SLOVENSKI STANDARD
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Reaktivnost sestavin cementa - Metoda za določanje hidracijske toplote in vsebnosti vezane vode

Reactivity of cement constituents - Heat of hydration and bound water content methods

Reaktivität von Zementbestandteilen - Verfahren zur Bestimmung der Hydratationswärme und des chemisch gebundenen Wassers

Réactivité des constituants du ciment - Méthodes de détermination de la chaleur d'hydratation et de la teneur en eau liée

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ICS:

91.100.10	Cement. Mavec. Apno. Malta	Cement. Gypsum. Lime. Mortar

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Reactivity of Cement Constituents - Heat of Hydration and Bound Water methods

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 51.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 17979:2023) has been prepared by Technical Committee CEN/TC 51 “Cement and Building Limes”, the secretariat of which is held by NBN.

This document is currently submitted to the CEN Enquiry.

1 Scope

This document specifies two complementary test methods to assess the chemical reactivity of a pozzolanic or latent hydraulic cement constituent by measurements of heat of hydration (see Clause 5 and 8.3 Method A, Heat of Hydration) or bound water content (see Clause 5 and 8.4 Method B, Bound Water Content) of hydrated pastes composed of the cement constituent, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide cured at 40 °C for 72 h and 168 h (3 days and 7 days).

These two test methods do not distinguish between latent hydraulic and pozzolanic reactivity. Therefore, these methods are used for measuring the chemical reactivity of following cement constituents as specified under EN 197-1 and EN 197-5: S, D, P, Q, V, W and T.

These test methods are used in complement with the current specifications on cement constituent reactivity given by EN 197-1 and EN 197-5, i.e. the reactive silicon dioxide content measured according to EN 196-2 for cement constituents P, Q and V; the compressive strength of specified test mortars determined according to EN 196-1 for cement constituents W and T, and the pozzolanicity of pozzolanic cements according to EN 196-5 for CEM IV type cements according to EN 197-1.

The test methods are used for qualification purposes if the cement constituents are tested at the fineness of the intended use.

NOTE In case the test methods are used for purposes of comparison of intrinsic reactivity, cement constituents are tested at similar fineness, where possible.

The test methods are also used for testing other new constituents that are latent hydraulic or pozzolanic and that are not covered by EN 197-1 and EN 197-5. However, for such new constituents the validity of the underlying correlations with strength development have not been verified; in consequence the test results can only be used for informative and indicative purposes.

Furthermore, these test methods are used in manufacturing control of cement constituents for assessing their latent hydraulic or pozzolanic reactivity.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 196-2, *Method of testing cement - Part 2: Chemical analysis of cement*

EN 196-6, *Methods of testing cement - Part 6: Determination of fineness*

EN 196-11, *Methods of testing cement - Part 11: Heat of hydration - Isothermal Conduction Calorimetry method*

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

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 9277, *Determination of the specific surface area of solids by gas adsorption — BET method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 197-1, EN 196-2, EN 196-11 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at https://www.electropedia.org/

3.1
chemically bound water
water, in hardened cement paste, that has reacted and is part of the structure of hydrated reaction products; chemically bound water for a specific time is taken as the mass loss when a paste specimen, dried previously at 40 °C, is heated in a furnace to 350 °C

Note 1 to entry: Some natural pozzolans can contain bound water and lose mass over this temperature range. This mass loss shall be determined and used to correct the bound water value for the paste.

3.2
constituent
main cement constituent, other than clinker

4 General requirements for testing

4.1 Number of tests

For each determination, one or more tests shall be carried out in which the number of measurements to be taken shall be as specified in the relevant clause of this document (see also 8.3 and 8.4).

4.2 Repeatability and reproducibility

4.2.1 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.

4.2.2 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, factors and results

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

Express the results, where a single test result has been obtained, to one decimal for the method A and to two decimal places for the method B (see Clause 9).

Express the results, where two test results have been obtained, as the mean of the results, as a percentage generally to two decimal places.

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.

The results of all individual tests shall be recorded.

5 Principle of test methods (A and B)

This document describes two test methods used to assess the chemical reactivity of a pozzolanic or latent hydraulic cement constituent over a curing time of 168 h.

Method A – Isothermal calorimetry is used to determine the heat of hydration of hydrating pastes composed of the cement constituent, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide. The heat of hydration value is used to determine the chemical reactivity of the cement constituent.

Method B – Chemically bound water of pastes composed of the cement constituent, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide is determined as a measure of the chemical reactivity of the cement constituent.

The results of these test methods can be used to estimate, at a given fineness, the potential contribution of a constituent to the development of strength, or other properties such as lower permeability, when used in common cement. The calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide are combined in proportions to provide a paste where the dissolved ions from these components simulate the pore solution in a portland cement system.

The pastes are cured at 40 °C to accelerate the rate of reaction of slowly constituents.

These test methods allow for the direct measurement of the hydraulic or pozzolanic reactivity of a cement constituent at a given fineness.

NOTE These test methods are based on established correlations between strength development and evolution of heat and chemically bound water for constituents covered by EN 197-1 and EN 197-5. For other (pozzolanic or latent hydraulic) reactive materials, the validity of such correlations has not been established.

There is no requirement to use Method A and Method B for a given application. In many instances the choice is based on the user's determination of available equipment. Method A can also provide an indication of rate of reactivity because measurements are taken continuously during the test period, while Method B provides the level of reactivity up to a single point in 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 ≤ 0,5 mS/m.

6.2 Calcium hydroxide (Ca(OH)2)

The calcium hydroxide should be protected from exposure to carbon dioxide. Material remaining in an open container after a test should not be used for subsequent tests.

6.3 Potassium hydroxide (KOH)

Potassium hydroxide from open containers should be protected from exposure to moisture and dried for at least 8 h at 105 °C prior to use.

Potassium hydroxide should be stored dry and protected from exposure to moisture or humidity.

6.4 Potassium sulfate

6.5 Calcium carbonate ($CaCO_3$) dried to constant mass at (200 ± 10) °C

6.6 Potassium solution

A potassium solution shall be prepared by dissolving 4,00 g of potassium hydroxide and 20,0 g of potassium sulfate in 1 000 ml of water.

7 Apparatus

7.1 balance, capable of weighing to an accuracy of ± 0,000 5 g.

7.2 mixer, a high-shear blender mixer capable of maintaining a no-load speed of at least 1 600 rpm.

7.3 isothermal heat conduction calorimeter, an isothermal heat conduction calorimeter conforming to EN 196-11.

7.4 calorimeter specimen containers and lids, that can be sealed air-tight.

7.5 laboratory oven, capable of being set at the following temperatures: (40 ± 2) °C

7.6 furnace, capable of being set at the following temperatures: (350 ± 10) °C.

7.7 crucibles, porcelain crucibles complying with the crucibles for measuring loss on ignition in EN 196-2.

7.8 petri dishes, glass petri dishes of at least 50 mm in diameter.

7.9 crushing equipment, mortar and pestle, disk pulverizer, rotary mill or crusher capable of decreasing the size of the paste particles to meet the size requirements given in Clause 8.3.3.

7.10 desiccator, containing anhydrous magnesium perchlorate (Mg(ClO4)2) or silica gel. Where self-indicating silica gel is used, a non-toxic indicator is recommended.

7.11 test sieves 125 µm, wire cloth test sieve conforming to ISO 3310-1 in accordance with ISO 565:1990, Table 1.

7.12 cylindrical polyethylene container, of about a volume of 50 ml with a pressure seal-plug locked by a screw plug, capable of preventing evaporation during storage.

7.13 wax paper, paper that has been made moisture-proof through the application of wax.

8 Procedures

8.1 Preparation of a test cement constituent specimen

Cement constituents subjected to testing according to method A and method B shall be tested at cement fineness. To qualify for testing the test specimen shall be dry-sieved on a 125 µm (7.11) sieve and the mass fraction retained on the sieve shall not be larger than 5 %, by mass.

Before testing the cement constituent (method A and method B), treat the laboratory sample, of appropriate fineness, as follows to obtain a homogeneous test sample.

Subsample approximately 100 g of the laboratory sample by means of a sample divider or by quartering.

Transfer the sample to a clean dry container with an airtight closure and shake vigorously to mix it thoroughly.

8.2 Testing Paste

For each method, prepare a testing paste using the proportioning test mixtures as described in Table 1.

Table 1 — Proportioning of test mixtures

Method	Constituent	$Ca(OH)_2$	$CaCO_3$	Potassium solution
Mass	g	g	g	g
Method A	2,00 ± 0,01 g	6,00 ± 0,01 g	1,00 ± 0,01 g	10,80 ± 0,01 g
Method B	4,00 ± 0,01 g	12,00 ± 0,01 g	2,00 ± 0,01 g	21,60 ± 0,01 g

NOTE 1 The total mass of the test mixtures is in excess of the required test specimen masses to account for losses during transfer of the test mixtures into the specimen containers.

Weigh the designated amounts (Table 1) of the dry reactive cement constituent, calcium hydroxide (6.2) and calcium carbonate (6.5), then combine and mix until a homogeneous colour is achieved.

NOTE 2 Mixing the dry mixture by hand with a spatula, passing through sieves multiple times, or use of a gyratory type mixer until a homogeneous colour is attained, are methods that have been found to be satisfactory. Gentle mixing is often required to avoiding creating dust.

Store the dry mixture in an air-tight container in a laboratory oven (7.5) at (40 ± 2) °C until the temperature of the dry mixture is stabilized at (40 ± 2) °C.

At the same time, place and seal the potassium solution in an air-tight container in a laboratory oven (7.5) at (40 ± 2) °C until the solution is at (40 ± 2) °C.

Mix the dry mixture and the potassium solution, at 1 600 ± 50 rpm for 2 min using the high-shear blender (7.2) so that a homogeneous paste is achieved.

Alternatively, use the procedure for mixing paste specified in EN 196-3.

Record the time at start of mixing and use as the time zero.

8.3 Method A, Heat of Hydration

8.3.1 Preparation of the apparatus

The isothermal heat conduction calorimeter (7.3) shall be calibrated at 40 C and set at (40 ± 0,5) °C for at least 16 h before the use.

Prior to mixing the testing pastes (8.1), place the calorimeter specimen containers, lids and pipettes in laboratory oven (7.5) at (40 ± 2) °C for 3 ± 1 h.

If the isothermal calorimeter has multiple test chambers, and a sufficient volume of paste is prepared, more than one test specimen can be tested simultaneously. However, inserting new test specimens into the calorimeter while other test specimens are in test can create significant additional noise depending on the ambient temperature, the reactivity of the materials tested and the specific design of the calorimeter. It is not recommended to insert new test specimens or otherwise open the calorimeter beyond 1 h after the mix time of the first test specimens inserted into the calorimeter.

Insert sealed, air-tight containers filled with (9,40 ± 0,05) g of deionized water into the reference channels of the calorimeter.

Because the total specimen mass to be placed in the calorimeter specimen chamber is instrument specific, to ensure that the thermal signal is within the measurement range of the instrument, the manufacturer's recommendations for reference and test specimen masses and should be followed.