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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
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Contents

Foreword

This document (FprEN 196-8:2009) 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 Unique Acceptance Procedure.

This document will supersede EN 196-8:2003.

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*

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*

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

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 7 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, *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. 7 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 one hour. 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 (eg. 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 kelvins 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