INTERNATIONAL STANDARD

ISO 29582-1

First edition
2009-07-15

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

Part 1:
Solution method

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

Partie 1: Méthode par dissolution

© ISO 2009

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

---

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 29582-1:2009
https://standards.iteh.ai/catalog/standards/sist/db303850-fd4d-4e53-b5da-
60b1b6c6049a/iso-29582-1-2009

Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 29582-1:2009
https://standards.iteh.ai/catalog/standards/sist/db303850-fd4d-4e53-b5da-
60b1b6c6049a/iso-29582-1-2009

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 29582-1 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*

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

Part 1:
Solution method

1 Scope

This part of ISO 29582 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 part of ISO 29582 is applicable to cements and hydraulic binders, whatever their chemical composition.

NOTE 1   Another procedure, called the semi-adiabatic method, is described in ISO 29582-2. Either procedure can be used independently.

NOTE 2   It has been demonstrated that the best correlation between the two methods is obtained at 7 d for the solution method in this part of ISO 29582 compared with 41 h for the semi-adiabatic method in ISO 29582-2.

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 of 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 of 0,40;

⸺ use of neat cement paste;

⸺ storage at a constant temperature of $(20,0 \pm 0,2)$ °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   Reagents and materials

4.1   **Acid mixture**, analytical-reagent quality, obtained by adding 2,760 g of 40 % hydrofluoric acid (HF) per 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.

4.2   **Acid mixture**, proposed alternative, containing ammonium fluoride (NH$_4$F) instead of hydrofluoric acid, prepared as follows.

Place about 400 g of (2,00 ± 0,01) mol/l nitric acid and 7,2 g of ammonium fluoride in the flask. Then, add (2,00 ± 0,01) mol/l nitric acid to make a total mass of 425,0 g ± 0,1 g. Any other procedure in the preparation of acid mixture can be adopted, provided that the same mass ratio of nitric acid to ammonium fluoride is conserved.

The quantity (mass or volume) of acid used, which is common to all tests, shall be measured to ± 0,2 %.

In the case of a dispute , only the reference acid mixture containing hydrofluoric acid is used.

4.3   **Zinc oxide** (ZnO), analytical quality, used to determine the thermal capacity of the calorimeter and prepare as follows.

Weigh 40 g to 50 g. Ignite at (950 ± 25) °C for 1 h. Cool in a desiccator. Grind to pass a 125 µm sieve. Store in a desiccator.

4.4   **Anhydrous cement**, from which metallic iron has been removed with a magnet, stored 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.

4.5   **Hydrated cement**, test sample prepared as follows.

Vigorously mix, either manually or mechanically, (100,0 ± 0,1) g of anhydrous cement with (40,0 ± 0,1) g of distilled or deionized water for 3 min at ambient temperature. Place the resulting paste in plastic or glass cylindrical vials (three for each hydration period being 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.
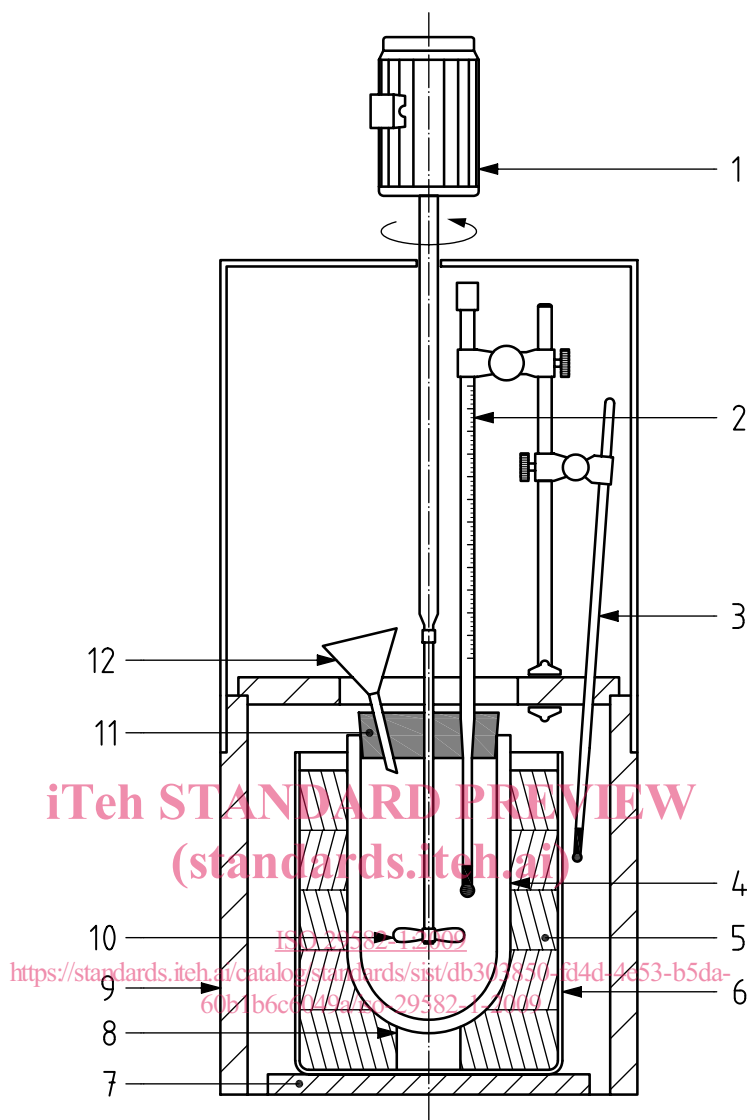
5   Apparatus

5.1   **Calorimeter**, comprised of the following (see Figure 1).

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.

5.1.1   **Dissolution vessel**, consisting of an insulated flask (e.g. 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 plastic) 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 K 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.

Key

1	stirrer motor	7	support
2	thermometer	8	flask support
3	ambient thermometer	9	box
4	flask	10	stirrer
5	insulating material	11	stopper
6	container	12	funnel

Figure 1 — Typical heat-of-solution calorimeter apparatus

5.1.2 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.

5.1.3 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.

5.1.4 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 have a low power rating (e.g. a motor of a few watts) so as to prevent any excessive heat emission from affecting measurements.

5.2 Thermostatic bath, e.g. water bath, for storing the hydrated samples at a temperature of $(20,0 \pm 0,2)$ °C.

5.3 Mortar or **electric grinder**, for crushing the hydrated samples.

5.4 Plastic or **glass vials**, of capacity approximately 20 ml, for storing the hydrated paste.

5.5 Sieve, of mesh size 125 µm.

5.6 Sieve, of mesh size 600 µm.

5.7 Chronometer, graduated in seconds, for timing the temperature readings.

5.8 Two platinum crucibles, of capacity approximately 20 ml, for ignition of samples.

5.9 Electric furnace, naturally ventilated, capable of operating at (950 ± 25) °C, for ignition of samples.

5.10 Analytical balance, capable of weighing to an accuracy of $\pm 0,000\,1$ g.

5.11 Balance, of capacity 2 kg, capable of weighing to an accuracy of $\pm 0,2$ g.

6 Calorimeter calibration

6.1 Principle

Calibration of the calorimeter is carried out in order to determine its thermal capacity and thermal leakage coefficient. These characteristics are determined by dissolving the ignited zinc oxide (4.2) in the acid mixture (4.1) and measuring the temperature of the calorimeter at fixed intervals of time. The temperature of the acid mixture shall be so set that after the dissolution reaction, the calorimeter temperature is at least 0,5 °C below the ambient temperature. Where a water bath is used, the temperature of the bath is considered to be the ambient temperature for the calorimeter.

6.2 Procedure

Measure a quantity of acid mixture (4.1) by mass or volume to $\pm 0,2$ % such that the liquid level is approximately 2 cm below the calorimeter stopper. Place the acid mixture in the flask. Immediately before the determination of the thermal capacity, ignite the zinc oxide at (950 ± 25) °C for a maximum of 5 min and cool in a desiccator to room temperature. The mass of zinc oxide, m_{ZnO}, being used, weighed to $\pm 0,000\,1$ g, is that required to satisfy Equation (1):

$$\frac{m_{acid}}{m_{ZnO}} = 60 \pm 1 \tag{1}$$

where m_{acid} is the mass of the acid mixture (4.1).

Carry out the procedure as follows.

a) Preliminary period: Stir the acid mixture for 40 min to 50 min.

b) Pre-period: When the rate of temperature increase is constant, start the timing using the chronometer (5.7) and record the initial temperature.

c) Sample introduction: After 15 min, record the temperature, T_0, and immediately add the zinc oxide sample to the acid mixture, taking not more than 1 min.

d) Dissolution period: Stir the mixture for 30 min, after which the dissolution is considered as being complete, and then record the temperature, $\bar{T}_{30}$. Record the ambient temperature, T_a. If the difference between T_a and $\bar{T}_{30}$ is less than 0,5 °C, then repeat the test.

e) Post-period: Record the final temperature, $\bar{T}_{45}$, after an additional 15 min.

In order to reduce reading errors, determine temperatures $\bar{T}_{-15}$, $\bar{T}_{30}$ and $\bar{T}_{45}$ as the average of five different readings recorded at intervals of 1 min over the period 2 min before to 2 min after the prescribed time, T_i (i.e. T_{i-2}, T_{i-1}, T_i, T_{i+1}, T_{i+2}). Determine the value of T_0 by extrapolation of the function of temperature against time in the period T_{-4} to T_{-1}. If the extrapolated value differs from the T_0 reading by more than $\pm 0,002$ °C, then replace T_0 by the extrapolated value. If more than a trace of zinc oxide is found adhering to the tip of the funnel or to the stopper when the calorimeter is opened, repeat the calibration.

NOTE The temperature readings, therefore, effectively start 17 min before the zinc oxide sample is introduced and the last reading takes place 47 min after its introduction. The total duration of the calibration is 64 min.

6.3 Calculation of calibration characteristics

6.3.1 Corrected temperature increase, ΔT_c

Calculate the corrected temperature increase, ΔT_c, expressed in kelvin, from Equation (2):

$$\Delta T_c = (\bar{T}_{30} - T_0) - 2(\bar{T}_{45} - \bar{T}_{30})$$ (2)

where $\bar{T}_{30}$ and $\bar{T}_{45}$ are the average values of five measurements made at intervals of 1 min.

6.3.2 Thermal leakage coefficient, K

Calculate the thermal leakage coefficient, K, in kelvin per 15 min per kelvin temperature difference, (K/15 min·K^{-1}), from Equation (3):

$$K = \frac{(T_0 - \bar{T}_{-15}) - (\bar{T}_{45} - \bar{T}_{30})}{(\bar{T}_{30} - T_0)}$$ (3)