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Cement — Test methods — Pozzolanicity test for pozzolanic cements

Ciments — Méthodes d'essai — Essai de pouzzolanicité des ciments pouzzolaniques

Reference number
ISO 863:2008(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 863 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

This second edition cancels and replaces the first edition (ISO 863:1990), which has been technically revised as follows, based on comments received by the Secretariat.

a) The procedure, reagents and layout of this International Standard have been aligned with the relevant clauses of ISO 29581-1.

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

c) Patton and Reeders reagent has been included as an additional, optional indicator for the 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.

Cement — Test methods — Pozzolanicity test for pozzolanic cements

1 Scope

This International Standard describes the method of measuring the pozzolanicity of pozzolanic cements. It does not apply to Portland pozzolana cements or to pozzolanas.

This International Standard 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.

ISO 385:2005, *Laboratory glassware — Burettes*

ISO 835:2007, *Laboratory glassware — Graduated pipettes*

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.

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

When 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 International Standard are expressed as repeatability standard deviation(s) and reproducibility standard deviation(s) and defined as follows.

a) Repeatability:

Precision under repeatability conditions where independent test results are obtained with the same method on identical test items (material) in the same laboratory by the same operator using the same equipment within short intervals of time.

b) Reproducibility:

Precision under reproducibility conditions where test results are obtained with the same method on identical test items (material) in different laboratories with different operators using different equipment.

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 by 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 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 for only the minimum time.

6 Reagents

6.1 General

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

Unless otherwise stated, “percentage” means percent by mass.

6.2 Hydrochloric acid (HCl), concentrated, ($\rho =$ 1,18 g/cm^3 to 1,19 g/cm^3).

6.3 Hydrochloric acid, dilute, about 0,1 mol/l, prepared by adding 8,5 ml of concentrated hydrochloric acid (6.2), measured using the 50 ml burette (7.8), to a 1 l volumetric flask (7.10) containing about 500 ml of water and making up to 1 000 ml with water.

6.4 Hydrochloric acid, dilute (1 + 2), prepared by adding 250 ml of concentrated hydrochloric acid (6.2) to 500 ml water.

6.5 Methyl orange, (dimethylaminoazobenzene *p*-sodium sulfonate).

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

6.7 Sodium hydroxide, (NaOH).

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

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

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

6.11 Murexide, (ammonium purpurate).

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

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

6.14 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.

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

6.16 Mixed calcein and methylthymol blue indicator, prepared by grinding (0,20 ± 0,02) g calcein (bis [bis (carboxymethyl)-amino-methyl)] –2', 7'-fluorescein [Fluoresceindi-(methylimino diacetic acid) sodium salt] and (0,10 ± 0,01) g methylthymol blue, sodium salt of 3', 3''-bis- [bis (carboxy-methyl)-aminomethyl]-thymolsulfophthalein, ($C_{37}H_{41}N_2O_{13}SNa_3$) with (100 ± 1) g of potassium nitrate (KNO_3).

6.17 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[1]) with (100 ± 1) g of anhydrous sodium sulfate (Na_2SO_4).

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

7.2 Funnel, wide stem.

7.3 Funnel, Büchner, porcelain, 60 mm inner diameter.

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

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[1] Eriochrome® Blue-Black R is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

7.5 **Vacuum flask**, 250 ml.

7.6 **Beakers**, 250 ml and 400 ml.

7.7 **Pipettes**, 50 ml and 100 ml, class A in accordance with ISO 835:2007.

7.8 **Burette**, 50 ml , class A in accordance with ISO 385:2005.

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

7.10 **Volumetric flasks**, 500 ml and 1 000 ml.

7.11 **Conical flask**, 250 ml.

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.

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 (6.9), 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) (6.4). 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 (6.8).

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 (6.12) or of mixed indicator (6.16). 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 (6.14). 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 Equation (1):

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

where

m_1 is the mass of calcium carbonate, expressed in grams;

V_1 is the volume of EDTA solution used for the titration, expressed 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 (6.17), or Patton and Reeders reagent mixture (6.18). Stir and titrate with the 0,03 mol/l EDTA solution (6.14) until the colour changes from pink to blue for the calcon indicator or 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 Equation (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 (6.15), 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 (6.6) to the solution and titrate with the 0,1 mol/l dilute hydrochloric acid (6.3) until the colour changes from yellow to orange.

Calculate the factor, f_2, of the hydrochloric acid solution from Equation (2):

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

where

m_2 is the mass of sodium carbonate, expressed in grams;

V_2 is the volume of hydrochloric acid used for the titration, expressed 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 being 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 from 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.

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 homogenize the filtrate and pipette 50 ml of the solution into the 250 ml beaker (7.6). Add five drops of methyl orange indicator (6.6) and determine the total alkalinity with the 0,1 mol/l dilute hydrochloric acid (6.3). 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, $c_{[\mathrm{OH}]}$, expressed in millimoles per litre, from Equation (3):

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

where

V_3 is the volume of 0,1 mol/l hydrochloric acid solution used for the titration, expressed 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 (6.8), using the pH meter (7.15). Titrate with 0,03 mol/l EDTA solution (6.14) determining the end-point by one of the methods in 8.1.

Calculate the calcium oxide concentration, $c_{[\mathrm{CaO}]}$, expressed in millimoles per litre, from Equation (4):

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

where

V_4 is the volume of EDTA solution used for the titration, expressed 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.

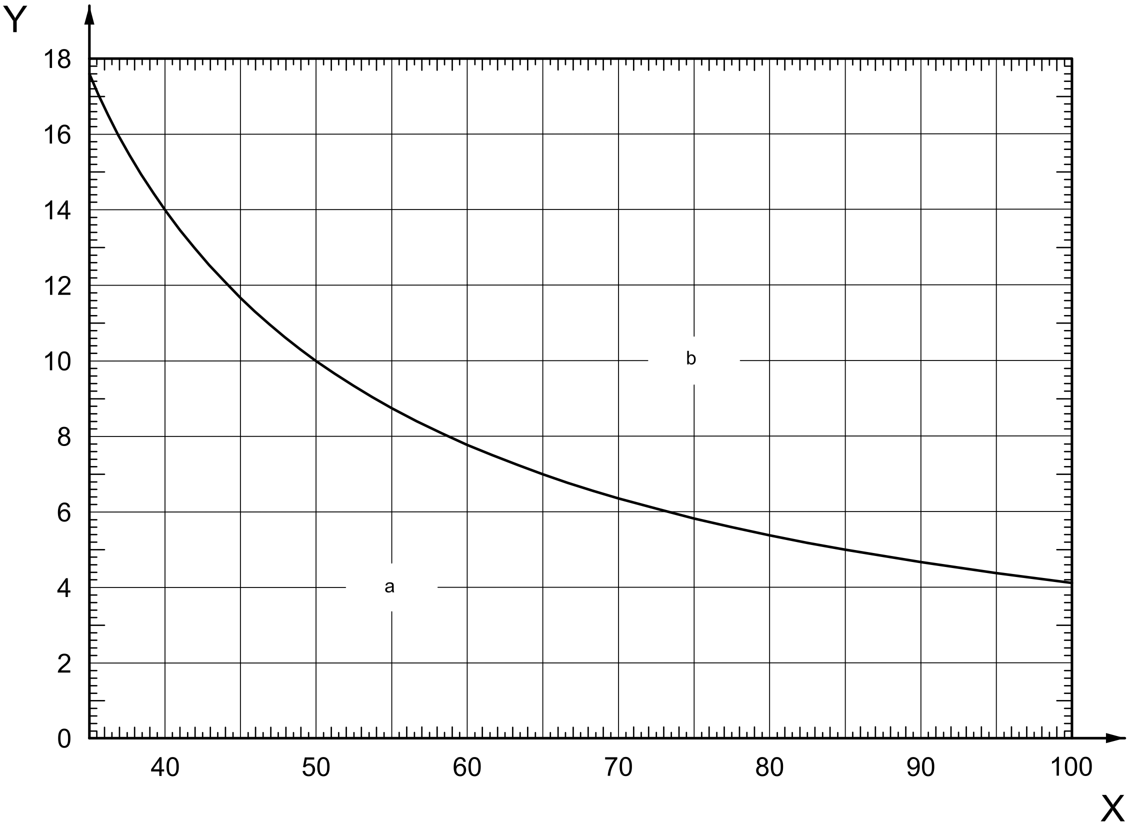
10.2 Assessment of pozzolanicity

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 $c_{[\mathrm{OH}]}$ by Equation (5):

$$c_{[\mathrm{CaO}]} = \frac{350}{\left(c_{[\mathrm{OH}]} - 15{,}0\right)} \tag{5}$$

where the calcium ion (expressed as calcium oxide) and hydroxyl ion concentrations are expressed 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

X hydroxyl ion concentration, expressed in millimoles per litre

Y calcium ion concentration (expressed as CaO), expressed in millimoles per litre

[a] Pass.

[b] Fail.

Figure 1 — Diagram for assessing pozzolanicity

10.3 Repeatability and reproducibility

a) The standard deviation for repeatability is

- hydroxyl ion: 0,5 mmol/l;
- calcium ion (expressed as calcium oxide): 0,2 mmol/l.

b) 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. When 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 in accordance with ISO 863:2008”.

Bibliography

[1] ISO 29581-1, *Cement — Test methods — Part 1: Analysis by wet chemistry*

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