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**Methods of testing cement —  
Determination of the heat of hydration —  
Part 2:  
Semi-adiabatic method**

*Méthodes d'essai des ciments — Détermination de la chaleur  
d'hydratation*

**iTeh STANDARD PREVIEW**  
*Partie 2: Méthode semi-adiabatique*  
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ISO 29582-2:2009

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 29582-2 was prepared by Technical Committee ISO/TC 74, *Cement and lime*.

ISO 29582 consists of the following parts, under the general title *Methods of testing cement — Determination of the heat of hydration*:

— *Part 1: Solution method*

— *Part 2: Semi-adiabatic method*

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# Methods of testing cement — Determination of the heat of hydration —

## Part 2: Semi-adiabatic method

### 1 Scope

This part of ISO 29582 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 part of ISO 29582 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 ISO 29582-1. 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 in this part of ISO 29582 compared with 7 d for the heat of solution method in ISO 29582-1.

### 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.

ISO 679<sup>1)</sup>, *Cement — Test methods — 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 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.

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1) To be published. (Revision of ISO 679:1989)

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).

The calorimeter shall have the following performance characteristics. The coefficient of total heat loss of the calorimeter shall not exceed  $100 \text{ J}\cdot\text{h}^{-1}\cdot\text{K}^{-1}$  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 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.

Both the calorimeter used for the test and that for the reference (see 4.2) shall have the following construction and characteristics.

a) **Flask**, insulated (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 the external diameter, approximately 120 mm. A rubber disc of approximately 85 mm in diameter and 20 mm thick 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) **Casing**, very rigid, 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 an air space of approximately 5 mm 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 which, and in contact with it, shall be a ring not less than 5 mm thick, made of a 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.

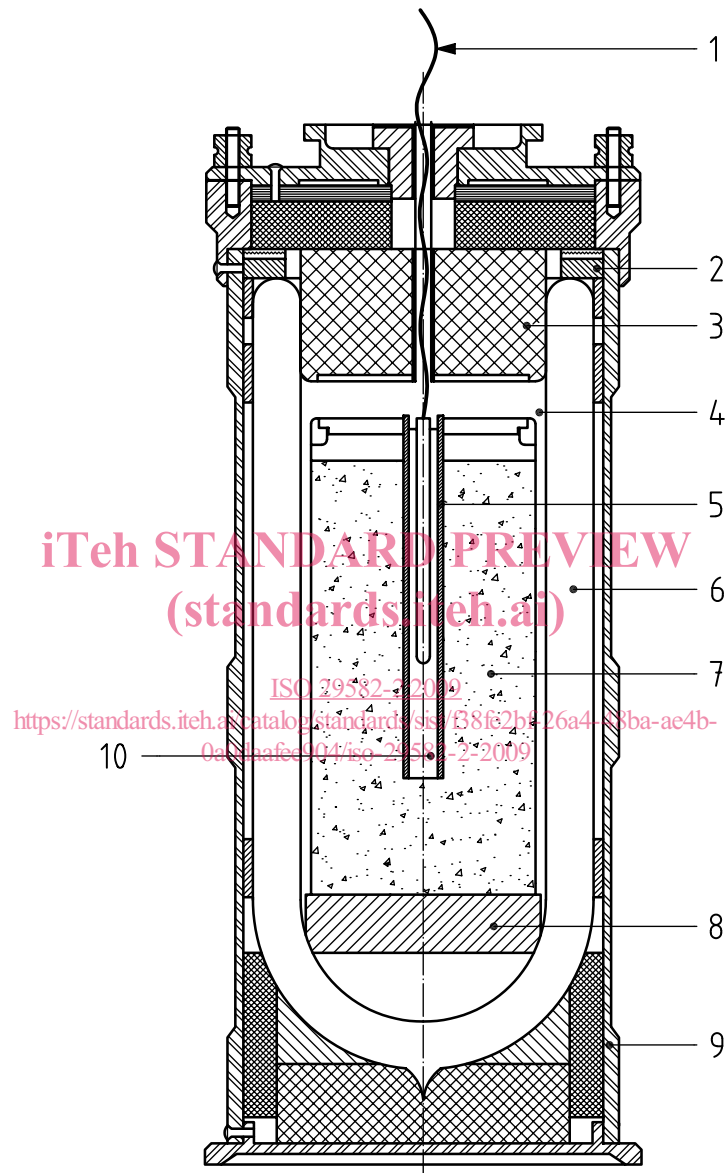
c) **Stopper**, insulating, made of the following three parts.

- 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 approximately 50 mm thick. It shall be made of expanded polystyrene (class 20  $\text{kg}/\text{m}^3$  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.

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

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

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 | 6  | dewar flask   |
| 2 | gasket                          | 7  | mortar sample |
| 3 | insulating stopper              | 8  | rubber disc   |
| 4 | mortar box                      | 9  | rigid casing  |
| 5 | thermometer pocket              | 10 | oil           |

**Figure 1 — Typical calorimeter**

**4.3 Thermometers**, platinum resistance, for the reference calorimeter and each test calorimeter, having a minimum range of 19 °C to 75 °C. If the conductors of the electrical resistor are made of copper, they shall have a cross-sectional area not greater than 0,25 mm<sup>2</sup> 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·mW<sup>-1</sup> (thermal resistance equivalent to that of a copper conductor with a sectional area of 0,25 mm<sup>2</sup> and 1 cm long).

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 cross-sectional area of the conductors will be a maximum of 0,80 mm<sup>2</sup> (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·h<sup>-1</sup>·K<sup>-1</sup> 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<sup>3</sup>, 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 with a nominal thickness of 0,3 mm and shall 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. (standards.iteh.ai)

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 ISO 679.

## 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·s<sup>-1</sup>.

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 ISO 679 and the test sample shall have a total mass of  $(1\,575 \pm 1)$  g. Each batch of mortar being mixed shall consist of  $(360,0 \pm 0,5)$  g of cement;  $(1\,080 \pm 1)$  g of sand from a sample of standard sand in accordance with the requirements of ISO 679; and  $(180,0 \pm 0,5)$  g of distilled or deionized water.

Since it is not possible to recover all the material added to the mixer bowl, the mortar batch being 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; homogenize 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.

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 ISO 679 has been shortened.

### 5.2.3 Positioning of the test sample

Immediately after mixing, weigh  $(1\,575 \pm 1)$  g of mortar into the box (4.4), which has previously been weighed, with its lid, to an accuracy of  $\pm 0,5$  g. Place the lid in position, making sure that it seals tightly. Fill the thermometer pocket with  $(2,5 \pm 0,5)$  cm<sup>3</sup> 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  $\pm 0,5$  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 (4.1) and close with the stopper. Immediately place the thermometer (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.

Do not take more than 6 min for the mixing and the positioning of the test sample.

At the end of the test, weigh the mortar box with its contents again, to an accuracy of  $\pm 0,5$  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 (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; every 4 h during the second day; and every 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,  $\theta_t$ , 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,  $Q_{\text{hyd}}$ , expressed in joules per gram of cement, at elapsed time,  $t$ , is calculated from Equation (1):

$$Q_{\text{hyd}} = \frac{c}{m_c} \theta_t + \frac{1}{m_c} \int_0^t \alpha \cdot \theta_t dt \quad (1)$$

where

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

$t$  is the hydration time, expressed in hours;

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

$\alpha$  is the coefficient of heat loss of the calorimeter (see 6.3), expressed in joules per hour per kelvin;

$\theta_t$  is the difference in the temperature of the test calorimeter compared with that of the reference calorimeter (see 5.3) at time,  $t$ , expressed in kelvin.

The first term in Equation (1) represents the heat,  $Q_A$ , accumulated in the calorimeter, and the second term represents the heat,  $Q_B$ , lost into the ambient atmosphere, both expressed in joules per gram of cement.

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

$$Q_{\text{hyd}} = \frac{c}{m_c} \theta_t + \frac{1}{m_c} \sum_{i=1}^n \bar{\alpha}_i \cdot \bar{\theta}_i \cdot \Delta t_i \quad (2)$$

where

$\bar{\alpha}_i$  is the mean value of the coefficient of heat loss of test sample during period of time,  $\Delta t_i$ ;

$\bar{\theta}_i$  is the mean value of the temperature rise of test sample during period of time,  $\Delta t_i$ .

### 6.2 Calculation of the heat accumulated in the calorimeter

The heat,  $Q_A$ , expressed in joules per gram of cement, accumulated in the calorimeter shall be calculated from the total thermal capacity,  $c$ , of the calorimeter, the mass of cement,  $m_c$ , and the difference in the temperature of the test calorimeter compared with that of the reference calorimeter,  $\theta_t$ , at point in time,  $t$ , by Equation (3):

$$Q_A = \frac{c}{m_c} \theta_t \quad (3)$$

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

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

where

0,8 is the thermal capacity per unit of mass of cement plus sand, expressed in joules per kelvin per gram;

- 3,8 is the average thermal capacity per unit of mass of water, expressed in joules per kelvin per gram;
- 0,50 is the thermal capacity per unit of mass of the mortar box, expressed in joules per kelvin per gram;
- $\mu$  is the thermal capacity of the empty calorimeter, expressed in joules per kelvin;
- $m_c$  is the mass of cement, expressed in grams;
- $m_s$  is the mass of sand, expressed in grams;
- $m_w$  is the mass of water, expressed in grams;
- $m_b$  is the mass of empty mortar box plus lid, expressed in grams.

NOTE Bound water has a thermal capacity per unit of mass lower than  $4,18 \text{ J}\cdot\text{K}^{-1}\cdot\text{g}^{-1}$ .

### 6.3 Calculation of heat lost to ambient atmosphere

Heat lost shall be calculated for known periods of hydration represented by the time elapsed between successive measurements of the temperature of the test sample; see 5.3.

The coefficient of heat loss,  $\alpha$ , expressed in joules per hour per kelvin, of the calorimeter is given by Equation (5):

$$\alpha = a + b \cdot \theta \quad (5)$$

where

- $a$  and  $b$  are the calorimeter calibration constants; see A.3.1.1,
- $\theta$  is the temperature rise, expressed in kelvin.

The heat lost into the ambient atmosphere,  $Q_B$ , expressed in joules per gram of cement, during the period of hydration,  $t_n$ , is given by Equation (6):

$$Q_B = \frac{1}{m_c} \sum_{i=1}^n \bar{\alpha}_i \cdot \bar{\theta}_i \cdot \Delta t_i \quad (6)$$

where

- $\Delta t_i$  is the elapsed time between the measurement of temperature at point in time,  $t_{i-1}$ , and the next measurement at point in time,  $t_i$ , expressed in hours;
- $\bar{\theta}_i$  is the average of the temperature rise of the test sample, between times  $t_i$  and  $t_{i-1}$ , expressed in kelvin, as given by Equation (7):

$$\bar{\theta}_i = \frac{\theta_i + \theta_{i-1}}{2} \quad (7)$$

where  $\bar{\alpha}_i$  is the average coefficient of total heat loss of the calorimeter (see Equation 5) in the period of time,  $\Delta t_i$ , expressed in joules per hour per kelvin, as given by Equation (8):

$$\bar{\alpha}_i = a + b \cdot \bar{\theta}_i \quad (8)$$