

Methods of testing cement —

Part 9: Heat of hydration — Semi-adiabatic method

The European Standard EN 196-9:2003 has the status of a
British Standard

ICS 91.100.10

National foreword

This British Standard is the official English language version of EN 196-9:2003.

The UK participation in its preparation was entrusted by Technical Committee B/516, Cement and lime, to Subcommittee B/516/12, Sampling and testing, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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This British Standard, was published under the authority of the Standards Policy and Strategy Committee on 16 February 2004

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 21 and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments

© BSI 16 February 2004

ISBN 0 580 43409 5

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.

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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.



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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.
 - $\frac{3}{4}$ 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/m³ 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.
 - $\frac{3}{4}$ 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.
 - $\frac{3}{4}$ 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).

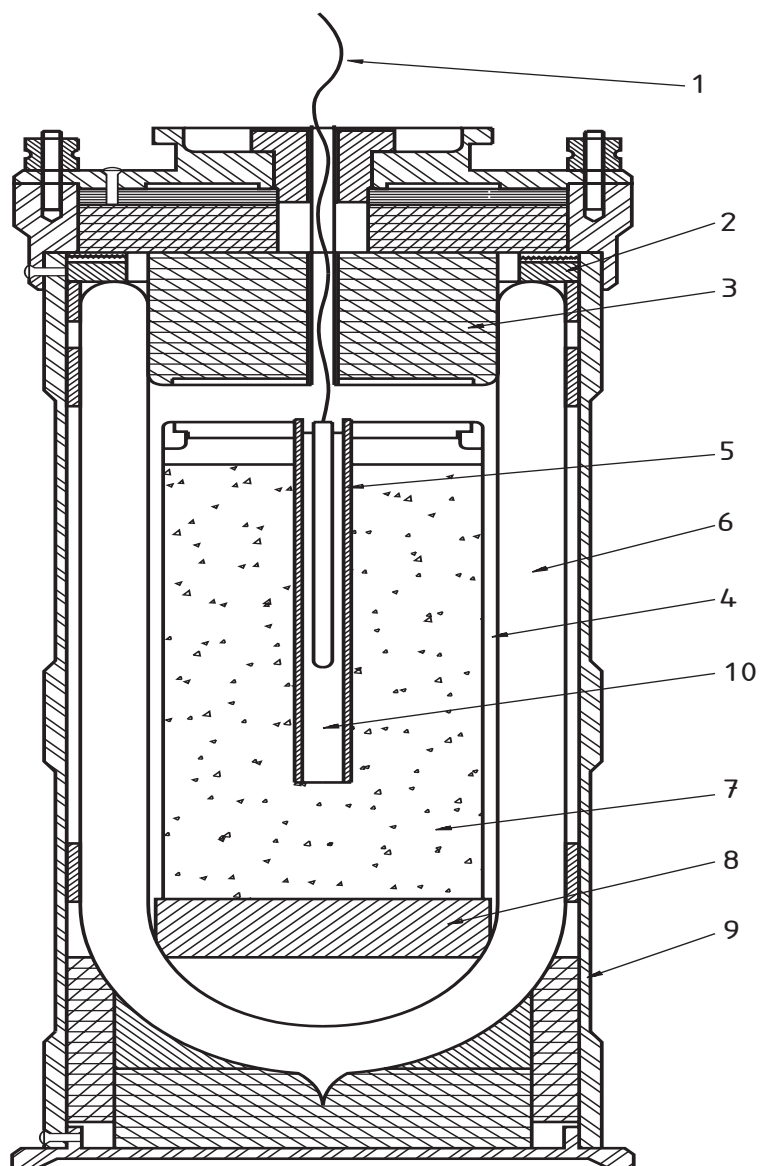
Recalibration is necessary:

- $\frac{3}{4}$ at least every 4 years or after 200 tests;
- $\frac{3}{4}$ 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 | 5 Thermometer pocket | 9 Rigid casing |
| 2 Gasket | 6 Dewar flask | 10 Oil |
| 3 Insulating stopper | 7 Mortar sample | |
| 4 Mortar box | 8 Rubber disc | |

Figure 1 — Typical calorimeter