



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

This standard is issued under the fixed designation C1753; 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 and health 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

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 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

C494/C494M Specification for Chemical Admixtures for Concrete

C1005 Specification for Reference Masses and Devices for Determining Mass and Volume for Use in the 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.

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.

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.

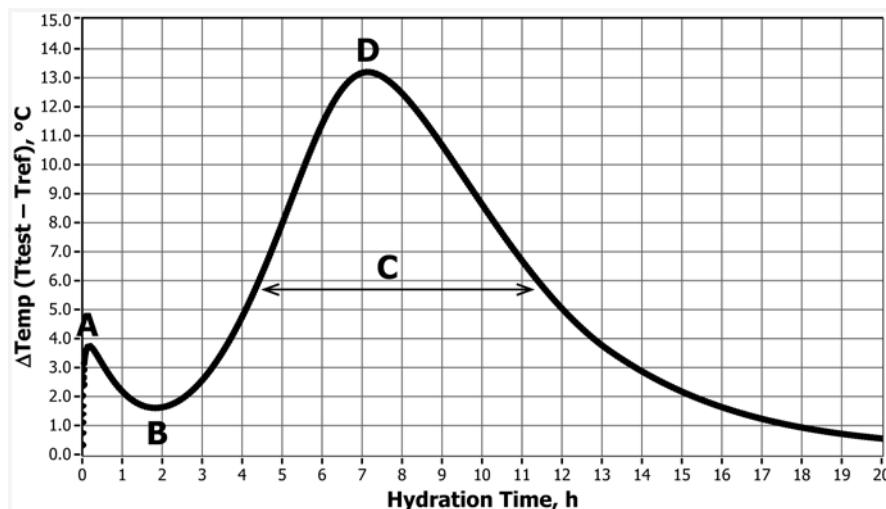
3.2.10 *thermal profile, n*—the temperature of a hydrating mixture (before or after subtraction of the reference temperature), plotted as a function of hydration time, that provides an indication of the rate of hydration over time.

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.

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.



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)

4.2 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 $\pm 1.0^\circ\text{C}$ [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).

NOTE 2—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,

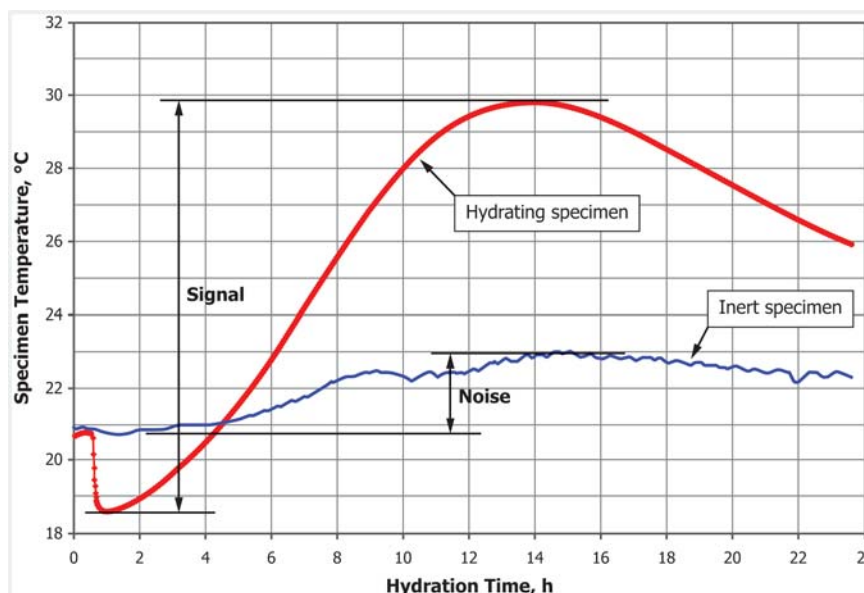


FIG. 2 Examples of Signal and Noise Determination for Verification of Signal-to-Noise Ratio

specimen mass, and initial mixture and test temperatures. A satisfactory inert specimen may be obtained using quantities of sand and water having masses within $\pm 10\%$ of the 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 specimen containers. The intent of a minimum signal-to-noise ratio requirement is to assure a well-defined thermal profile that is influenced minimally by ambient temperature changes and the presence of other test specimens, yet having a maximum main peak temperature that is 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 6.2.1 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 $3\text{ }^{\circ}\text{C}$ [$5\text{ }^{\circ}\text{F}$] during testing, unless deliberate change of ambient conditions during the period of temperature measurement is part of the test program.

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 $\pm 3.0\text{ }^{\circ}\text{C}$ [$5\text{ }^{\circ}\text{F}$].

8.1.2 *Materials and Initial Mixture Temperatures*—Precondition all materials as necessary to achieve an initial mixture temperature of $23.0 \pm 2.0\text{ }^{\circ}\text{C}$ [$73.5 \pm 3.5\text{ }^{\circ}\text{F}$] 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 $\pm 2.0^\circ\text{C}$ [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 to 1000 g [0.7 to 2.2 lb] for paste and 1500 to 4000 g [3.3 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 to 600 mL [10 to 35 in.³] for paste and 650 to 1650 mL [40 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 slump⁴ 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.

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, i.e., 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 SO₃ 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.

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 be reduced and signal-to-noise increased by testing mortar sieved from the concrete in accordance with Practice C172/C172M, although sieving of mortar from the concrete will increase the elapsed hydration time prior to first measurement of temperatures.

X1.3.2 Various laboratory mixing methods for mortar and paste have been used successfully, including methods presented in Practice C305 and other methods as indicated in Practice C1679. Modifications of these and other methods have been found advantageous, as well. Some method variations include shorter mixing times (often 60 seconds or less) that facilitate earlier data collection during initial hydration. Mixing devices and hardware may include kitchen mixers, paint stirrers, and simple mechanical agitation. For short duration mixing, cementitious materials are often introduced to the mixing bowl in a dry state, and mix water with dispersed

chemical admixtures is added subsequently as mixing begins. Hand-held kitchen mixers can also be used in such a way that separate scraping of the sides of the mixing bowl is unnecessary. Specific protocols can be adjusted to include delayed admixture additions to simulate what might occur at the concrete plant, as an experiment variable.

X1.3.3 Regardless of the specific mixing protocol used in a test series, it is important that the method produce homogeneous mixtures, that the method is repeatable, resulting in identical mixing for each mixture to be compared, and that timing of each stage of mixing be controlled to reproduce the mixing process as accurately as possible for each test mixture.

X1.3.4 It is possible that mixing intensity may affect results, especially when the shear applied by mixing differs significantly from that in concrete mixing. Thus test comparisons using different mixing methods for the same mixture proportions may be warranted to verify the suitability of the mixing protocol selected.

X1.4 Evaluation of Results

X1.4.1 Results of thermal measurement testing can be evaluated effectively only if comparative graphs include a control mixture and other mixtures of significance. Evaluation of results from a single test series may require multiple comparison graphs, each featuring a certain category of mixture variation (such as admixture dosage, SCM replacement rate, cement sample or SO_3 content). The scale used for hydration time axes should generally be the same for all graphs unless expanded scales of data segments are used to highlight periods of rapid hydration. Corresponding bar graphs of the results of other tests for each mixture (such as mixture flow or mini-slump, time of initial setting, compressive strength) may also be useful in evaluating thermal measurement results and correlating performance trends with thermal profiles.

X1.4.2 Relative times of setting and influences on setting can be studied by comparing the hydration times of main peak response among mixtures in the same test series. Because there may be variability in the magnitudes and shapes of these main

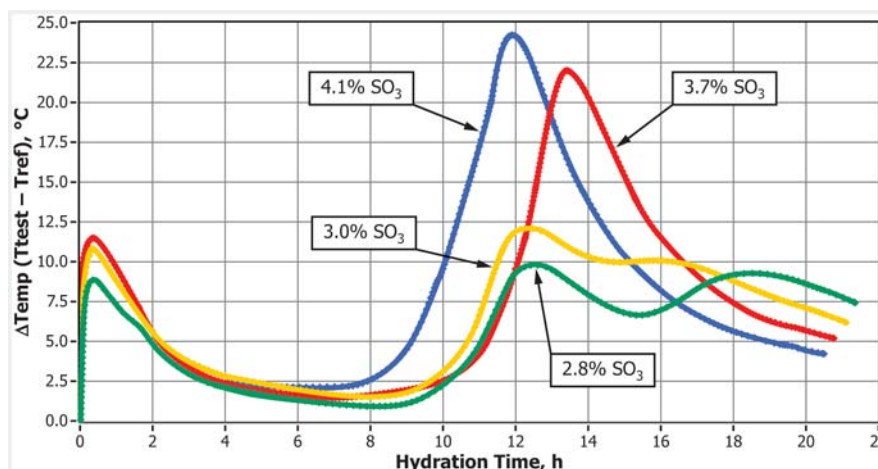


FIG. X1.1 The Effects of Incremental Cement SO_3 Content on Main Peak Response for a Mixture with 25% Class C Fly Ash Replacement and Water Reducing Admixture at 35 °C [95 °F] Initial Mixture and Test Temperatures, $w/cm = 0.40$

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										

peak responses, due to chemistry or fineness changes associated with different materials or proportions, the most consistent method is to compare hydration times corresponding to a selected fraction or percentage of the main peak response temperature rise. Fig. X1.2 shows a thermal profile with 20% and 50% fraction time of setting markers, which occur at approximately 4.9 and 6.4 hours of hydration time, respectively. A minimum temperature during the dormant period (see Fig. X1.2) may not be clearly evident in some thermal profiles or may be missing from data (often the case for tests of actual project concrete), and in such cases the reliability of relative times of setting based on fraction times determined from the thermal profiles is questionable.

X1.4.2.1 The hydration times at which a given fraction of the main peak response temperature rise occur can be computed from a data record using a spreadsheet or can be estimated from the graphs (50% fraction times are convenient for scaling). The hydration times corresponding to a selected fraction may differ somewhat from actual times of initial or final setting of concrete, as defined by Test Method C403/C403M, but trends of changes in fraction times observed in comparisons of different mixtures are generally consistent with trends based on penetration resistance testing.

X1.4.2.2 Comparison of relative times of setting by this process will become less reliable if there are significant differences between the maximum temperatures of main peak

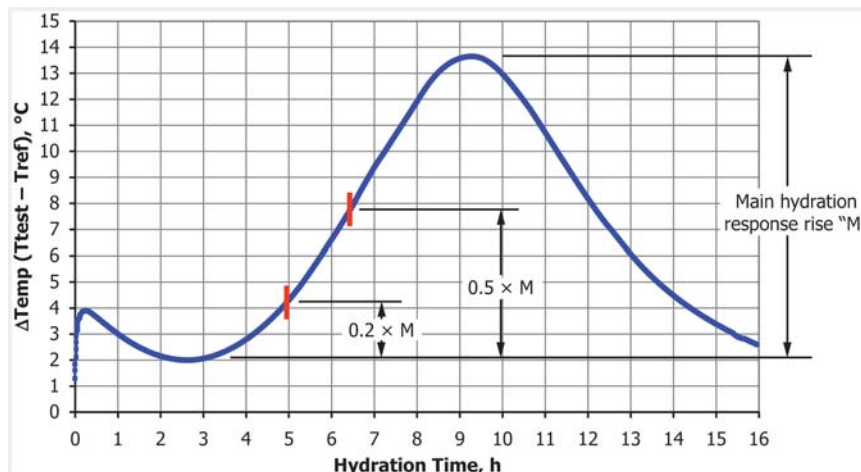


FIG. X1.2 Example – Time of Setting Markers Shown at 20% and 50% Fractions of Main Peak Response Temperature Rise for a Typical Mixture Thermal Profile

responses, which may indicate changes in hydration behavior, such as that seen in Fig. X1.1. Extreme caution should be exercised in drawing conclusions in these circumstances.

X1.4.3 In many cases, the times of initial and final setting of concrete as determined by Test Method C403/C403M can be approximated using thermal profiles of concrete, or mortar sieved from concrete in accordance with Practice C172/C172M. The actual fractions used may be based on empirical relationships established from experience with various materials, or can be refined for a given set of materials. This approach may be useful as an alternative to penetration resistance testing, as in Test Method C403/C403M, for development of QC data by the concrete producer or contractor. When paste of otherwise similar proportions as the paste fraction of mortar or concrete and the same % fraction is used, the associated indication of time of setting will generally be longer.

X1.4.4 Thermal profiles of mixtures that are affected by sulfate imbalance will differ in shape, timing, and magnitude of peaks, as compared with normal hydration profiles. Depending on the severity of imbalance and the time of soluble sulfate depletion, excessive hydration of calcium aluminates will usually be indicated as a latent exotherm after the main peak response has begun or as a sudden exotherm before the main peak response. The time when the main peak starts, its duration, and maximum temperature of the main peak response are also usually affected. If this indication of excessive calcium aluminate hydration takes place before the main peak response begins, the hydration of calcium silicates that normally results in main peak response (along with associated time of setting and strength gain) may be interrupted for an extended period of time. In such cases, data collection for 48 to 72 hours may be useful for a complete understanding, and time of setting evaluation using the fraction method may not be appropriate. Parallel compressive strength testing of such mixtures at early ages (usually 1 to 3 days) may also be especially useful in confirming this behavior, as interrupted calcium silicate hydration clearly results in significant strength gain delays. Fig. X1.3 shows thermal profiles for a series of paste mixtures, using a

single cement sample, that range in sulfate balance from normal (A) to severely affected (D) and (E), due to the increasing admixture dosages. The mixture-to-mixture variation in initial peak (during the first hour of hydration time) and subsequent temperature drop as the dormant period begins are the result of changes in the extent to which levels of sulfates in solution are adequate for control of initial aluminate hydration. As more extreme initial aluminate hydration occurs due to this sulfate starvation, main peak response is affected and components of recurring aluminate hydration can be seen in the thermal profiles, later in time. Compressive strengths for each of the paste mixtures at 1 day were determined by testing the hardened cylindrical paste specimens that were used in thermal testing in accordance with Test Method C39/C39M. These strength results are shown in the inset bar chart, and this protocol was also used in some of the application examples to follow.

X1.5 Examples

X1.5.1 Effects of Specimen Type, Mass, and Insulation:

X1.5.1.1 Selection of optimum specimen size and surrounding insulation, if any, depending on the type of test specimens used (concrete, mortar, or paste) will help assure adequate signal-to-noise ratio and reasonable peak hydration temperatures, as discussed in X1.2.5. The following examples demonstrate these effects.

X1.5.1.2 *Paste vs. Mortar*—Fig. X1.4 shows thermal profiles produced from laboratory mixtures of the paste and mortar fractions of a concrete mixture of interest containing Type II cement with 25% Class F fly ash replacement and a Type A water reducing admixture, $w/cm = 0.52$, at 32 °C [90 °F] initial mixture temperature. Mortar was made with the same proportions of cementitious materials used for the paste, with the addition of concrete sand at approximately 2.6 times the mass of the cementitious materials. Equal volume (approximately 500 mL [30 in.³]) specimens of paste and mortar were tested using non-insulated 76 × 114 mm [3 × 4.5 in.] specimen containers at a 32°C [90 °F] test temperature. Fig. X1.4(a) shows the thermal profiles plotted on the same scale, while Fig.

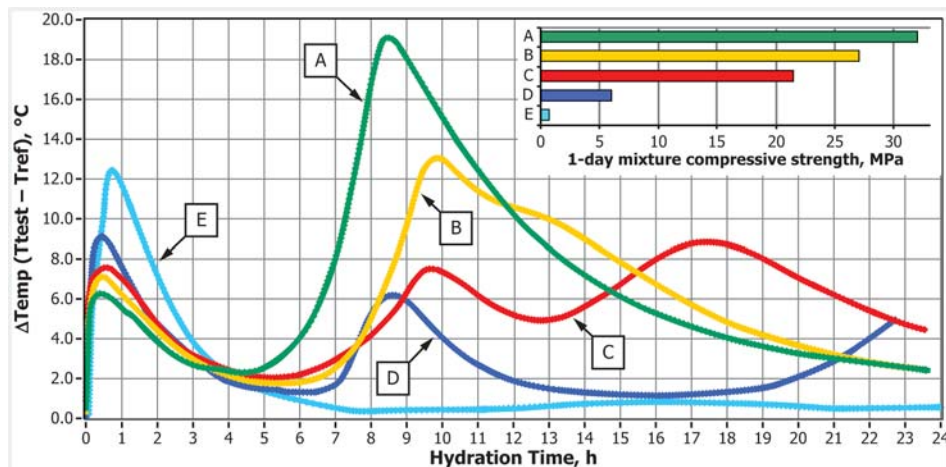


FIG. X1.3 Thermal Profiles and 1-Day Strengths for Paste Mixtures Indicating Normal Hydration (A) and Sulfate Imbalance of Increasing Severity (B through E) (Note: 1 MPa = 145 psi)

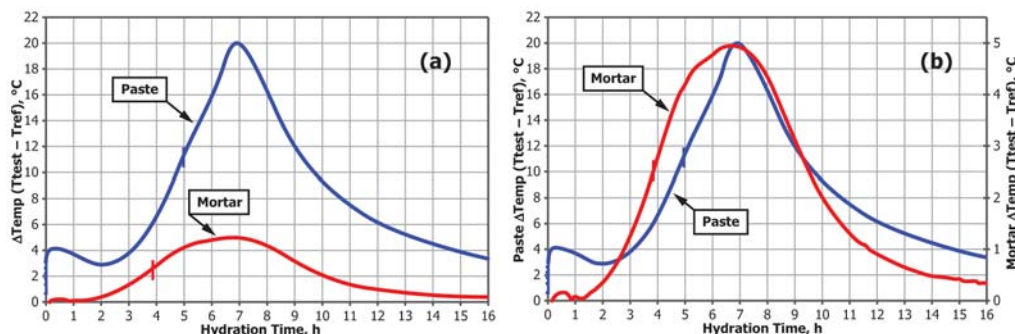


FIG. X1.4 (a) Equal Volumes of Similar Paste and Mortar Mixtures Compared; (b) Same Comparison Using an Expanded Temperature Scale for the Mortar Profile

X1.4(b) uses an expanded temperature scale for the mortar, to better define and compare its shape. The 50% fraction time of setting markers are shown on each thermal profile for reference. Note that the aggregate content of the mortar has resulted in significantly reduced peak hydration temperature, and a resulting lower signal-to-noise ratio. The use of insulation around specimen containers of this size for mortar can improve the signal-to-noise and repeatability of measurements by increasing the peak hydration temperature.

X1.5.1.3 *Specimen Mass*—Fig. X1.5 shows thermal profiles for laboratory-made mortar or paste mixtures of the same proportions as the paste fraction of the mortar, but varying specimen mass and size, tested without insulation at 23°C [73 °F] initial mixture and test temperatures. The paste mixture featured in Fig. X1.5(a) was made using Type II cement with 25% Class C fly ash replacement, a Type A water reducer, and $w/cm = 0.51$. Incrementally larger cylindrical specimen containers of approximately 2:1 height-to-diameter were used, resulting in specimen mass that varied as indicated from 240 to 1900 g [0.5 to 4.2 lb]. The mortar used for mixtures featured in Fig. X1.5(b) was made with identical materials and proportions as used for the paste, plus the addition of concrete sand at 2.75 times the mass of cementitious materials. Cylindrical specimen molds of 76 × 152 mm [3 × 6 in.] and 102 × 203 mm [4 × 8 in.] were used, and resulting specimen masses were 1560 and 3700 g [3.4 to 8.2 lb], respectively. Note that peak hydration temperatures increased and indicated main peak response was slightly delayed as specimen mass was increased. Also, the time of setting based on the 50% fraction was affected to a minor extent.

X1.5.1.4 *Insulation and Combinations of Insulation and Specimen Mass*—Thermal profiles of mortar and paste mixtures that demonstrate effects of changes in insulation around specimen containers and effects of different combinations of insulation and specimen mass are presented in Fig. X1.6. These thermal profiles have been plotted without subtraction of corresponding inert specimen temperatures in order to call attention to measured peaks. The mortar used for Fig. X1.6(a) thermal profiles was made using Type II cement with 25% Class F fly ash replacement, a Type A water reducing admixture, $w/cm = 0.51$, and concrete sand at 2.75 times the mass of cementitious materials. The approximate initial mixture and test temperature was 23° C [73 °F]. Replicate batches of mortar of the same proportions were tested in identical 76 × 152 mm [3 × 6 in.] cylindrical specimen molds that varied only with respect to the insulation provided – no insulation and rigid foam insulation of either 15 or 50 mm [0.6 or 2.0 in.] thickness surrounding the containers on all sides. Note that peak temperatures increase with the amount of insulation used – effects similar to those resulting from changes in specimen mass, as in X1.5.1.3.

X1.5.1.5 Paste mixtures featured in Fig. X1.6(b) were made using Type I cement with 25% Class C fly ash, a Type A water reducing admixture, and $w/cm = 0.51$. Initial mixture and test temperatures were approximately 20°C [68 °F]. Paste batches were repeated, using the same proportions but with different variations of mass, specimen container, and surrounding insulation. These conditions included a 620 g [1.4 lb] specimen in a paper coffee cup approximately 64 × 102 mm [2.5 × 4 in.] with no insulation, an identically sized specimen in a similar

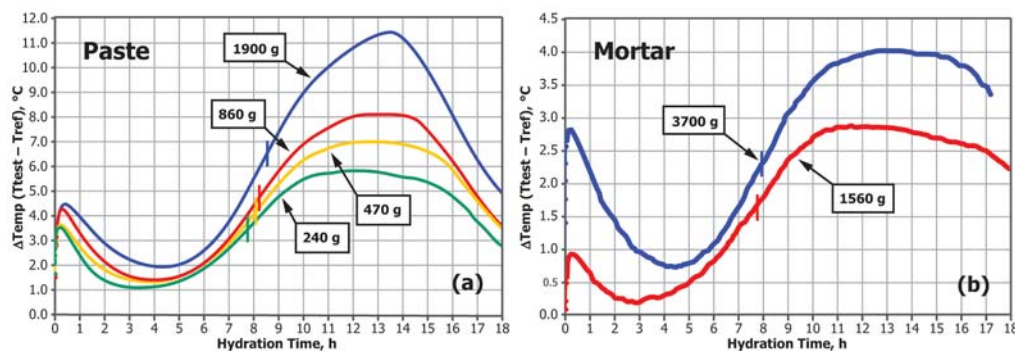


FIG. X1.5 (a) Effects of Specimen Mass in Paste; (b) Effects of Specimen Mass in Mortar

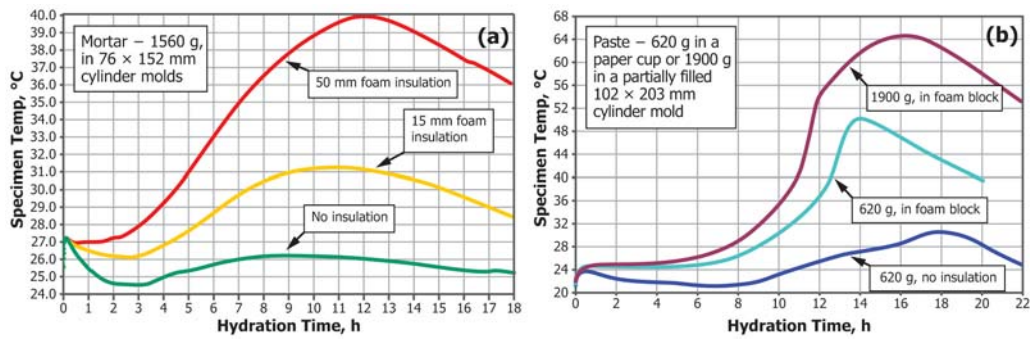


FIG. X1.6 (a) Effects of Changes in Surrounding Insulation on Repeated Mortar Mixtures; (b) Effects of Varied Insulation and Specimen Mass on Repeated Paste Mixtures

paper cup placed in a foam block with at least 65 mm [2.5 in.] of insulation on all sides, and a 1900 g [4.2 lb] specimen in a 102 × 203 mm [4 × 8 in.] specimen container (partially filled) placed in a foam block with similar insulation conditions except for no insulation covering the top of the container. Note that peak hydration temperatures increase with specimen mass and insulation, as illustrated in the other examples. As these peak temperatures become more extreme, however, the thermal profile shape is considerably altered from the increased hydra-

tion rate of calcium aluminate, which can be disproportionately accelerated at higher temperatures. This can potentially affect the usefulness of such profiles in comparing mixtures. For the most useful data, therefore, peak hydration temperatures measured in a test series should not exceed those expected for in-place concrete in the field.

X1.5.2 Effects of w/c:

X1.5.2.1 Fig. X1.7 shows the effects of changes in water-

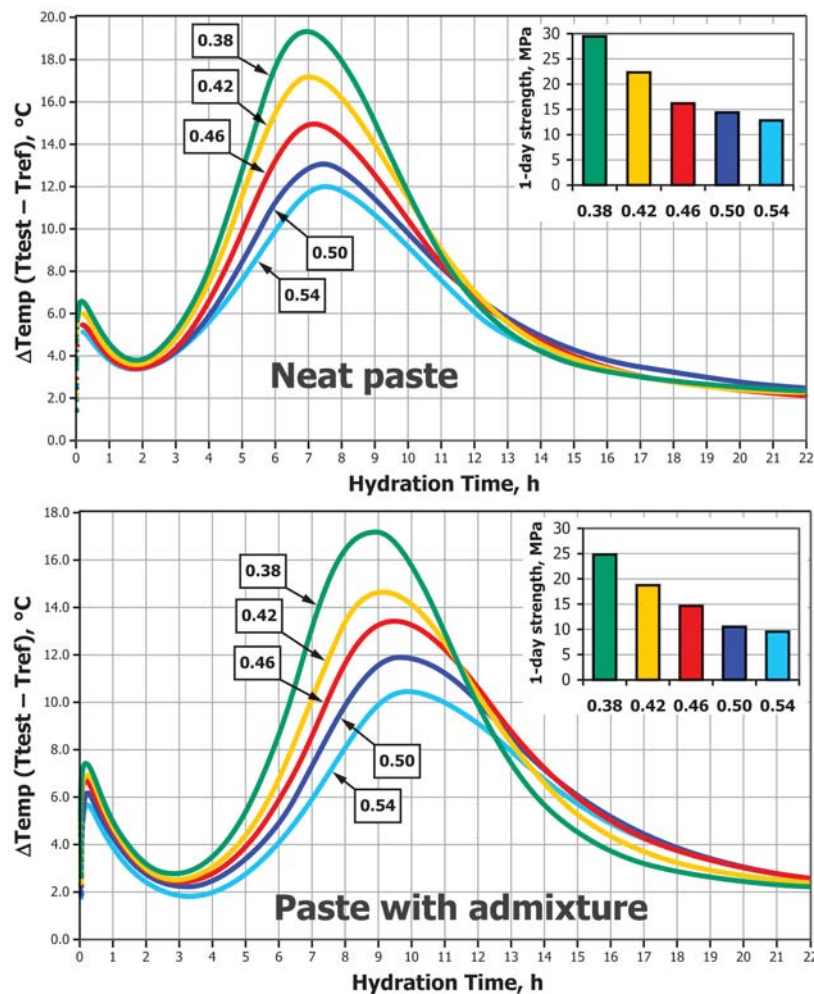


FIG. X1.7 Effects of w/c on Paste Thermal Profiles and Strength (Note: 1 MPa = 145 psi)

cement ratio (w/c) on thermal profiles for paste mixtures made using a single sample of Type II cement without admixtures (upper graph) and with a Type A/D water-reducing admixture (lower graph). Approximately 900 g [2.0 lb] of paste for each mixture was tested in non-insulated 76 × 114 mm [3 × 4.5 in.] specimen containers. Initial mixture and test temperatures were approximately 23 °C [73 °F], and w/c was varied from 0.38 to 0.54. The 1-day compressive strengths determined according to Test Method C39/C39M are shown in the corresponding bar charts. Note that as w/c is increased, the peak temperatures decrease. The time when the main peak response starts is not affected significantly, though there is indication of increased time of setting in the mixtures with admixture compared to those without. The same w/c changes in concrete mixtures, however, would produce noticeably different concrete times of setting as determined by Test Method C403/C403M. For this reason, w/c is not a variable that can be evaluated for effects on concrete time of setting using thermal testing. For a constant mass of paste, the actual mass of hydrating cement is reduced as the w/c increases. Thus much of the difference in thermal profiles is due simply to the amount of cement available for hydration per unit mass of paste. This is in contrast to isothermal calorimetric methods, where the heat flow can be expressed as heat flow per mass of cement, thus correcting for this effect. For this reason and to simplify evaluation of the effects of other mixture variables, w/c is not usually an experiment variable in thermal measurement testing. Test series should generally be planned using a fixed, representative w/c , or one selected to assure reasonable and uniform mixture consistency without significant bleeding. If w/c is to be investigated, the best comparison will be achieved if the paste or mortar specimen masses are adjusted to maintain similar masses of cementitious materials.

X1.5.3 Effects of Initial Mixture and Test Temperatures:

X1.5.3.1 Just as initial concrete temperatures influence concrete performance, initial mixture temperatures and the test temperature have significant influences on the results of thermal testing. Ideally, for optimum data quality, the initial mixture temperature should be the same as that of the test temperature, especially if the testing plan calls for little or no insulation. If equipment for controlling test temperature (such as a temperature-controlled laboratory cabinet) is not available, a testing protocol that includes insulated specimens of appropriate mass will generally produce more useful data, though setting trends indicated by the thermal profiles may be less consistent with trends established using penetrometer testing. If sulfate balance effects are being evaluated, initial mixture temperature and test temperature extremes have a critical influence on performance.

X1.5.3.2 Fig. X1.8 presents thermal profiles from a series of similar laboratory mortar mixtures with three different initial mixture and test temperatures (15, 22, and 32 °C [59, 72, and 90 °F]). A single sample of Type II cement was used throughout, along with a single sample of Class C fly ash at 25% replacement (in 3 of 4 mixtures), an admixture that is marketed for either Specification C494/C494M Type A (water reduction) or Type D (water reduction and retardation) applications, depending on the dosage rate used (in 2 of 4

mixtures), and project concrete sand. Mixture proportions are summarized in Table X1.2. Each mortar specimen was approximately 1560 g [3.4 lb] in mass and was tested in a 76 × 152 mm [3 × 6 in.] cylindrical specimen container, without insulation. The 22 °C [72 °F] condition was the ambient laboratory temperature, while 15 and 32 °C [59 and 90 °F] conditions were accomplished using a temperature controlled curing tank for heating or cooling materials before mixing as well as for controlling the test temperature throughout data collection. The test specimens are not placed in the curing tank water, but are stored on a platform above the water. Thermal profiles are shown with 50% fraction time of setting markers, for reference. Temperature conditions influence the time of setting indications, as expected, with greater influence when the admixture is used. A significant sulfate imbalance is indicated for mixtures made with the admixture and tested at 32 °C [90 °F], especially for the higher (Type D) dosage. The time of setting markers for these mixtures are omitted because normal setting did not occur. No such issues were experienced at lower temperatures. This illustrates the importance of using extreme field temperatures in testing.

X1.5.3.3 Fig. X1.9 shows thermal profiles for specimens from the same mixtures of paste made at initial temperatures of approximately 32 °C [90 °F] but subjected to different test temperatures (32 and 20 °C) [90 and 68 °F]. Three different Type II cements (A, B, and C) were compared in otherwise identical mixtures with 25% Class F fly ash replacement and a Type A water reducing admixture, at $w/cm = 0.40$. Paste specimens were each approximately 360 g [0.8 lb] and were tested using non-insulated cylindrical 51 × 102 mm [2 × 4 in.] specimen containers. Materials were pre-heated to slightly over 32 °C [90 °F] before mixing. A temperature controlled curing tank was used for the 32 °C [90 °F] test temperature, and the 20 °C [68 °F] condition was room ambient. Fig. X1.9(a) shows thermal profiles without subtraction of corresponding inert specimen (reference) temperatures, which are plotted separately on the same graph. Fig. X1.9(b) shows the same data, plotted with subtraction of the reference temperatures. While either plotting approach could be used for data evaluation, note that profile shapes are affected by the initial change in specimen temperature for the ambient (20 °C [68 °F]) test temperature (mixture temperature begins to drop before the effects of hydration become evident), complicating evaluation somewhat. The main peak response has been delayed and reduced as expected, but the indicated initial (calcium aluminate) peak is unrealistically exaggerated relative to the main peak response. This might be mistaken for incompatibility behavior or otherwise misinterpreted, because the initial portions of the thermal profiles are affected by differences between initial mixture temperatures and the test temperature, rather than hydration kinetics. Similar initial mixture and test temperatures produce generally more useful data. If it is necessary to use a different test temperature than the initial mixture temperature, this should be clearly noted in reporting.

X1.5.4 Studies of the Effects of Changes in Materials, Proportions, and Temperature:

X1.5.4.1 Effects of Different Admixtures, Dosages, and Test Temperatures—Fig. X1.10 shows thermal profiles for paste

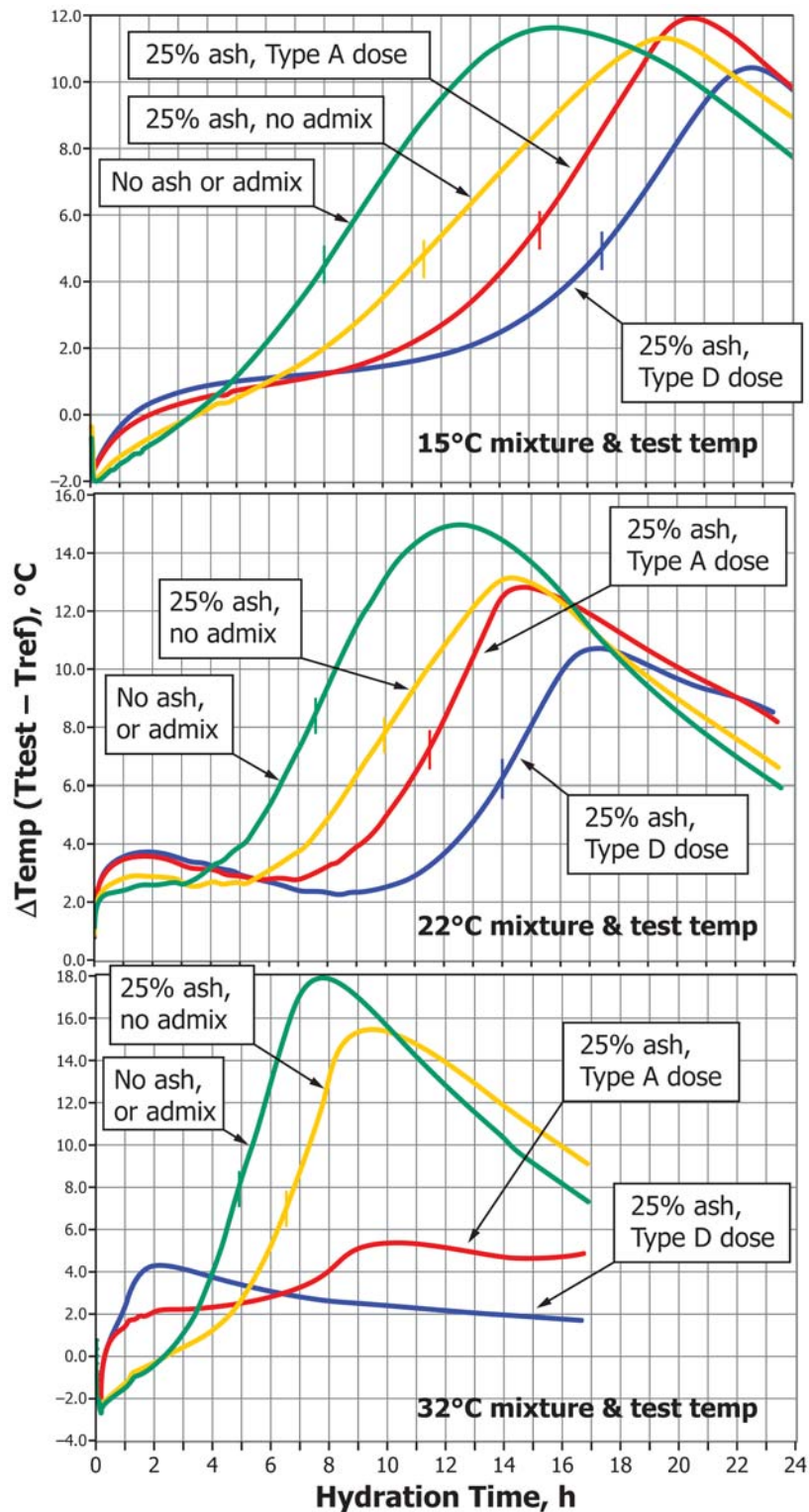


FIG. X1.8 Effects of Different Temperatures (Initial Mixture and Test Temperature) on Mortar

mixtures that were tested at two different initial mixture and test temperatures in order to compare the retarding influences and temperature sensitivity effects of different admixtures. Paste mixtures made without SCMs at $w/c = 0.40$ were tested in non-insulated paper coffee cups approximately 64×102 mm [2.5×4 in.], each specimen was approximately 600 g [1.3 lb].

All mixtures were made with the same sample of Type II cement and were of identical proportions except for the admixture products and dosages. Fig. X1.10(a) compares results at 23 °C [73 °C] and Fig. X1.10(b) shows those at 13 °C [55 °C]. Thermal profiles are shown with 50% fraction time of setting markers. A temperature-controlled curing tank was used

TABLE X1.2 Mixture Proportions for Temperature Effects Example Mixtures (Fig. X1.8)

NOTE 1—1 mL/100 kg = 0.0154 oz/100 lb; 1 mL = 0.0338 oz.

Mixture description	cement, g	fly ash, g	w/cm	water, g	admix dosage rate	dose, mL	sand, g
No ash or admix	500		0.45	225			1360
25% ash, no admix	375	125	0.45	225			1360
25% ash, Type A dose	375	125	0.40	200	Type A, 260 mL/100 kg	1.30	1360
25% ash, Type D dose	375	125	0.40	200	Type D, 390 mL/100 kg	1.95	1360

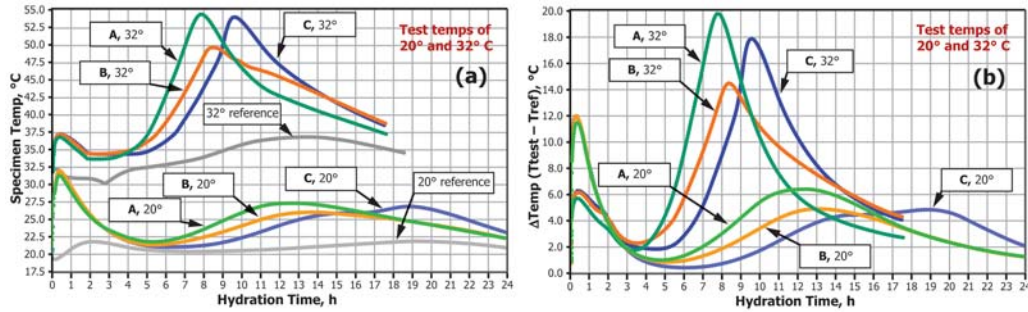
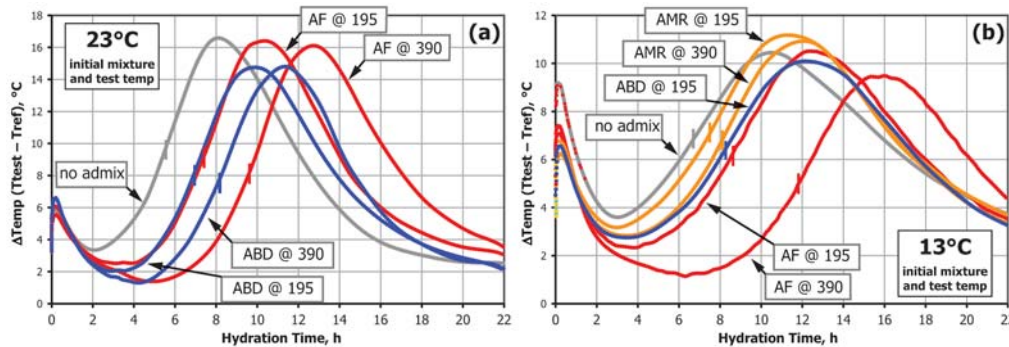


FIG. X1.9 Effects of Different Test Temperatures for Paste Specimens Mixed at 32 °C [90 °F] (a) Plotted without Subtraction of Reference Temperatures; (b) Same Data Plotted with Subtraction of Reference Temperatures



NOTE 1—See Table X1.3 for descriptions of admixtures; indicated dosage rates are mL/100 kg of cement.

FIG. X1.10 Effects of Admixtures and Dosages on Paste (a) at 23 °C [73 °F]; (b) at 13 °C [55 °F]

for the 13 °C [55 °C] test temperature and cooling of materials before mixing; the 23 °C [73 °C] test temperature condition was ambient room temperature. Descriptions and dosages of the admixture products are summarized in Table X1.3. Note that with this cement, the AF admixture appeared to result in more retardation at the higher dosage than even the ABD product, and also contributed more retarding influence at the lower temperature, even though it is marketed as a “set neutral” product. The AMR product included among the 13 °C [55 °C] mixtures shown in Fig. X1.10(b) showed little associated

retardation or temperature sensitivity for the same dosages, which were on the lower end of the recommended range for each product.

X1.5.4.2 Evaluation of Fly Ash Replacement Level—Thermal measurement testing can be useful in evaluating SCM effects in a proposed mixture for a given set of materials and expected field temperatures, to aid in proportioning decisions. Fig. X1.11 shows thermal profiles from a series of laboratory paste mixtures ($w/cm = 0.40$) made and tested at the highest expected project field temperature of 34°C [93 °F], using a

TABLE X1.3 Admixtures and Dosages as Used in Example Test Series (Fig. X1.10)

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

Product ID in graphs	Manufacturer's product information			Test dosages used, mL/100 kg cement	
	water reduction categories	setting influences	recommended dosage range, mL/100 kg cement	low dose	high dose
AMR	Type A & mid-range	set neutral	195 to 980	195	390
AF	Type A, F, & mid-range	set neutral	195 to 780	195	390
ABD	Type A, B, & D	some retardation	195 to 460	195	390

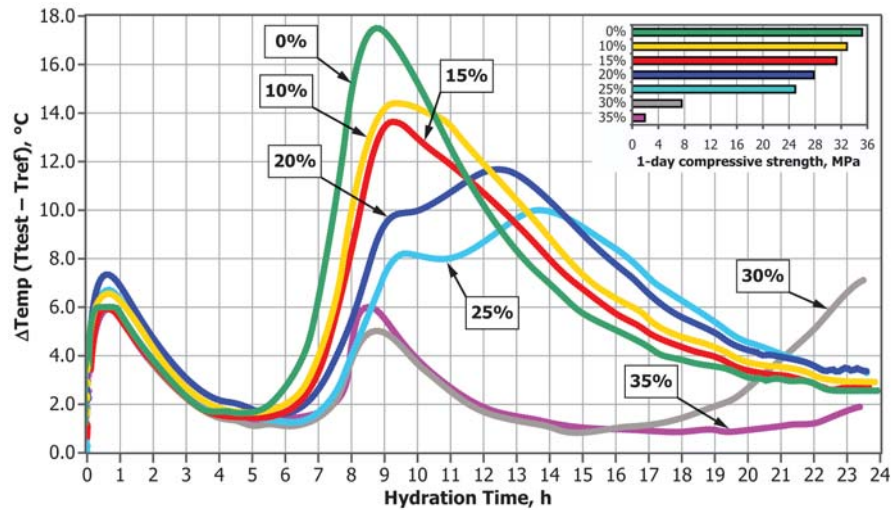


FIG. X1.11 Effects of Fly Ash Replacement Level in Paste Mixtures Made with a Type A/D Admixture at 34°C [93 °F] Initial Mixture and Test Temperature (Note: 1 MPa = 145 psi)

single sample of the Type II cement to be used and the highest expected dosage (390 mL/100 kg [6 oz/100 lb], based on total cementitious materials content) of a Type A/D admixture. Fly ash replacement level was incrementally increased from 0% to 35% using a single sample of Class C fly ash from the source planned for use in the project. Paste specimens were each about 350 g, tested in non-insulated 51 × 102 mm [2 × 4 in.] specimen containers. The fly ash replacement levels used for each mixture are indicated on the thermal profiles, and corresponding 1-day compressive strengths of the same specimens are shown in the inset bar chart. Note that sulfate imbalance begins to be indicated as fly ash replacement level is increased above 15%. Considering the possible effects of variability of other mixture components not included as test variables, it might be prudent to limit the project fly ash replacement level to around 15% with this set of materials and other proportions, if concrete temperatures in the field are to be this high.

X1.5.4.3 Evaluation of Sulfate Imbalance Effects of Admixture Dosage—The effects of possible sulfate imbalance resulting from higher admixture dosages can also be evaluated using thermal measurement testing. Fig. X1.12 shows thermal profiles of laboratory paste mixtures made and tested at 32 °C [90 °F] using a single sample of Type II cement, 25% replacement with a single sample of Class C fly ash, and dosages of a Type A/D admixture ranging from 0 to 520 mL/100 kg [0 to 8 oz/100 lb], based on total cementitious materials content. Paste specimens were each about 350 g [0.8 lb], tested in 51 × 102 mm [2 × 4 in.] non-insulated specimen containers. Admixture dosage is indicated for each thermal profile, and corresponding 1-day compressive strengths of the same specimens are shown in the inset bar chart. Note that effects of dosage increases up to 260 mL/100 kg [4 oz/100 lb] appear to result only in expected retardation, but sulfate imbalance indication is evident at the

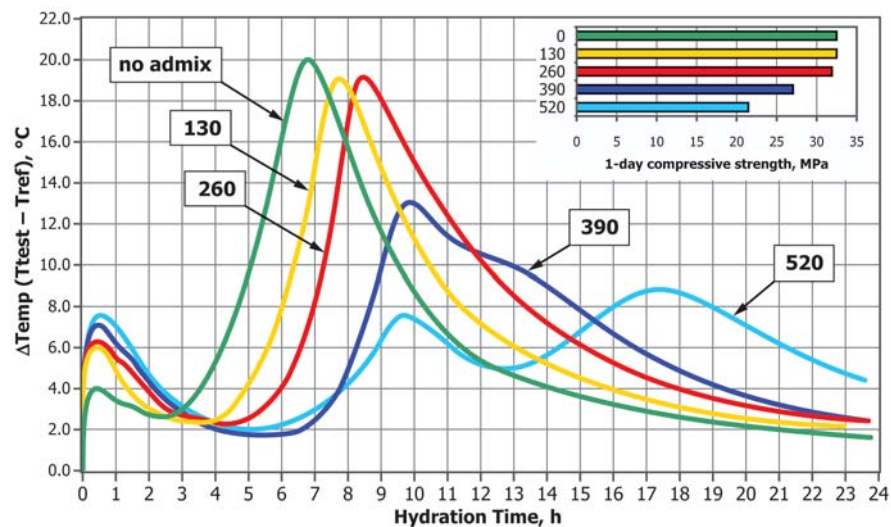


FIG. X1.12 Effects of Admixture Dosage (as Indicated, in mL/100 kg of Total Cementitious Materials) for 32°C Paste Mixtures Made with 25% Class C Fly Ash Replacement of Cement (Note: 1 mL/100 kg = 0.0154 oz/100 lb., 1 MPa = 145 psi)

390 mL/100 kg [6 oz/100 lb] dosage, becoming more significant at the 520 mL/100 kg [8 oz/100 lb] dosage.

X1.5.5 Studies Comparing the Performance of Different Materials Sources:

X1.5.5.1 Evaluation of Different Cements for Sulfate Adequacy—The performance of different cement sources in a proposed concrete mixture having potentially high demand for sulfate can be evaluated using thermal measurement testing. The paste fraction for such a concrete mixture was modeled in laboratory mixtures comparing the performance of seven cement sources at the expected extreme field temperature of 34 °C [93 °F] (initial mixture and test temperature). Thermal profiles and 1-day compressive strengths of cylindrical specimens of the same paste batches used for thermal testing are shown in Fig. X1.13. The mixtures were made using samples of Types I and II cements from seven sources, 25% Class C fly ash replacement, and a Type A water reducing admixture, at $w/cm = 0.40$. Paste specimens were approximately 600 g [1.3 lb] each and were tested in non-insulated paper coffee cups approximately 64 × 102 mm [2.5 × 4 in.]. Note that the different cement sources result in different indications of times of setting and measured peak temperatures even when profiles suggest normal performance (A, E, D), due to expected differences in cement chemistry and fineness. Note also that mixtures with cements C, G, and possibly B indicate mild sulfate imbalance while the mixture with cement F has a severe sulfate imbalance under the test conditions.

X1.5.5.2 Comparison of Different Fly Ash Sources—The performance of different SCMs can be similarly evaluated for use in a project concrete mixture where potential sulfate imbalance is of concern. Fig. X1.14 includes thermal profiles and 1-day compressive strengths of additional (cylindrical) specimens of the same paste batches from three otherwise identical paste mixtures, comparing different sources of Class C fly ash. The mixtures used a single source of Type I cement and a Type A admixture. Fly ash replacement was 30% and w/cm was 0.40. Initial mixture and test temperatures of 32 °C [90 °F] were obtained using a temperature-controlled curing tank, and 600 g [1.3 lb] paste specimens from each mixture

were tested in non-insulated paper coffee cups approximately 64 × 102 mm [2.5 × 4 in.]. The mixture made with fly ash source “A” indicates no sulfate balance issues, while mixtures with the other sources each suggest slight imbalance. Additional mixtures at varying replacement rates can be used to further define the sensitivity of each source for more guidance on the use of these materials at different proportions.

X1.5.6 Evaluation of Sulfate Balance:

X1.5.6.1 Test Series Design—Concrete field issues relating to slump loss, abnormal setting tendencies, and poor early-strength gain may be related to sulfate balance issues, which have often been misdiagnosed. Thermal measurement testing is well suited for the evaluation of such issues, in the field or laboratory, including verification of proposed remedial measures. Design of a test series for this purpose may be approached from either incremental sulfate *demand* mixtures or incremental sulfate *level* mixtures. Incremental sulfate *level* is where mixtures that are otherwise similar contain increasing levels of soluble calcium sulfate content. Incremental sulfate *demand* is where mixtures that are otherwise similar contain increasing levels of SCMs, increasing dosages of admixtures, or increasing initial mixture and test temperatures. The examples presented in X1.5.4.2 and X1.5.4.3 represent cases of otherwise similar mixtures with increasing sulfate demand as influenced by these variables. An effective test series with incremental sulfate level mixtures may be planned using multiple samples of the same cement source selected to include a range of measured total SO₃ levels. If only a single sample of the cement source of interest is available, a series of laboratory mixtures with incremental sulfate levels can be planned using additions of a calcium sulfate source such as calcium sulfate dihydrate, hemihydrate or Terra Alba⁵ gypsum.

X1.5.6.2 Example of Test Series with Laboratory Calcium Sulfate Additions—Fig. X1.15 shows thermal profiles and 1-day compressive strengths of the same specimens for two

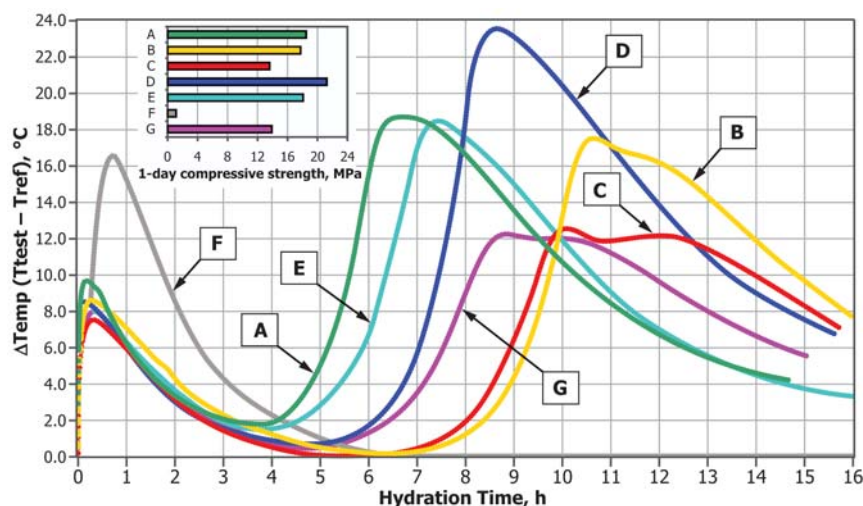


FIG. X1.13 Cement Sources Compared in Laboratory Paste Mixtures, Test Temperature = 34 °C [93 °F] (Note: 1 MPa = 145 psi)

⁵ Terra Alba No. 1 gypsum is supplied by the U.S. Gypsum Co., sourced from the Southard, OK plant.

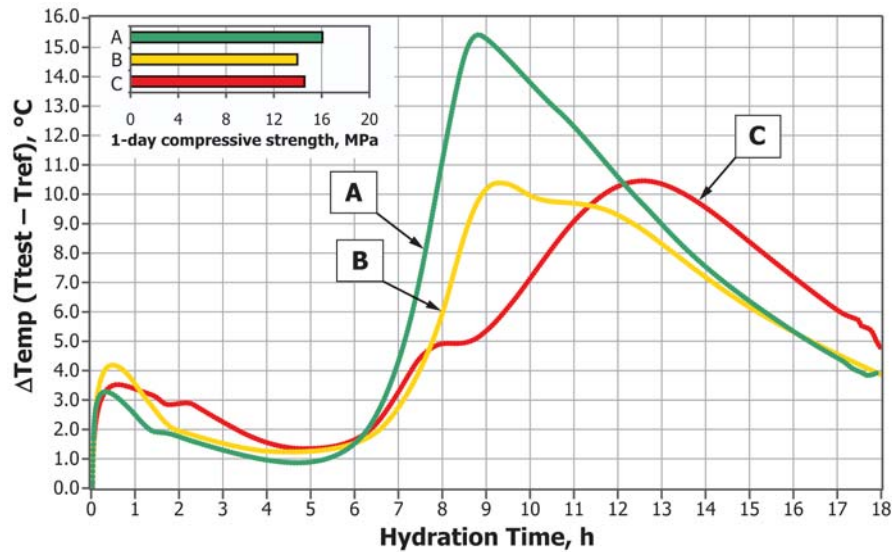


FIG. X1.14 Three Fly Ash Sources Compared in Paste Mixtures of Identical Proportions at 32 °C [90 °F] (Note: 1 MPa = 145 psi)

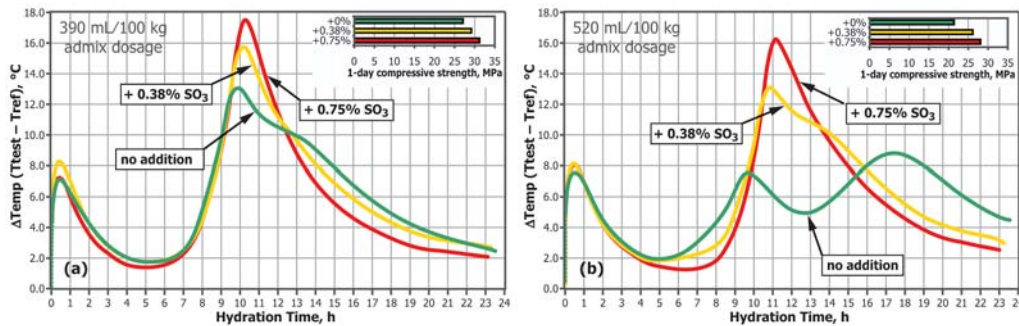


FIG. X1.15 Effects of Incremental Addition of Calcium Sulfate to 32 °C [90 °F] Paste Mixtures Made with 25% Class C Fly Ash Replacement and (a) 390 mL/100 kg [6 oz/100 lb.] and (b) 520 mL/100 kg [8 oz/100 lb.] of Type A/D admixture (Note: 1 MPa = 145 psi)

series of laboratory paste mixtures designed to help evaluate whether field reports of occasional concrete slump loss and somewhat erratic time of setting and early strengths may be sulfate balance related. The paste fraction proportions of the concrete mixture were used in laboratory paste mixtures at a test temperature representative of the field concrete. This was done using a single sample of Type II cement from the project with 25% Class C fly ash replacement at $w/cm = 0.40$ and 32 °C [90 °F] initial mixture and test temperatures, using a temperature-controlled curing tank. Paste specimens were each about 350 g [0.8 lb], tested in non-insulated 51 × 102 mm [2 × 4 in.] specimen containers. The series shown in Fig. X1.15(b) differs from that in Fig. X1.15(a) only in the use of a higher dosage of the Type A/D admixture, as indicated. Each series includes mixtures at three different sulfate levels: no addition, and additions of Terra Alba⁵ gypsum to raise the SO₃ levels by

0.38% and 0.75% of the cementitious materials. A slight sulfate imbalance is suggested in the mixture without sulfate addition at the lower admixture dosage; this becomes more extreme with the higher admixture dosage. In each case, performance is improved with calcium sulfate addition, confirming the suspicion that the reported field performance issues may be sulfate balance related. These additions were relatively slight, so this set of materials and proportions may be near the imbalance threshold. In such cases, abnormal behavior may be erratic in a project, as the mixture responds to normal variability of component materials and actual temperatures. This illustrates the need to include test dosages and temperature conditions beyond those contemplated in the field, as this may be the only way to determine how close the system is to an out-of-balance condition.

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