



Designation: C1679 – 22

Standard Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry¹

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 This practice does not cover the measurement of heat of hydration. Heat of hydration can be determined according to Test Method [C1702](#).

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

1.4 *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, health, and environmental 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)*

1.5 *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.*

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.

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

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 and Other Inorganic Cements](#)

[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 Physical Testing of Hydraulic Cements](#)

[C1602/C1602M Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete](#)

[C1702 Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry](#)

[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

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

calcium-alumino-silicate glasses also occurring in some 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.

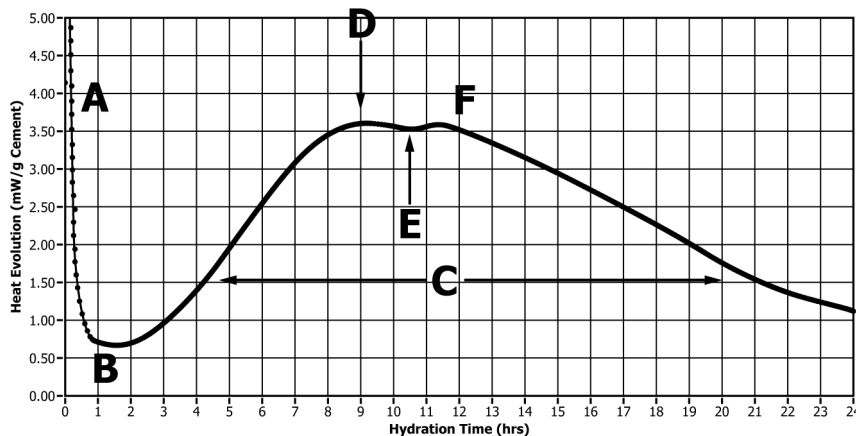
3.2.13 *stock solution, n*—a solution of admixture in water prepared to enable more precise volumetric addition of small quantities of admixture, typically made by pipetting known volumes of admixture into a volumetric flask and diluting it to the flask’s fixed volume.

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, apthitalite, 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



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,⁴ followed by (F) accelerated calcium aluminate activity.

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

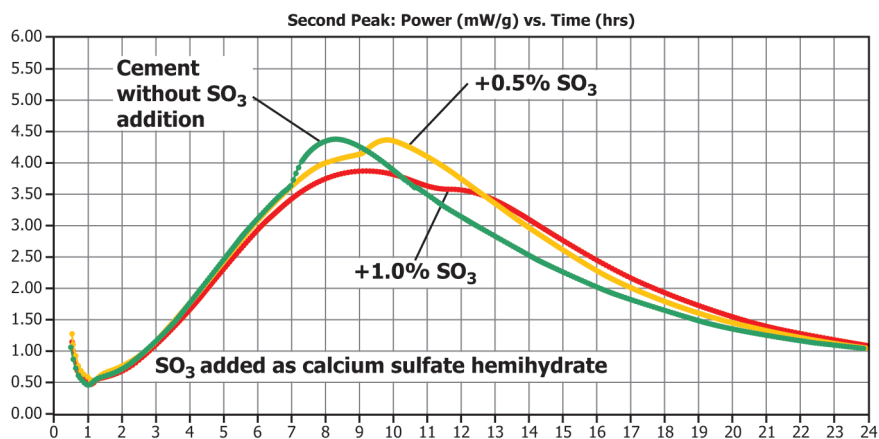


FIG. 2 Example of the Effect of Soluble Calcium Sulfate Addition on the Timing of the Sulfate Depletion Point for a Type I Portland Cement Mixed with Water Only at w/c = 0.45

the main hydration peak. When a combined mixture is at sulfate balance, further addition of soluble sulfate will not increase the size, or accelerate the onset, of the main hydration peak.

3.2.17 *thermal equilibrium time, n*—the elapsed hydration time when the thermal power of replicate mixtures do not differ by more than 0.2 mW/g of dry material.

3.2.18 *thermal indicator of setting time, n*—the hydration time to reach a thermal power of 50 % of the maximum value of the main hydration peak.

3.2.19 *thermal mass, n*—the amount of thermal energy that can be stored by a material (J/K).

3.2.19.1 *Discussion*—The thermal mass of a given material is calculated by multiplying the mass by the specific heat capacity of the material. For the purpose of calculating the thermal mass used in this standard, the following specific heat capacities can be used: The specific heat capacity of a typical unhydrated portland cement and water is 0.75 and 4.18 J/(g·K), respectively. Thus a mixture of A g of cement and B g of water has a thermal mass of $(0.75 \times A + 4.18 \times B)$ J/K. The specific heat capacity of typical quartz and limestone is 0.75 and 0.84 J/(g·K), respectively. The specific heat capacity of most amorphous supplementary cementitious material such as fly ash or slag is approximately 0.8 J/(g·K).

3.2.20 *thermal power, n*—heat production rate measured in watts (W) or joules per second (J/s), usually expressed in relation to the mass of cementitious material, as mW/g or J/s/g.

3.2.20.1 *Discussion*—The thermal power is an indicator of the rate of various chemical reactions between cementitious materials, other fine particles, mix water and admixtures.

3.2.21 *vial, n*—container into which the freshly mixed cementitious mixture is placed for a measurement.

4. Summary of Practice

4.1 An isothermal calorimeter consists of heat sink with a thermostat, two heat flow sensors and a specimen vial holder attached to each sensor. A vial containing a freshly prepared mixture is placed in contact with one of the vial holders and a thermally inert material is placed in contact with the other. The heat of hydration released by the reacting cementitious speci-

men is transferred and passes across a heat flow sensor. The calorimeter output is calculated from the difference between the outputs from the test specimen heat flow sensor and the inert specimen heat flow sensor. Because the heat is allowed to flow away from the specimen, the measurement will take place at essentially constant temperature (isothermal conditions).

4.2 Mixtures with cement, SCM, admixtures, water and optional fine aggregate are prepared and introduced into an isothermal calorimeter. Isothermal calorimetry tests are performed on a series of different mixtures for relative comparison of the hydration kinetics. The output of the calorimeter is evaluated by graphical and mathematical means to evaluate retarding and accelerating effects of different combinations of materials. Calcium sulfate may be added as a probe to determine if the addition of admixture, SCMs, or both have increased the mixture’s demand for sulfate beyond that which is available in the cement.

5. Significance and Use

5.1 Thermal power curves are used to evaluate the isothermal hydration kinetics of the combined mixture of different materials during the early period after being mixed with water. These isothermal power curves, or hydration profiles, may provide indications relative to setting characteristics, compatibility of different materials, sulfate balance and early strength development. The isothermal hydration profiles can also be used to evaluate the effects of compositions, proportions, and time of addition of materials as well as curing temperature. Special care must be used in evaluating extended retardation with paste specimens, which have been shown to overestimate the retardation of some mixtures containing cement, SCM, and admixtures.

5.2 This procedure can be used to measure the effect of chemical admixtures on the cement hydration profile. In many cases, the addition of chemical admixture changes the kinetics of cement hydration.

5.3 Although this technique has been used historically to understand issues related to setting and slump loss, it must be emphasized that isothermal calorimetry results cannot predict concrete performance definitely, either positively or negatively.