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Cementprovning –
Del 9: Värmeutveckling – Semiadiabatisk metod

Methods of testing cement –
Part 9: Heat of hydration – Semi-adiabatic method

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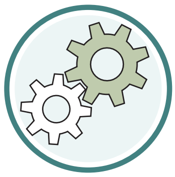

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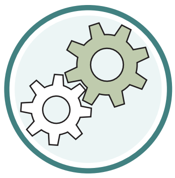

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Europastandarden EN 196-9:2010 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 196-9:2010.

Denna standard ersätter SS-EN 196-9, utgåva 1.

The European Standard EN 196-9:2010 has the status of a Swedish Standard. This document contains the official English version of EN 196-9:2010.

This standard supersedes the Swedish Standard SS-EN 196-9, edition 1.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Cement och byggkalk, SIS/TK 185.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 196-9

March 2010

ICS 91.100.10

Supersedes EN 196-9:2003

English Version

Methods of testing cement - Part 9: Heat of hydration - Semi-adiabatic method

Prüfverfahren für Zement - Teil 9: Hydratationswärme - Teiladiabatisches Verfahren

This European Standard was approved by CEN on 21 February 2010.

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 196-9:2010: E

Contents

Foreword

This document (EN 196-9:2010) 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.

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

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

This document supersedes EN 196-9:2003.

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*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard describes a method of measuring the heat of hydration of cements by means of semi-adiabatic calorimetry, also known as the Langavant method. The aim of the test is the continuous measurement of the heat of hydration of cement during the first few days. The heat of hydration is expressed in joules per gram of cement.

This standard is applicable to all cements and hydraulic binders, whatever their chemical composition, with the exception of quick-setting cements.

NOTE 1 An alternative procedure, called the solution method, is described in EN 196-8. Either procedure can be used independently.

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

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 196-1, *Methods of testing cement — Part 1: Determination of strength*

EN 573-3:2009, *Aluminium and aluminium alloys — Chemical composition and form of wrought products — Part 3: Chemical composition and form of products*

3 Principle

The semi-adiabatic method consists of introducing a sample of freshly made mortar into a calorimeter in order to determine the quantity of heat emitted in accordance with the development of the temperature. At a given point in time the heat of hydration of the cement contained in the sample is equal to the sum of the heat accumulated in the calorimeter and the heat lost into the ambient atmosphere throughout the period of the test.

The temperature rise of the mortar is compared with the temperature of an inert sample in a reference calorimeter. The temperature rise depends mainly on the characteristics of the cement and is normally between 10 K and 50 K.

4 Apparatus

4.1 Calorimeter, consisting of an insulated flask sealed with an insulated stopper and encased in a rigid casing which acts as its support (see Figure 1). Both the calorimeter used for the test and that for the reference (see 4.2) shall have the following construction and characteristics:

a) ***Insulated flask*** (e.g. Dewar flask), made of silver plated borosilicate glass; cylindrical in shape with a hemispherical bottom. The internal dimensions shall be approximately 95 mm in diameter and 280 mm in depth; and external diameter of approximately 120 mm. A rubber disc of approximately 85 mm diameter and 20 mm thickness shall be placed at the bottom of the flask to act as support for the sample container and evenly distribute the load on the glass wall.

b) ***Very rigid casing***, having a sufficiently wide base to ensure good stability of the whole unit (e.g. made of duralumin, 3 mm thick). The flask shall be separated from the lateral walls of the casing by approximately 5 mm air space and rest on a support 40 mm to 50 mm thick made of a material having low thermal conductivity (e.g. expanded polystyrene). The upper edge of the flask shall be protected by a rubber gasket above, and in contact with, which shall be a ring, not less than 5 mm thick, made of low thermal conductivity material, fixed to the calorimeter casing. The ring shall serve to locate the flask in position and provide a bearing surface for the stopper so as to ensure the tightness of the locking device.

c) ***Insulating stopper***, made of three parts:

1) the lower part, which is inserted into the flask and which serves to provide a maximum prevention of heat loss into the external atmosphere. It shall be cylindrical in shape, of diameter equal to the internal diameter of the flask, and in thickness approximately 50 mm. It shall be made of expanded polystyrene (class 20 kg/m^3 approximately) or of another material of similar thermal characteristics. Its base can be protected by a layer of plastic (e.g. polymethyl methacrylate), approximately 2 mm thick;

2) the central part, which serves to ensure the tightness of the calorimeter whilst contributing to the reduction of losses, shall consist of a foam rubber disc 120 mm in diameter;

3) the upper part, which is intended to ensure the correct and consistent positioning of the stopper unit against the Dewar flask, shall consist of a rigid casing incorporating a snap locking device in such a way as to compress the foam rubber central part ensuring the tight fitting of the stopper.

d) ***Performance characteristics.*** The coefficient of total heat loss of the calorimeter shall not exceed 100 $J{\cdot}h^{-1}{\cdot}K^{-1}$ for a temperature rise of 20 K. This value, together with the thermal capacity, shall be determined in accordance with the calibration procedure given in Annex A (see A.3.1).

Recalibration is necessary:

— at least every four years or after 200 tests;

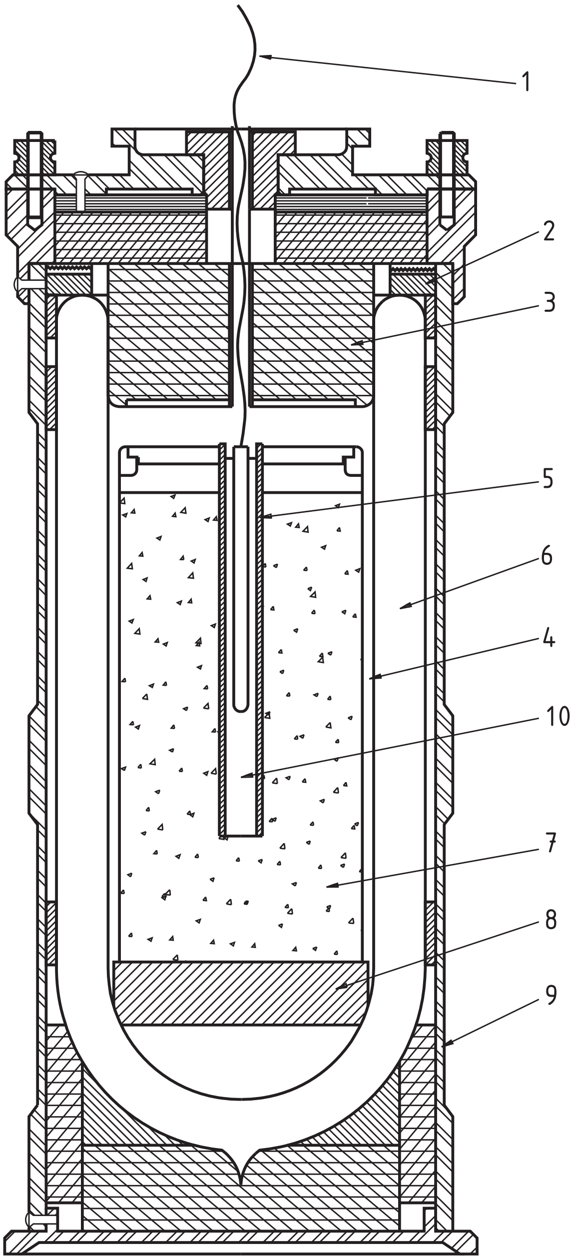
— whenever deterioration occurs in the calorimeter or an insulating component.

In order not to impair the insulation of the calorimeter, the temperature of the mortar under test shall not exceed 75 °C.

4.2 Reference calorimeter, having the same construction and characteristics as the test calorimeter (see 4.1).

It shall contain a mortar box in which is a sample of mortar mixed at least 12 months previously (and is considered to be inert).

NOTE Where an inert sample is not available an aluminium cylinder of the same thermal capacity as the mortar box and mortar sample may be used.



Key

1 platinum resistance thermometer

2 gasket

3 insulating stopper

4 mortar box

5 thermometer pocket

6 dewar flask

7 mortar sample

8 rubber disc

9 rigid casing

10 oil

Figure 1 — Typical calorimeter

4.3 Platinum resistance thermometers, for the reference calorimeter and each test calorimeter, having a minimum range 19 °C to 75 °C.

If the conductors of the electrical resistor are made of copper they shall have a sectional area not greater than 0,25 mm^2 in the part which passes through the stopper. If they are made of another metal the total thermal resistance per centimetre of conductor shall be greater than 0,10 $K \cdot mW^{-1}$ (thermal resistance equivalent to that of a copper conductor with a sectional area of 0,25 mm^2 and 1 cm in length).

The thermal output of the thermometer shall not exceed 3 mW. Direct current supply, which constitutes a power input, shall be avoided if the thermal output exceeds 0,2 mW. It is advisable to ensure the accuracy of the overall temperature measuring and recording equipment.

The temperature of the test sample shall be measured to an accuracy of ± 0,3 °C.

Where the calorimeter is calibrated in situ with the conductors used for the tests of heat of hydration, the total sectional area of the conductors will be a maximum of 0,80 mm^2 (four wires 0,5 mm in diameter), but shall be such that the coefficient of heat loss of the calorimeter is less than 100 $J \cdot h^{-1} \cdot K^{-1}$ for a temperature rise of 20 K (see A.3.1.1).

The protective sheath of these conductors shall be made of a material having a low thermal conductivity.

4.4 Mortar box, consisting of a cylindrical container fitted with a cover, having a volume of approximately 800 cm^3, designed to contain the sample of mortar under test.

The mortar box, discarded after each test, shall be impermeable to water vapour. This shall be checked in use by weighing the mortar box after each test (see 5.2.3). It shall be made of electrically counter welded tin plate of nominal thickness 0,3 mm; have a diameter of approximately 80 mm and a height of approximately 165 mm. Its height shall be designed to provide an air space of approximately 10 mm between the top of the mortar box and the stopper.

The lid of the mortar box shall be fitted with a central thermometer pocket in the form of a cylindrical pipe, closed at its base. The internal diameter of the pocket shall be slightly greater than that of the thermometer. Its length shall be approximately 100 mm to 120 mm and enable it to extend to the centre of the test sample.

4.5 Temperature recording apparatus, capable of recording the measurements taken by each thermometer.

4.6 Mortar mixing apparatus, conforming to EN 196-1.

5 Determination of the heat of hydration

5.1 Laboratory

The laboratory where the mortar is mixed shall be maintained at a temperature of (20 ± 2) °C.

The room where the test is carried out shall be maintained at a temperature of (20,0 ± 1,0) °C. The measured temperature of the reference calorimeter shall be considered to be the ambient temperature and shall be maintained during the test within ± 0,5 °C. The distance between each of the calorimeters shall be approximately 120 mm. The velocity of the ventilation air around the calorimeters shall be less than 0,5 $m \cdot s^{-1}$.

When several tests are being carried out simultaneously, at least one reference calorimeter shall be provided for every six test calorimeters; where several test calorimeters are used with one reference calorimeter, a hexagonal arrangement shall be used with the reference calorimeter in the centre.