SLOVENSKI STANDARD

SIST EN 196-9:2004

01-januar-2004

Metode preskušanja cementa – 9. del: Toplota hidratacije – Semiadiabatska metoda

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

Prüfverfahren für Zement - Teil 9: Hydratationswärme - Teildiabetisches Verfahren

Méthodes d'essais des ciments - Partie 9: Chaleur d'hydratation - Méthodes semi-adiabatique

Ta slovenski standard je istoveten z: EN 196-9:2003

ICS:

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

SIST EN 196-9:2004 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 196-9

October 2003

ICS 91.100.10

English version

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

Méthodes d'essai des ciments - Partie 9: Chaleur d'hydratation - Méthode semi-adiabatique

Prüfverfahren für Zement - Teil 9: Hydratationswärme - Teildiabetisches Verfahren

This European Standard was approved by CEN on 25 March 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 196-9:2003 E

Contents

Foreword

This document (EN 196-9:2003) has been prepared by Technical Committee CEN/TC 51, '*Cement and building limes*', the secretariat of which is held by IBN.

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of an identical text or by endorsement, at the latest by April 2004, and conflicting national standards shall be withdrawn at the latest by April 2004.

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 time and soundness*

EN 196-5 *Methods of testing cement — Part 5: Pozzolanicity test for pozzolanic cements*

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 196-21: *Methods of testing cement — Part 21: Determination of the chloride, carbon dioxide and alkali content of cement*

EN 196-21 is currently being revised and incorporated into EN 196-2.

Another document, ENV 196-4 *Methods of testing cement — Part 4: Quantitative determination of constituents*, has been drafted and will be published as a CEN Technical Report.

Annex A is normative and annex B is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, 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 7 days for the heat of solution method (EN 196-8).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

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

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) *an 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) *a 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) *an insulating stopper*, made of three parts.

— 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.

— 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.

— 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·h^{-1}·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:

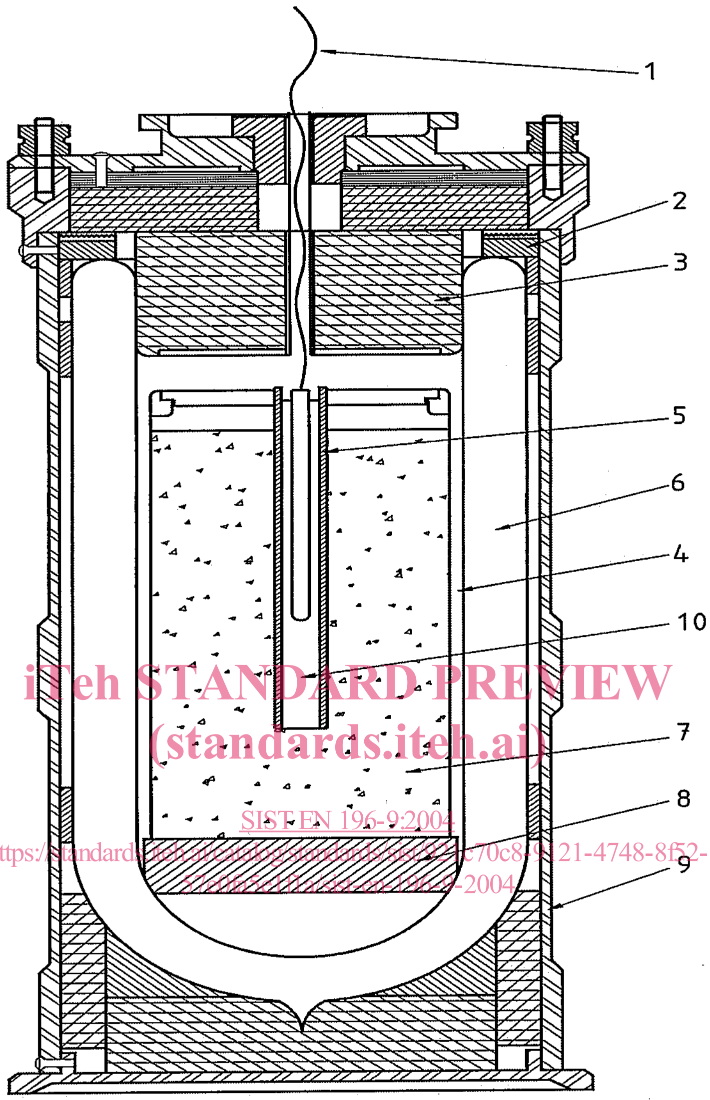
— at least every 4 years or after 200 tests;

— 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 (4 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 12 cm. 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.

5.2 Procedure

5.2.1 Mortar composition

The composition of the mortar shall be in accordance with EN 196-1 and the test sample shall have a total mass of (1 575 ± 1) g. Each batch of mortar to be mixed shall consist of (360,0 ± 0,5) g cement; (1 080 ± 1) g sand from a sample of CEN standard sand complying with the requirements in EN 196-1; and (180,0 ± 0,5) g distilled or deionised water.

NOTE Since it is not possible to recover all the material added to the mixer bowl the mortar batch to be mixed should be slightly more than 1 575 g, the proportions by mass of the various constituents being maintained.

5.2.2 Mixing

The cement, the water, the sand, the mortar box, the mixer bowl and the other instruments coming into contact with the mortar shall be stored in the test room.

With the mixer in the operating position, pour the sand and then the cement into the mixer bowl; homogenise the mixture of sand and cement for 30 s at low speed; pour in the water, record the time, and mix immediately at low speed for 60 s; set the mixer to high speed and mix for a further 60 s.

NOTE In order to avoid thermal losses, it is recommended to carry out the mixing in a relatively short time. It is for this reason that the mixing time prescribed in standard EN 196-1 has been shortened.

5.2.3 Positioning of the test sample

Immediately after mixing weigh (1 575 ± 1) g of mortar into the box (see 4.4) which has previously been weighed, with its lid, to an accuracy of ± 0,5 g. Place the lid in position making sure that it seals tightly. Fill the thermometer pocket with (2,5 ± 0,5) cm^3 of oil (e.g. thin mineral oil) in order to improve the thermal contact between the test sample and the thermometer.

Weigh the filled mortar box, to an accuracy of ± 0,5 g in order to be able to check at the end of the test for any leakage of water vapour. Immediately after weighing place the mortar box in the test calorimeter (see 4.1) and close with the stopper. Immediately place the thermometer (see 4.3) in position in the thermometer pocket, so that it is approximately in the centre of the test sample. Seal the opening across the stopper by means of the locking device.

NOTE The stopper can also be sealed by means of a flexible sealant or mastic.

Not more than 6 min shall be taken for the mixing and the positioning of the test sample.

At the end of the test the mortar box with its contents shall be weighed again, to an accuracy of ± 0,5 g. If a reduction in mass of more than 2 g is found the test is not valid and shall be repeated.

5.3 Measurement of heating

The time of addition of water shall be taken as the start of timing. The measurement of heating consists of reading, at specific moments in time, the temperature of the test sample and that of the inert sample located in the reference calorimeter (see 4.2).

At least one reading shall be taken in the first 30 min followed by readings at least every: 1 h for the first 24 h; 4 h during the second day; and 6 h until the expiry of the selected test period. The frequency of these measurements may be increased according to the characteristics of the cement being tested. The time of each temperature reading shall be recorded in hours and minutes.

At each temperature reading the temperature rise of the test sample, θ_t, shall be determined as the difference between the temperature of the test sample, T_s, and that of the inert sample, T_r, in the reference calorimeter.

6 Calculation of the heat of hydration

NOTE A worked example is given in annex B.

6.1 Principles of calculations

The heat of hydration, Q, in joules per gram of cement, at elapsed time, t, is calculated from equation (1):

$$Q = \frac{c}{m_c}\theta_t + \frac{1}{m_c}\int_0^t \alpha \cdot \theta_t \cdot dt \qquad (1)$$

where

m_c is the mass of cement contained in the test sample, in grams;

t is the hydration time, in hours;

c is the total thermal capacity of the calorimeter (see 6.2), in joules per kelvin;

α is the coefficient of heat loss of the calorimeter (see 6.3), in joules per hour per kelvin;

θ_t is the temperature rise of the test sample (see 5.3) at time t, in kelvin.

The first term in equation (1) represents the heat accumulated in the calorimeter A, and the second term represents the heat lost into the ambient atmosphere B (each in joules per gram of cement).

Equation (1) can be simplified to equation (2):

$$Q = \frac{c}{m_c}\theta_k + \frac{1}{m_c}\sum_{i=0}^{i=n}\overline{\alpha_i}\cdot\overline{\theta_i}\cdot\Delta t_i \qquad (2)$$

where $\overline{\alpha_i}$ and $\overline{\theta_i}$ are the mean values of the coefficient of heat loss and temperature rise of test sample during period of time, Δt_i.

6.2 Calculation of the heat accumulated in the calorimeter

Heat accumulated in the calorimeter, A, in joules per gram of cement, shall be calculated from the total thermal capacity, c, of the calorimeter, the mass of cement, m_c, and the temperature rise of the test sample, θ_t, at point in time, t by equation (3):

$$A = \frac{c}{m_c}\theta_t \qquad (3)$$

where the total thermal capacity of the calorimeter, c, including the mortar box and mortar sample under test is expressed by equation (4):

$$c = 0{,}8(m_c + m_s) + 3{,}8m_w + 0{,}50m_b + \mu \qquad (4)$$