SLOVENSKI STANDARD

SIST EN 13454-2:2004+A1:2007

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Veziva, sestavljena veziva in industrijsko pripravljene mešanice za estrihe na osnovi kalcijevega sulfata - 2. del: Preskusne metode

Binders, composite binders and factory made mixtures for floor screeds based on calcium sulfate - Part 2: Test methods

Calciumsulfat-Binder, Calciumsulfat-Compositbinder und Calciumsulfat-Werkmörtel für Estriche - Teil 2: Prüfverfahren

Liants, liants composites et mélanges fabriqués en usine à base de sulfate de calcium pour chapes de sol - Partie 2: Méthodes d'essai

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91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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This European Standard was approved by CEN on 1 September 2003 and includes Amendment 1 approved by CEN on 22 June 2007.

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Foreword

This document (EN 13454-2:2003+A1:2007) has been prepared by Technical Committee CEN/TC 241 “Gypsum and gypsum based products”, the secretariat of which is held by AFNOR.

This document shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2008 and conflicting national standards shall be withdrawn at the latest by January 2008.

This document includes Amendment 1 approved by CEN on 2007-06-22.

This document supersedes EN 13454-2:2003.

The start and finish of text introduced or altered by amendment is indicated in the text by tags [A1] [A1].

[A1] *deleted text* [A1]

The European Standard EN 13454 for binders, composite binders and factory made mixtures for floor screeds based on calcium sulfate consists of two parts:

Part 1: Definitions and requirements

Part 2: Test methods

The requirements in prEN 13454-1 are based on the results of tests according to EN 13454-2 on binders, composite binders and factory made mixtures for floor screeds based on calcium sulfate.

This European Standard describes test methods for binders, composite binders and factory made mixtures where the principal active component is calcium sulfate.

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

1 Scope

This European Standard describes the test methods for binders and composite binders for floor screeds based on calcium sulfate specified in prEN 13454-1.

This European Standard describes the test methods for factory made mixtures for floor screeds based on calcium sulfate specified in EN 13813.

This European Standard describes reference test methods. If other than these methods and conditions are used, it is necessary 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.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

[A1⟩ EN 196-1:2005 ⟨A1], *Methods of testing cement – Part 1 : Determination of strength*

[A1⟩ EN 196-3:2005, *Methods of testing cement - Part 3: Determination of setting times and soundness* ⟨A1]

EN 196-7:1989, *Methods of testing cement – Part 7: Methods of taking and preparing samples of cement*

EN 459-2:2001, *Building lime – Part 2: Test methods*

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 Test conditions and sampling

3.1 General requirements for testing

3.1.1 Water

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

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

3.1.2 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 sulfate (e.g. glass, brass, stainless steel, hardened steel, hard rubber and plastics etc.).

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

3.2 Sampling for binders (CAB, CAC)

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

3.3 Sampling for factory made mixtures (CA)

Sampling for factory made mixtures shall be carried out in accordance with EN 13892-1.The composition of samples shall always be representative of the average composition of the material, the possibility of segregation being taken into consideration. The test report shall state whether the samples taken are spot samples or composite samples (See 3.6 and 3.7 of EN 196-7:1989).

Samples which are likely to change in air shall be placed in airtight containers, such as cans, immediately after they have been taken.

Wherever possible tests shall be carried out on specimens prepared immediately after obtaining the sample, if necessary on site. Specimens shall be prepared during working time but not later than 2 h after gauging. This period shall also be observed when fresh samples are supplied to a testing centre, and may be appropriately decreased or increased for samples containing accelerators or retarders.

If transportation of fresh samples cannot be avoided, they shall be supplied immediately for testing, accompanied by a certificate stating which tests are to be carried out, and protected from changes, such as loss of water, entry of water etc., as may occur during transportation. Specimens shall be prepared immediately on receipt of the sample, after the sample has been mixed again manually.

Specimens shall be prepared within the working time after gauging. For this reason, they shall as a rule (e.g. when prepared in situ) be stored under vibration-free conditions and protected from climatic effects (e.g. frigobox etc.) for at least 24 h before they are conveyed in the moulds for testing. If special requirements in respect of the preparation and storage of the particular sample are specified for the tests envisaged these shall be observed.

4 Test methods for binders (CAB, CAC)

4.1 Content of calcium sulfate

4.1.1 Principle

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

4.1.2 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.

4.1.3 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.

4.1.4 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 the boil and maintain this for 10 min. Add 150 ml of hot water and allow to boil 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 deionised water until the filtrate is free of chloride.

Make up to approximately 350 ml using distilled water. Add a few drops of indicator and neutralise with ammonia solution. Add 20 ml 2 N HCl. Bring to the boil again and add 50 ml of hot barium chloride solution stirring vigorously during the addition. Bring to the 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 equation :

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

where

W_1 is the mass of sample, in grams ;

W_2 is the mass of precipitate, in grams.

4.2 Determination of pH

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

4.3 Determination of setting time

4.3.1 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 clause 5 of [A1⟩ EN 196-3:2005 ⟨A1] 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.

4.3.2 Apparatus

a) Mixer : As described in 4.4 of [A1⟩ EN 196-1:2005 ⟨A1].

b) Vicat penetration apparatus : As described in 5.1 of [A1⟩ EN 196-3:2005 ⟨A1].

4.3.3 Standard consistence test

Use the Vicat apparatus as shown in Figure 1(a) and 1(b) with the plunger shown in Figure 1(c). The plunger (Figure 1(c)) 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 and bottom of (70 ± 5) mm and (80 ± 5) mm respectively. 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 may 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.

4.3.4 Procedure

4.3.4.1 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, may be used provided that it can be shown to give the same test results as the specified method.

4.3.4.2 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 1c) 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.

4.3.5 Setting time test

4.3.5.1 Determination of initial setting time

Remove the plunger and replace it by the needle (Figure 1 (d)) 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 1d), 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 4.3.4.1 and 4.3.4.2.

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 may be assured by reducing the time interval between penetration tests near the end point and observing that successive results do not fluctuate excessively.

4.3.5.2 Determination of final setting time

Invert the filled mould used in 4.3.4 on its baseplate so that the tests for final set are made on the face of the specimen originally in contact with the baseplate. Fit the needle with a ring attachment (Figure 1 (e)) to facilitate accurate observation of small penetrations. Use the procedure described in 4.3.4. The intervals of time between penetration tests may be increased to e.g. 30 min.

Record, to the nearest 15 min, the time measured from zero at which the needle first penetrates only 0,5 mm into the specimen as the final setting time of the binder. This time is that which the ring attachment first fails to mark the specimen and may be accurately established by reducing the time interval between tests near the end point and observing that successive test results do not fluctuate excessively.

NOTE Automatic setting time machines are commercially available and may be used provided that they can be shown to give the same test results as the specified apparatus and procedure.

4.4 Determination of strengths

Determine the flexural and the compressive strength in accordance with EN 196-1, "binder" being substituted, where "cement" is mentioned.

4.4.1 Apparatus

a) Mixer : As described in 4.4 of A1⟩ EN 196-1:2005 ⟨A1.

b) Moulds with base : As described in 4.5 of A1⟩ EN 196-1:2005 ⟨A1 for material see 3.1.3.

c) Vibrating table : As described in 5.1.2.2.1 of EN 459-2:2001.

d) Flexural strength, testing machine : As described in 4.7, 4.8, 4.9 of A1⟩ EN 196-1:2005 ⟨A1.

e) Compressive strength, testing machine : As described in clause 9 of A1⟩ EN 196-1:2005 ⟨A1.

f) Flow table, mould, cam and tamper : As described in 5.5.2.1.2 of EN 459-2:2001