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Metode preskušanja cementa - 8. del: Toplota hidratacije - Metoda raztapljanja

Methods of testing cement - Part 8: Heat of hydration - Solution method

Prüfverfahren für Zement - Teil 8: Hydratationswärme - Lösungsverfahren

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Methods of testing cement - Part 8: Heat of hydration - Solution method

Prüfverfahren für Zement - Teil 8: Hydratationswärme - Lösungsverfahren

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 196-8:2010) 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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

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This document supersedes EN 196-8:2003.

EN 196, *Methods of testing cement*, consists of the following parts:

— *Part 1: Determination of strength*

— *Part 2: Chemical analysis of cement*

— *Part 3: Determination of setting times and soundness*

— *Part 5: Pozzolanicity test for pozzolanic cement*

— *Part 6: Determination of fineness*

— *Part 7: Methods of taking and preparing samples of cement*

— *Part 8: Heat of hydration — Solution method*

— *Part 9: Heat of hydration — Semi-adiabatic method*

— *Part 10: Determination of the water-soluble chromium (VI) content of cement*

— CEN/TR 196-4, *Methods of testing cement — Part 4: Quantitative determination of constituents*

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1 Scope

This European Standard describes a method of determining the heat of hydration of cements by means of solution calorimetry, also known as the solution method. The heat of hydration is expressed in joules per gram of cement.

This standard is applicable to cements and hydraulic binders whatever their chemical composition.

NOTE 1 Another procedure, called the semi-adiabatic method, is described in EN 196-9. Either procedure can be used independently.

NOTE 2 It has been demonstrated that the best correlation between the two methods is obtained at seven days for the solution method (EN 196-8) compared with 41 h for the semi-adiabatic method (EN 196-9).

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 197-1:2000, *Cement — Part 1: Composition, specifications and conformity criteria for common cements*

3 Principle

The method consists in measuring the heats of solution, in an acid mixture, of anhydrous cement and cement hydrated under standardized conditions for a predetermined period of time, e.g. seven days.

These standardized hydration conditions are as follows:

— water/cement ratio 0,40;

— use of neat cement paste;

— storage at constant temperature of (20,0 ± 0,2) °C during the whole hydration process.

The heat of hydration for each period, H_i, is obtained from the difference between the heat of solution of anhydrous cement, Q_a, and that of hydrated cement, Q_i.

4 Materials

4.1 Acid mixture

Analytical reagent quality acid mixture, obtained by adding 2,760 g of 40 % hydrofluoric acid (HF) for every 100,0 g of (2,00 ± 0,01) mol/l nitric acid (HNO_3), or 2,600 ml of hydrofluoric acid for every 100,0 ml of nitric acid.

WARNING — Hydrofluoric acid can cause painful skin burns which heal only with difficulty and precautions in handling this very corrosive substance should be strictly observed.

The quantity (mass or volume) of acid to be used, which is common to all tests, shall be measured to ± 0,2 %.

4.2 Zinc oxide (ZnO)

Use zinc oxide of analytical quality to determine the thermal capacity of the calorimeter. Weigh 40 g to 50 g. Ignite at (950 ± 25) °C for 1 h. Cool in a desiccator. Grind to pass a 125 µm sieve. Store in a desiccator.

4.3 Anhydrous cement

Store anhydrous cement, from which metallic iron has been removed with a magnet, in a sealed container to avoid absorption of water or carbon dioxide. Bring the test sample to ambient temperature and carefully homogenize it before use.

4.4 Hydrated cement

Prepare the hydrated cement test sample by vigorously mixing, either manually or mechanically, (100,0 ± 0,1) g of anhydrous cement with (40,0 ± 0,1) g of distilled or deionised water for 3 min at ambient temperature. Place the resulting paste in plastics or glass cylindrical vials (three for each hydration period to be tested) so that each vial contains 15 g to 20 g of material. Seal the vials by means of a stopper and, if necessary, with paraffin wax or similar material and store them horizontally in a thermostatic bath maintained at a temperature of (20,0 ± 0,2) °C.

5 Apparatus

5.1 Calorimeter.

NOTE The method does not deal with the standardization of the calorimetric apparatus, or the measuring instruments. Insulated flasks with a volume of about 650 ml have proved to be suitable.

A suitable calorimeter (see Figure 1) comprises the following:

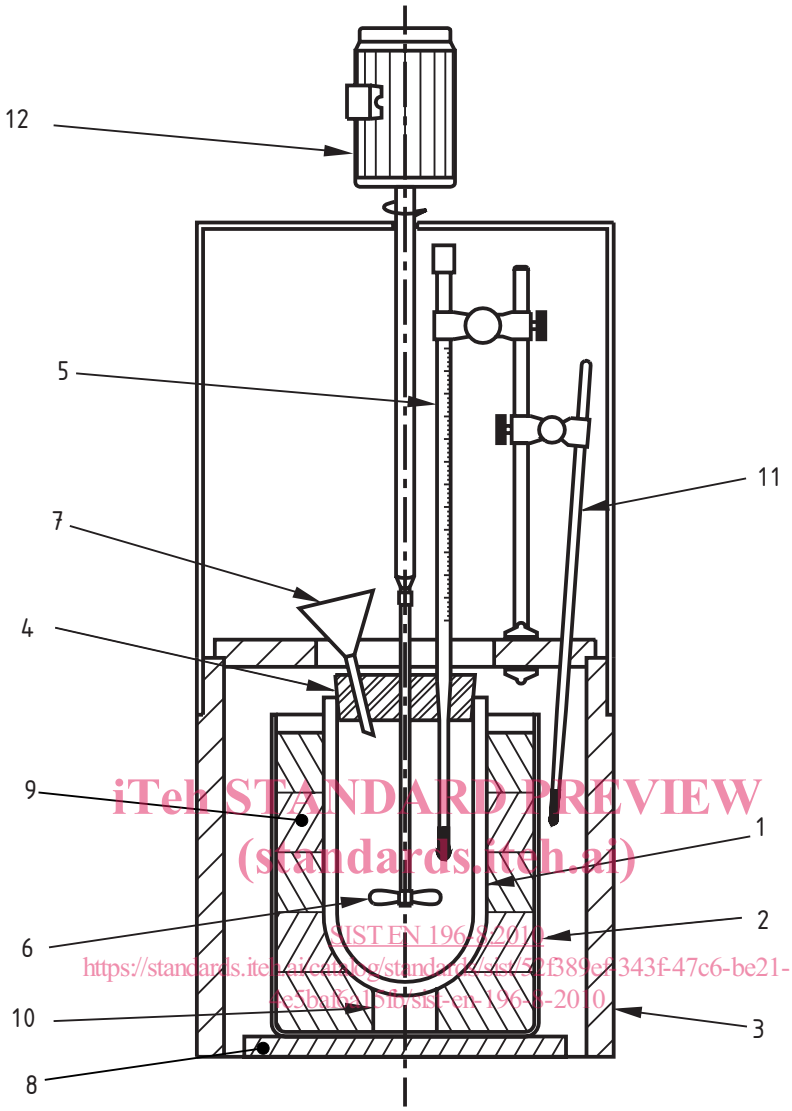
a) ***Dissolution vessel***, consisting of: an insulated flask (e.g. Dewar flask), placed either in a heat insulated container set inside a box constructed of insulating material (e.g. wood, plastics), or immersed in a thermostatic water bath regulated to ± 0,2 °C; and an insulated stopper (made of cork or plastics) through which holes are provided for the thermometer, the stirrer and the funnel used for introducing the sample. The insulation of the calorimeter shall ensure that the thermal leakage coefficient, *K* (determined in accordance with 6.3), is less than 0,06 K per 15 min for each Kelvin above ambient temperature. The internal surface of the flask, that part of the thermometer immersed in the acid mixture and the lower part of the stopper, shall be acid mixture resistant.

b) ***Thermometer***, either a Beckmann thermometer with a 5 °C to 6 °C scale and subdivisions every 0,01 °C or other measurement apparatus of an equal or higher accuracy such as a thermistor or platinum resistance thermometer, positioned such that the end of the thermometer is at least 4 cm below the level of the liquid surface.

Express temperature readings with a resolution of ± 0,002 °C. Adjust the zero of the Beckmann thermometer so that the upper limit of the scale is approximately the ambient, or water bath, temperature. Calibrate the thermometer in a thermostatic bath against a 0,01 °C graduated and calibrated thermometer.

c) ***Funnel***, of acid mixture resistant plastics, through which the sample is introduced into the flask and which extends below the lower part of the stopper by 5 mm to 6 mm and is sealed during the test.

d) ***Stirrer***, of acid mixture resistant plastics, positioned such that the blades are as close as possible to the bottom of the flask and rotated by a motor at a speed of (450 ± 50) min^{-1}. The motor shall be low power rated (e.g. a motor of a few watts) so as to prevent any excessive heat emission from affecting measurements.



Key

1 flask	5 thermometer	9 insulating material
2 container	6 stirrer	10 flask support
3 box	7 funnel	11 ambient thermometer
4 stopper	8 support	12 stirrer motor

Figure 1 — Typical heat of solution calorimeter apparatus

5.2 Thermostatic bath, e.g. water bath, for storing the hydrated samples at a temperature of (20,0 ± 0,2) °C.

5.3 Mortar or electric grinder, for crushing the hydrated samples.

5.4 Plastics or glass vials, of capacity approximately 20 ml, for storing the hydrated paste.

5.5 Sieve, of mesh size 125 µm.

5.6 Sieve, of mesh size 600 µm.

5.7 Chronometer, graduated in seconds, for timing the temperature readings.

5.8 Two platinum crucibles, of capacity approximately 20 ml, for ignition of samples.

5.9 Electric furnace, naturally ventilated, capable of operating at (950 ± 25) °C, for ignition of samples.

5.10 Analytical balance, capable of weighing to an accuracy of ± 0,000 1 g.

5.11 Balance, of capacity 2 kg, capable of weighing to an accuracy of ± 0,2 g.

6 Calorimeter calibration

6.1 Principle

Calibration of the calorimeter is carried out in order to determine its thermal capacity and thermal leakage coefficient. These characteristics are determined by dissolving the ignited zinc oxide (4.2) in the acid mixture (4.1) and measuring the temperature of the calorimeter at fixed intervals of time. The temperature of acid mixture shall be so set that after the dissolution reaction the calorimeter temperature is at least 0,5 °C below the ambient temperature. Where a water bath is used the temperature of the bath is considered to be the ambient temperature for the calorimeter.

6.2 Procedure

Measure a quantity of acid mixture (4.1) by mass or volume to ± 0,2 % such that the liquid level will be approximately 2 cm below the calorimeter stopper. Place the acid mixture in the flask. Immediately before the determination of the thermal capacity, ignite the zinc oxide at (950 ± 25) °C for a maximum of 5 min and cool in a desiccator to room temperature. The quantity of zinc oxide to be used, weighed to ± 0,000 1 g, is that required to satisfy Equation (1):

$$\frac{Mass\ of\ acid\ mixture}{Mass\ of\ zinc\ oxide} = 60 \pm 1 \qquad (1)$$

Carry out the procedure as follows:

a) Preliminary period
Stir the acid mixture for 40 min to 50 min.

b) Pre-period
When the rate of temperature increase is constant, start the timing using the chronometer (5.7) and record the initial temperature, $\overline{T}_{-15}$

c) Sample introduction
After 15 min record the temperature, T_0, and immediately add the zinc oxide sample to the acid mixture, taking not more than 1 min.