
**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



Reference number
ISO 29582-1:2009(E)

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Published in Switzerland

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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 29582-1 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 $(20,0 \pm 0,2) ^\circ\text{C}$ 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 $(2,00 \pm 0,01)$ 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_4F) instead of hydrofluoric acid, prepared as follows.

Place about 400 g of $(2,00 \pm 0,01)$ mol/l nitric acid and 7,2 g of ammonium fluoride in the flask. Then, add $(2,00 \pm 0,01)$ mol/l nitric acid to make a total mass of $425,0 \text{ g} \pm 0,1 \text{ g}$. 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 $\pm 0,2 \%$.

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 $(950 \pm 25) ^\circ\text{C}$ 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, $(100,0 \pm 0,1)$ g of anhydrous cement with $(40,0 \pm 0,1)$ 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 $(20,0 \pm 0,2) ^\circ\text{C}$.

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 $\pm 0,2 ^\circ\text{C}$; 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.