SLOVENSKI STANDARD
SIST EN 13454-2:2019

01-april-2019

Nadomešča:
SIST EN 13454-2:2004+A1:2007

Veziva za estrihe na osnovi kalcijevega sulfata - 2. del: Preskusne metode

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

Ta slovenski standard je istoveten z: EN 13454-2:2019

ICS:

91.100.10	Cement. Mavec. Apno. Malta	Cement. Gypsum. Lime. Mortar
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

SIST EN 13454-2:2019 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13454-2

January 2019

ICS 91.100.10; 91.100.50

Supersedes EN 13454-2:2003+A1:2007

English Version

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

Calciumsulfat-Binder für Estriche - Teil 2: Prüfverfahren

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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of an identical text or by endorsement, at the latest by July 2019, and conflicting national standards shall be withdrawn at the latest by July 2019.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;

j) 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 ± 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 ± 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 ± 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 ± 25) °C. Cool in a desiccator and weigh. Calculate the $CaSO_4$ content as a percentage from the formula:

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

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 ± 0,5 pH.

5.3 Determination of setting time

5.3.1 Determination of CAB and CAC setting time

5.3.1.1 Principle

Determine setting time using the method given in EN 196-3, “binder” being substituted, where “cement” is mentioned.

The setting time is determined by observing the penetration of a needle into a binder paste of standard consistence according to EN 196-3:2016, Clause 5 until it reaches a specified value.

Binder paste of standard consistency has a specified resistance to penetration by a standard plunger. The water required for such a paste is determined by trial penetrations of pastes with different water contents.

5.3.1.2 Apparatus

a) Mixer: As described in EN 196-1:2016, 4.4.

b) Vicat penetration apparatus: As described in EN 196-3:2016, 5.1.

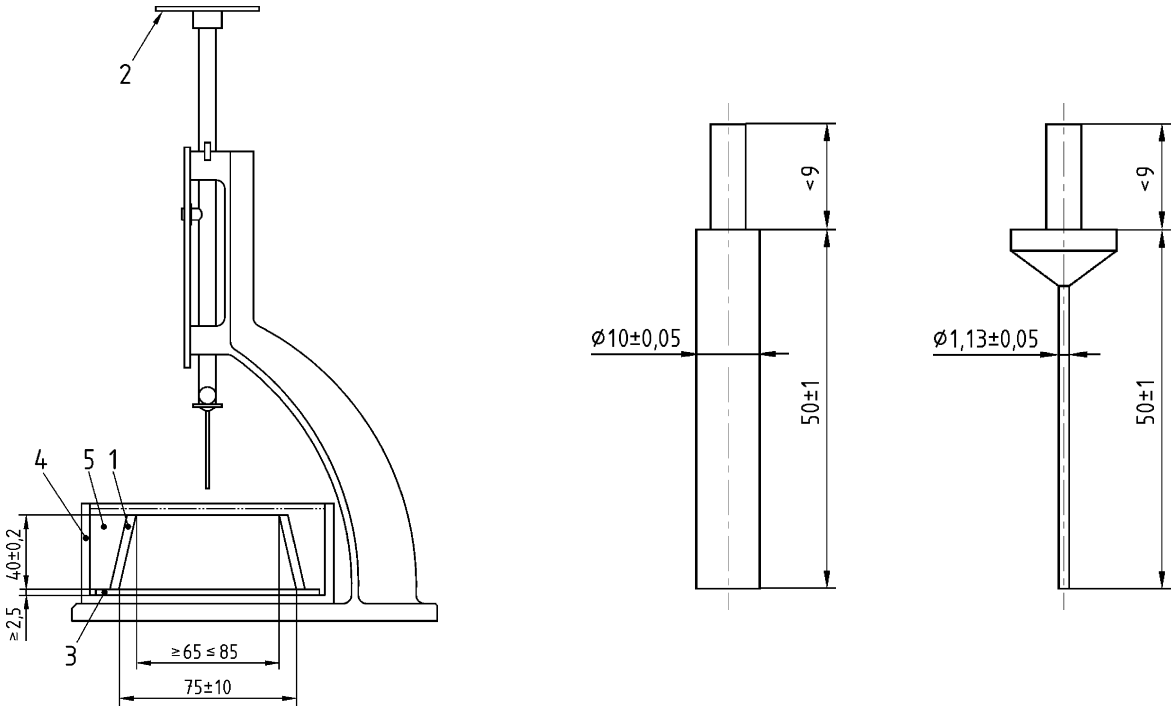
5.3.1.3 Standard consistency test

Use the Vicat apparatus as shown in Figure 1 a) with the plunger shown in Figure 1 b). The plunger (Figure 1 b)) shall be of non-corrodible metal in the form of a right cylinder of (50 ± 1) mm effective length and of (10,00 ± 0,05) mm diameter. The total mass of moving parts shall be (300 ± 1) g. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the plunger.

The Vicat mould (see Figure 1 a)) to contain the paste under test shall be of hard rubber. It shall be of truncated conical form (40,0 ± 0,2) mm deep and shall have internal diameters at top of (70 ± 5) mm and at bottom of (80 ± 5) mm. It shall be adequately rigid and shall be provided with a plane glass base-plate larger than the mould and at least 2,5 mm thick.

NOTE Moulds of metal or plastics or of cylindrical form can be used provided that they are of the specified depth and that they can be shown to give the same test results as the specified hard rubber mould of truncated conical form.

Dimensions in millimetres



a) Side view with mould in upright position for initial setting time determination

b) plunger for standard consistency

c) needle for initial test

Key

1 hard rubber mould
2 platform for correcting weights
3 basis plate
4 test container
5 water

The specified dimensions should be observed. If the plunger and needle are all adjusted to have the same mass, e.g. (9 ± 0,5) g, one correcting weight is sufficient for each apparatus.

Figure 1 — Vicat apparatus for determination of the standard consistency and setting time

5.3.1.4 Procedure

5.3.1.4.1 Mixing the binder paste

Weigh, to the nearest 1 g, 500 g of binder. Weigh a quantity of water, e.g. 125 g, in the mixer bowl or measure the water from the graduated cylinder or burette into the mixer bowl. Possible admixture should already be diluted in water.

Add the binder carefully to the water in order to avoid loss of water or binder. The time of addition shall not be less than 5 s nor more than 10 s. Note the time of completion of the addition as zero time from which later measurements of time shall be made. Start the mixer immediately and run at low speed for 90 s.

Stop the machine after 90 s for 15 s during which remove with a suitable scraper any paste adhering to the bowl outside the mixing zone and return it to the mix. Restart the machine and run at low speed for a further 90 s. The total mixer running time shall be 3 min.

NOTE Any other mixing method, whether by machine or hand, can be used provided that it can be shown to give the same test results as the specified method.

5.3.1.4.2 Filling the mould and carrying out the test

Transfer the paste after mixing immediately to the mould, which has previously been placed on a lightly greased plan glass base plate, and fill it to excess without undue compaction or vibration. Remove the excess by a gentle sawing motion with a straight-edged implement in such a way as to leave the paste filling the mould and having a smooth upper surface.

Adjust the Vicat apparatus with the plunger (Figure 1 b)) attached in advance of the test, by lowering the plunger to rest on the baseplate to be used and adjusting the pointer to read zero on the scale. Raise the plunger to the stand-by position.

Immediately after levelling the paste, transfer the mould and baseplate to the Vicat apparatus and position it centrally under the plunger. Lower the plunger gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the plunger to penetrate vertically into the centre of the paste. The release of the plunger shall occur 4 min after zero time. Read the scale when penetration has ceased or 30 s after the release of the plunger, whichever is the earlier.

Record the scale reading, which indicates the distance between the bottom face of the plunger and the baseplate, together with the water content of the paste expressed as a percentage by mass of the binder. Clean the plunger immediately after each penetration.

Repeat the test with pastes containing different water contents until one is found to produce a distance between plunger and baseplate of (6 ± 3) mm. Record the water content of that paste to the nearest 0,5 % as the water for standard consistency.

5.3.1.5 Setting time test

Remove the plunger and replace it by the needle (Figure 1 c)) which shall be of steel and in the form of a right cylinder of effective length (50 ± 1) mm and diameter (1,13 ± 0,05) mm. The total mass of moving parts shall be (300 ± 1) g. Their movement shall be truly vertical and without appreciable friction, and their axis shall coincide with that of the needle.

Adjust the Vicat apparatus with the needle (Figure 1 d)), attached in advance of the test, by lowering the needle to rest on the baseplate to be used and adjusting the pointer to read zero on the scale. Raise the needle to the stand-by position.

Fill a Vicat mould with paste of standard consistency and level it, in accordance with 5.3.1.4.1 and 5.3.1.4.2.

Place the filled mould and baseplate in the room and, after a suitable time, transfer to the Vicat apparatus and position under the needle. Lower the needle gently until it is in contact with the paste. Pause in that position for between 1 s and 2 s in order to avoid initial velocity or forced acceleration of the moving parts. Then release the moving parts quickly and allow the needle to penetrate vertically into the paste. Read the scale when penetration has ceased, or 30 s after the release of the needle, whichever is the earlier.

Record the scale reading, which indicates the distance between the end of the needle and the baseplate, together with the time from zero. Repeat the penetration test on the same specimen at conveniently spaced positions, not less than 10 mm from the rim of the mould or from each other, at conveniently spaced intervals of time, e.g. at 10 min intervals. Between penetration tests keep the specimen in a room. Clean the Vicat needle immediately after each penetration.

Record the time measured from zero at which the distance between the needle and the baseplate is (6 ± 3) mm as the initial setting time of the binder to the nearest 5 min. The required accuracy can be ensured by reducing the time interval between penetration tests near the end point and observing that successive results do not fluctuate excessively.