relative carbonation resistance	CEN/TS 12390-10	XC
Chloride diffusion resistance	EN 12390-11	XS, XD, XF2, XF4
Sulphate resistance	Described in Annex A	XA3

The reference and the assessed concretes are to be specified based on the following characteristics:

- Main characteristics of reference concrete (to which performance is equivalent):
  - Type, source and dosage of all constituents
  - Exposure class according to EN 206
  - Minimum compressive strength for the exposure class (\*)
  - Maximum w/c ratio (\*)
  - Minimum cement ( $c_r$ ) or cement + addition content (\*)

- Main characteristics of assessment concrete:
  - Same type and source of main constituents
  - The maximum addition/cement ratio ( $r_{a/c}$ )
  - Minimum k-value
  - Type II addition fineness
  - Maximum water content ( $w_a/(c_a + a)$ ), including type II addition aggregate moisture content
  
- (\*) Values for these characteristics are defined in EN 206 Annex F for each exposure class. Other values can be defined by the manufacturer, if considered appropriate.
 

Criteria for determination of the reference value, as defined in CEN/TR 16563 chapter 6 shall be taken into account.

**Table 6:** Concretes information for the assessment of durability.

Reference concrete	Assessment concrete
CEM XX – YY R (Type and resistance)	
Aggregates (Type and amount)	
Cement amount: $c_r \geq x \text{ kg/m}^3$ (x = minimum value defined in EN 206, table F.1, for the exposure class to be assessed)	Cement amount: $c_a = c_r / (1 + k \cdot r_{a/c}) = \_ \text{ kg/m}^3$ Type II addition amount: $a = r_{a/c} \cdot c_a = \_ \text{ kg/m}^3$ (maximum allowed acc. to manufacturer)
Water / cement ratio: $w_0 \leq y$ (y = maximum value defined in EN 206, table F.1, for the exposure class to be assessed)	$w_a / (c_a + a) = w_0 \cdot (c_a + k \cdot a) / (c_a + a) = \_$
Concrete compressive strength $f_{ck} \geq z$ (z = minimum value defined in EN 206, table F.1, for the exposure class to be assessed)	$f_{ck} \geq z$
Notes: $r_{a/c}$ is the maximum addition/cement ratio defined by the manufacturer. k value is indicated by the manufacturer according to its experience (not the value obtained in 2.2.9)	

For each couple of reference and assessment concretes, the following tests shall be carried out:

1. Compressive strength, according to EN 12390-3 after 7 and 28 days, as shown in table 7.
2. Determination of durability performance according to table 8.

**Table 7:** Compressive strength test scheme.

<b>CEM XX – YY R (Type and resistance) / Exposure class XX</b>				
Sample age	7 days		28 days	
Concrete	Reference	Assessed	Reference	Assessed
$\omega_1$	$\sigma_{r17}$	$\sigma_{a17}$	$\sigma_{r128}$	$\sigma_{a128}$
$\omega_2$	$\sigma_{r27}$	$\sigma_{a27}$	$\sigma_{r228}$	$\sigma_{a228}$
$\omega_3$	$\sigma_{r37}$	$\sigma_{a37}$	$\sigma_{r328}$	$\sigma_{a328}$

Notes:  
water/cement ratios can be defined as follows:

- $\omega_1 = \omega_0 - 0,02$
- $\omega_2 = \omega_0$
- $\omega_3 = \omega_0 + 0,02$

Other different ratios could be used if more adequate.

**Table 8:** Test scheme for each durability performance (see table 5).

<b>CEM XX – YY R (Type and resistance) / Exposure class XX</b>								
Sample age	t1 days		t2 days		...		tn days	
Concrete	Reference	Assessed	Reference	Assessed	R	A	Reference	Assessed
$\omega_1$	$\bar{\delta}_{r1t1}$	$\bar{\delta}_{a1t1}$	$\bar{\delta}_{r1t2}$	$\bar{\delta}_{a1t2}$	...	...	$\bar{\delta}_{r1n}$	$\bar{\delta}_{a1n}$
$\omega_2$	$\bar{\delta}_{r2t1}$	$\bar{\delta}_{a2t1}$	$\bar{\delta}_{r2t2}$	$\bar{\delta}_{a2t2}$	...	...	$\bar{\delta}_{r2n}$	$\bar{\delta}_{a2n}$
$\omega_3$	$\bar{\delta}_{r3t1}$	$\bar{\delta}_{a3t1}$	$\bar{\delta}_{r3t2}$	$\bar{\delta}_{a3t2}$	...	...	$\bar{\delta}_{r3n}$	$\bar{\delta}_{a3n}$

Notes:

- 1) water/cement ratios can be defined as follows:
  - $\omega_1 = \omega_0 - 0,02$
  - $\omega_2 = \omega_0$
  - $\omega_3 = \omega_0 + 0,02$
 Other different ratios could be used if more adequate.
- 2) Sample ages as defined in the assessment method standard (see table 5).

For each cement type and exposure class, results for reference and assessed concretes shall be determined.

The equivalent durability of the product for each cement type and exposure class shall be expressed in the ETA by means of a level.

### 3 ASSESSMENT AND VERIFICATION OF CONSTANCY OF PERFORMANCE

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

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

The system is: 1+.

#### 3.2 Tasks of the manufacturer

The cornerstones of the actions to be undertaken by the manufacturer of the product in the procedure of assessment and verification of constancy of performance are laid down in Table 9.

**Table 9:** Control plan for the manufacturer; cornerstones.

No	Subject/type of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
<b>Factory production control (FPC) including testing of samples taken at the factory in accordance with a prescribed test plan</b>					
1	Composition	2.2.1	Table 2	1	2 / month <sup>1)</sup> 1 / month <sup>2)</sup>
2	Activity index	2.2.2	≥ 75% for 28 days samples ≥ 85% for 90 days samples	1	4 / month <sup>1)</sup> 2 / month <sup>2)</sup>
3	Fineness	2.2.3	Description	1	2 / day <sup>1)</sup> 1 / day <sup>2)</sup>
4	Soundness	2.2.4	Description	1	2 / week <sup>1)</sup> 1 / week <sup>2)</sup>
5	Moisture content	2.2.5	Description	1	1 / day
6	Loss on ignition	2.2.6	Level	1	2 / day <sup>1)</sup> 1 / day <sup>2)</sup>
7	Particle density	2.2.7	2.2.7	1	2 / month <sup>1)</sup> 1 / month <sup>2)</sup>
8	Initial setting time	2.2.8	2.2.8	1	2 / month <sup>1)</sup> 1 / month <sup>2)</sup>
9	Compressive strength and k value	2.2.9	Level	1	1 / year <sup>3)</sup>
10	Depth of penetration of water under pressure	2.2.10	Level	1	1 / year <sup>3)</sup>
11	Relative carbonation resistance		Level	1	1 / 3 years <sup>3)</sup>
12	Chloride diffusion resistance		Level	1	1 / 3 years <sup>3)</sup>
13	Sulphate resistance		Level	1	1 / 3 years <sup>3)</sup>
<sup>1)</sup> Initial period (3 months) <sup>2)</sup> Routine situation <sup>3)</sup> For each type of cement included in the ETA					

### 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 type II addition are laid down in Table 10.

**Table 10:** Control plan for the notified body; cornerstones.

No	Subject/type of control	Test or control method	Criteria, if any	Minimum number of samples	Minimum frequency of control
<b>Initial inspection of the manufacturing plant and of factory production control</b>					
1	The notified product certification body shall verify the ability of the manufacturer for a continuous and orderly manufacturing of the product according to the manufacturer's Control Plan. In particular the following items shall be appropriately considered <ul style="list-style-type: none"> <li>• personnel and equipment</li> <li>• the suitability of the factory production control established by the manufacturer</li> <li>• full implementation of the prescribed test plan</li> </ul>				---
<b>Continuous surveillance, assessment and evaluation of factory production control</b>					
2	The notified product certification body shall verify that <ul style="list-style-type: none"> <li>• the manufacturing process</li> <li>• the system of factory production control</li> <li>• the implementation of the prescribed test plan</li> </ul> are maintained.				Once a year
<b>Audit-testing of samples taken by the notified product certification body at the manufacturing plant or at the manufacturer's storage facilities</b>					
1	Composition	2.2.1	Table 2	1	Once a year
2	Activity index	2.2.2	≥ 75% for 28 days samples ≥ 85% for 90 days samples	1	
3	Fineness	2.2.3	> 275 m <sup>2</sup> /kg	1	
4	Soundness	2.2.4	Description	1	
5	Moisture content	2.2.5	Description	1	
6	Loss on ignition	2.2.6	Level	1	
7	Particle density	2.2.7	2.2.7	1	
8	Initial setting time	2.2.8	2.2.8	1	
9	Compressive strength and k value	2.2.9	Level	1	
10	Depth of penetration of water under pressure	2.2.10	Level	1	1 / 3 years
11	Relative carbonation resistance		Level	1	
12	Chloride diffusion resistance		Level	1	
13	Sulphate resistance		Level	1	

## 4 REFERENCE DOCUMENTS

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

EN 450-1	Fly ash for concrete. Part 1: Definition, specifications and conformity criteria.
EN 15167-1	Ground granulated blast furnace slag for use in concrete, mortar and grout. Part 1: Definitions, specifications and conformity criteria.
EN 13263-1	Silica fume for concrete. Part 1: Definitions, requirements and conformity criteria.
EN 206	Concrete. Specification, performance, production and conformity
EN 196-1	Methods of testing cement. Part 1: Determination of strength.
EN 196-2	Method of testing cement. Part 2: Chemical analysis of cement.
EN 196-3	Methods of testing cement. Part 3: Determination of setting times and soundness.
EN 196-6	Methods of testing cement. Part 6: Determination of fineness.
EN 1744-1	Tests for chemical properties of aggregates. Part 1: Chemical analysis.
EN 1744-7	Tests for chemical properties of aggregates. Part 7: Determination of loss of ignition of Municipal Incinerator Bottom Ash Aggregate (MIBA Aggregate).
EN 451-1	Method of testing fly ash. Part 1: Determination of free calcium oxide content.
EN 451-2	Method of testing fly ash. Part 2: determination of fineness by wet sieving.
EN 1015-7	Methods of test for mortar for masonry. Part 7: determination of air content of fresh mortar.
EN 1097-7	Tests for mechanical and physical properties of aggregates. Part 7: Determination of the particle density of filler - Pycnometer method.
EN 413-2	Masonry cement. Part 2: Test methods.
EN 12390-3	Testing hardened concrete. Part 3: Compressive strength of test specimens.
EN 12390-8	Testing hardened concrete. Part 8: Depth of penetration of water under pressure.
CEN/TS 12390-10	Testing hardened concrete. Part 10: Determination of the relative carbonation resistance of concrete.
EN 12390-11	Testing hardened concrete. Part 11: Determination of the chloride resistance of concrete, unidirectional diffusion.
CEN/TR 16639	Use of k-value concept, equivalent concrete performance concept and equivalent performance of combinations concept.
CEN/TR 16563	Principles of the equivalent durability procedure.
ISO 13320	Particle size analysis. Laser diffraction methods.
ASTM C827M - 10	Standard Test Method for Change in Height at Early Ages of Cylindrical Specimens of Cementitious Mixtures

## ANNEX A – SULPHATE RESISTANCE TEST METHODS

There are two methods for the determination of sulphates resistance:

1. Sulfate resistance (Flat prism method) -  $S_{FPM}$
2. Sulfate resistance (Square prism method) -  $S_{SPM}$

### A.1 Method 1: Sulfate resistance (Flat prism method) - SFPM

#### A.1.1 Making of test specimens

For each mortar to be tested 24 flat prisms with dimensions 10 mm x 40 mm x 160 mm shall be made in accordance with EN 196-1 and compacted on the vibrating table.

#### A.1.2 Storage of test specimens

Time (days)	Number of samples	Storage conditions
2	24	(20 ± 2)°C and > 95% RH (climate 20/95) Stored in the mould. After this period samples are demoulded.
14	24	In saturated Ca(OH) <sub>2</sub> solution at (20 ± 2)°C
Until test	6	In a 4,4 % Na <sub>2</sub> SO <sub>4</sub> solution at 20°C (sulfate storage at 20°C)
	6	In a 4,4 % Na <sub>2</sub> SO <sub>4</sub> solution at 5°C (sulfate storage at 5°C)
	6	In a saturated Ca(OH) <sub>2</sub> solution at 20°C (reference storage 20°C)
	6	In a saturated Ca(OH) <sub>2</sub> solution at 5°C (reference storage 5°C)

In all storages samples are stored on edge, standing on gratings with a ratio of volume of solution/solid matter from 3:1 to 5:1. The Na<sub>2</sub>SO<sub>4</sub> solution is to be replaced every 14 days with a new Na<sub>2</sub>SO<sub>4</sub> solution. Temperature shall be controlled at 5 °C and 20°C. The saturated Ca(OH)<sub>2</sub> solution is to be checked every 14 days for its saturation. If needed, it has to be concentrated.

#### A.1.3 Tests

After a period of storage of 0, 14, 28, 56, 90, and 180 days, the following tests are performed:

1. Length of samples
2. Dynamic modulus of elasticity
3. Change in mass

Photos of the specimens will be taken after every testing to illustrate the formations of the cracks.

#### A.1.4 Analysis

Elongation of the flat prisms is to be determined as mean value of the measured values from 3 specimens and the difference in elongation between the sulfate storage and the reference storage is to be reported.

The dynamic modulus of elasticity is to be determined as mean value from the measured values from 3 specimens and reported.

## **A.2 Method 2: Sulfate resistance (Square prism method) – SSPM**

This test method is adapted from the CUR – Civiltechnisch Centrum Uitvoering Research en Regelgeving – Recommendation 48 testing procedure.

### **A.2.1 Apparatus and solution**

#### **A.2.1.1 Containers**

Containers for storage of distilled water and sulfate solution must have a capacity of 1.5 and 2.5 litres and measure at least 180 mm x 80 mm. Each container must be capable of containing  $1.0 \pm 0.1$  litres of liquid, so that the depth of the liquid reaches at least 25 mm. All the containers must be fitted with light-proof lids and must be manufactured in a material that does not react with its content.

It is allowed to put specimens of different cement types in a single container provided that the chemical composition of the cement is equivalent. In this case  $1.0 \pm 0.1$  litres of liquid are used per three specimens.

#### **A.2.1.2 Sulphate Solution**

The sulphate solution must have a concentration of  $16 \pm 0.5$  g/l of  $\text{SO}_4$ , and is prepared by adding  $\text{Na}_2\text{SO}_4$  or  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  of analytical purity to distilled water, or to water of the same purity.

Note: The  $\text{SO}_4$  content of the  $\text{Na}_2\text{SO}_4$  can be measured before the solution is prepared, or the  $\text{SO}_4$  content of the solution can be measured and corrected, if necessary.

### **A.2.2 Manufacturing of test specimens**

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

### **A.2.3 Conditioning**

The specimens shall be stored according to clause 8.3 of EN 196-1. Immediately after demoulding the specimens shall be placed in 2 containers (2 x 3 specimens) containing 1 litre of distilled water each.

The specimens must be placed along each other with at least 5 mm space between them, at least 5 mm water above and at least 5 mm distance from the sides of the containers. The specimens must be placed on supports at least 2 mm clear from the bottom of the containers.

### **A.2.4 Testing procedure**

At the age of 28 days the length of each specimen shall be measured.

Before carrying out the measurements, the measuring apparatus must be calibrated using the reference bar. Note the result or adjust the measuring apparatus to the standard value.

Remove one specimen at a time and clean the measuring points with a damp cloth. Note the measured value  $L(0)$ .



After measuring each specimen, replace immediately 3 bars in the container with distilled water (set 1) while the 3 others are put into a new container containing 1 litre of sulfate solution (set 2) for the next storage period.

All the containers shall be stored with a sealed lid at  $(20 \pm 2)^\circ\text{C}$ .

The distilled water shall not be changed during the whole storage period but fill up the water level with additional water if necessary.

The sulfate solution shall be replaced every 28 days.

### **A.2.5 Test**

Measure the length of the prisms  $L(t)$  in the same way after 4, 8, 12, 16, 20, 26, 29, 40 and 52 weeks in the container.

Photos of the specimens will be taken after every testing to illustrate the formations of the cracks. Record any visible degradation of the specimens.

### **A.2.6 Analysis**

For each storage period ( $t$ ), measure the changes in the length of each specimen in relation to the length  $L(0)$ , as a percentage of the standard length of 160 mm, round to an accuracy of 0,005%. The percentage expansion of the prisms is to be calculated as mean value for the three specimens stored in distilled water and for the three specimens stored in the sulphate storage.

The difference between the average of the three specimens stored in distilled water and the three specimens stored in the sulphate solution is reported for each storage period.