

Institut luxembourgeois de la normalisation de l'accréditation, de la sécurité et qualité des produits et services

ILNAS-EN 13454-2:2019

# Binders for floor screeds based on calcium sulphate - Part 2: Test methods

Calciumsulfat-Binder für Estriche - Teil 2: Prüfverfahren

Liants à base de sulfate de calcium pour chapes - Partie 2 : Méthodes d'essai

#### **National Foreword**

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# EUROPEAN STANDARD ILNAS-EN 13454-2:2016 EN 13454-2

# NORME EUROPÉENNE

**EUROPÄISCHE NORM** 

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#### **English Version**

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Calciumsulfat-Binder für Estriche - Teil 2: Prüfverfahren

This European Standard was approved by CEN on 19 November 2018.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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# **European foreword**

This document (EN 13454-2:2019) has been prepared by Technical Committee CEN/TC 241 "Gypsum and gypsum based products", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2019, and conflicting national standards shall be withdrawn at the latest by July 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document will supersede EN 13454-2:2003+A1:2007.

The main differences between this document and EN 13454-2:2003+A1:2007 are:

- a) the title was adapted;
- b) the normative references were updated;
- c) the terms 'flow diameter' and 'spread' were interchanged;
- d) the clause on sampling for factory made mixtures was deleted;
- e) a differentiation between binders for stiff and flowing mixtures was introduced;
- f) the setting time for binders for flowing mixtures was introduced;
- g) the final setting time was deleted;
- h) the working time for binders and factory made mixtures were introduced;
- i) the test methods for factory made mixtures (CA) were deleted;
- i) the document was editorially revised.

EN 13454, *Binders for floor screeds based on calcium sulphate* consists of two parts:

- Part 1: Definitions and requirements
- Part 2: Test methods

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## 1 Scope

This document describes the test methods for binders for floor screeds based on calcium sulphate specified in EN 13454-1. In addition, some of the described test methods apply for factory made mixtures for floor screeds based on calcium sulphate specified in EN 13813.

This document describes reference test methods. If methods and conditions other than these are used, it is important to show that they give results equivalent to those given by the reference methods. In the event of a dispute, only the reference test method is used.

This document describes test methods for binders and made mixtures where the principal active component is calcium sulphate.

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 196-1:2016, Methods of testing cement - Part 1: Determination of strength

EN 196-3:2016, Methods of testing cement - Part 3: Determination of setting times and soundness

EN 196-7, Methods of testing cement - Part 7: Methods of taking and preparing samples of cement

EN 459-2:2010, Building lime - Part 2: Test methods

EN 13279-2, Gypsum binders and gypsum plasters - Part 2: Test methods

EN 13454-1, Binders for floor screeds based on calcium sulphate - Definitions and requirements

EN 13813, Screed material and floor screeds - Screed material - Properties and requirements

EN 13892-1, Methods of test for screed materials - Part 1: Sampling, making and curing specimens for test

#### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

#### 4 Test conditions and sampling

#### 4.1 General requirements for testing

#### 4.1.1 Water

The water used for testing and chemical analyses shall be distilled or deionized.

Unless otherwise specified, the water temperature shall be the same as the air temperature in the laboratory.

#### 4.1.2 Apparatus

The apparatus used for gauging and the moulds used for preparing the test specimens shall be free from leaks and shall be manufactured from a water proof material which is non-reactive to calcium sulphate (e.g. glass, brass, stainless steel, hardened steel, hard rubber and plastics, etc.).

Since the characteristics of calcium sulphate are strongly influenced by the presence of particles of calcium sulphate dihydrate which can influence the setting time, all equipment used in the tests shall be kept in a perfect state of cleanliness.

### 4.2 Sampling for binders

Sampling for binders shall be carried out in accordance with EN 196-7 and EN 459-2.

#### 5 Test methods for binders

### 5.1 Content of calcium sulphate

# 5.1.1 Principle

The calcium sulphate is decomposed by digestion with hydrochloric acid solution. Insoluble impurities are removed by filtration. The sulphate in the filtrate is determined gravimetrically as barium sulphate.

#### 5.1.2 Apparatus

- a) Sieve 0,1 mm mesh.
- b) 600 ml and 800 ml beakers.
- c) Rapid filtration funnels.
- d) Muffle furnace.
- e) Ignition crucible, porosity 4.
- f) Filter paper capable of retaining particles greater than 2,5 μm.
- g) Porous porcelain or silica crucible.

#### 5.1.3 Reagents

a) Hydrochloric acid solution: 2 N;

b) Ammonia solution: (50 % by volume)

c) Barium chloride: (3 % by mass solution in water)

d) Methyl orange or other suitable indicator.

#### **5.1.4 Procedure**

Grind the dried sample until it passes completely through a sieve with a mesh of 0,1 mm. Weigh accurately about 0,5 g into a 600 ml beaker, add 25 ml of hot distilled water.

Mix the sample and water with a glass rod until all the lumps are dispersed. Add 25 ml of 2 N HCl. Bring to boil and maintain this for 10 min. Add 150 ml of hot water and allow boiling for 15 min. Remove the beaker from the heat and filter through the filter paper into an 800 ml beaker. Wash out the beaker and filter paper using hot distilled or deionized water until the filtrate is free of chloride.

Make up to approximately 350 ml using distilled water. Add a few drops of indicator and neutralize with ammonia solution. Add 20 ml 2 N HCl. Bring to boil again and add 50 ml of hot barium chloride solution stirring vigorously during the addition. Bring to boil and allow to settle just below boiling for at least 30 min.

Filter by one of the following methods:

a) Filtration using filter crucibles.

Ignite a clean ignition crucible, porosity 4, or a porous porcelain filter crucible at  $(800 \pm 25)$  °C for 10 min.

Cool in a desiccator to room temperature and weigh.

Decant the clear liquid through the crucible using a slight suction. Wash the precipitate 3 times by decantation with hot water, transfer to the crucible and wash 6 times with small quantities of hot water. The last wash should be chloride free. Dry the crucible and contents and ignite at  $(800 \pm 25)$  °C for 10 min.

b) Filtration using filter paper.

Use filter paper as described. Decant the clear liquid through the filter paper. Wash the precipitate 3 times by decantation with hot water, transfer to the filter paper and wash with small quantities of hot water until the wash is chloride free.

After filtration and washing fold the paper around the precipitate and place it in a weighed porcelain or silica crucible, previously ignited at  $(800 \pm 25)$  °C and cooled in a desiccator. Heat gently to char the paper and expel the volatile matter. Do not allow the paper to burst into flames. Raise the temperature slowly to burn off the carbon with free access of air.

Finally ignite for 10 min at  $(800 \pm 25)$  °C. Cool in a desiccator and weigh. Calculate the CaSO<sub>4</sub> content as a percentage from the formula:

$$[CaSO_4] = W_2 \times 58,33/W_1 \tag{1}$$

where

 $W_1$  is the mass of sample, in grams;

 $W_2$  is the mass of precipitate, in grams.

#### 5.2 Determination of pH-value

Disperse 1 part by mass of the pulverized sample in 10 parts by mass of deionized or distilled water. Stir it for 5 min and then measure the pH-value by a pH-meter or pH-paper to the nearest  $\pm$  0,5 pH.