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Standard Practice for
Evaluating Early Hydration of Hydraulic Cementitious Mixtures Using Thermal Measurements[1]

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.*

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

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[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.

[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

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

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.

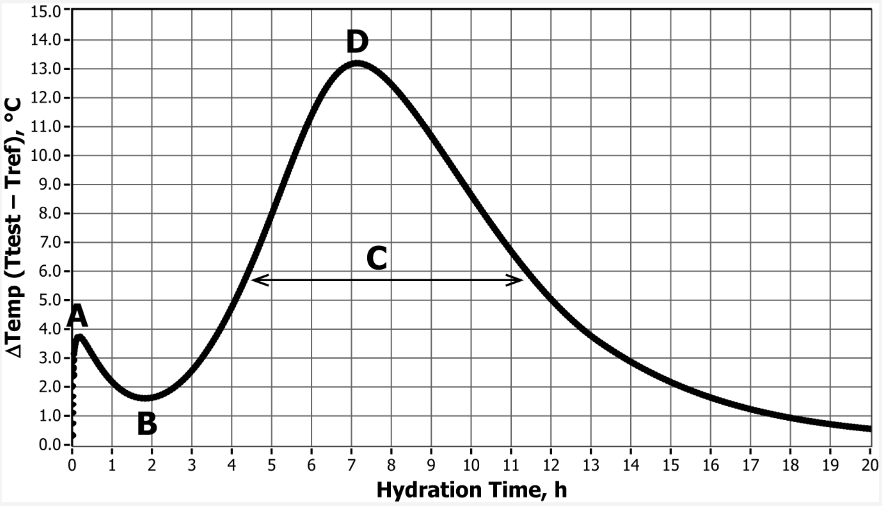
3.2.8 *test specimen, n*—a hydraulic cementitious mixture being evaluated for its thermal response.

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.



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)

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.

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 ± 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 ± 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 ± 1.0 °C [2 °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 measured during the time period in which the signal is established (Fig. 2) for an inert specimen having a mass similar to that of the test specimens. The inert specimen shall remain in the same environment as the test specimens to indicate both the effects of changes in ambient temperature as well as any thermal influences of adjacent test specimens (see also 6.2.5).

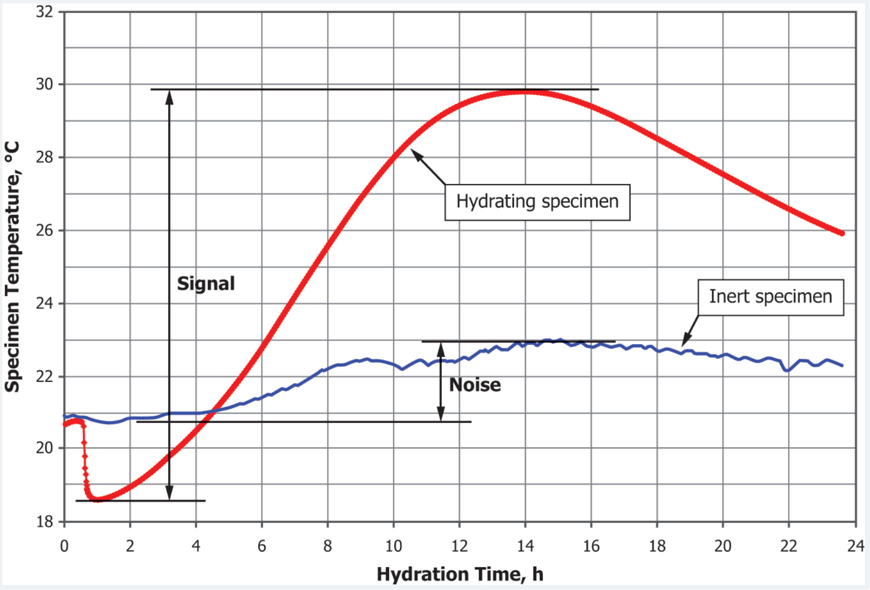


FIG. 2 Examples of Signal and Noise Determination for Verification of Signal-to-Noise Ratio

NOTE 2—Exceeding the minimum signal-to-noise ratio is more important than specific requirements for insulation value of the specimen container or environment (see Appendix for guidance). Selected specimen containers and insulation configurations (if any) may vary with mixture type, specimen mass, and initial mixture and test temperatures. A satisfactory inert specimen may be obtained using quantities of sand and water having masses within ± 10 % of the combined solids and water contents of the test specimens. Thermal influences from other test specimens may be reduced by providing adequate spaces between specimens in the test environment, depending on the insulating values of the specimen containers. The intent of a minimum signal-to-noise ratio requirement is to assure a well-defined thermal profile that is minimally influenced by ambient temperature changes and the presence of other test specimens. The maximum main peak temperature should be similar to the maximum temperature that would be expected for in-place concrete in the application of interest. Because the type, shape, and mass of the test specimen, insulation around the specimen container, and initial mixture and test temperatures all influence main peak response levels, it is important to balance these factors to meet the requirements of 8.2.2 without causing unrealistic main peak response temperatures (see Note 4 and the Appendix for guidance).

6.2.3 The air space or insulation, or both, surrounding the specimen containers, whether the test specimen is stored under ambient conditions or inside a conditioned chamber intended to replicate field conditions of interest, shall be controlled to ensure that the measured temperature of the inert specimen (reference temperature) does not vary from the test temperature by more than 3 °C [5 °F] during testing, unless deliberate change of ambient conditions during the period of temperature measurement is part of the test program.

6.2.3.1 A conditioned chamber may be a laboratory test chamber, portable curing chamber, or other temperature-controlled container capable of maintaining the test temperature in the ambient space around the hydrating test specimens without the use of forced air circulation. Portable concrete cylinder curing boxes that employ circulating temperature-conditioned water below a specimen shelf have been successfully used. Other types of chambers that use forced air in the regulation of internal temperature should not be used, however, as air movement around the specimen containers can adversely influence test results.

6.2.4 The data acquisition equipment shall be capable of performing continuous logging of the temperatures with a time interval between recorded measurements not greater than 60 s.

6.2.5 *Specimen Containers* of volume and insulating value as needed to meet the requirements of 6.2.1 for the test mixtures and conditions that can be sealed while providing access for the temperature sensors of the thermal measurement system, if required (see Note 4). For systems without continuous insulation between specimen containers, provide a clear distance of at least 70 mm [3 in.] between individual specimen containers.

6.2.6 The location of temperature sensors relative to specimen containers shall be similar for all test specimens and for the inert specimen.

7. Materials

7.1 *Mixture Materials:*

7.1.1 Mixture materials, including cementitious materials and admixtures, shall be obtained from the concrete producer, or otherwise obtained to be representative of those specific to the purpose of the test.

7.2 *Calcium Sulfate:*

7.2.1 Use reagent grade calcium sulfate dihydrate or hemihydrate prepared from reagent grade calcium sulfate dihydrate or calcium sulfate anhydrite to verify whether a mixture is in sulfate balance. See the Appendix for examples of sulfate addition for evaluation of sulfate balance.

7.2.2 It is permissible to use a source-specific calcium sulfate for performing a test series that is related to a specific cement production source.

8. Procedure

8.1 *Temperature Conditions:*

8.1.1 *Specimen Preparation Temperature*—Maintain the temperature of the air in the vicinity of all equipment and materials used in specimen preparation at the test temperature to within ± 3.0 °C [5 °F].

8.1.2 *Materials and Initial Mixture Temperatures*—Precondition all materials as necessary to achieve an initial mixture temperature of 23.0 °C ± 2.0 °C [73.5 °F ± 3.5 °F] or other specific initial mixture temperature according to test objectives.

NOTE 3—Depending on test objectives, a test temperature representative of typical or extreme field conditions may be selected. For other evaluations, a test temperature equal to the laboratory temperature is typically used. Regardless of test temperature, the initial mixture and specimen temperatures should usually be controlled to be as close to the test temperature as possible so that measured changes in specimen temperature over time result essentially only from hydration influences, and so that the initial (calcium aluminate) hydration and dormant periods are captured in the thermal profile. If the initial mixture temperature differs from the test temperature, it becomes difficult to use the thermal profile for a relative indication of time of setting.

8.1.3 *Thermal Measurement System and Ambient Temperature*—The temperature of the thermal measurement system and the surrounding ambient environment shall be within ± 2.0 °C [3.5 °F] of the test temperature before beginning a test. Allow sufficient time for the temperature measurement system to stabilize to the ambient temperature.

8.2 *Test Specimens:*

8.2.1 The number of specimens and number of test batches depend on the purpose of the test program (see the Appendix for examples of test programs).

8.2.2 The volume and mass (see Note 4 and the Appendix) of the test specimen depend on the thermal measurement equipment, insulating value of the specimen container and any surrounding insulation, test temperature, the type of mixture (paste, mortar, or concrete), and the test objectives. Masses of specimens that will be compared with each other shall not differ by more than 5% of the average.

NOTE 4—Typical specimen mass is 300 g to 1000 g [0.7 to 2.2 lb] for paste and 1500 g to 4000 g [3.3 lb to 8.8 lb] for mortar or concrete, though acceptable temperature measurements have been reported with mortar specimens of as little as 750 g [1.7 lb]. Corresponding container volumes are approximately 150 mL to 600 mL [10 in. to 35 in.^{3}] for paste and 650 mL to 1650 mL [40 in. to 100 in.^{3}] for mortar or concrete. The selection of specimen mass and the use of insulation around specimen containers must be balanced; specimens with greater mass require less insulation. Thermal testing with concrete or mortar specimens is usually preferred when time of setting trends are being evaluated, but testing with paste specimens of similar proportions may be equally useful and may be more convenient. Thermal profiles for paste specimens with the same proportions as the paste fractions of concrete mixtures being evaluated, without the aggregates, have been shown to consistently produce indications of longer times of setting than those for concrete or mortar specimens, but trends are similar.

8.3 *Mixing:*

8.3.1 Any effective mixing procedure is allowed; various suitable mixing methods are described in the Appendix. Depending on the method used, the order of the introduction of materials to the mixing bowl or container may differ. Dispense liquid admixtures into mixing water to form a solution before introduction into the cementitious materials. The solution containing admixtures may consist of all of the mix water or some portion, if admixture addition is to be delayed. Liquid admixtures may be introduced directly to mixing water using a graduated syringe or obtained from a stock solution at appropriate dilution. Inspect stock solutions for separation and remix, if necessary. Record the time of initial mixing (when wetting of cementitious materials first occurs), to the nearest minute.

8.3.2 Because mixing intensity is a variable that may influence the interaction of materials used to prepare test specimens, in many cases different mixing procedures (speeds or durations) may be needed, depending on the goal of the testing. Unless mixing intensity is a defined variable in a testing program, mixtures prepared using different mixing procedure shall not be compared.

8.4 *Mortar:*

8.4.1 If mortar is to be tested, it can be prepared independently or obtained from fresh concrete by wet sieving in accordance with Practice C172/C172M.

8.5 *Transferring Mixture to Specimen Container and Test Environment:*

8.5.1 Place the appropriate mass of the batch contents into the specimen container, using a suitable clean spatula, spoon, or scoop; pouring is permitted if the batch is sufficiently fluid (see Note 5). If necessary, consolidate the specimen by rodding, tamping, or tapping. Cover and seal the specimen container, providing access for temperature sensors (such as thermocouples) that must be inserted into the test specimen.

NOTE 5—It may be useful to measure slump, flow, mini slump[4] or other properties for comparing consistency. Specimen type and consistency govern which method(s) could be used.

8.5.2 Immediately place the specimen container in the test environment and begin recording specimen temperature.

8.6 *Thermal Measurements:*

8.6.1 Ensure that temperature sensors are in contact with the specimen or container as required for the equipment used. Record, to the nearest minute, the time at the start of mixing (time of initial contact of water with cementitious materials) and the time at which temperature measurements are initiated or when the specimen temperature is first measured using continuously logged data (see Note 6).

8.6.2 The time delay between the start of mixing and initial measurement of specimen temperature may vary according to test series and specimen details but the extent of this delay shall be controlled to within ± 15 seconds for all specimens being compared.

NOTE 6—The time delay between the start of mixing and initial measurement of specimen temperature should be as short as possible.

8.6.3 For typical test durations of less than 48 hours, measure the specimen temperatures at intervals of no greater than 60 seconds until at least two hours after the maximum temperature of the main peak response has been reached (Fig. 1). Alternatively, greater intervals are permitted to simplify data management for extremely gradual rates of specimen temperature change and/or test durations in excess of 48 hours.

9. Evaluation of Test Results

9.1 Test results are evaluated typically by comparing differences in thermal profiles from different test mixtures. See examples in the Appendix.

9.2 Plot specimen temperature as a function of time, using a common time scale relative to time at the start of mixing ($t = 0$ at the time of first wetting of cementitious materials, to the nearest minute) for all mixtures to be compared. Optionally, plot specimen temperature after subtraction of the temperature of the inert specimen at the corresponding elapsed time for each data point to isolate temperature changes due to hydration. It is permissible to plot segments of the thermal profile for special evaluation (see Note 7). Smoothing of temperature data is permissible if errant data points were logged that can be reasonably attributed to spurious data or any type of malfunction of measurement equipment.

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[4] Kantro, D.L. (1980). "Influence of water-reducing admixtures on properties of cement paste—a miniature slump test," *Cement, Concrete, and Aggregates*, 2, pp. 95-102.

NOTE 7—It may be useful to separately plot temperature data during the first 30 to 60 minutes of hydration, or other time period showing rapid temperature change, in order to expand the time scale for better display of rapid temperature changes due to calcium aluminate hydration. The usefulness of such early data may depend on timing of initial temperature measurements, insulation properties of specimen container and environment, specimen mass, and other equipment configuration details.

9.3 Indications of relative time of setting for different mixtures, if called for, shall be evaluated using the same fraction of the main peak response temperature rise (maximum temperature minus minimum dormant period temperature). See Note 8 and examples in the Appendix.

NOTE 8—A fixed fraction or percentage of the main peak response temperature rise is used as a temperature indication of relative time of setting when comparing different mixtures. For the given conditions, a fraction that approximates the times of initial or final setting of concrete, as defined by Test Method C403/C403M, may be selected to permit correlation with penetration resistance data. It is, however, often convenient to select a percentage that can be used easily in visual evaluation of thermal profiles, such as 50 %. In such cases, the thermal indication of times of setting may differ significantly from times of setting based on Test Method C403/C403M, but can still be useful in evaluation of the relative effects of different mixture variables on setting. See the Appendix for examples of use of thermal profiles for evaluation of setting trends and the influences of variables related to materials, proportions, and temperature conditions.

10. Report

10.1 Report the following information:

10.1.1 Type of equipment used including descriptions of specimen containers, layout and spacing of individual specimen containers, type and locations of temperature sensors, and any insulation used.

10.1.2 Signal-to-noise ratio as determined from test data for the specimens and test equipment and conditions, noting compliance with 6.2.1.

10.1.3 Source and identity of all materials tested, method of conditioning them to test temperature, and temperature prior to mixing.

10.1.3.1 If calcium sulfate was added, describe the specific type of calcium sulfate used and its source, as well as the timing of addition. If calcium sulfate hemihydrate was used, evidence shall be supplied of its hydration form before testing.

10.1.4 Mixture proportions, including the concentrations of any stock solutions used.

10.1.5 Mixing method and duration, including sequences and timing of mixing and scraping down, volume of mixing bowl or container used for mixing, and speed of mixer.

10.1.6 Addition sequence for all materials, and method of addition of admixture(s).

10.1.7 Method or description of any consolidation effort used.

10.1.8 Any unusual behavior, such as early stiffening during specimen preparation.

10.1.9 Mass of the test specimens placed in the specimen containers.

10.1.10 Test temperatures, initial mixture temperatures at the conclusion of mixing, date, time at the start of mixing and elapsed time to the first recorded specimen temperature, and duration of thermal measurements for each test mixture.

10.1.11 If thermal indication of relative time of setting is used, the fraction or percentage of main peak response temperature rise used in evaluation.

10.1.12 The results and test method used to measure fluidity or consistency of specimen, if applicable.

10.1.13 Plots of thermal profiles for all test mixtures and the temperature history of the inert specimen from the start of testing. See Appendix for examples.

10.1.14 Explanation of any periods of missing or flawed temperature data affecting individual thermal profiles, including any non-uniformity of the elapsed time from the start of mixing to first recorded specimen temperature.

10.1.15 Statement that the test was carried out in accordance with this practice and notes of any deviations from intended test conditions.

11. Keywords

11.1 cement – admixture interactions; hydration; setting; sulfate balance; thermal measurement testing; thermal profiles

APPENDIX

(Nonmandatory Information)

X1. TYPICAL APPLICATIONS

X1.1 Introduction

X1.1.1 Thermal measurement testing can be used to study setting characteristics, relative early-age hydration efficiency, and the potential for abnormal behavior in paste, mortar, or concrete mixtures. As such it can be used as part of concrete quality control, for the evaluation of candidate materials sources or materials variability, and to investigate the influences of different component materials, proportions, and concrete temperatures.

X1.1.2 Several examples of experimental evaluations are shown in this appendix. Each example represents a specific set of materials, and the results cannot be extrapolated to other sets of materials.

X1.2 Experiment Design and Planning

X1.2.1 While uses of thermal testing may include routine concrete or mortar mixtures for quality control or benchmarking of setting trends, many applications may be designed to answer questions about the influences of alternative materials sources, material variability, proportions, initial concrete temperature, and test temperatures. Thermal measurement testing should generally be planned to include a number of similar but distinct mixtures featuring specific variables, the performance effects of which are to be compared.

X1.2.1.1 Measurements of temperature according to this practice are typically subject to more variability than the temperature or heat measurements of more sophisticated calorimetry methods. Standardization of equipment is not usually warranted, and control of the test temperature may be approximate, affecting results to some extent. For these reasons, replicate test mixtures should include several increments of the variables of interest so that performance trends can be identified and inherent test variability evaluated and considered. Comparisons of thermal profiles obtained in different test series, at different locations, or using different equipment are not usually appropriate.

X1.2.2 The objectives of thermal measurement experiments may include evaluation of the effects of different cements, supplementary cementitious materials (SCMs), chemical admixtures, dosage rates, and addition sequences. Other parameters such as mix water source, presence of finely divided particles, materials variability, mixing method, initial mixture temperature, and test temperature can be studied as well. Experiments may be intended to evaluate the sulfate balance of a mixture, that is, whether the soluble calcium sulfate (contributed typically by the portland or blended cement) in a mixture is adequate for the materials, proportions, and project temperatures of interest.

X1.2.3 Chemical admixtures and SCMs may be selected and dosed based on submitted or envisioned concrete mixtures or supplier recommendations. It is recommended to include dosages that are both lower and higher than the envisioned dosage, in order to establish the mixture sensitivity to those materials. The dosage sequencing protocol for concrete batches may also be a variable of interest, as delayed addition of chemical admixtures, seconds or minutes after initial introduction of mix water and mixing effort, can be useful in avoiding sulfate-balance issues.

X1.2.4 Variation of both the initial mixture temperature and test temperature (usually simulating field temperatures of interest) are important to include in the experiment, because time of setting can vary unpredictably and sulfate-balance effects can change unexpectedly with temperature changes. While the effects of these variations can be evaluated using field testing at actual ambient temperatures, it is often useful to simulate field temperatures in laboratory experiments. Depending on the laboratory equipment and the number and distance between test specimens during testing, precise control of test temperatures is often a challenge due to the collective contribution of hydration heat from the test specimens. In such cases, the number of test specimens contained in a temperature-controlled cabinet or vessel may need to be limited in order to meet the reference temperature requirement of 6.2.3. The plotting of specimen temperature after the subtraction of reference temperature (see 9.2) usually helps to minimize the unwanted influences of changes in ambient temperature during testing.

X1.2.5 Cementitious mixtures of all types, including concrete, mortar, soil stabilization mixtures, grout, and paste, can be used in thermal measurement testing. Depending on the configuration of the available test apparatus, size and shape of specimen containers, and the insulation that will surround hydrating specimens, if any, selection of the appropriate type of mixture may influence the applicability of data produced. Peak hydration temperatures during testing will typically be reduced as the proportions of aggregate in the mixture increases. Likewise, as the volume and mass of the test specimen increases, the peak hydration temperature increases, other factors being equal. The most useful data are generally produced by balancing these factors so that peak temperatures achieved during testing result in adequate signal-to-noise ratio (see 6.2.2, Note 2, and Fig. 2) without exceeding the expected peak temperatures of field concrete in place. Artificially high peak temperatures during testing will often result in unrealistic thermal profiles, because different chemical compounds in the hydrating mixture respond to temperature differently with respect to the rate of hydration.

X1.2.5.1 In general, concrete or other mixtures with high aggregate content require larger (more massive) test specimens surrounded by insulation. The quality of data for concrete mixtures can often be improved by testing only the mortar fraction, obtained in accordance with Practice C172/C172M, especially when specimens are smaller (less massive) than ideal. Likewise, laboratory testing of paste-only specimens is often done using smaller specimens, without insulation around specimen containers.

X1.2.5.2 There should be uniformity of mixture consistency for the test specimens in a test series. Consolidate test specimens, if needed, to remove excessive entrapped air. Mix water should be proportioned to result in a uniform mixture without excessive segregation.

X1.2.6 Experiments intended to evaluate mixture sulfate balance should include a range of key variables (usually including all possible combinations of admixtures, SCMs and extremes of field temperatures) sufficient to demonstrate the relative contribution of each variable to sulfate balance issues. This will typically require including overdoses of admixtures and SCMs and initial and test temperatures higher than those anticipated in the field. Sulfate balance-related abnormal behavior may occur with only a slight incremental change in a critical variable. As such, even normal variability of a component material should be anticipated as a possible source of performance issues when a mixture is near its sulfate imbalance threshold.

X1.2.6.1 The evaluation of sulfate balance for a given set of materials and proportions can also be approached using incremental sulfate contents (see X1.5.6), through the addition of calcium sulfate in replicate mixtures or the use of multiple cement samples from the same source that vary in SO_3 content, to determine if normal main peak response (or sulfate “balance” as defined in Practice C1679) can be restored (see 5.2 and 5.6). Additions of reagent grade calcium sulfate may not necessarily result in the same performance as the equivalent increments of calcium sulfate introduced during the cement grinding process, but performance trends will be similar. Fig. X1.1 shows an example of the influences of incremental cement sulfate content in paste made with 25 % Class C fly ash and water reducing admixture. The abnormally-shaped, dual peak thermal profiles, reduced peaks, and delayed setting evident in the mixtures with lower sulfate levels can be confirmed as effects of sulfate imbalance, because a single, higher peak and normal setting was restored as sulfate was increased.

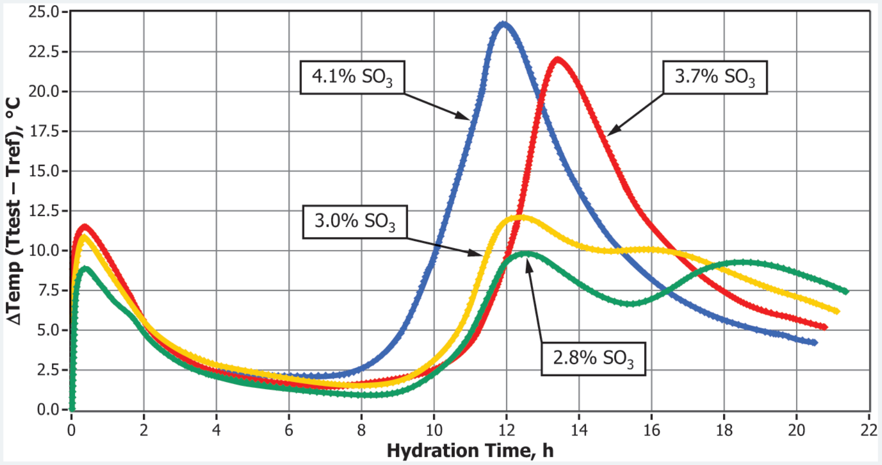

FIG. X1.1 The Effects of Incremental Cement SO_3 Content on Main Peak Response for a Mixture with 25 % Class C Fly Ash Replacement and Water Reducing Admixture at 35 °C [95 °F] Initial Mixture and Test Temperatures, *w/cm* = 0.40

X1.2.7 A mixture plan should be prepared before each test series, for efficiency of batching and mixing procedures during test execution and to serve as a record of the mixture materials and proportions. An example mixture plan is shown in Table X1.1. Test results from this mixture series are presented and discussed in X1.5.4.1.

X1.3 Mixing Methods

X1.3.1 Actual project concrete for thermal measurement testing can be sampled in the field according to Practice C172/C172M. Concrete mixed in the laboratory according to Practice C192/C192M can also be used. In either case, data variability can often

TABLE X1.1 Example Mixture Proportions for a Laboratory Paste Test Series

NOTE 1—1 mL/100 kg = 0.0154 oz/100 lb.

Channel	Temp, °C	Mixture description	Cement		SCM		Water		Admixture by product (rate, mL/100 kg) - dose, mL			time @data start
			Type & source	mass (g)	Type & source	mass (g)	w/cm	mass (g)	A/B/D	A/F	A/F/MR	
A1	23	No admix - 23 °C	Type II - project	500			0.40	200				
A2	23	ABD @ 195 - 23 °C	Type II - project	500			0.40	200	(195) - 0.98			
A3	23	ABD @ 390 - 23 °C	Type II - project	500			0.40	200	(390) - 1.95			
A4	23	AF @ 195 - 23 °C	Type II - project	500			0.40	200		(195) - 0.98		
A5	23	AF @ 390 - 23 °C	Type II - project	500			0.40	200		(390) - 1.95		
A6	23	not used										
A7	23	not used										
A8	23	23 °C reference - sand + water										
B1	13	No admix - 13 °C	Type II - project	500			0.40	200	(195) - 0.98			
B2	13	ABD @ 195 - 13 °C	Type II - project	500			0.40	200		(195) - 0.98		
B3	13	AF @ 195 - 13 °C	Type II - project	500			0.40	200		(390) - 1.95		
B4	13	AF @ 390 - 13 °C	Type II - project	500			0.40	200			(195) - 0.98	
B5	13	AMR @ 195 - 13 °C	Type II - project	500			0.40	200			(390) - 1.95	
B6	13	AMR @ 390 - 13 °C	Type II - project	500			0.40	200				
B7	13	not used										
B8	13	13 °C reference - sand + water										