DRAFT INTERNATIONAL STANDARD ISO/DIS 29582-1

Attributed to ISO/TC **74** by the Central Secretariat (see page iii)

Voting begins on **2007-03-05**

Voting terminates on **2007-08-05**

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

FAST-TRACK PROCEDURE

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

ICS 91.100.10

NOTE FROM THE ISO CENTRAL SECRETARIAT

This draft International Standard is submitted for voting to ISO member bodies under the fast-track procedure.

ISO 29582-1 was prepared by CEN (as EN 196-8) and is submitted for approval under a special “fast-track procedure”, by Technical Committee ISO/TC 74, *Cement and lime*, in parallel with its approval by the ISO member bodies (see the ISO/IEC Directives, Part 1, 2004, Annex F, F.2.1.1).

F.2 “Fast-track procedure”

F.2.1 Proposals to apply the fast-track procedure may be made as follows.

F.2.1.1 Any P-member or category A liaison organization of a concerned technical committee may propose that an **existing standard from any source** be submitted for vote as an enquiry draft. The proposer shall obtain the agreement of the originating organization before making a proposal. The criteria for proposing an existing standard for the fast-track procedure are a matter for each proposer to decide.

F.2.1.2 An international standardizing body recognized by the ISO or IEC council board may propose that a **standard developed by that body** be submitted for vote as a final draft International Standard.

F.2.1.3 An organization having entered into a formal technical agreement with ISO or IEC may propose, in agreement with the appropriate technical committee or subcommittee, that a **draft standard developed by that organization** be submitted for vote as an enquiry draft within that technical committee or subcommittee.

F.2.2 The proposal shall be received by the Chief Executive Officer, who shall take the following actions:

a) settle the copyright and/or trademark situation with the organization having originated the proposed document, so that it can be freely copied and distributed to national bodies without restriction;

b) for cases F.2.1.1 and F.2.1.3, assess in consultation with the relevant secretariats which technical committee/subcommittee is competent for the subject covered by the proposed document; where no technical committee exists competent to deal with the subject of the document in question, the Chief Executive Officer shall refer the proposal to the technical management board, which may request the Chief Executive Officer to submit the document to the enquiry stage and to establish an ad hoc group to deal with matters subsequently arising;

c) ascertain that there is no evident contradiction with other International Standards;

d) distribute the proposed document as an enquiry draft (F.2.1.1 and F.2.1.3) in accordance with 2.6.1, or as a final draft International Standard (case F.2.1.2) in accordance with 2.7.1, indicating (in cases F.2.1.1 and F.2.1.3) the technical committee/subcommittee to the domain of which the proposed document belongs.

F.2.3 The period for voting and the conditions for approval shall be as specified in 2.6 for an enquiry draft and 2.7 for a final draft International Standard. In the case where no technical committee is involved, the condition for approval of a final draft International Standard is that not more than one-quarter of the total number of votes cast are negative.

F.2.4 If, for an enquiry draft, the conditions of approval are met, the draft standard shall progress to the approval stage (2.7). If not, the proposal has failed and any further action shall be decided upon by the technical committee/subcommittee to which the document was attributed in accordance with F.2.2 b).

If, for a final draft International Standard, the conditions of approval are met, the document shall progress to the publication stage (2.8). If not, the proposal has failed and any further action shall be decided upon by the technical committee/subcommittee to which the FDIS was attributed in accordance with F.2.2 b), or by discussion between the originating organization and the office of the CEO if no technical committee was involved.

If the standard is published, its maintenance shall be handled by the technical committee/subcommittee to which the document was attributed in accordance with F.2.2 b), or, if no technical committee was involved, the approval procedure set out above shall be repeated if the originating organization decides that changes to the standard are required.

Contents

Foreword

This document (EN 196-8:2003) 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.

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 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 standard is applicable to cements and hydraulic binders whatever their chemical composition.

NOTE 1 Another procedure, called the semi-adiabatic method, is described in EN 196-9. Either procedure can be used independently.

NOTE 2 It has been demonstrated that the best correlation between the two methods is obtained at 7 days for the solution method (EN 196-8) compared with 41 h for the semi-adiabatic method (EN 196-9).

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

3 Principle

The method consists in 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 0,40;

— use of neat cement paste;

— storage at constant temperature of (20,0 ± 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 Materials

4.1 Acid mixture

Analytical reagent quality acid mixture, obtained by adding 2,760 g of 40 % hydrofluoric acid (HF) for every 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.

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

4.2 Zinc oxide (ZnO)

Use zinc oxide of analytical quality to determine the thermal capacity of the calorimeter. Weigh 40 g to 50 g. Ignite at (950 ± 25) °C for one hour. Cool in a desiccator. Grind to pass a 125 µm sieve. Store in a desiccator.

4.3 Anhydrous cement

Store anhydrous cement, from which metallic iron has been removed with a magnet, 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.

4.4 Hydrated cement

Prepare the hydrated cement test sample by vigorously mixing, either manually or mechanically, (100,0 ± 0,1) g of anhydrous cement with (40,0 ± 0,1) g of distilled or deionised water for 3 min at ambient temperature. Place the resulting paste in plastics or glass cylindrical vials (three for each hydration period to be 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

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.

A suitable calorimeter (see Figure 1) comprises the following:

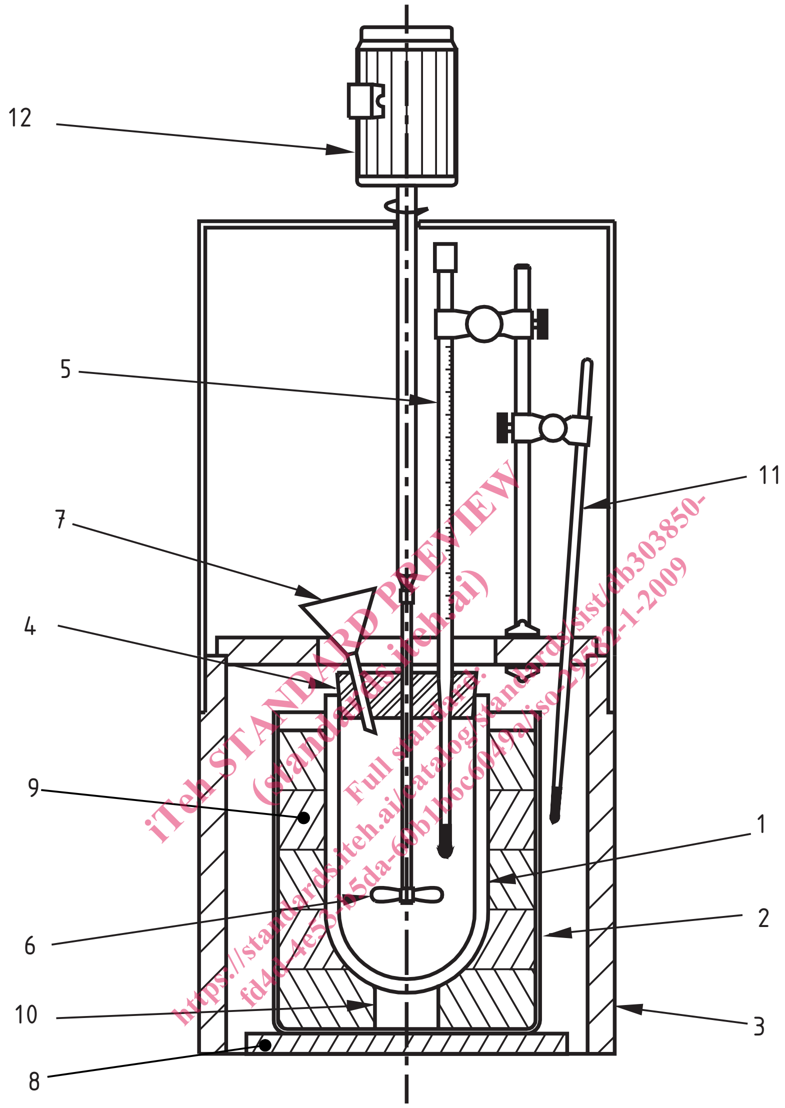
a) ***Dissolution vessel***, consisting of: an insulated flask (eg. Dewar flask), placed either in a heat insulated container set inside a box constructed of insulating material (e.g. wood, plastics), or immersed in a thermostatic water bath regulated to ± 0,2 °C; and an insulated stopper (made of cork or plastics) 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 kelvins 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.

b) ***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.

c) ***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.

d) ***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 be low power rated (e.g. a motor of a few watts) so as to prevent any excessive heat emission from affecting measurements.



Key

1 Flask
2 Container
3 Box
4 Stopper
5 Thermometer
6 Stirrer
7 Funnel
8 Support
9 Insulating material
10 Flask support
11 Ambient thermometer
12 Stirrer motor

Figure 1 — Typical heat of solution calorimeter apparatus

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

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

5.4 Plastics 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 ± 0,0001 g.

5.11 Balance, of capacity 2 kg, capable of weighing to an accuracy of ± 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 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 ± 0,2 % such that the liquid level will be 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 quantity of zinc oxide to be used, weighed to ± 0,0001 g, is that required to satisfy equation (1):

$$\frac{\textit{Mass of acid mixture}}{\textit{Mass of zinc oxide}} = 60 \pm 1 \qquad (1)$$

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, . T_{-15}

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.