



Designation: C1753/C1753M – 21a

Standard Practice for Evaluating Early Hydration of Hydraulic Cementitious Mixtures Using Thermal Measurements¹

This standard is issued under the fixed designation C1753/C1753M; 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 evaluating relative differences in early hydration of hydraulic cementitious mixtures such as paste, mortar, or concrete, including those containing chemical admixtures, various supplementary cementitious materials (SCMs), and other finely divided materials, by measuring the temperature history of a specimen.

1.2 Calorimetry is the measurement of heat lost or gained during a chemical reaction such as cement hydration; calorimetric measurements as a function of time can be used to describe and evaluate hydration and related early-age property development. Calorimetry may be performed under isothermal conditions (as described in Practice C1679) or under adiabatic or semi-adiabatic conditions. This practice cannot be described as calorimetry because no attempt is made to measure or compute the heat evolved from test specimens due to hydration, but it can in many cases be used for similar evaluations. Variables that should be considered in the application of this practice are discussed in the Appendix.

1.3 *Units*—The values stated in either SI units or inch-pound units shall be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in non-conformance with the standard. Some values have only SI units because the inch-pound equivalents are not used in practice.

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.*

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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NOTE 1—**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.*

2. Referenced Documents

2.1 *ASTM Standards:*³

- C39/C39M Test Method for Compressive Strength of Cylindrical Concrete Specimens
- C125 Terminology Relating to Concrete and Concrete Aggregates
- C172/C172M Practice for Sampling Freshly Mixed Concrete
- C192/C192M Practice for Making and Curing Concrete Test Specimens in the Laboratory
- 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
- C494/C494M Specification for Chemical Admixtures for Concrete
- C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in Physical Testing of Hydraulic Cements
- C1679 Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry

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

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology C125, Terminology C219, and Practice C1679.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *adiabatic, adj*—occurring without exchange of heat with the environment.

3.2.2 *exotherm, n*—heat evolution during hydration as evidenced by an increase in measured specimen temperature shown in the thermal profile.

3.2.3 *inert specimen, n*—specimen placed within the same thermal environment as the test specimen(s), made of a nonreactive material of similar heat capacity and the same mass as the reacting test specimen(s).

3.2.3.1 *Discussion*—The difference between the temperature of the hydrating test specimen(s) and the inert specimen represents the change in specimen temperature due to hydration. Interpretation can often be improved by comparing temperature histories after subtracting the temperature of the corresponding inert specimen (reference temperature), which tends to account for the effects of changing environment temperature during the measurement period.

3.2.4 *main peak response, n*—the initial temperature rise and subsequent temperature drop in the measured thermal profile that starts at the end of the dormant period and, for a mixture with normal sulfate balance, lasts for several hours.

3.2.5 *reference temperature, n*—the temperature of the inert specimen in a test series at the time corresponding to a particular temperature of the test specimen.

3.2.6 *sulfate demand, n*—the level of soluble calcium sulfate in a hydrating cementitious mixture required to maintain normal hydration behavior for a specific combination of

mixture proportions, materials properties, initial mixture temperature, and test temperature.

3.2.7 *sulfate imbalance threshold, n*—the condition of a cementitious mixture in terms of mixture proportions, materials properties, initial mixture temperature, and test temperature, for which a small change in any of these variables can result in abnormal hydration behavior due to depletion of calcium sulfate in solution.

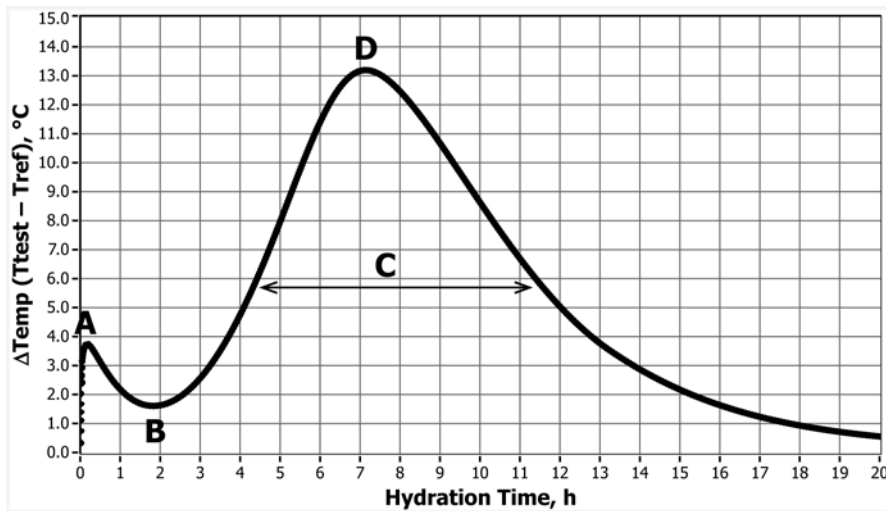
3.2.8 *test specimen, n*—a hydraulic cementitious mixture being evaluated for its thermal response.

3.2.9 *test temperature, n*—the temperature of the air or insulation, if any, surrounding the test specimen containers at the start of temperature measurement, normally intended to remain constant.

3.2.10 *thermal profile, n*—the temperature of a hydrating mixture (before or after subtraction of the reference temperature), plotted as a function of hydration time, that provides an indication of the rate of hydration over time.

3.2.10.1 *Discussion*—An example thermal profile is shown in Fig. 1. On the vertical axis T_{test} refers to the temperature of the test specimen and T_{ref} refers to the temperature of the inert (reference) specimen. The shape of the thermal profile is affected not only by mixture hydration but also by the specimen type and mass, mixture proportions, specimen initial temperature, specimen container size and shape, insulation (if any) provided around the specimen container, and the temperature of the surrounding environment. Additional guidance is provided in the Appendix.

3.2.11 *time of setting marker, n*—the point marked on the thermal profile indicating the hydration time when a selected fraction of the main peak amplitude is attained and that is used as a relative indicator of time of setting.



NOTE 1—(A) initial exotherm from dissolution of cement and initial hydration, principally of calcium aluminates; (B) dormant period temperature reduction associated with very low heat evolution indicating slow and well-controlled hydration; (C) main peak response associated primarily with hydration reactions contributing to setting and early strength development, with maximum temperature at (D). The maximum temperature (D) and the rates of temperature rise and fall that shape the main peak response (C) are affected not only by hydration but by the related cooling response of the specimen.

FIG. 1 Example Thermal Profile of a Portland Cement Paste Mixture (Inert Specimen Temperature Subtracted from Test Specimen Temperature)

4. Summary of Practice

4.1 A thermal measurement test system consists of temperature measuring devices, data collection equipment, and specimen containers of similar volume, shape, and material, capable of similarly isolating each test specimen and an inert specimen. The specific insulation values for specimen containers and the test temperature are selected based on the intended test objectives. Related guidance is provided in the Appendix.

4.2 Mixtures composed of cementitious materials, water, and optionally chemical admixtures, or aggregate, or both, are prepared and introduced into specimen containers for collection of temperature data.

4.3 Thermal profiles are plotted using a common time scale that begins at the time of initial mixing of water with cementitious materials, which is the start of hydration time. The measured thermal profiles allow qualitative comparison of early hydration kinetics, such as retarding or accelerating trends, as influenced by different combinations of materials, and abnormal hydration behaviors that can interfere with setting and strength development.

5. Significance and Use

5.1 This practice provides a means of assessing the relative early hydration performance of various test mixtures compared with control mixtures that are prepared in a similar manner.

5.2 Thermal profiles are used to evaluate the hydration behavior of hydraulic cementitious mixtures after the addition of water. They may provide indications concerning setting characteristics, compatibility of different materials, sulfate balance, relative heat of hydration, and early strength development. They can be used to evaluate the effects of compositions, proportions, and time of addition of materials as well as the initial mixture and test temperatures. Thermal profile testing is an effective tool for identifying performance sensitivities or trends, and may help to reduce the number of concrete test mixtures required to develop and qualify mixtures, especially those to be subject to variable ambient environments. It may be used by concrete producers, materials suppliers, and other practitioners to support mixture development, selection of material types or sources, optimization of proportions, or troubleshooting of field problems.

5.3 This practice can be used to understand concrete problems related to slump loss, setting, and early strength, but results may not predict field concrete performance. Performance verification with concrete is needed to quantify the trends identified using thermal testing.

5.4 This practice can be used to evaluate the effects of chemical admixtures on the thermal profiles of cementitious mixtures. This can be especially useful in selecting dosages appropriate for different ambient conditions.

5.5 Thermal measurement testing as described in this practice may have similar significance and use as isothermal calorimetry described by Practice C1679 or some types of near-adiabatic calorimetry. The selection of which practice or methods to use may depend on specific applications and circumstances. The thermal profiles obtained by this practice

may have similar shapes to isothermal hydration profiles as obtained by Practice C1679, but thermal profiles from this practice do not provide quantitative measurement of heat of hydration, are affected by various details of the test conditions and mixtures (see 3.2.10 and the Appendix), and are subject to greater variability. Equipment used for this practice is less expensive than isothermal or near-adiabatic calorimeters and may be more easily adapted for use in the field or where a large number of different specimens and mixtures must be evaluated in a short time period. Identification of the sulfate depletion point of a mixture (as described in Practice C1679) is not generally possible using thermal measurement testing.

5.6 To evaluate the potential for abnormal hydration, it is important that the test temperatures and the initial temperatures of the mixture be selected to represent the range of expected initial concrete field temperatures.

5.7 This practice is not intended to provide results that can be compared across laboratories using different equipment nor to provide quantitative measurements or corrected approximations of actual hydration heat. It should not be cited in project specifications or otherwise used for the purpose of acceptance or rejection of concrete. It is intended to serve as a simple and expedient tool for comparison of the relative early-age hydration performance of different specific combinations of materials that are prepared and stored under the same conditions.

6. Apparatus

6.1 Devices for Preparing Specimens:

6.1.1 *Weights and Weighing Devices*, used for preparation of laboratory test mixtures up to 5 kg [11 lb] total mass shall conform to the requirements of Specification C1005. For preparing test mixtures of greater total mass including concrete batches in the laboratory, weighing devices shall conform to the requirements of Practice C192/C192M.

6.1.2 *Graduated Cylinders*, shall conform to the requirements of Specification C1005. The permissible variation for graduated cylinders of less than 100-mL capacity shall be $\pm 1.0\%$ of the indicated capacity.

6.1.3 *Graduated Syringes*, if used, shall be of suitable capacities to contain the desired volume of liquid admixture and shall be accurate to $\pm 3\%$ of the required volume.

6.1.4 *Mixing Apparatus*, capable of producing a uniform mixture.

6.2 *Thermal Measurement Test Equipment and Data Acquisition System*—Actual design of the equipment, whether commercial or custom-built, may vary, but it shall meet the following requirements for the selected type, shape, and mass of the specimen, insulation (if any) surrounding the specimen container, initial mixture temperature, and test temperature.

6.2.1 Temperature sensors shall be thermistors or thermocouples with measurement accuracy of $\pm 1.0\text{ }^{\circ}\text{C}$ [$2\text{ }^{\circ}\text{F}$].

6.2.2 The signal-to-noise ratio shall be at least 5.0. Signal is defined as the difference between the highest and the lowest temperatures measured from the dormant period through the main peak response (Fig. 1) for a test specimen in the test series without admixture or SCMs (Fig. 2). Noise is defined as the difference between the highest and the lowest temperatures