Designation: C1679 – 17

Standard Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry[1]

This standard is issued under the fixed designation C1679; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This practice describes the apparatus and procedure for measuring relative differences in hydration kinetics of hydraulic cementitious mixtures, either in paste or mortar (see Note 1), including those containing admixtures, various supplementary cementitious materials (SCM), and other fine materials by measuring the thermal power using an isothermal calorimeter.

NOTE 1—Paste specimens are often preferred for mechanistic research when details of individual reaction peaks are important or for particular calorimetry configurations. Mortar specimens may give results that have better correlation with concrete setting and early strength development and are often preferred to evaluate different mixture proportions for concrete. Both paste and mortar studies have been found to be effective in evaluating concrete field problems due to incompatibility of materials used in concrete mixtures.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* (**Warning—**Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.[2]*)*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C125 Terminology Relating to Concrete and Concrete Aggregates

C172/C172M Practice for Sampling Freshly Mixed Concrete

C219 Terminology Relating to Hydraulic Cement

C305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency

C403/C403M Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance

C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes

C778 Specification for Standard Sand

C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the Physical Testing of Hydraulic Cements

C1602/C1602M Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete

C1738/C1738M Practice for High-Shear Mixing of Hydraulic Cement Pastes

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology C125 and Terminology C219.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *baseline, n*—the signal from the calorimeter when there is an inert specimen in the instrument.

3.2.2 *calcium aluminate, n*—various aluminate phases including but not limited to the tricalcium aluminate and ferrite phases in portland cement clinker, calcium aluminate phases occurring in some supplementary cementitious materials, and calcium-alumino-silicate glasses also occurring in some

[1] This practice is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.48 on Performance of Cementitious Materials and Admixture Combinations.

Current edition approved Dec. 1, 2017. Published January 2018. Originally approved in 2007. Last previous edition approved in 2014 as C1679 – 14. DOI: 10.1520/C1679-17.

[2] Section on Safety Precautions, Manual of Aggregate and Concrete Testing, *Annual Book of ASTM Standards*, Vol 04.02.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

supplementary cementitious materials, that are capable of consuming the sulfate phases present in hydrating cementitious systems.

3.2.3 *calibration coefficient, n*—a factor that relates the value recorded by the data acquisition system to the thermal power output.

3.2.3.1 *Discussion*—Normally recorded data are in volts and the calibration coefficient has units of watts per volt (W/V). Some calorimeters may have internal automatic calibration and will give the output in watts without the user having to specify the calibration coefficient.

3.2.4 *combined mixture, n*—combination of all the materials that are introduced into the calorimeter for measuring hydration kinetics.

3.2.5 *hydration time, n*—the elapsed time from initial contact between the cementitious materials and the mix water.

3.2.6 *inert specimen, n*—specimen placed within the isothermal calorimeter made of a non-reactive material of similar thermal properties (mainly heat capacity) as the reacting specimen made of the cementitious test mixture.

3.2.6.1 *Discussion*—The output from the calorimeter is the difference between the heat flow from the test specimen and the inert specimen. The use of an inert specimen substantially decreases the noise and drift of the measured heat flow.

3.2.7 *isothermal calorimeter, n*—a calorimeter that measures heat flow from a specimen maintained at a constant temperature by intimate thermal contact with a constant temperature heat sink.

3.2.8 *isothermal calorimetry, n*—an experimental technique to monitor the thermal power output from a specimen kept at near isothermal conditions.

3.2.9 *isothermal hydration profile, n*—the thermal power plotted as a function of hydration time, which provides an indication of the rate of hydration over time at a given temperature.

3.2.10 *main hydration peak, n*—the broadest peak in the isothermal hydration profile that starts at the end of the dormant period and for a well-balanced mixture lasts for several hours (see Fig. 1).

3.2.11 *near isothermal conditions, n*—a constant temperature with a permissible variation of ± 1.0 °C.

3.2.12 *specimen holder, n*—container within the isothermal calorimeter that conducts the heat from the specimen in the vial to the heat flow sensor.

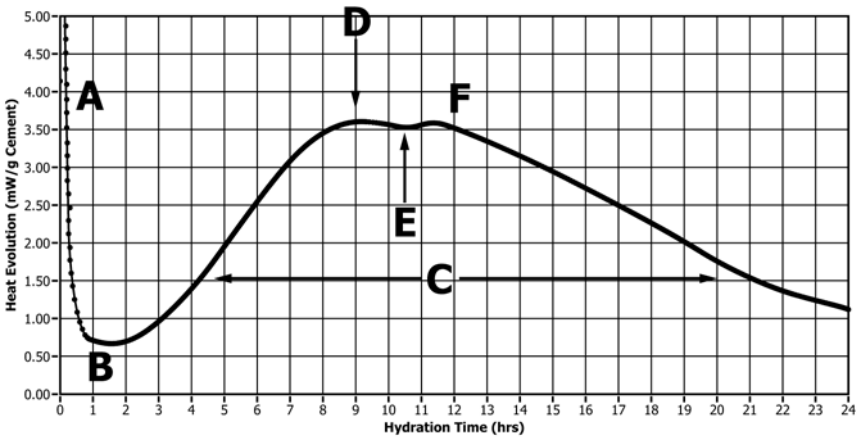
3.2.13 *stock solution, n*—a solution of admixture in water prepared to enable more precise volumetric addition of small quantities of admixture, typically made by pipetting known volumes of admixture into a volumetric flask and diluting it to the flask's fixed volume.

3.2.14 *sulfate addition, n*—the addition of a soluble sulfate source (such as gypsum, calcium sulfate hemihydrate, alkali sulfate) to a combined mixture to investigate whether a given combination of materials is in sulfate balance.

3.2.15 *sulfate balance of mixture, n*—the situation when the size of the main hydration peak is not increased by sulfate additions; in some cases where the main peak is increased in size by added sulfate, it will also be accelerated in time.

3.2.16 *sulfate depletion point, n*—the onset of accelerated calcium aluminate activity that for a portland cement in absence of supplementary cementitious material (SCM) and admixture may take place after the main hydration peak.

3.2.16.1 *Discussion*—The sulfate depletion point may become impossible to detect without further addition of soluble calcium sulfate for certain cements and more often in combined mixtures with admixtures or SCMs, or both. In some cases other sources of sulfate might be used to mimic potential conditions in the system. Among these are anhydrite, arcanite, calcium langbeinite, aphthitalite, syngenite, and others. Fig. 2 shows an example of the effect of added sulfate on the sulfate depletion point. Added sulfate may, in some combined mixtures with admixtures or SCMs, or both, accelerate the onset of the main hydration peak. When a combined mixture is at



NOTE 1—(A) initial thermal power by dissolution of cement and initial cement hydration; (B) dormant period associated with very low thermal power indicating slow and well-controlled hydration: (C) main hydration peak associated mainly with hydration reactions contributing to setting and early strength development, with maximum at (D); and (E) sulfate depletion point,[6] followed by (F) accelerated calcium aluminate activity.

FIG. 1 Example of Thermal Power Curve for Isothermal Hydration of Portland Cement