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Isothermal Conduction Calorimetry (ICC) for the determination of heat of hydration of cement: State of Art Report and Recommendations

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English Version

Isothermal Conduction Calorimetry (ICC) for the determination of heat of hydration of cement: State of Art Report and Recommendations

Bestimmung der Hydratationswärme von Zement durch isotherme Wärmeflusskalorimetrie: Stand der Technik und Empfehlungen

This Technical Report was approved by CEN on 26 November 2013. It has been drawn up by the Technical Committee CEN/TC 51.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (CEN/TR 16632:2014) has been prepared by Technical Committee CEN/TC 51 “Cement and building limes”, the secretariat of which is held by NBN.

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It is divided into two sections. The first section is a State of Art Report of the test method based on the collection of the technical data sheets of the calorimeters adopted in the European cement laboratories and also on the collection of the results of several experimental activities. The second section is made of recommendations for the measurement of heat of hydration of cement by ICC. Based on the State of Art Report, this section provides some basic elements of the test procedure with the aim to become a first guide for the laboratories that are currently using ICC or for those laboratories that would start to adopt this method. By using the information and adopting the procedures given in the document it will be possible to compare in a more reliable way both the performances of the different calorimeters and the test results.

Annex A (informative) provides a Glossary.

Introduction

In 2007, CEN/TC 51, through resolution 495, agreed that WG 12/TG 3 investigates the suitability for standardization of the test method based on isothermal conduction calorimetry (ICC). The Task Group 3 has been reactivated and held its first meeting in 2008.

Since no national standard on ICC for the determination of heat of hydration of cement was available, TG 3 started its activity on the item by gathering the available information on recommendations or published scientific papers, inter-laboratory experimental exercises. The available information, collected into a State of Art report, has been analysed and discussed in order to identify those aspects of the test method that can be already considered consolidated as well as those elements that still need further development.

The second step of the activity was the redaction of a Recommendations document including a testing procedure for the measuring of heat of hydration of cement by ICC. The circulation of this document in the laboratory actually involved in ICC testing, would lead to the application of uniform general principles and, therefore, to a better data reproducibility.

In this CEN/TR, the State of Art document and the Recommendations document are reviewed into a single document divided into two parts:

a) State of art report on the application of ICC for the determination of heat of hydration of cement;

b) Recommendations for the measurement of Heat of Hydration of Cement by Isothermal Conduction Calorimetry.

PART A
State of art report on the application of ICC for the determination of heat of hydration of cement

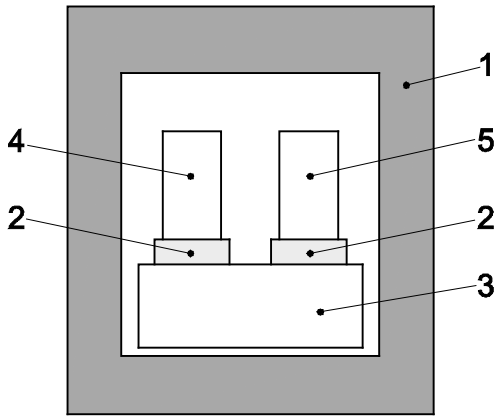
1 Basic principle and key points of ICC

1.1 Basic Principle

The test method is designed to measure the heat of hydration of cement when mixed with water. The measurement takes place at essentially constant temperature, if the instrument and the measurement are well designed, therefore it is assumed to be the “isothermal heat of hydration of cement”.

An isothermal heat conduction calorimeter (here called calorimeter) consists of a thermostatic heat sink upon which two heat flow sensors are placed. The sample is placed in an ampoule that is placed in an ampoule holder that is in contact with one of the heat flow sensors, and an inert reference is placed in contact with the other. The sample ampoule and the reference ampoule are thermally connected by heat flow sensors to a thermostatic heat sink. The output from the calorimeter is the difference between the outputs from the sample heat flow sensor and the reference heat flow sensor. A general scheme of a heat conduction calorimeter is given in Figure 1.

However the actual design of an individual instrument, whether commercial or home-built, may vary.



Key

1 thermostat

2 heat flow sensors

3 heat sunk

4 sample

5 reference

Figure 1 — A schematic drawing of a heat conduction calorimeter

Most part of the calorimeters can measure the heat of hydration of samples mixed outside from the instrument, therefore the heat produced during the mixing is not measured. It is not easy to solve this problem designing a calorimeter provided with an internally mixing device having the proper efficacy.

1.2 Key points of ICC

When performing ICC measurements on cement samples some key points have to be considered and correctly managed:

— Constant value of the temperature of the thermostat;

— Stability of the temperature of the thermostat all over the test duration;

— Control of the maximum difference between sample temperature and thermostat temperature (isothermal conditions);

— The baseline of the instrument (measured with an inert sample of similar thermal properties of test sample) should be both repeatable and stable;

— Calibration of the calorimeter. The method currently used is based on the joule effect produced by a resistor feed with an electrical current; no standard material for the calibration is available for the time being;

— Check that the ampoule is vapour tight enough (so that endothermic thermal powers of evaporation do not influence the measurements).

2 Normative references

Not applicable.