SLOVENSKI STANDARD

SIST EN 196-5

prva izdaja
september 1995

Metode preskušanja cementa - 5. del: Določanje pucolanske aktivnosti za pucolanske cemente (prevzet standard EN 196-5:1994 z metodo platnice)

Methods of testing cement - Part 5: Pozzolanicity test for pozzolanic cement

Méthodes d'essais des ciments - Partie 5: Essai de pouzzolanicité des ciments pouzzolaniques

Prüfverfahren für Zement - Teil 5: Prüfung der Puzzolanität von Puzzolanzementen

Deskriptorji: cement, pucolan, kemijski preskus, določanje sestavin, kalcijev hidroksid, EDTO, kemijski reagenti, aparatura

ICS 91.100.10

Referenčna številka
SIST EN 196-5:1995 (en)

Nadaljevanje na straneh od II do IV in 1 do 6

UVOD

Standard SIST EN 196-5 (en), Metode preskušanja cementa - 5. del: Določanje pucolanske aktivnosti za pucolanske cemente, prva izdaja, 1995, ima status slovenskega standarda in je z metodo platnice prevzet evropski standard EN 196-5, Methods of testing cement - Part 5: Pozzolanicity test for pozzolanic cement, 1994, v angleškem jeziku.

NACIONALNI PREDGOVOR

Evropski standard EN 196-5:1994 je pripravil tehnični odbor Evropske organizacije za standardizacijo CEN/TC 51 Cement in apno.

Odločitev za prevzem tega standarda po metodi platnice je sprejela delovna skupina USM/TC CAA/WG 1 Cement, potrdil pa tehnični odbor USM/TC CAA Cement, apno in vlaknatocementni izdelki.

Ta slovenski standard je dne 1995-08-29 odobril direktor USM.

SLOVENSKI STANDARD SIST EN 196 ZA PRESKUŠANJE CEMENTA OBSEGA NASLEDNJE DELE:

SIST EN 196-1:1995 (en)	Metode preskušanja cementa - 1. del: Določanje trdnosti
SIST EN 196-2:1995 (en)	Metode preskušanja cementa - 2. del: Kemijska analiza cementa
SIST EN 196-3:1995 (en)	Metode preskušanja cementa - 3. del: Določanje časa vezanja in prostorninske obstojnosti
SIST ENV 196-4:1995 (en)	Metode preskušanja cementa - 4. del: Kvantitativno določanje sestavin
SIST EN 196-5:1995 (en)	Metode preskušanja cementa - 5. del: Določanje pucolanske aktivnosti za pucolanske cemente
SIST EN 196-6:1995 (en)	Metode preskušanja cementa - 6. del: Določanje finosti
SIST EN 196-7:1995 (en)	Metode preskušanja cementa - 7. del: Metode odvzemanja in priprave vzorcev cementa
SIST EN 196-21:1995 (en)	Metode preskušanja cementa - 21. del: Določanje količine kloridov, ogljikovega dioksida in alkalij v cementu

PREDHODNA IZDAJA

- JUS B.C1.018:1959 (sh) Pucolani - Kvalitet i ispitivanje

OSNOVA ZA IZDAJO STANDARDA

- Prevzem standarda EN 196-5:1994

OPOMBI

- Povsod, kjer se v besedilu standarda uporablja izraz "evropski standard", v SIST EN 196-5:1995 to pomeni "slovenski standard".

- Uvod in nacionalni predgovor nista sestavni del standarda.

VSEBINA **stran**

Po mnenju Ministrstva za informiranje Republike Slovenije z dne 18. februarja 1992, štev. 23/96-92, spada ta publikacija med proizvode informativne narave iz 13. točke tarifne številke 3, za katere se plačuje 5-odstotni prometni davek.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 196-5

December 1994

ICS 91.100.10

Supersedes EN 196-5:1987

Descriptors: Cements, pozzolans, chemical tests, determination of content, calcium hydroxide, EDTA, chemical reagents, apparatus

English version

Methods of testing cement - Part 5: Pozzolanicity test for pozzolanic cement

Méthodes d'essais des ciments - Partie 5: Essai de pouzzolanicité des ciments pouzzolaniques

Prüfverfahren für Zement - Teil 5: Prüfung der Puzzolanität von Puzzolanzementen

This European Standard was approved by CEN on 1994-12-12. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Contents

Foreword

This European Standard was drawn up by Technical Committee CEN/TC 51 'Cement and building limes', of which the secretariat is held by IBN.

The European Standard on the methods of testing cement comprises the following Parts:

EN 196-1	Methods of testing cement — Part 1: Determination of strength
EN 196-2	Methods of testing cement — Part 2: Chemical analysis of cement
EN 196-3	Methods of testing cement — Part 3: Determination of setting time and soundness
ENV 196-4	Methods of testing cement — Part 4: Quantitative determination of constituents
EN 196-5	Methods of testing cement — Part 5: Pozzolanicity test for pozzolanic cements
EN 196-6	Methods of testing cement — Part 6: Determination of fineness
EN 196-7	Methods of testing cement — Part 7: Methods of taking and preparing samples of cement
EN 196-21	Methods of testing cement — Part 21: Determination of the chloride, carbon dioxide and alkali content of cement

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 June 1995, and conflicting national standards shall be withdrawn at the latest by June 1995.

This European standard supersedes EN 196-5:1987.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

1 Scope

This European Standard describes the method of measuring the pozzolanicity of pozzolanic cements conforming to ENV 197-1. This standard does not apply to Portland pozzolana cements or to pozzolanas.

This method constitutes the reference procedure.

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.

EN 196-2	*Methods of testing cement — Part 2 : Chemical analysis of cement*
EN 196-7	*Methods of testing cement — Part 7 : Methods of taking and preparing samples of cement*
ENV 197-1	*Cement — Composition, specifications and conformity criteria — Part 1 : Common cements*
ISO 385-1 : 1984	*Laboratory glassware — Burettes — Part 1 : General requirements*
ISO 835-1 : 1981	*Laboratory glassware — Graduated pipettes — Part 1 : General requirements*
ISO 3534 : 1977	*Statistics — Vocabulary and symbols*

3 General requirements for testing

3.1 Expression of masses, volumes and factors

Express masses in grams to the nearest 0,0001 g and volumes from the burette in millilitres to the nearest 0,05 ml. Express the factors of solutions, given by the mean of three determinations, to three places of decimals.

3.2 Number of tests

The number of tests shall be two (see also **3.3**).

3.3 Expression of results

Express the results of the determinations in millimoles per litre to the nearest 0,1 mmol/l.

Give the final result as the mean of two determinations to one place of decimals.

If the difference between two determinations is more than twice the standard deviation for repeatability, repeat the test and take the mean of the two closest values.

3.4 Repeatability and reproducibility

The standard deviation of repeatability gives the closeness of agreement between successive results obtained with the same method on identical material tested under the same conditions (same operator, same apparatus, same laboratory and short intervals of time[1]).

The standard deviation of reproducibility gives the closeness of agreement between individual results obtained with the same method on identical material but tested under different conditions (different operators, different apparatus, different laboratory and/or different time[1]).

The standard deviations of repeatability and reproducibility are expressed in millimoles per litre.

4 Preparation of a cement sample

Take a sample by the method described in EN 196-7. Treat this laboratory sample as described in EN 196-2.

5 Principle

The pozzolanicity is assessed by comparing the quantity of calcium hydroxide present in the aqueous solution in contact with the hydrated cement, after a fixed period of time, with the quantity of calcium hydroxide capable of saturating a solution of the same alkalinity. The test is considered positive if the concentration of calcium hydroxide in the solution is lower than the saturation concentration.

Experiment has shown that a mixture of 20 g of cement and 100 ml of water at 40 °C achieves equilibrium after a period of 8 days or 15 days[2].

To evaluate the results it is therefore necessary to know the solubility at 40 °C of calcium hydroxide in a solution of which the alkalinity varies from 35 to about 100 mmol OH^- per litre.

6 Reagents

Use only reagents of recognized analytical quality and freshly boiled water, distilled or of equivalent purity, during the analysis.

6.1 *Concentrated hydrochloric acid (HCl)*, approximately 12 mol/l (ρ = 1,18 g/cm^3 to 1,19 g/cm^3).

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[1] Definitions taken from ISO 3534.

[2] 8 days are sufficient if the test is positive at this stage (see **10.2**).

6.2 *Dilute hydrochloric acid : about 0,1 mol/l.* Using the 50 ml precision burette (**7.8**), add 8,5 ml of concentrated hydrochloric acid (**6.1**) to a 1 litre volumetric flask (**7.10**) containing about 500 ml of water. Then make up the volume with water.

6.3 *Dilute hydrochloric acid (1 + 2)* : add 250 ml of concentrated hydrochloric acid to 500 ml water.

6.4 *Methyl orange* (dimethylaminoazobenzene p-sodium sulfonate).

6.5 *Methyl orange indicator* : dissolve 0,02 g of methyl orange in water and make up to 1000 ml.

6.6 *Sodium hydroxide (NaOH).*

6.7 *Sodium hydroxide solution* : dissolve 100 g of sodium hydroxide in water and make up to 1000 ml.

6.8 *Calcium carbonate ($CaCO_3$)* : dried at 110 °C.

6.9 *Potassium chloride (KCl)* : dried at 110 °C.

6.10 *Murexide* (ammonium purpurate).

6.11 *Murexide indicator* : grind and mix 1 g of murexide with 100 g of dry potassium chloride.

6.12 *EDTA* (disodium dihydrate salt of ethylenediaminetetra-acetic acid).

6.13 *EDTA solution about 0,025 mol/l* : dissolve 9,306 g of EDTA in water and make up to 1000 ml.

6.14 *Sodium carbonate (Na_2CO_3)* : dried at 260 °C.

7 Apparatus

7.1 *500 ml cylindrical polyethylene container* of about 70 mm diameter with a pressure seal-plug locked by a screw plug.

7.2 *Wide stem funnel.*

7.3 *Porcelain Buchner funnel* of 60 mm inner diameter.

7.4 *Filter paper with low porosity* (mean pore diameter of about 2µm).

7.5 *250 ml vacuum flask.*

7.6 *250 ml and 400 ml beakers.*

7.7 *50 ml and 100 ml precision pipettes* (class A of ISO 835-1 : 1981).

7.8 *50 ml precision burette* (class A of ISO 385-1 : 1984).

7.9 *Uniform temperature enclosure* controlled thermostatically at (40 ± 0,5) °C.

7.10 *500 ml and 1000 ml volumetric flasks.*

7.11 *250 ml conical flask.*

8 Standardization of solutions

8.1 Standardization of the EDTA solution

Weigh approximately 1 g of calcium carbonate (**6.8**) to the nearest 0,0001 g and introduce into the 250 ml beaker (**7.6**). Add approximately 100 ml of water and, very carefully, 50 ml of dilute hydrochloric acid (**6.3**) keeping the beaker covered with a watch glass.

Stir with a glass rod and ensure that dissolution is complete. Then transfer the solution into the 500 ml volumetric flask (**7.10**), wash the beaker and watch glass carefully with water, adding the washings to the solution and make up the volume with water.

Pipette 50 ml of the solution into the 400 ml beaker (**7.6**), dilute with approximately 150 ml of water and add the quantity of sodium hydroxide solution (**6.7**) necessary to achieve pH 13 (check the pH by means of a pH meter or by indicator papers).

Add approximately 50 mg of murexide indicator (**6.11**) and titrate by means of the burette (**7.8**) against the EDTA solution (**6.13**) until there is a steady colour change from purple to violet. From the volume of EDTA solution used, calculate the factor f_1 of the EDTA solution by the formula:

$$f_1 = \frac{m_1}{100,09} \times \frac{1000}{10 \times 0,025 \times V_1}$$

$$= \frac{m_1}{V_1} \times 39,96 \qquad (1$$

where

f_1 is the factor of the ETDA solution;

m_1 is the mass of calcium carbonate, in grams;

V_1 is the volume of EDTA solution used for the titration, in millilitres;

100,09 is the molecular mass of calcium carbonate.

8.2 Standardization of the 0,1 mol/l solution of hydrochloric acid

Weigh approximately 0,2 g of sodium carbonate (**6.14**), add it to the 250 ml flask (**7.11**) and dissolve it in 50 ml to 75 ml of water. Add five drops of the methyl orange indicator (**6.5**) to the solution and titrate with the 0,1 mol/l dilute hydrochloric acid (**6.2**) until the colour changes from yellow to orange.

Calculate the factor f_2 of the hydrochloric acid solution by the formula:

$$f_2 = \frac{2 \times m_2}{105{,}989} \times \frac{1000}{0{,}1 \times V_2} = \frac{m_2}{V_2} \times 188{,}70 \qquad (2)$$

where

f_2 is the factor of the hydrochloric acid;

m_2 is the mass of sodium carbonate, in grams;

V_2 is the volume of hydrochloric acid used for the titration, in millilitres;

105,989 is the molecular mass of sodium carbonate.

9 Procedure

9.1 Storage and filtration

Pipette 100 ml of freshly boiled water into the polyethylene container (**7.1**) and place the sealed container in the thermostatic enclosure (**7.9**) until equilibrium is reached (about 1 h). Remove the container from the thermostatic enclosure. Pour (20 ± 0,01) g of the cement to be examined into it, using the wide stem funnel (**7.2**). Immediately seal the container hermetically.

Shake vigorously for about 20 s to avoid formation of cement lumps. A horizontal rotary motion has to be used to prevent any part of the sample or liquid being thrown up and remaining separated from the rest of the solution.

Replace the container in the thermostatic enclosure, making sure that its base is perfectly horizontal so that the deposited layer of cement has a uniform thickness. Perform all operations outside the thermostatic enclosure as quickly as possible (in 1 min maximum) to avoid any appreciable lowering in temperature of the contents of the container.

After a period of 8 days or 15 days[2] in the thermostatic enclosure, remove the container and filter the solution immediately under vacuum through the Buchner funnel (**7.3**) into the vacuum flask (**7.5**) using dry double filter paper (**7.4**) in less than 30 s (to avoid absorption of atmospheric carbon dioxide and any appreciable lowering in temperature of the solution). Seal the vacuum flask immediately and let the filtrate cool to room temperature.

9.2 Determination of the hydroxyl ion concentration

Shake the vacuum flask (**7.5**) to homogenise the filtrate and pipette 50 ml of the solution into the 250 ml beaker (**7.6**). Add five drops of methyl orange indicator (**6.5**) and determine the total alkalinity with the dilute hydrochloric acic (**6.2**). The titration end-point corresponds to the colour change from yellow to orange.

Calculate the hydroxyl ion concentration $[OH^-]$ by the formula:

$$[OH^-] = \frac{1000 \times 0{,}1 \times V_3 \times f_2}{50} = 2 \times V_3 \times f_2 \qquad (3)$$

where

$[OH^-]$ is the hydroxyl ion concentration in millimoles per litre;

V_3 is the volume of 0,1 mol/l hydrochloric acid solution used for the titration, in millilitres;

f_2 is the factor of 0,1 mol/l hydrochloric acid solution, in grams per millilitre.

9.3 Determination of the calcium oxide concentration

To the same solution remaining after completing **9.2**, add 5 ml of sodium hydroxide solution (**6.7**), approximately 50 mg of murexide indicator (**6.11**) and titrate the calcium oxide with EDTA solution (**6.13**) by means of the burette (**7.8**) until there is a steady colour change from purple to violet.

Before and after titration, the pH value of the solution shall be at least 13, if not, add the requisite amount of sodium hydroxide solution.

Calculate the calcium oxide concentration [CaO] by the formula:

$$[CaO] = \frac{1000 \times 0{,}025 \times V_4 \times f_1}{50} = 0{,}5 \times V_4 \times f_1 \qquad (4)$$

where

[CaO] is the calcium oxide concentration in millimoles per litre;

V_4 is the volume of EDTA solution used for the titration, in millilitres;

f_1 is the factor of the EDTA solution.

[2] 8 days are sufficient if the test is positive at this stage (see **10.2**).