INTERNATIONAL STANDARD

ISO
863

First edition
1990-04-01

Cement — Test methods — Pozzolanicity test for pozzolanic cements

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

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

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 method constitutes the reference procedure.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 680 : 1989, *Cement — Test methods — Chemical analysis.*

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 0,000 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 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).

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).

These deviations are exactly defined in ISO 3534.

The standard deviations of repeatability and reproducibility are expressed in absolute percentages.

4 Preparation of cement sample

Take a laboratory sample: the exact method will be described in a future International Standard. Treat this sample as described in clause 6 of ISO 680 : 1989.

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.

Experience 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. [1]

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 mmol OH^- per litre to about 100 mmol OH^- per litre.

---

1) 8 days are sufficient if the test is positive at this stage (see 10.2).

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 ($\varrho = 1{,}18$ g/cm^3 to $1{,}19$ g/cm^3).

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 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 1 000 ml.

6.6   Sodium hydroxide (NaOH).

6.7   Sodium hydroxide solution.

Dissolve 100 g of sodium hydroxide in water and make up to 1 000 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 salt) [(ethylenedinitrilo)tetraacetic acid, disodium salt, dihydrate].

6.13   EDTA (disodium salt) solution about 0,025 mol/l.

Dissolve 9,306 g of EDTA (disodium salt) in water and make up to 1 000 ml.

6.14   Sodium carbonate (Na$_2$CO$_3$): 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 2 µm).

7.5   250 ml vacuum flask.

7.6   250 ml and 400 ml beakers.

7.7   50 ml and 100 ml precision pipettes (ISO class A).

7.8   50 ml precision burette (ISO class A).

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

7.10   500 ml and 1 000 ml volumetric flasks.

7.11   250 ml conical flask.

8   Standardization of solutions

8.1   Standardization of EDTA solution

Weigh approximately 1 g of calcium carbonate (6.8) 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 (7.7) 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 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:

$$f_1 = \frac{m_1}{100{,}09} \times \frac{1\,000}{10 \times 0{,}025 \times V_1} = \frac{m_1}{V_1} \times 39{,}96 \quad \ldots (1)$$

where

m_1 is the mass of calcium carbonate;

V_1 is the volume of EDTA solution used for the titration;

100,09 is the molecular mass of calcium carbonate.

8.2 Standardization of 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:

$$f_2 = \frac{2m_2}{105,989} \times \frac{1\,000}{0,1 \times V_2} = \frac{m_2}{V_2} \times 188,70 \qquad \ldots (2)$$

where

m_2 is the mass of sodium carbonate;

V_2 is the volume of hydrochloric acid used for the titration;

105,989 is the molecular mass of sodium carbonate.

9 Procedure

9.1 Storage and filtration

Pipette (7.7) 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 g ± 0,01 g of the cement to be examined into it, using the wide stem funnel (7.2). Seal the container immediately.

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 thermoplastic 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[1] in the thermostatic enclosure, remove the container and filter the solution immediately under vacuum through the Buchner funnel (7.3) into

---

[1] 8 days are sufficient if the test is positive at this stage (see 10.2).

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 hydroxyl ion concentration

Shake the vacuum flask (7.5) to homogenize the filtrate and pipette (7.7) 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 acid (6.2). The titration end-point corresponds to the colour change from yellow to orange.

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

$$[OH^-] = \frac{1\,000 \times 0,1 \times V_3 \times f_2}{50} = 2 \times V_3 \times f_2 \qquad \ldots (3)$$

where

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

f_2 is the factor of 0,1 mol/l hydrochloric acid solution.

9.3 Determination of 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 should be at least 13; if it is not, add the requisite amount of sodium hydroxide solution.

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

$$[CaO] = \frac{1\,000 \times 0,025 \times V_4 \times f_1}{50} = 0,5 \times V_4 \times f_1 \quad \ldots (4)$$

where

V_4 is the volume of EDTA solution used for the titration;

f_1 is the factor of the EDTA solution.

10 Results

10.1 Expression of results

Plot the mean concentrations (see 3.3) of hydroxyl ions and calcium oxide in the solution, obtained in accordance with 9.2

and 9.3, as a point on figure 1 which shows the saturation concentration of calcium oxide in the solution as a function of the hydroxyl ion concentration at 40 °C.

10.2 Assessment of results

The cement satisfies the pozzolanicity test when the point plotted is under the curve of calcium oxide saturation concentration shown in figure 1.

10.3 Repeatability and reproducibility

The standard deviation for repeatability is as follows:

for calcium oxide: 0,2 mmol/l

for hydroxyl ion : 0,5 mmol/l

The standard deviation for reproducibility is as follows:
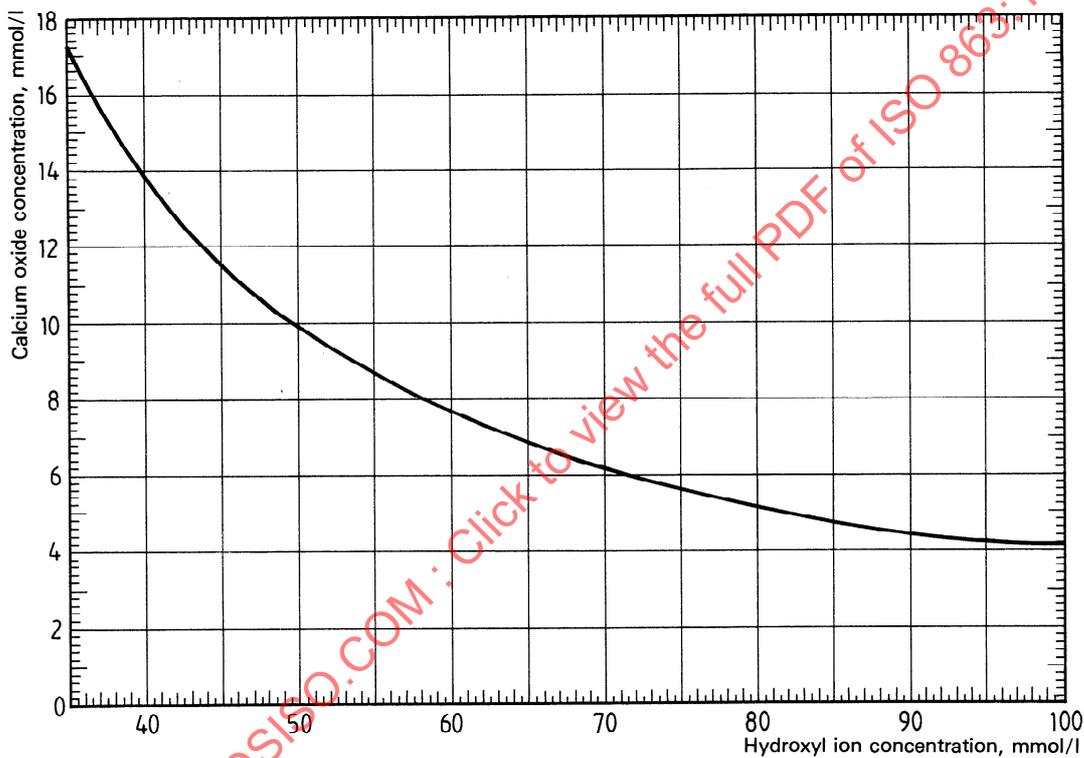
for calcium oxide: 0,5 mmol/l

for hydroxyl ion : 1,0 mmol/l

Figure 1 — Diagram for assessing pozzolanicity