BS EN 196-5:2011

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Methods of testing cement

Part 5: Pozzolanicity test for pozzolanic cement

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National foreword

This British Standard is the UK implementation of EN 196-5:2011. It supersedes BS EN 196-5:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/516/12, Sampling and testing.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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Foreword

This document (EN 196-5:2011) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by NBN.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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This document supersedes EN 196-5:2005.

This 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 times and soundness*;
- CEN/TR 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 cement*;
- 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 196-8, *Methods of testing cement — Part 8: Heat of hydration — Solution method*;
- EN 196-9, *Methods of testing cement — Part 9: Heat of hydration — Semi-adiabatic method*;
- EN 196-10, *Methods of testing cement — Part 10: Determination of the water-soluble chromium (VI) content of cement*.

NOTE A previous part, EN 196-21: *Methods of testing cement — Part 21: Determination of the chloride, carbon dioxide and alkali content of cement*, has been revised and incorporated into EN 196-2.

This edition introduces the following technical changes based on comments received by the secretariat:

a) the procedure, reagents and layout of the standard have been aligned with the relevant clauses of EN 196-2;

b) the procedure for preparation of a test sample has been clarified;

c) Patton and Reeders reagent has been included as an additional, optional indicator for visual determination of EDTA titrations;

d) the specification for apparatus has been extended to include a balance of specified accuracy; apparatus for measuring the absorbance of a solution whilst being stirred and a pH meter of specified accuracy.

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1 Scope

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

This method constitutes the reference procedure.

2 Normative references

The following referenced documents are indispensable for the application 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 196-7, *Methods of testing cement — Part 7: Methods of taking and preparing samples of cement*

EN ISO 385:2005, *Laboratory glassware — Burettes (ISO 385:2005)*

EN ISO 835:2007, *Laboratory glassware — Graduated pipettes (ISO 835:2007)*

3 Principle

The pozzolanicity is assessed by comparing the concentration of calcium ion, expressed as calcium oxide, present in the aqueous solution in contact with the hydrated cement, after a fixed period of time, with the quantity of calcium ion capable of saturating a solution of the same alkalinity. The cement is considered to satisfy the test, i.e. gives a positive result, if the concentration of calcium ion in the solution is lower than the saturation concentration.

NOTE 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 between 8 d and 15 d. If the cement satisfies the test at 8 d (see 10.2) it is not necessary to continue to 15 d.

4 General requirements for testing

4.1 Number of tests

Where the determination is one of a series subject to statistical control, determination by a single test shall be the minimum required.

Where the determination is not part of a series subject to statistical control, the number of tests shall be two (see also 10.1).

In the case of dispute, the number of tests shall be two.

4.2 Repeatability and reproducibility

Repeatability and reproducibility in this document are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s).

4.3 Expression of masses, volumes and factors

Express masses in grams to the nearest 0,000 1 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 decimal places.

4.4 Determination of constant mass

Determine constant mass by drying for successive periods at the stated temperature, or making successive 15 min ignitions, followed each time by cooling and then weighing. Constant mass is reached when the difference between two successive weighings is less than 0,000 5 g.

5 Preparation of a test sample of cement

Before starting the determinations, treat the laboratory sample, taken in accordance with EN 196-7, as follows to obtain a homogenous test sample.

Take approximately 100 g of the sample using a sample divider or by quartering. Sieve this portion on a 150 µm or 125 µm sieve until the residue remains constant. Grind the retained material so that it completely passes the 150 µm or 125 µm sieve. Transfer the sample to a clean dry container with an airtight closure and shake vigorously to mix it thoroughly.

Carry out all operations as quickly as possible to ensure that the sample is exposed to ambient air only for the minimum time.

6 Reagents

Use only reagents of analytical quality. References to water mean distilled or de-ionised water having an electrical conductivity ≤ 0,5 mS/m. The quantities of reagents listed are to indicate concentrations; actual quantities to be prepared shall be adjusted according to the amounts required.

Unless otherwise stated (%) means percent by mass.

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

6.2 Hydrochloric acid, about 0,1 mol/l, prepared by measuring with a graduated cylinder (7.16) 8,5 ml of concentrated hydrochloric acid (6.1) to a litre volumetric flask (7.10) containing about 500 ml of water and make up to 1 000ml with water. Determine the factor of normality of the solution as indicated in 8.2.

6.3 Dilute hydrochloric acid (1 + 2), prepared by adding 250 ml of concentrated hydrochloric acid (6.1) to 500 ml water.

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

6.5 Methyl orange indicator, prepared by dissolving (0,020 ± 0,002) g of methyl orange (6.4) in water and making up to 1 000 ml.

6.6 Sodium hydroxide, (NaOH).

6.7 Sodium hydroxide solution, prepared by dissolving (100 ± 1) g of sodium hydroxide (6.6) in water and making up to 1 000 ml.

6.8 Calcium carbonate, ($CaCO_3$), dried to constant mass at (200 ± 10) °C (purity greater than 99,9 %).

6.9 Sodium chloride, (NaCl), dried to constant mass at (110 ± 5) °C.

6.10 Murexide, (ammonium purpurate).

6.11 Murexide indicator, prepared by grinding (1,0 ± 0,1) g of murexide with (100 ± 1) g of dry sodium chloride (NaCl).

6.12 EDTA, (dihydrated disodium salt of ethylenediaminetetra-acetic acid).

6.13 EDTA solution, about 0,03 mol/l, prepared by dissolving (11,17 ± 0,01) g of EDTA in water and making up to 1 000 ml. Store in an air-tight polyethylene container. Determine the factor of molarity of the solution as indicated in 8.1.

6.14 Sodium carbonate, (Na_2CO_3), dried to constant mass at (250 ±10) °C.

6.15 Mixed calcein and methylthymol blue indicator, prepared by grinding (0,20 ± 0,02) g calcein (bis [bis (carboxymethyl)-amino-methyl]) –2', 7'-fluorescein (fluorescein, Flurorescein di-(methylimino diacetic acid) sodium salt) and (0,10 ± 0,01) g methylthymol blue, sodium salt of 3', 3''-bis- [bis (carboxy-methyl)-aminomethyl]-thymolsulfophthalein, ($C37H_{41}N_2O_{13}SNa_3$) with (100 ± 1) g of potassium nitrate (KNO_3).

6.16 Calcon indicator, prepared by grinding (1,0 ± 0,1) g of calcon, sodium 2-hydroxy-4-(2-hydroxy-1-napthylazo) napthalene-1-sulfonate, (Eriochrome Blue-Black R) with (100 ± 1) g of anhydrous sodium sulfate (Na_2SO_4).

6.17 Patton and Reeders reagent, prepared by mixing (1,0 ± 0,1) g of Calcon carboxylic acid, (2-hydroxy-1-(2-hydroxy-4-sulfo-1-napthylazo)-3-napthoic acid, ($C_{21}H_{14}N_2O_7S$), with (100 ± 1) g of anhydrous sodium sulfate (Na_2SO_4).

7 Apparatus

7.1 500 ml cylindrical polyethylene container, of about 70 mm diameter with a pressure seal-plug locked by a screw plug, capable of preventing evaporation during storage.

7.2 Wide stem funnel.

7.3 Porcelain Büchner funnel, of 60 mm inner diameter.

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

7.5 250 ml vacuum flask.

7.6 250 ml and 400 ml beakers.

7.7 50 ml and 100 ml pipettes, class A of EN ISO 835:2007.

7.8 50 ml burette, class A of EN ISO 385:2005.

7.9 Uniform temperature enclosure, controlled thermostatically at (40 ± 1) °C.

7.10 500 ml and 1 000 ml volumetric flasks.

7.11 250 ml conical flask.

7.12 Balance, capable of weighing to an accuracy of ± 0,000 5 g.

7.13 Apparatus for measuring the absorbance, at 520 nm and 620 nm of a solution contained in a titration beaker, while stirring.

7.14 Stirrer, e.g. magnetic stirrer, with inert, e.g. PTFE, covered bar.

7.15 pH meter, capable of measuring to an accuracy of ± 0,05.

7.16 Graduated cylinder of 10ml or 20 ml.

8 Standardization of solutions

8.1 Standardization of the EDTA solution

Weigh to an accuracy of ± 0,000 5 g (1,00 ± 0,01) g of calcium carbonate (6.8), m_1, and place it in a 400 ml beaker (7.6) with approximately 100 ml of water. Cover the beaker with a watch glass and carefully introduce approximately 10 ml of hydrochloric acid (1 + 2) (6.3). Stir with a glass rod and ensure that dissolution is complete, bring to the boil in order to expel the dissolved carbon dioxide. Cool to room temperature, transfer to a volumetric flask (7.10), wash the beaker and watch glass carefully with water, adding the washings to the solution and make up to 1 000 ml with water.

Pipette 50 ml of the calcium solution into a beaker suitable for the measuring apparatus (7.13). Then dilute with water to a volume suitable for the operation of the apparatus. Using a pH meter (7.15), adjust the pH of this solution to (12,5 ± 0,2) with the sodium hydroxide solution (6.7).

Determine the end-point using one of the following two methods.

a) Photometric determination of the end-point (reference method):

Add, without weighing, approximately 0,1 g of murexide indicator (6.11) or of mixed indicator (6.15). Place the beaker in the apparatus (7.13) set at 620 nm when using murexide or at 520 nm when using the mixed indicator and, while stirring continuously, titrate with 0,03 mol/l EDTA solution (6.13). In the vicinity of the colour change, construct a curve giving the absorbance values as a function of the volume of EDTA added. The volume V_1 used is determined from the intersection of the line of greatest slope near the colour change and the line of almost constant absorbance after the colour change.

Calculate the factor, f_1, of the EDTA solution from the formula:

$$f_1 = \frac{m_1 \times 50}{100{,}09 \times 0{,}03 \times V_1} = \frac{m_1}{V_1} \times 16{,}652 \qquad (1)$$

where

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.

b) Visual determination of the end-point (alternative method)

Add, without weighing, about 0,1 g of the calcon indicator (6.16), or Patton and Reeders reagent mixture (6.17). Stir and titrate with the 0,03 mol/l EDTA solution (6.13) until the colour changes from pink to blue (purple to clear blue for Patton and Reeders reagent), and one drop in excess does not further increase the intensity of the blue colour. The volume V_1 is used to calculate the standardization factor f_1 using the formula (1).

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

Weigh, to an accuracy of ± 0,000 5 g, (0,200 ± 0,001) g of sodium carbonate (6.14), m_2, add it to the 250 ml conical 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 from 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

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), seal and place in the uniform temperature enclosure (7.9) until equilibrium is reached (about 1 h). Remove the container from the uniform temperature enclosure. Pour (20,00 ± 0,01) g of the cement to be tested 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. Use a horizontal rotary motion 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 uniform temperature enclosure, making sure that its base is horizontal so that the deposited layer of cement has a uniform thickness. Perform all operations outside the uniform temperature 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 d or 15 d in the uniform temperature enclosure, remove the container and filter the solution immediately under vacuum through the Büchner 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.

NOTE If the cement satisfies the test at 8 d (see 10.2) it is not necessary to continue to 15 d.

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 0,1 mol/l dilute hydrochloric acid (6.2). The titration end-point corresponds to the colour change from yellow to orange. Keep the titrated solution, A, for the determination of calcium oxide concentration (9.3).

Calculate the hydroxyl ion concentration, $[OH]^-$, in millimoles per litre, from the formula:

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

where

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.

9.3 Determination of the calcium oxide concentration

Using the titrated solution, A, remaining after completing 9.2 adjust the pH to (12,5 ± 0,2), with sodium hydroxide solution (6.7), using the pH meter (7.15). Titrate with 0,03 mol/l EDTA solution (6.13) determining the end-point by one of the methods in 8.1.

Calculate the calcium oxide concentration, [CaO], in millimoles per litre, from the formula:

$$[\mathrm{CaO}] = \frac{1000 \times 0{,}03 \times V_4 \times f_1}{50} = 0{,}6 \times V_4 \times f_1 \quad (4)$$

where

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

f_1 is the factor of the EDTA solution.

10 Results

10.1 Calculation and expression of results

Calculate the concentrations of hydroxyl ion and calcium ion (expressed as calcium oxide), each expressed to the nearest 0,1 mmol/l.

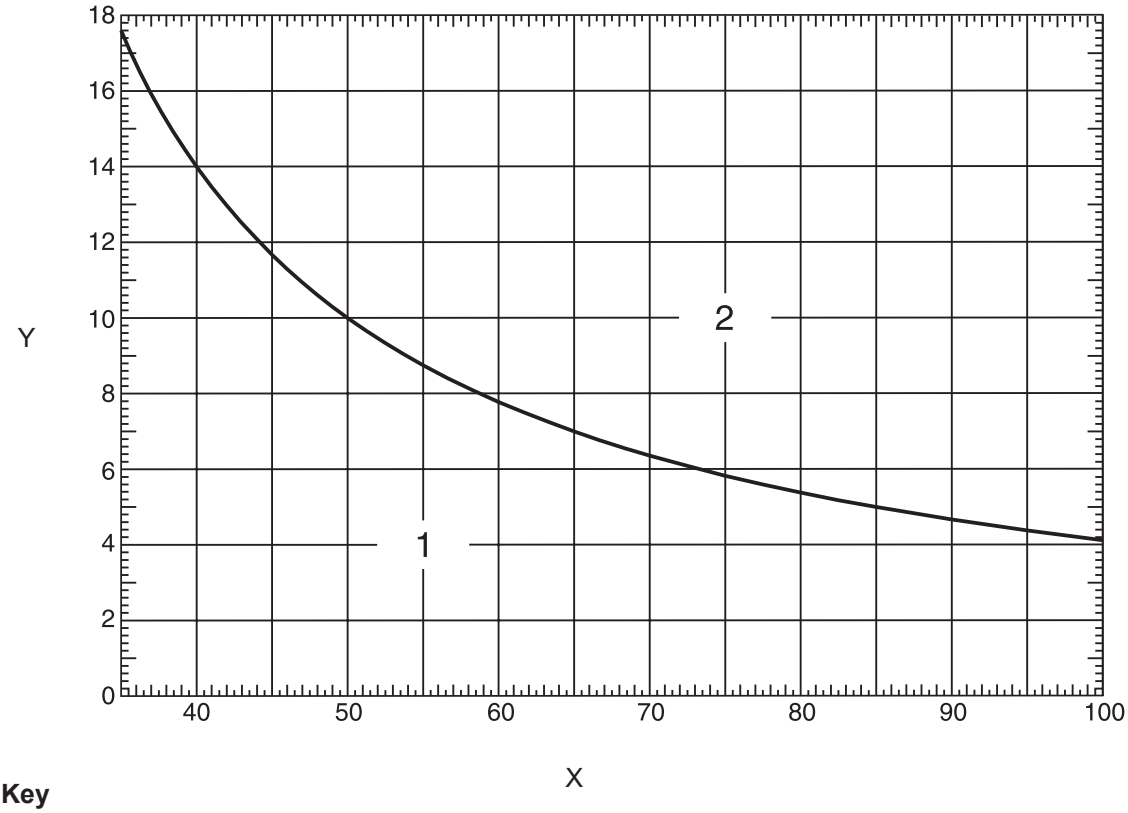
Express the results, where two test results have been obtained, as the mean of the results to the nearest 0,1 mmol/l.

If the two test results differ by more than twice the standard deviation of repeatability, repeat the test and take the mean of the two closest test results.

10.2 Assessment of pozzolanicity

Plot the concentrations of hydroxyl ion and calcium ion (expressed as calcium oxide) in the solution, obtained in accordance with 10.1, as a point on Figure 1 which shows the saturation concentration of calcium ion (expressed as calcium oxide) as a function of the hydroxyl ion concentration at 40 °C. The curve in Figure 1 may be expressed mathematically over the range 45 mmol/l to 90 mmol/l [OH] by [CaO] = 350 / ([OH] - 15,0) where the calcium ion (expressed as calcium oxide) and hydroxyl ion concentrations are given in millimoles per litre.

The cement satisfies the test for pozzolanicity when the point plotted is below the curve of calcium ion (expressed as calcium oxide) saturation concentration shown on Figure 1.



Key

1 Pass

2 Fail

X Hydroxyl ion concentration, mmol/l

Y Calcium ion concentration (expressed as calcium oxide), mmol/l

Figure 1 — Diagram for assessing pozzolanicity

10.3 Repeatability and reproducibility

The standard deviation for repeatability is:

hydroxyl ion : 0,5 mmol/l;

calcium ion (expressed as calcium oxide) : 0,2 mmol/l.

The standard deviation for reproducibility is:

hydroxyl ion : 1,0 mmol/l;

calcium ion (expressed as calcium oxide) : 0,5 mmol/l.

11 Reporting of results

Record all individual results. Where the cement has been shown to satisfy (or not satisfy) the test for pozzolanicity as determined in Clause 10, it shall be reported that:

This cement satisfies (does not satisfy) the test for pozzolanicity according to EN 196-5.

Bibliography

[1] EN 197-1, *Cement — Part 1: Composition, specifications and conformity criteria for common cements*

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