Evaluation of Semi-Adiabatic Calorimetry to Quantify Concrete Setting

by

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A thesis submitted to the Graduate Faculty of
Auburn University
in partial fulfillment of the
requirements for the Degree of
Master of Science

Auburn, Alabama
May 14, 2010

Keywords: concrete, setting, semi-adiabatic,
calorimetry, thermal

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Abstract

Although setting is routinely measured in practice, current methods for determining concrete setting have been criticized for being impractical, arbitrary, and lacking correlation to field data. In recent years attempts have been made to develop an alternate method for quantifying concrete setting based on semi-adiabatic calorimetry results during the hydration of a concrete sample.

In this study, methods to estimate concrete setting times based on data obtained from thermal testing were examined and compared to currently accepted penetration resistance-based test results. Maturity concepts were applied throughout the study to allow accurate comparisons between methods and to allow for a better correlation to in-place concrete measurements. It was found that set times that compare to those obtained as per ASTM C 403 can be accurately obtained through semi-adiabatic calorimetry. Initial and final set times were determined to occur at pre-defined adiabatic temperature rises, and a method for the calculation of these times is presented.

Acknowledgments

I would like to especially thank Dr. Anton Schindler for his guidance, time, and patience on this project and throughout my graduate career. Also, concrete lab technician Billy Wilson, as well as fellow students Ben Byard, Paul Coulston, Daniel Mundie, and the undergraduate research assistants deserve thanks for all their hard work, for without them, this project would not have been possible. I would also like to thank my parents, Benny and Pam Weakley for their support and guidance throughout my life, and special thanks should go to my wife, Alison Weakley for her love, support, and patience throughout this process.

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Chapter 1

Introduction

1.1 Background

Setting of concrete is generally described as the gradual increase in rigidity due to the hydration of cement (Mindess, Young, and Darwin 2003). Scripture (1956) further defined the setting process as one that begins with the loss of workability of a concrete mixture and ends with the onset of “appreciable” strength gain, commonly referred to as hardening. Since both setting and hardening are gradual processes controlled by cement hydration, the time of set has long been defined only in terms of specific test methods (Scripture 1956). Initial set has historically been linked to the point after which concrete can no longer be consolidated by vibration and final set to the start of strength development (Tuthill and Cordon 1956; Sprouse and Peppler 1978). For a schematic representation of the setting and hardening process see Figure 1.1. The degree of hydration of a concrete sample can be linked to the setting and hardening processes. As the cement paste gradually hydrates over time (this process is represented in the figure by the solid line), cement molecules form hydration products. The interlocking of these products eventually leads to the setting and later the hardening of a concrete sample. In Figure 1.1, simplified illustrations of the various hydration products and their interaction are depicted above their respective hydration state. All symbols used in the illustrations are defined within the figure.

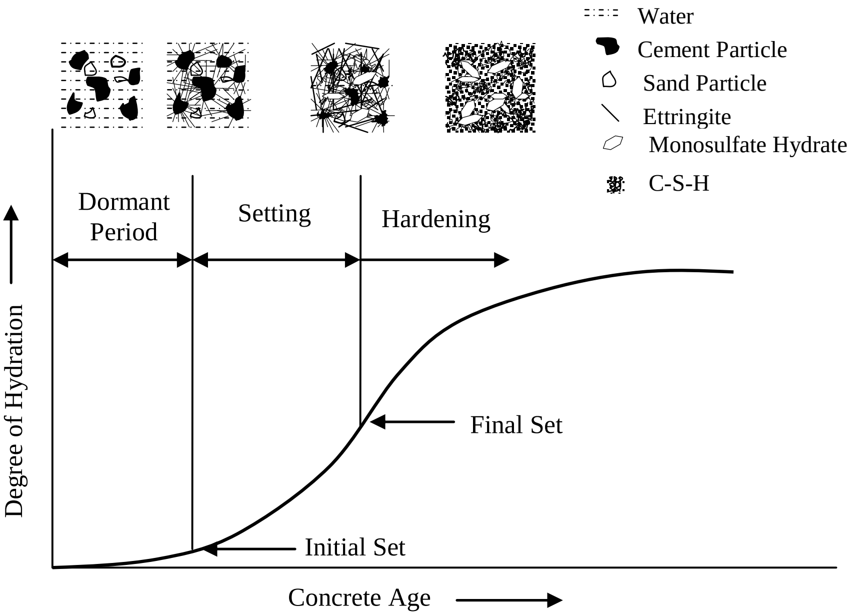


Figure 1.1: Schematic showing periods of setting and hardening (adapted from Soroka 1980 and Schindler 2004b)

For contractors, knowing the setting times of a particular mixture is important in order to plan for placement, finishing, texturing, and form removal, all of which are dependent on the rate of setting and placement conditions (Scripture 1956). In the laboratory, setting times are routinely measured as a standard fresh concrete property and are commonly used as a means of comparison between mixtures with different cement properties, mixture proportions, material types, and environmental conditions (Dobson 1994). Additionally, as the start of stress development, the final set point is especially important when determining stresses due to shrinkage and relaxation within concrete (Weiss 2002).

Although initial set is generally thought to occur within 2 to 5 hours of mixing and final set is thought to occur within 5 to 10 hours of mixing, setting times may vary over a much broader time span. It has long been noted that the setting times of a particular concrete mixture are dependent on the properties of the cement used in the mixture, concrete mixture proportions, the presence of chemical admixtures, and environmental conditions (including concrete temperature) during the setting process (Sprouse and Peppler 1978; Dobson 1994). In order to quantify setting in light of these influences, several test procedures have been proposed over the years to determine mixture-specific setting times. These include penetration resistance-based methods, electrical measurements, ultrasonic testing, shrinkage measurements, and measurements of the heat evolution of a sample (Dobson 1994).

Recently there has been increased interest in the use of the measurement and analysis of the heat evolution of a sample as a means of quantifying concrete setting. These “thermal methods” have become popular due to the relative ease of use as compared to established testing techniques and the fact that they allow for the testing of concrete samples, as opposed to mortar or cement paste samples. Examples of thermal methods as well as some sample results can be seen in Figure 1.2.

Thermal data and points of initial and final set as defined by Sandberg and Liberman (2007) as a percentage of the maximum temperature rise recorded can be seen in Figure 1.2 a, and results from this analysis as compared to penetration resistance testing can be seen in Figure 1.2 b. It should be noted that final set generally compared to that obtained through penetration resistance testing within ± 20 percent. Similarly, initial and final setting as defined by Schindler (2004b) as the degree of hydration of a concrete sample obtained through thermal testing can be seen in Figure 1.2 c. Results from this analysis can be seen (notated "predicted") along with final set as measured through penetration resistance in Figure 1.2 d. Again, final set was generally predicted within ± 20 percent of that measured through penetration resistance testing.

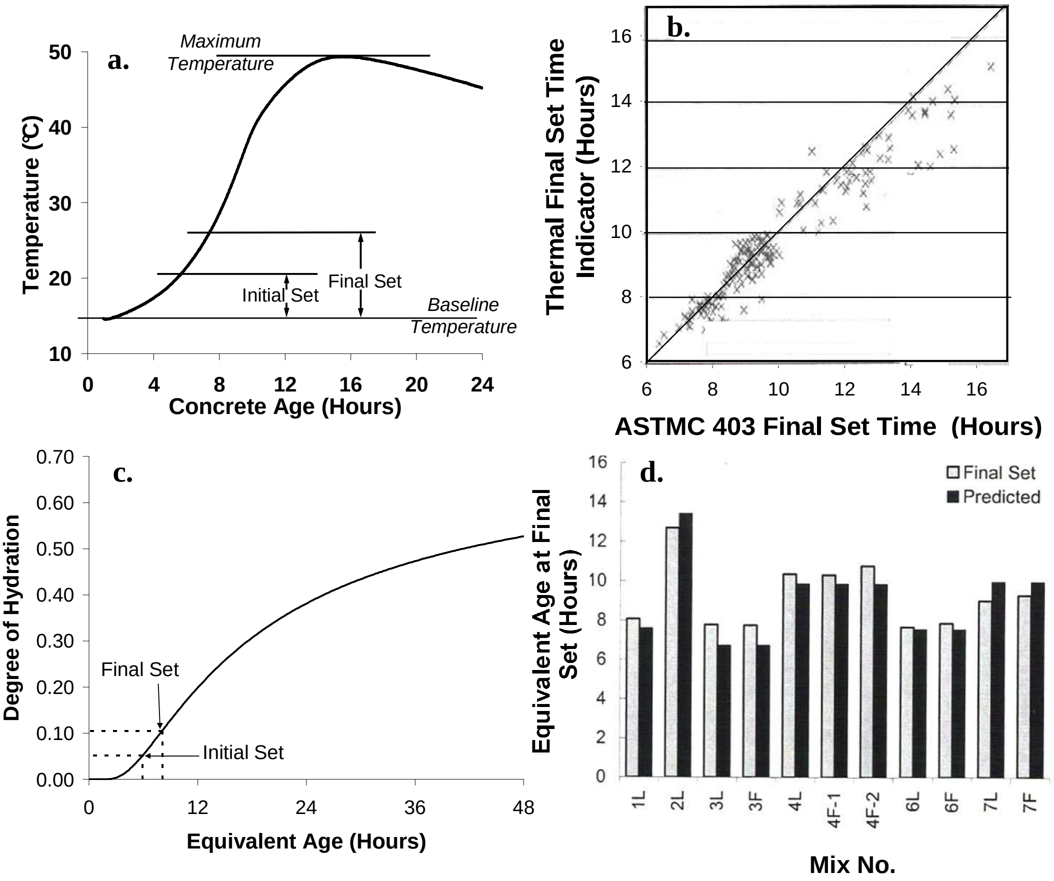


Figure 1.2: Examples of thermal methods from a) Sandberg and Liberman (2007) and c) Schindler (2004b) and sample results from b) Sandberg and Liberman (2007) and d) Schindler (2004b)

In light of these results and others, a draft specification for the determination of setting through the analysis of thermal data from insulated concrete specimen was proposed to the American Society for Testing and Materials (ASTM) entitled the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method*. Based on work by Sandberg and Liberman (2007), initial and final setting were defined in terms of the second and first derivative of concrete temperature, respectively.

It has been well documented that environmental conditions, specifically temperature, have a substantial impact on setting. The reactions involved in hydration are temperature dependent, therefore, the rate of setting is much faster at higher temperatures and much slower at lower temperatures. Temperature effects on the hydration of cement have been successfully approximated by the use of an approach referred to as the maturity method. Although the maturity method has been applied to other areas of concrete research, most notably prediction of concrete strength, application to concrete setting measurements has been minimal (Weiss 2002; Pinto and Hover 1999).

1.2 Research Significance

ASTM C 403 (2008) is the current standard method for determining the setting time of concrete. This method, which is based on work by Tuthill and Cordon (1956), defines points of initial and final set as certain penetration resistance values obtained from a sieved-mortar sample. Although initially calibrated to physical properties of concrete, the points chosen as initial and final set have been described as somewhat arbitrary, yet convenient for comparison purposes (Dobson 1994). This method has been criticized lately due to the need for constant human monitoring and the lack of correlation between setting results of sieved-mortar samples and in-place concrete. Recent work has addressed the former of these concerns with the application of various methods for determining "thermal" set using semi-adiabatic calorimetry as a means of quantifying the hydration process within a concrete sample (Christianson 2006). Separately, lack of correlation to field data has been partially addressed by the application of maturity concepts to penetration resistance based setting methods (Pinto and Hover 1999; Garcia et al. 2008).

In this study, the possibility of quantifying setting times based on thermal data is examined more thoroughly and compared to the currently accepted penetration resistance methods. Maturity concepts are applied throughout the study in order to account for the effect of temperature on the degree of hydration and thus setting. The use of the maturity method avoids inconsistent comparisons between methods of determining setting caused by varying temperature histories. The maturity method also allows for the possibility of better correlation to in-place concrete measurements.

1.3 Research Objectives

This study was initiated to evaluate the use of thermal methods to accurately and consistently predict the setting of concrete. The primary objectives of this research are as follows:

(a) Evaluate the current standard for determining setting times of concrete, ASTM C 403 (2008), to establish a basis of comparison for future work,

(b) Determine the applicability and accuracy of a currently proposed ASTM standard, the *Standard Test Method for Determining Setting Time of Concrete* *by the Temperature Method* to determine setting by thermal methods,

(c) Develop and analyze alternative thermal methods to determine the setting times of concrete, and

(d) If possible, prepare a test protocol for this thermal method that accurately and reliably estimates the setting times of concrete.

1.4 Report Scope

The organization of the remainder of this thesis can be seen in the organizational chart presented in Figure 1.3.

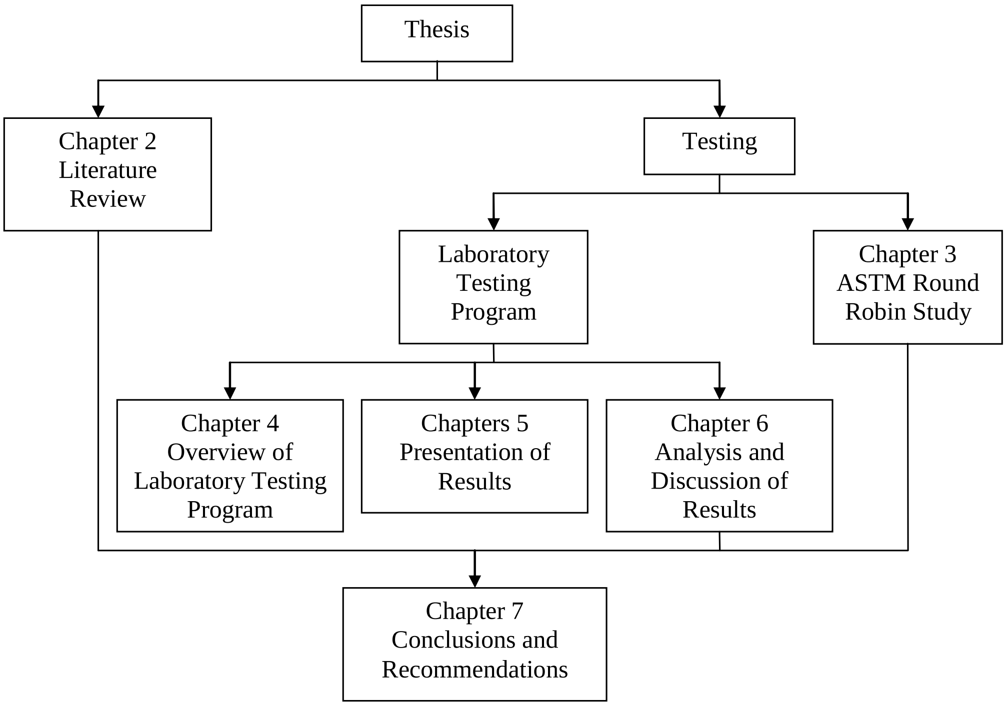


Figure 1.3: Thesis organization

An overview of concrete setting behavior and past research on the subject is presented in Chapter 2. An overview of the chemistry of cement paste hydration and its effect on concrete setting is presented. Current cement paste and concrete setting determination methods are summarized and new research related to thermal methods to determine setting times is presented. Influences on setting times of concrete are discussed, and finally, a brief summary of the maturity method is presented, and its application to concrete setting is examined.

A summary of a recent round robin study on concrete setting is presented in Chapter 3. An ASTM round robin study was conducted in November of 2008 to determine the precision and bias of ASTM C 403 (2008), and to evaluate a previously proposed ASTM specification for determining setting times of concrete by temperature methods. The round robin was documented, and the results of this study as well as discussion and recommendations are presented.

In Chapter 4, a laboratory testing procedure developed in order to evaluate the measurement of concrete setting through thermal methods is presented. The overall testing program is discussed along with testing apparatus and methodology, concrete production methods, mixture proportions for concrete evaluated in the study, and properties of raw materials used. Sample results from this testing as well as a brief discussion are presented in Chapter 5.

Data analysis and the evaluation of thermal methods for calculating concrete setting can be found in Chapter 6. Adjustments to thermal and penetration resistance data are discussed and six independent analysis methods are presented. Each thermal analysis method is evaluated as compared to ASTM C 403 (2008) data, and the results are presented. These results are discussed, and conclusions and recommendations for future work are presented. Conclusions and recommendations for the determination of concrete setting through thermal testing are presented in Chapter 7.

The aforementioned draft ASTM specification (discussed in Chapter 3) is presented in Appendix A. Details concerning experimental procedure as well as analysis of thermal data are presented. Appendix B contains supplementary information concerning the ASTM round robin testing. Pictures of all Category I, II, and III devices are presented. Appendix C contains concrete compressive strength results for all concrete batches tested in the laboratory testing program. Similarly, Appendices D and E contain results from all batches tested in the laboratory testing program for penetration resistance testing, and semi-adiabatic calorimetry and isothermal calorimetry testing, respectively.

Chapter 2

Literature Review

The cement hydration process and the factors that influence the setting of cement and concrete must be studied in order to understand the mechanism of concrete setting. In this chapter, the historical and technical information required to understand current methods of quantifying setting and to evaluate thermal setting methods are presented. Topics covered include portland cement hydration, mechanical and thermal methods for determining set, and the application of the maturity method to setting.

2.1 Setting Mechanisms

Portland cement, the most common binder used in concrete production, is a composition of several compounds that react simultaneously during cement hydration to form a hardened product. When the water is added to a concrete mixture, the compounds within cement immediately begin to hydrate at varying rates. It is these reactions and their respective rates coupled with the effects of various admixtures that determine the setting and hardening characteristics of a particular concrete mixture (Mehta and Monteiro 2006). In the following sections, the chemistry behind cement hydration is examined. The effect of these reactions and their relative rates on setting is discussed and a framework for quantifying these effects is presented.

2.1.1 Chemistry of Portland Cement

The four main compounds in portland cement are tricalcium silicate (C_3S), dicalcuim silicate (C_2S), tricalcium aluminate (C_3A), and teracalcium aluminoferrite (C_4AF). The process of hydration and the properties of the resulting concrete are highly influenced by the relative proportion of these compounds, as well as the water-cement ratio used (w/c), the fineness of the cement, the curing temperature, and the presence of chemical and mineral admixtures (Older 1998). Chen and Older (1992) found that in "normal" portland cement concrete mixtures, setting appears to be primarily the consequence of the hydration of C_3S and C_3A yielding the formation of calcium silicate hydrate (C-S-H) and calcium trisulfoaluminate hydrate, commonly referred to as ettringite. The hydration of C_3S and C_3A will be discussed in more detail in the following section.

2.1.1.1 Hydration of C_3A and C_3S

The reaction of C_3A and water occurs when the water first contacts the cement yielding crystalline hydrates. This reaction occurs immediately, and would lead to a very fast set if it were not slowed down by the presence of sulfate in the form of gypsum. Gypsum is added in portland cement production phase where the sulfate to aluminate ratio is balanced to ensure adequate setting times and maximum performance. Once the dormant period is reached, C_3A, gypsum, and water react at a moderate rate to form ettringite, a needle like crystal, while releasing little heat (See Reaction 1). Once the gypsum is consumed, ettringite becomes unstable and allows the C_3A to again rapidly hydrate, gradually converting the ettringite to a monosulfate hydrate and releasing a considerable amount of heat (see Reaction 2) (Mehta and Monteiro 2006; Mindess, Young, and Darwin 2003).

$$\mathbf{C_3A + gypsum + water \longrightarrow ettringite} \qquad \textbf{(Reaction 1)}$$

$$\mathbf{C_3A + ettringite + water \longrightarrow monosulfate\ hydrate} \qquad \textbf{(Reaction 2)}$$

C_3S hydrates at a slower rate than the aluminates (see Reaction 3), but its primary hydration product, C-S-H, is the largest contributor to strength in the hydrated cement paste. Calcium hydroxide (CH) is also produced, but has much less of an effect on concrete setting.

$$\mathbf{C_3S + water \longrightarrow C\text{-}S\text{-}H + CH} \qquad \textbf{(Reaction 3)}$$

Silicate hydration is generally broken down into five stages and these can be seen on a heat evolution curve in Figure 2.1. In the first stage, generally lasting only minutes, the ions present dissolve into solution. This is followed by a dormant period, stage two, in which hydration products begin to form. In stage three and four, silicates hydrate rapidly reaching a peak and then slowing down until reaching a steady state in stage five. Generally, only stages two and three contribute to setting behavior (Mindess, Young, and Darwin 2003).

2.1.1.2 Heat Evolution

The reactions that take place in cement paste are exothermic, therefore heat is released during the process. If measured, this temperature rise can be related to cement hydration (Lawrence 1998). One way the combined effect of these separate reactions has been studied has been to examine of the heat evolution of each compound as it hydrates. The rate of heat evolution of the C_3S and C_3A reactions can be seen in Figure 2.1 and Figure 2.2 respectively. Since the C_3A reaction is highly dependent on the amount of sulfate present in the portland cement, curves are plotted for 10 percent, 15 percent, and 20 percent by weight of gypsum.

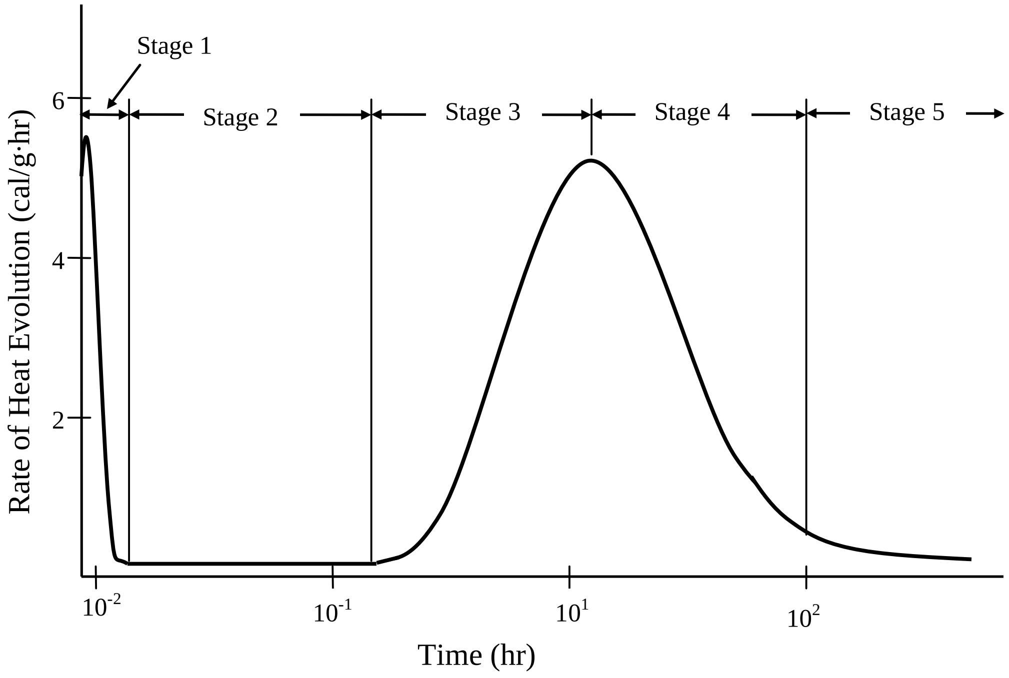


Figure 2.1 Rate of heat evolution of C_3S reaction (adapted from Mindess, Young, and Darwin 2003)

The five stages of hydration can be seen for both C_3S hydration (Figure 2.1), and sulfate-balanced C_3A hydration (Figure 2.2). The beginning and end of each stage can be quantified by the relative rates of heat evolution associated with each stage. Stage one (the pre-induction period) is associated with a brief, extremely high rate of hydration. This peak in the rate is followed immediately by the dormant stage (a.k.a. the induction period), stage two, in which very little heat is evolved. In stage three (the acceleration stage), the rate gradually increases, reaching a peak between stage three and four (the post-acceleration period). In stage four, the rate of heat evolution gradually decreases until it reaches a steady state at stage five.

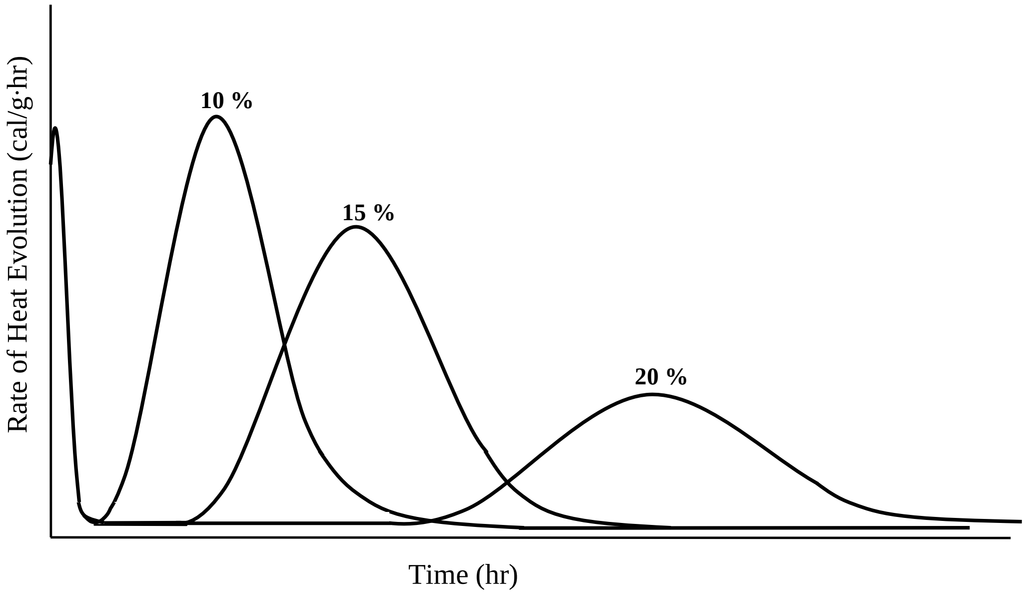


Figure 2.2: Rate of heat evolution of C_3A+ gypsum reaction. % weight of gypsum is given for each curve. (Adapted from Mindess, Young, and Darwin 2003)

2.1.1.3 Portland Cement Hydration

The simultaneous reaction of C_3A and C_3S are the major contributors to the loss of workability and start of strength gain that combine to define the setting of concrete. As can be seen in Figure 2.3, the heat evolution of portland cement paste resembles both the heat evolution of C_3S and sulfate-balanced C_3A. The hydration process can again be divided into 5 general stages where the stages represent a brief period of high activity and heat evolution, followed by a dormant period, periods of rapidly increasing and decreasing hydration product formation, and finally a steady state (Mindess, Young, and Darwin 2003).

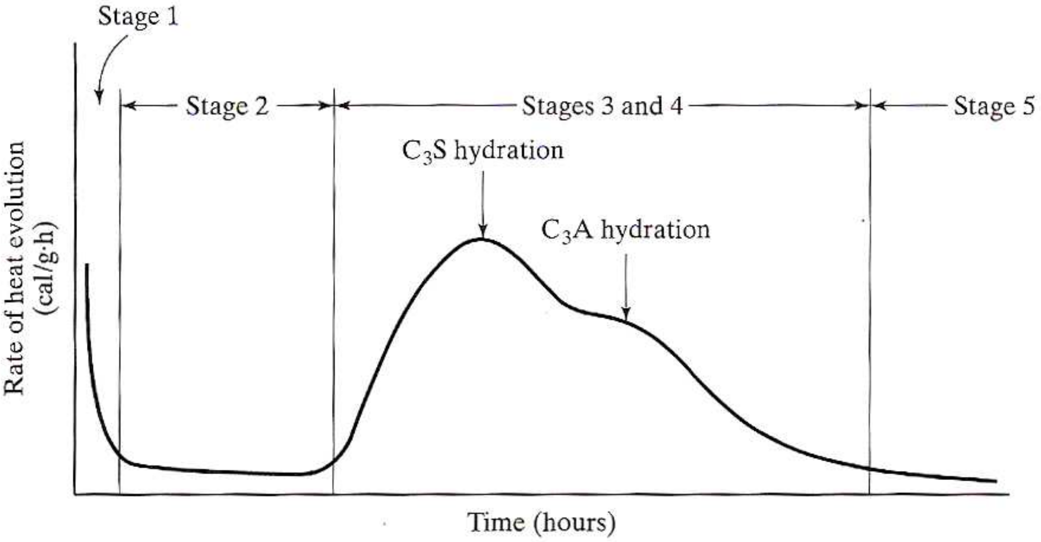


Figure 2.3: Rate of heat evolution during the hydration of portland cement (Mindess, Young, and Darwin 2003)

Dalgleish et al. (1982) state that both initial and final set can be linked to the formation of C-S-H, and only abnormal set is controlled by the formation of ettringite. However, in a study that examined cement pastes with different quantities of C_3S, C_3A, and gypsum as well as varying w/c, Chen and Older (1992) found that setting can be controlled by either the aluminates or the silicates. It was determined that setting appears to occur when a certain amount of hydration product was formed regardless if it is predominately ettringite or C-S-H. Figure 2.4 illustrates typical rates of hydration product formation during these processes.

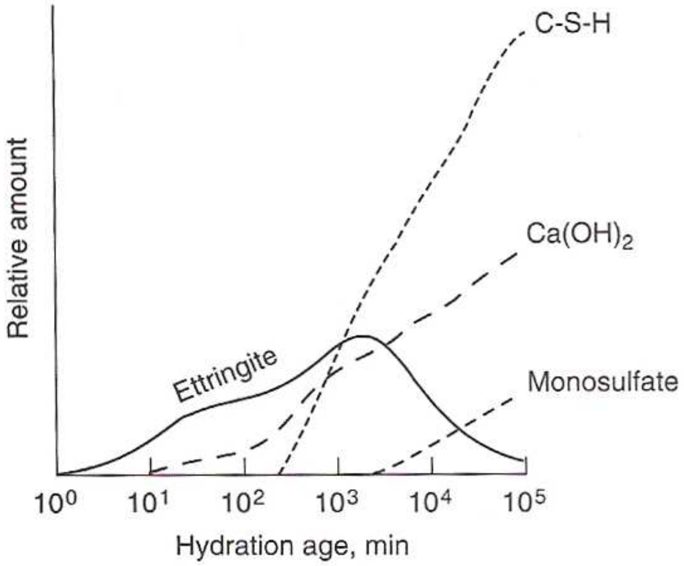


Figure 2.4: Relative amount of hydration products formed in portland cement hydration (Mehta and Monteiro 2006)

2.1.2 Supplementary Cementing Materials

Supplementary cementing materials (SCM's), or mineral admixtures, are finely ground solid materials added primarily as a cement replacement (Mindess, Young, and Darwin 2003). These are added as a cost reduction, and to lower water demand, improve durability, and lower the heat of hydration of a concrete. Two of the more common

SCM's are fly ash, a bi-product from coal production, and ground-granulated blast-furnace (GGBF) slag a bi-product from the production of cast iron and steel. When used as a cement replacement, both of these materials will alter the setting-hardening properties of a concrete mixture (Mehta and Monteiro 2006).

Although fly ash has some cementitious properties, it is generally referred to as a pozzolanic material. A pozzolan is defined as a siliceous material which, in the presence of moisture, will react with calcium hydroxide to form compounds with cementitious properties (Mehta and Monteiro 2006). When added as a cement replacement, fly ash reacts with calcium hydroxide from the silicate stage of hydration to form C-S-H as shown in Reaction 4. This pozzolanic reaction improves concrete durability and long term strength and reduces the overall heat of hydration.

$$\textbf{Pozzolan} + \textbf{CH} + \textbf{water} \longrightarrow \textbf{C-S-H} \qquad \textbf{(Reaction 4)}$$

There are two classes of fly ash used in concrete production today defined by their chemical composition. Class F fly ash has less than 10 percent of CaO and is considered strictly pozzolanic. Class C fly ass typically contains between 15 and 40 percent CaO and has both pozzolanic and cementitious properties due to the presence of crystalline compounds that behave similarly to the aluminates in portland cement during hydration. Both of these materials will alter the hydration process of a concrete mixture, generally delaying the setting process (Mindess, Young, and Darwin 2003).

Unlike fly ash, ground-granulated blast-furnace slag is a strictly cementitous compound. Although GGBF slag will hydrate by itself, this process is much too slow for concrete applications. When hydrated along with cement, the alkaline compounds present in the hydrated cement ($Ca(OH)_2$) activate the slag yielding a rate of hydration slightly slower than plain portland cement. A mixture containing a substantial replacement of GGBF slag will have a delayed set and a lower heat of hydration when compared to traditional concrete; however, the effect of GGBF slag may vary depending on its fineness and composition. For more information on the effect of SCM's on concrete setting see Section 2.3.2.4.

2.1.3 Degree of Hydration

Once the chemistry of setting is understood, it is helpful to be able to quantify the process of hydration. The term degree of hydration (α), as it pertains to cement paste and concrete, refers to the progress of the hydration reactions between the cement and water contained in a sample. An α of 0.0 represents the state in which no reactions have occurred where an α of 1.0 represents the state in which complete hydration has been reached (CEA 42 1981). A typical plot of degree of hydration versus concrete age can be seen in Figure 2.5.

As the degree of hydration increases and hydration products form, they can be assumed to have an equal contribution to the development of concrete properties (Byfors 1980). Taplin (1959) found a linear relationship between the development of compressive strength of concrete and the measured degree of hydration. This relationship implies that the development of physical concrete properties, including concrete setting, can be estimated through the measurement of degree of hydration.

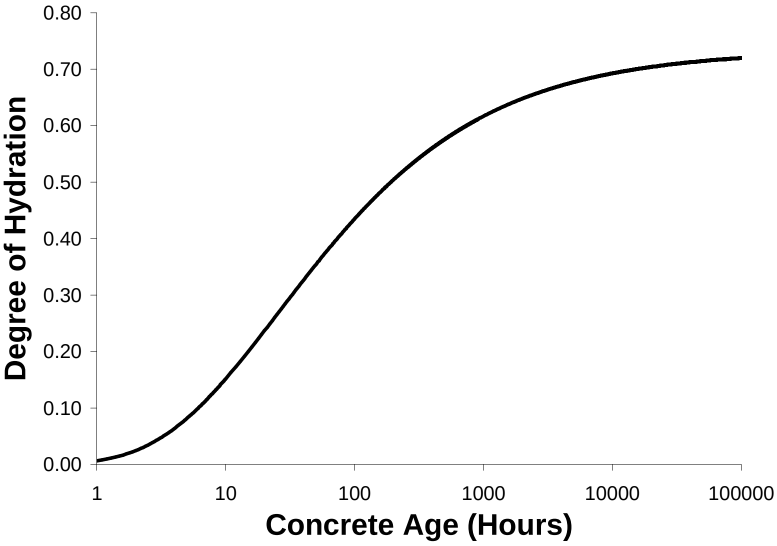


Figure 2.5: Typical plot of degree of hydration versus concrete age

2.1.3.1 Degree of Hydration Formulations

It is difficult to account for the exact hydration of each compound within cement due to the varying rates of reaction of each cement compound. Not only does each compound hydrate independently, but the chemical composition of cement and the cement content within a concrete mixture are highly variable as well. Several broader formulations for degree of hydration have been proposed. One widely accepted definition for the degree of hydration of concrete is presented in a document by the International Union of Laboratories and Experts in Construction Materials, Systems, and Structures (RILEM, from *Reunion Internationale des Laboratoires et Experts des Materiaux, Systemes de Construction et Ouvrages*). This RILEM state-of-the-art report concerning properties of set concrete at early ages (CEA 42 1981), defines degree of hydration as follows:

$$\alpha(t) = \frac{C(t)}{C}$$ Equation 2.1

where:

α(t) = degree of hydration at time t,

C(t) = quantity of hydrated cement at time t, and

C = original quantity of cement.

Although this definition seems relatively simple, CEA 42 (1981) states that it is "almost impossible to make a direct determination of the quantity of cement gel formed or the quantity of hydrated cement." Due to this fact, α is usually quantified indirectly. This can be accomplished through analysis of the chemically bound water, determination of the quantity of unhydrated cement through x-ray analysis, or based on the total heat of hydration of a sample (RILEM CEA 42 1981).

One of the more common methods of quantifying α is through analysis of chemically bound water. Powers and Brownyard (1948) demonstrated that a direct relationship exists between the amount of cement gel formed, and the quantity of chemically bound water within cement paste. They also found that portland cement will bind about 25% by weight of water when complete hydration is reached. Therefore, based on this information, an indirect formulation of degree of hydration can be expressed as follows:

$$\alpha(t) = \frac{w_n(t)}{0.25 \cdot C}$$ Equation 2.2

where:

$\alpha(t)$ = degree of hydration at time t,

$w_n(t)$ = quantity of bound water at time t, and

C = original quantity of cement.

This formulation is fairly common due to its relative ease of measurement. The quantity of bound water (w_n) is usually defined as “the quantity of water which is emitted from a dried (105° C) specimen when it is subjected to ignition (1050° C)” (Byfors 1980). Bound water can be measured by simply recording the difference in weight between the dried specimen and the specimen after subjected to ignition at 1050 °C.

Powers and Brownyard (1948) also found that quantity of heat developed during hydration is directly proportional to the quantity of bound water (w_n). Therefore, a formulation for degree of hydration similar to that above can be developed for a heat of hydration based model. In this formulation, the quantity of heat developed at a given time is divided by either the measured or theoretical total quantity of heat available in the system (i.e. the quantity of heat at complete hydration). This definition can be expressed as follows (Powers and Brownyard 1948):

$$\alpha(t) = \frac{H(t)}{H_T} \qquad \text{Equation 2.3}$$

where:

$\alpha(t)$ = degree of hydration at time t,

$H(t)$ = quantity of heat developed at time t, and

H_T = quantity of heat developed at complete hydration.

Methods for determining *H(t)* include the use of an adiabatic calorimeter, the measurement of the heat of solution, and the use of an isothermal calorimeter (also known as a conduction calorimeter). Of these methods, the calorimetry based methods are preferred due to the unreliability of the heat of solution measurements (CEA 42 1981). H_T can be measured in a similar fashion or can be approximated directly from the cement compounds present in a given cement (Taylor 1997). Schindler and Folliard (2005) proposed a method for approximating the total heat of hydration of a concrete sample based on properties of the cement and mixture proportions. Their formulation can be expressed as follows:

$$H_u = 461 \cdot p_{Slag} + 1800 \cdot p_{FACaO} \cdot p_{FA} + p_{cem} \cdot H_{cem} \qquad \text{Equation 2.4}$$

where:

H_u = total heat of hydration of cementitious materials at 100% hydration, (J/g),

p_{Slag} = slag weight ratio in terms of total cementitious content,

p_{FACaO} = weight ratio of CaO in terms of total fly ash content, and

p_{FA} = fly ash weight ratio in terms of total cementitious content.

p_{cem} = cement weight ratio in terms of total cementitious content,

H_{cem} = total heat of hydration of cement, (J/g), expressed as follows:

$$H_{cem} = 500 \cdot p_{C_3S} + 260 \cdot p_{C_2S} + 866 \cdot p_{C_3A} + 420 \cdot p_{C_4AF} + 624 \cdot p_{SO_3} + 1186 \cdot p_{FreeCaO} + 850 \cdot p_{MgO} \quad \text{Equation 2.5}$$

where:

H_{cem} = total heat of hydration of cement, (J/g), and

p_i = weight ratio of the i^{th} compound in terms of total cement content.

2.1.3.2 Ultimate Degree of Hydration

It has long been noted that concrete, even when well cured, contains a substantial amount of unhydrated cement. Therefore, in light of the above definitions of degree of hydration, complete hydration ($\alpha = 1.0$) may never be reached unless the water-cement ratio is very high. In a study of the chemically bound water of fully hydrated specimen containing a range of cementious materials, Mills (1966) found “in most, if not all, cement pastes hydration stops before the cement is totally consumed.” An equation for the approximation of the ultimate degree of hydration was developed and can be expressed as follows:

$$\alpha_{ult} = \frac{1.031 \cdot w/c}{0.194 + w/c} \quad \text{Equation 2.6}$$

where:

α_{ult} = ultimate degree of hydration, and

w/c = water-cement ratio by weight.

Schindler and Folliard (2005) expanded this model to include the use of supplementary cementing materials. The resulting equation can be expressed as follows:

$$\alpha_{ult} = \frac{1.031 \cdot w/c}{0.194 + w/c} + 0.5 \cdot p_{FA} + 0.3 \cdot p_{SLAG} \leq 1.0 \qquad \text{Equation 2.7}$$

where:

α_{ult} = ultimate degree of hydration,

w/c = water-cement ratio by weight,

p_{FA} = weight ratio of fly ash in terms of the total cement content, and

p_{SLAG} = weight ratio of GGBF slag in terms of the total cement content.

The dependency of rate of hydration and ultimate degree of hydration on w/c has been noted by many researchers (Powers and Brownyard 1948; Taplin 1959; Byfors 1980; Chen and Older 1992). It should be noted that, although the rate of hydration may be affected by curing temperature (see Section 2.3.3 and 2.4), the ultimate degree of hydration, as calculated above, is independent of curing temperature. This is in agreement with the findings of Kjellsen et al. 1991. This effect is illustrated in Figure 2.6 where the degree of hydration of three samples cured at differing temperatures converges after approximately 130 hours when tested with an isothermal calorimeter.

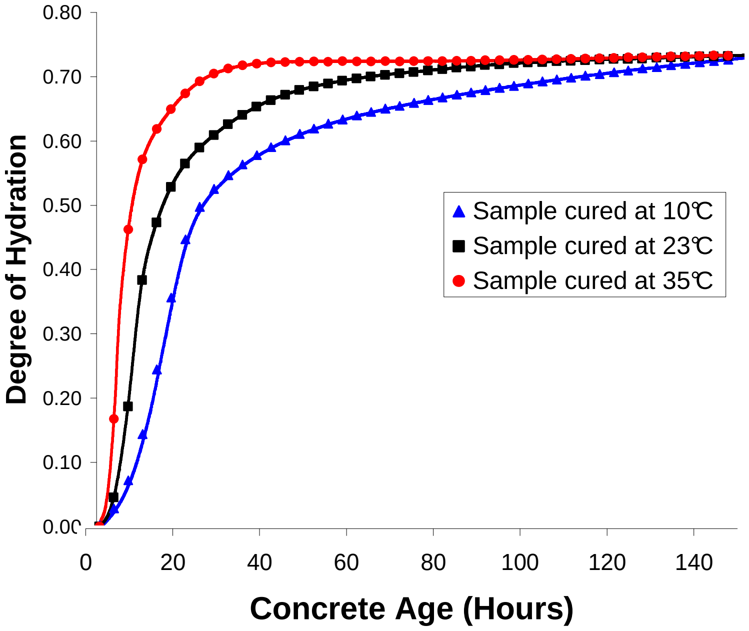


Figure 2.6: Degree of hydration plots for concrete samples cured at differing temperatures

2.1.3.3 Hydration equation

In 1984, Freiesleben, Hansen, and Petersen proposed the use of a three parameter exponential equation to represent the heat development of a portland cement concrete sample. This function has been shown to successfully approximate the shape of a hydration curve and the three parameters can be manipulated to fit most hydration data sets. Pane and Hansen (2002) used the same function to model the relationship between degree of hydration and time. This function can be expressed as follows:

$$\alpha(t) = \alpha_{ult} \cdot \exp\left(-\left(\frac{\tau}{t}\right)^{\beta}\right) \qquad \text{Equation 2.8}$$

where:

$\alpha(t)$ = degree of hydration at time t,

α_{ult} = ultimate degree of hydration as defined in eq. 2.4,

τ = hydration time parameter,

β = hydration shape parameter, and

t = concrete age or equivalent age.

2.1.3.4 Critical Degree of Hydration

By definition, degree of hydration is a measure of the process of the reactions that make up cement hydration. It is these very reactions that determine the setting and hardening characteristice of a concrete mixture (Mehta and Monteiro 2006). Both Taplin (1959) and Powers and Brownyard (1948) noted the relationship between concrete compressive strength and degree of hydration, but little was said about concrete setting. Byfors (1980) defined the term critical degree of hydration (α_{cr}) as the degree of hydration at which strength development begins (α corresponding to final set). Using data collected by both Powers and Brownyard (1948) and Taplin (1959), Byfors formulated α_{cr} as a linear function of the water-cement ratio as presented in the following expression:

$$\alpha_{cr} = k_s \cdot w/c \qquad \text{Equation 2.9}$$

where:

α_{cr} = critical degree of hydration,

k_s = constant which varies from 0.40 to 0.46, and

w/c = water-cement ratio by weight.

These constants were derived through the extrapolation of strength versus degree of hydration data. Schindler (2004b) expanded this work obtaining constants through degree of hydration calculations calibrated to current penetration based setting methods as defined in ASTM C 403. An overview of these penetration methods as well as work in thermal determination of setting times can be found in the following sections.

2.2 Determination of Setting Times

In 1956, Scripture described a need for a testing method that would quantify the time of set of a concrete mixture. He defined setting as the time between loss of workability and significant strength gain, and determined that setting should be defined in terms of a particular test method. Over the years several methods have been proposed including penetration models, electrical measurements, ultrasonic testing, shrinkage measurements, and measurements of the heat evolution of a sample (Dobson 1994). Currently, the most common method for defining setting is by penetration resistance, but there has recently been a renewed interest in the use of thermal methods for determining set. The background and use of both of these methods is presented below.

2.2.1 Penetration Resistance Methods

Cement-based materials are highly visco-elastic during the setting and hardening stages, therefore their rheological properties are difficult to measure traditionally (Jousset et al. 2007). In order to study the setting and hardening process, several empirical tests have been used to infer these properties, the most common of which include the Vicat needle for cement paste, and the Proctor penetrometer for a mortar sample. Both of these tests examine the ability of a material to resist the penetration of a tool that is forced into a sample, and setting characteristics can be inferred from the results (Lootens et al. 2009).

2.2.1.1 Summary of Penetration Testing Methods

The Vicat test, standardized by ASTM in 1944 (ASTM C 191 1944), is used to determine the setting time of hydraulic cement. In this testing method, a single 300 gram (10.6 oz) needle (1.0 mm in diameter) is allowed to penetrate a cement paste sample of “normal” consistency and the depth of penetration is recorded at 15 minute time intervals. Depths of 25 mm (1 inch) and 0 mm, respectively, are defined as initial and final set, and the time at which these are obtained are determined to be the setting times of the cement paste. These two points are arbitrary, and are indirect indicators of setting, however they serve as convenient points of comparison between samples (Lootens et al. 2009). It should be noted that “normal” consistency cement paste usually has a w/c of around 0.25. For an example of a Vicat penetration resistance plot for three paste samples see Figure 2.7.

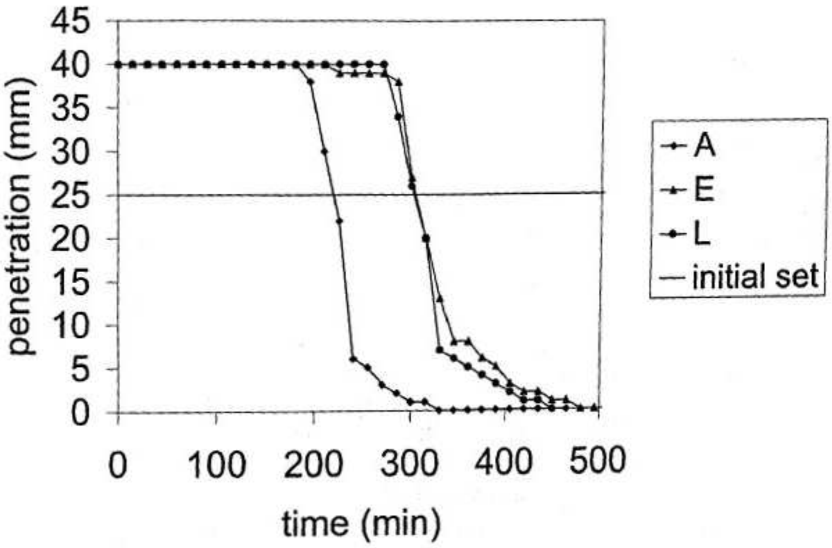


Figure 2.7: Vicat penetration depth versus time plots (Struble et al. 2001)

Struble et al. (2001) noted that, in light of the effect of w/c on setting times in concrete (see Section 2.3.2.2), a penetration method for cement paste should be developed to be used on pastes of a consistency more typical of pastes used in concrete. Struble et al. (2001) demonstrated the use of the vicat needle on cement paste of varying consistency, w/c from 0.25 to 0.40, and found no problems with the more fluid pastes.

The proctor penetrometer was first used to find setting of a concrete mixture by Tuthill and Cordon (1956). They outlined a testing procedure in which setting can be inferred through the perpetration resistance of a sieved-mortar sample. In their testing a concrete sample is sieved through a number four sieve to obtain a mortar specimen. This mortar is then placed, consolidated, and covered in a container that is at least 150 mm (6.0 in.) deep with a diameter large enough for at least six undisturbed readings. It was noted by Tuthill and Cordon (1956) that the temperature of the specimen should remain at the average temperature at which the concrete will be placed and harden for the duration of testing. After removing bleed water, a series of penetration readings are taken in which the force required for a needle to penetrate the concrete 25 mm (1 inch) in 10 seconds is recorded. (Examples of Proctor penetration devices can be seen in Figure 2.8.) The readings are taken at equal time intervals with progressively smaller needles starting at 645 mm^2 (1 in^2) and ending at 16 mm^2 (1/40 in^2) until a reading of at least 445 newtons (100 pounds) is recorded using the 16 mm^2 needle.

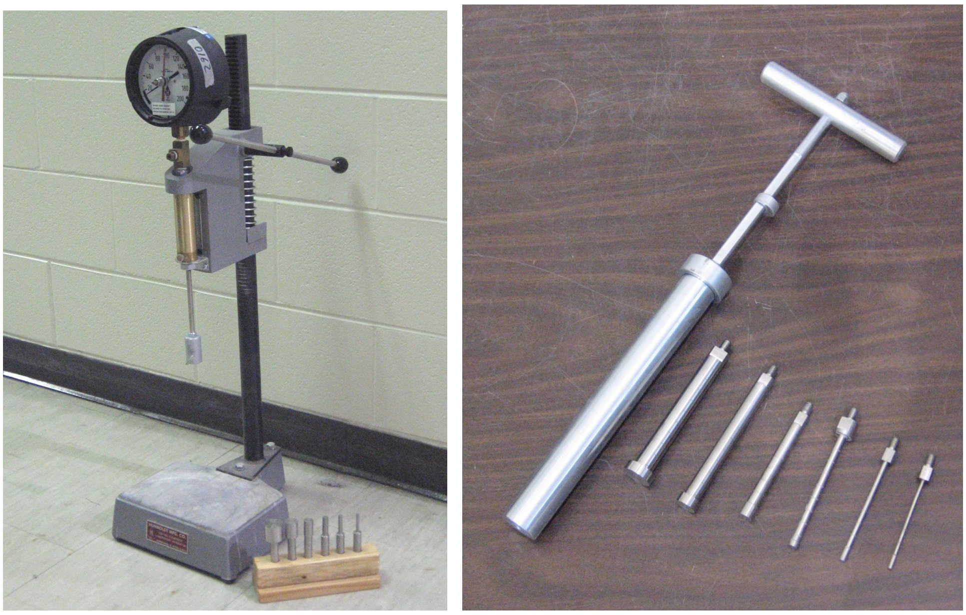


Figure 2.8 Examples of Proctor penetration devices

Penetration resistance in terms of stress can then be calculated by dividing the force required to penetrate the sample by the area of the needle. Tuthill and Cordon (1956) defined a vibration limit, or a mortar penetration resistance beyond which concrete can no longer be made plastic by vibration, as 3.5 MPa (500 psi). It was found that when mortar penetrations reached this point, a vibrator no longer sinks into concrete under its own weight, therefore concrete layers can no longer me made monolithic and cold joints are possible. They also defined the point at which concrete can be considered completely set as a resistance of 27.6 MPa (4000 psi). It was found that at this point, concrete will have a compressive strength of approximately 0.7 MPa (100 psi). These limits, although somewhat arbitrary, seem to fit the criteria for the bounds of setting that Scripture defined in 1956.

A specification for testing based on this method was adopted by ASTM as ASTM C 403 in 1957. The points previously referred to as the vibration limit and end of setting (Tuthill and Cordon 1956) have come to be known as initial set and final set, respectively. Due to the fact that it is unlikely that readings of exactly 3.5 and 27.6 MPa will be taken, these values are obtained through interpolation (Dobson 1994). The current testing specification, ASTM C 403 (2008), allows users to use regression analysis, a hand-fit smooth curve, or a linear fit on a log-log scale to obtain initial and final set points. Current users of this specification are not in complete agreement that these values truly represent the vibration limit and point of appreciable strength gain, therefore test results are used mainly for comparison purposes between different mixtures and/or curing conditions (Dobson 1994). Although the physical definitions of concrete setting may not be represented by this testing method, setting characteristics relative to other mixtures and/or curing conditions can be inferred from results. For example penetration resistance curves obtained as per ASTM C 403 (2008) see Figure 2.9.

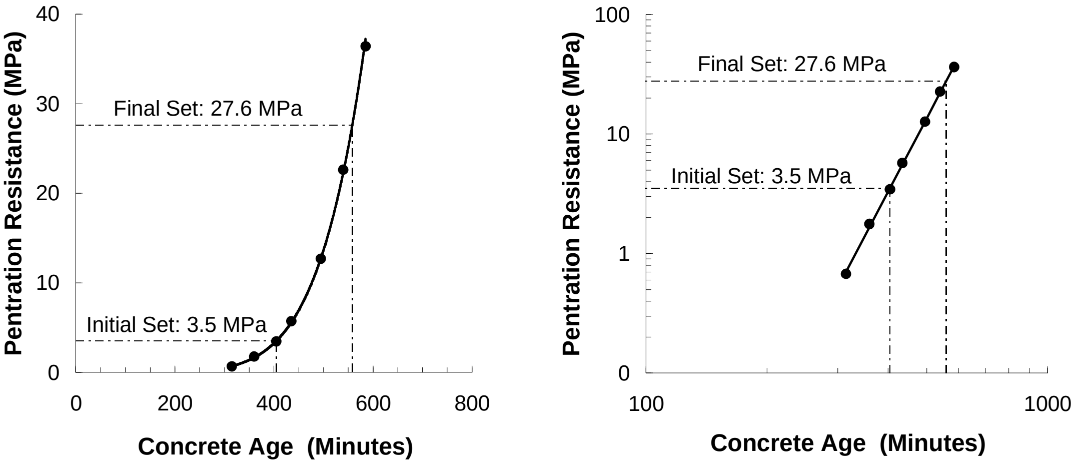


Figure 2.9: Typical penetration resistance versus concrete age plots

Much like the *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle,* the points of initial and final set have been described as arbitrary, but are convenient for comparison between mixtures with different mixture proportions, cement properties, material types, and environmental conditions (Dobson 1994). Although initial set is sometimes regarded as the time at which finishing can begin for in-place concrete, Abel and Hover (2000) found that initial set, as defined by ASTM C 403 (2008), occurs much later than the point at which finishing operations traditionally begin. Similarly, although final set has traditionally been associated with the start of concrete strength gain, when penetration resistance results have been compared directly with strength development, it has been noted that concrete strength development frequently lags penetration resistance (Malhotra, 1982). This lack of correlation of ASTM C 403 (2008) results to field concrete as well as the need for constant human monitoring are often cited as this method's main disadvantages (Christensen 2006).

2.2.1.2 Theoretical Background

Penetration-based methods are commonly used as implicit tests to study the setting or consistency of cementitious materials (Jousset et al. 2007). Although these methods provide valuable information on the setting process, until recently there was very little literature linking the penetration resistance and physical properties of the material being tested.

Pinto (1997) related the penetration resistance of concrete to the bearing capacity of soil. As can be seen in Figure 2.10, when a force is applied to a sample, shear stresses develop in a predictable pattern. Therefore, as the needle penetrates it is resisted by a combination of the bearing capacity of the mortar and the friction between the mortar and the needle.

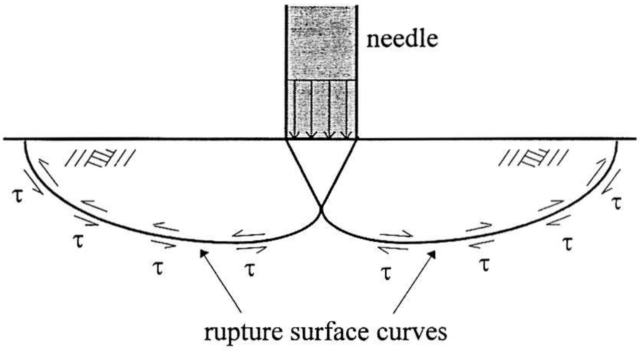


Figure 2.10: Shear stresses in mortar analogous to bearing in soil (Jumikis 1987)

Lootens et al. (2007) demonstrated a similar link between penetration resistance and shear yielding of a mortar sample with the use of ultrasound spectroscopy and finite element analysis. The finite element model was based on Roussel's (2006) segregation model of an aggregate particle moving through a non-Newtonian fluid. As can be seen in Figure 2.11, development of shear stress predicted in the model is similar to the soil bearing analogy made by Pinto (1997). It was shown that friction on a penetrometer needle had a negligible effect on the values obtained. It was then found that the force resisted from a penetration test scales directly with the material's yield stress (therefore with results from ultrasonic techniques). An analytical correlation was derived to allow calculation of yield stress from several types of penetration measurements (proctor penetration resistance was not included in those derived).

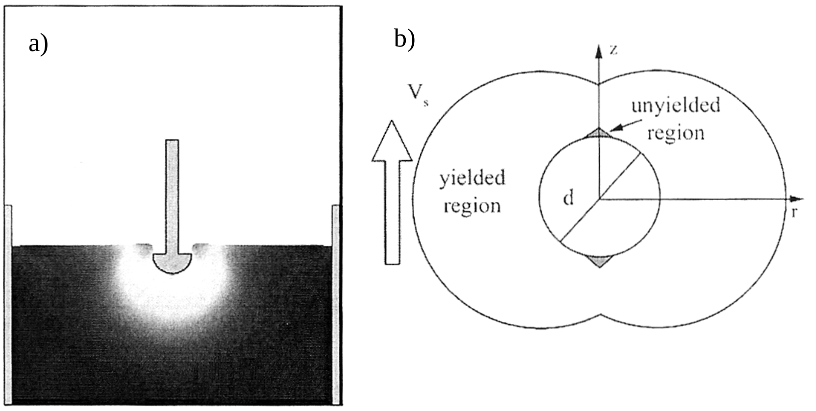


Figure 2.11: a) FEM model of penetration of a hemispherical tip (Lootens et al. 2007). b) Model used in analysis for yielded and unyielded zones for a sphere in a non-Newtonian fluid (Roussel 2006).

2.2.1.3 Inferring Concrete Setting from Mortar/Cement Paste Testing

In 1956, Scripture noted that penetration based methods were not a reliable means of predicting concrete set due to the discrepancies between Vicat penetration results for a mortar pats sieved from a concrete source, and neat cement pats. Although penetration based methods now exist for both mortar and paste samples, there is little literature dealing with the differences observed in the two measurements. Garcia et al. (2008) stated that any comparison made between the Vicat and Proctor test would be impossible unless both have the same w/c. Struble et al. (2001) noted that the ASTM testing method for determining time of set of a cement paste (ASTM C 191) calls for a w/c that is not practical for use in a traditional concrete mixture. Testing was done to show the applicability of the Vicat penetration test to cement pastes with higher w/c. It was also determined that the proctor penetration test can be comparably applied to cement pastes. Due to the arbitrary set times defined by each method the actual setting times did not agree, but similar trends were noted.

Tuthill and Cordon (1956), noted that mortar sieved from a concrete sample and the concrete sample itself hydrated at differing rates. This was attributed to a number of things, including differing heats of hydration (due to the larger mass of concrete and relatively small mortar sample size), and a difference in perceived workability due to the presence of coarse aggregate. It was concluded that, although mortar and concrete may not be at exactly the same degree of hydration at any given time, their rate of setting and hardening should be proportionate, therefore mortar penetration resistance can be a reliable indicator of concrete setting.

Abel and Hover (2000) compared penetration resistance development in mortar and concrete with the development of a “finisher’s foot.” The theory behind this device was based on a rule of thumb in field placement that “a bull-floated concrete surface is ready for subsequent finishing operations when a worker standing on the fresh concrete leaves a boot print about 6 mm (0.25 in.) deep" (Abel and Hover 2000; ACI 302.1R-96). A penetration needle intended to mimic the boot of an adult man, was used to penetrate a concrete specimen to a depth of 6 mm (0.25 in.) and setting was inferred from these readings. It was found that the penetration resistance developed in a similar manner in the concrete and mortar as can be seen by the natural continuation of the penetration resistance curves from one test method to the other in Figure 2.12.

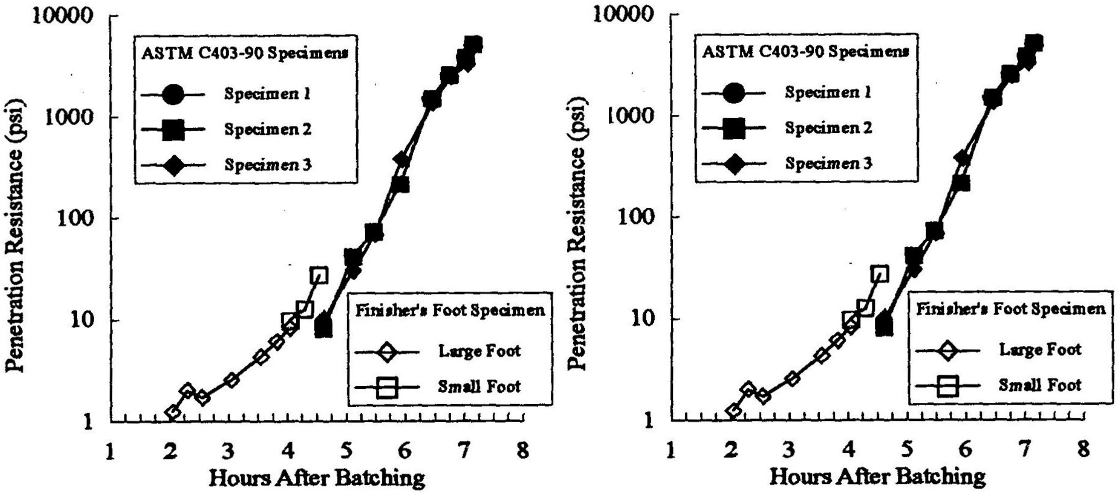


Figure 2.12: Penetration resistance versus concrete age for finisher's foot and ASTM C 403 (Abel and Hover 2000)

2.2.2 Heat of Hydration

One of the older methods for examining the hydration process of cement paste, mortar, or concrete is to examine the heat evolution of a sample (see Section 2.1.1.2) (Weiss 2002). Isothermal calorimetry has been applied in the past to research the energy liberated in the hydration process and has been used to quantify the phases of cement hydration as well as the effects of various additives (Sandberg and Liberman 2007).

Recently, due to high cost of isothermal calorimeters in comparison to other calorimeters and sample size restrictions of such devices (most isothermal devices limit a sample to approximately 100 g where other thermal devices allow for the use of standard concrete cylinders), several researchers have investigated the use of semi-adiabatic calorimetry to develop a thermal based method for determining the setting of concrete.

2.2.2.1 Background

In his overview of concrete setting, Scripture (1956) mentions the use of the rate of heat evolution as a method for determining set. This method was ruled out at the time due to the fact that it is a measure of a chemical property while setting is a physical phenomenon. According to Weiss (2002), initial set has traditionally been linked to somewhere between the dormant stage (stage two) and the rapid hydration of the silicates (stage three). Similarly, final set has been associated with a point near the peak rate of hydration (between stage three and stage four). See Figure 2.13 for a graphical representation of these points for a cement paste sample.

Taplin (1959) and others (Powers and Brownyard 1948; Byfors 1980), have established a link between strength development of concrete and its degree of hydration. As discussed previously (Section 2.1.3.1), the heat of hydration of a sample is often used as an indicator of its degree of hydration (Powers and Brownyard 1948). Since it has been determined that setting occurs when a certain amount of hydration products have formed (Chen and Older 1992), the use of degree of hydration, or an indicator of degree of hydration, to define setting has been investigated.

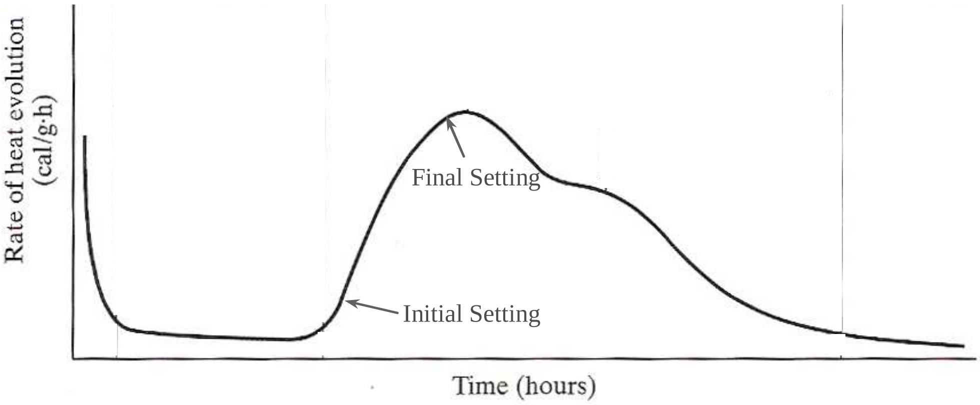


Figure 2.13: Initial and final set points of cement paste as defined by rate of heat evolution (adapted from Weiss 2002)

In order to obtain repeatable and reliable results from thermal testing, thermal devices must be able to accurately record the heat development of concrete. According to the RILEM Technical Committee (TC) 119 - Avoidance of thermal cracking in concrete at early ages (TCE), Isothermal Calorimetry, also known as conduction calorimetry, is the measure of the heat development required to maintain a sample at a constant temperature. Adiabatic calorimetry is the measure of cumulative heat development as a sample hydrates under conditions where there is no heat exchange to or from the testing environment (RILEM TC 119 TCE 1 1997).

Examples of semi-adiabatic and fully-adiabatic temperature profiles for a concrete mixture can be seen in Figure 2.14. A fully-adiabatic profile is obtained by adjusting the measured semi-adiabatic profile for device specific thermodynamic losses as well as for the effect that this temperature difference has on the hydration process. As can be seen below, these curves are initially identical, and diverge as thermal losses increase. The isothermal response of the sample also differs from the fully-adiabatic response. Although the general shape of the profiles is similar, the adiabatic sample hydrates at a faster rate and reaches a higher degree of hydration in a given time than the isothermal sample due to the increasingly higher temperature of the adiabatic calorimeter.

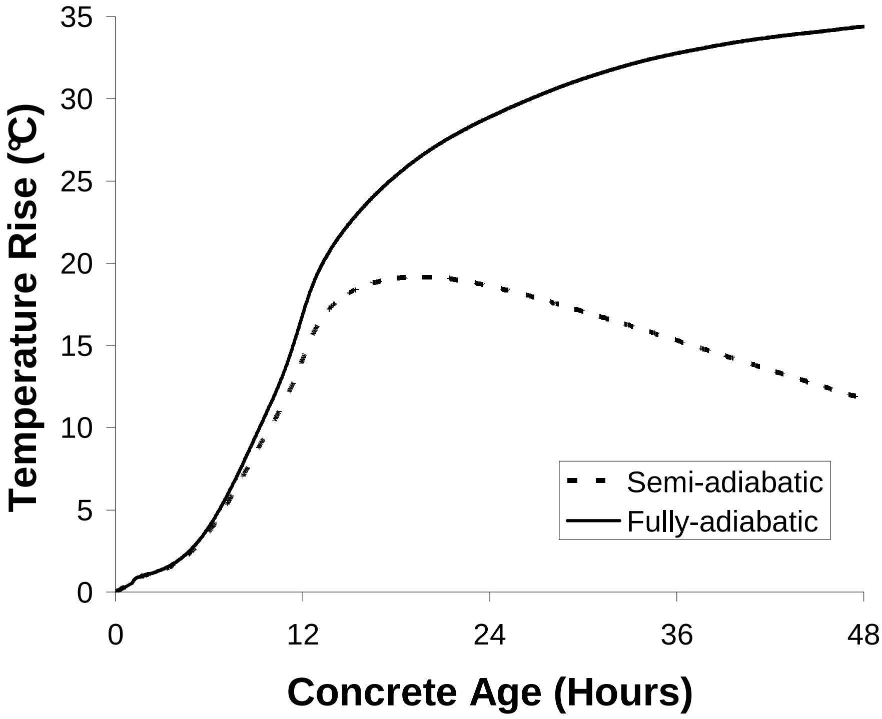


Figure 2.14: Example semi-adiabatic and fully-adiabatic temperature profiles

Isothermal calorimetry methods have historically been preferred to test cement pastes due to the fact that they offer superior accuracy and repeatability in testing. Although useful for cement quality control testing, these methods have downfalls when it comes to concrete testing. Isothermal devices are not only very costly, but they require a sample size impractical for concrete applications (Sandberg and Liberman, 2007). A fully-adiabatic response of concrete is difficult to obtain due to the effects of temperature loss, but it is possible to obtain a semi-adiabatic response with equipment that is readily available and much less expensive than isothermal calorimeters (Christensen 2006). Due to the cost and practicality of semi-adiabatic devices, recent work on thermal set methods for determining setting has primarily focused on semi-adiabatic calorimeter testing methods.

2.2.2.2 Thermal Methods to Determine Setting

In 2007, Sandberg and Liberman made use of methods previously proposed by Bruce Christensen (yet not formerly published) in which temperature data collected with the use of a minimally insulated thermal testing device was used to predict concrete setting. It should be noted that the thermal device used did not meet the requirements set by RILEM TC 119 TCE 1 (1997) to be considered semi-adiabatic. Sample temperature profiles for a mixture tested using a device similar to Sandberg and Liberman (2007) and device considered semi-adiabatic by RILEM TC 119 TCE 1 (1997) are shown in Figure 2.15. It should be noted that the temperatures for the first few hours of testing are relatively independent of device properties, but as time and temperature increase, the insulation or the calorimeter has a substantial effect on the temperature profile obtained.

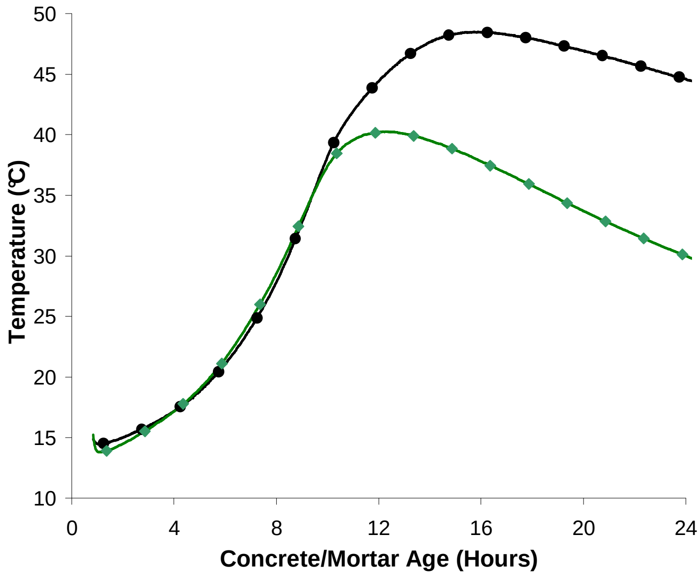


Figure 2.15: Sample temperature profiles from devices of varying properties

Two methods of obtaining setting times comparable to those of ASTM C 403 from temperature histories of concrete samples were proposed, the “Fractions Method”, and the “Derivatives Method.” For the *Derivatives Method*, final set was defined as the maximum slope (first derivative) of the main alite (C_3S) hydration peak and initial set was defined as the maximum curvature (second derivative) of the main alite (C_3S) hydration peak. A plot of the first and second derivative of hydration data can be seen in Figure 2.16 along with points defined as initial and final set. In the *Fractions Method*, initial and final set were defined as percentages of the total semi-adiabatic temperature rise of a specimen (see Figure 2.17). Under standard laboratory curing conditions, default values of 21% and 42% were defined as initial and final set respectively, and “refined” fractions for each cement type and w/c were computed.

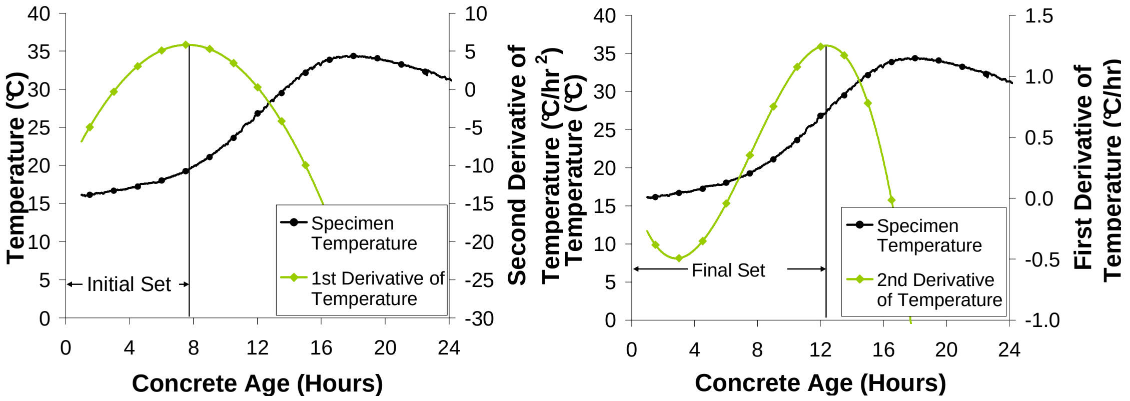


Figure 2.16: Initial and final set as defined by the *Derivatives Method* (adapted from Sandberg and Liberman 2007)

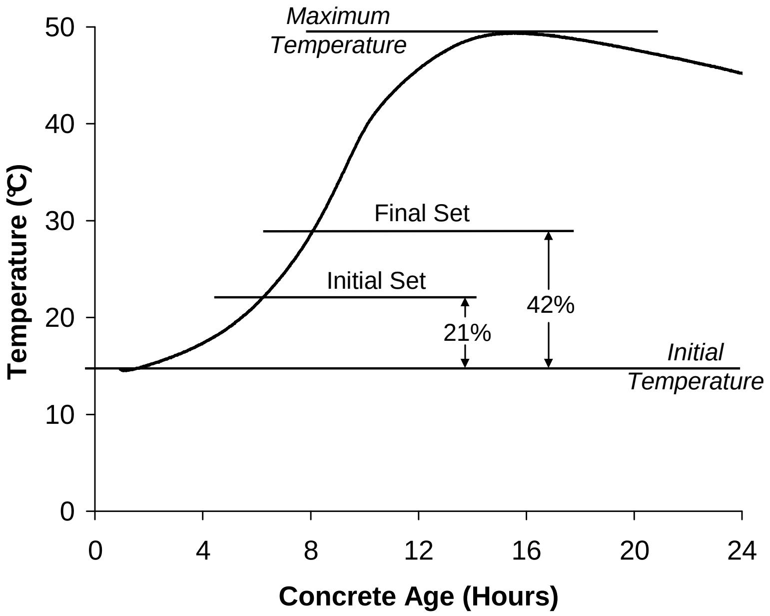


Figure 2.17: Initial and final set as defined by the *Fractions Method* (adapted from Sandberg and Liberman 2007)

It was concluded that thermal set times that correlate to ASTM C 403 were a possibility, but that various mixtures (cured at different temperatures) should be examined. It was also noted that, since minimally insulated devices were used, and no adjustment was made for thermal losses the setting times obtained by this method were heat-loss and device dependent. Therefore, varying concrete and ambient temperatures may have a substantial impact on setting results obtained through these methods.

Schindler (2004b) used semi-adiabatic calorimetry to develop a model in which a maturity-based definition of setting was developed in terms of the degree of hydration of a sample. Setting was determined to occur when a certain percentage of hydration products have formed. These points, similar in concept to the point referred to by Byfors (1980) as the critical degree of hydration, were defined in terms of the w/c of the specific mixture being tested. Initial and final set were defined as the maturity (equivalent age) needed in order to obtain a degree of hydration of 0.15 and .26 times the w/c respectively. The equivalent age at setting obtained through thermal methods were compared to those obtained as per ASTM C 403 (after adjusting for maturity). Both field and laboratory concrete was examined and it was concluded that the model proved reasonably accurate for all mixtures studied excluding those containing GGBF slag. These results were supported by a separate study by Edison (2007), although the critical degree of hydration for initial and final set were found to be 0.12 and 0.22 times the w/c, respectively.

In 2007, a draft ASTM specification was proposed for determining the setting times of concrete using thermal methods. Based on the previously mentioned research , this draft ASTM specification outlines a method for determining final and initial setting times of concrete by the maximum first and second derivatives, respectively. Derivatives were calculated using a direct-slope approach and a 200-minute smoothing interval. Specifications for an insulated device was outlined and no adjustments were made for maturity or device thermal losses. For discussion and further data relating to this proposed specification see Chapter 3.

2.3 Factors That Influence Time of Setting

Time of setting of a concrete mixture is affected by a number of factors. In order to accurately predict concrete setting, it is helpful to understand the influences of these factors. Properties of the cement itself, such as type, brand, and fineness, influence the chemical composition of cement and the way and rate in which it hydrates. Properties of the mixture design such as total cement content, w/c, SCM's, and the presence of admixtures also have a significant affect on the chemical reactions involved. In addition, since the concrete hydration process is a result of a series of temperature dependent reactions, placement and curing conditions, especially temperature, have a significant impact on setting times obtained (Dobson 1994).

2.3.1 Cement Properties

Specific properties of cement used in concrete can have a significant impact on the setting characteristics of a concrete mixture. Cement type, brand, and fineness have all been found to affect the setting times obtained as per ASTM C 403 (Dobson 1994).

2.3.1.1 Cement Fineness

The effects of the chemical compounds that make up portland cement on setting have been discussed in Section 2.1.1. It should be logical then that the relative amounts of these compounds can make a substantial difference in the setting times of a mixture. Less obvious is the effect of the fineness of a cement. As can be seen in Figure 2.18, setting is accelerated as the fineness of the cement used increases, all other properties being held constant.

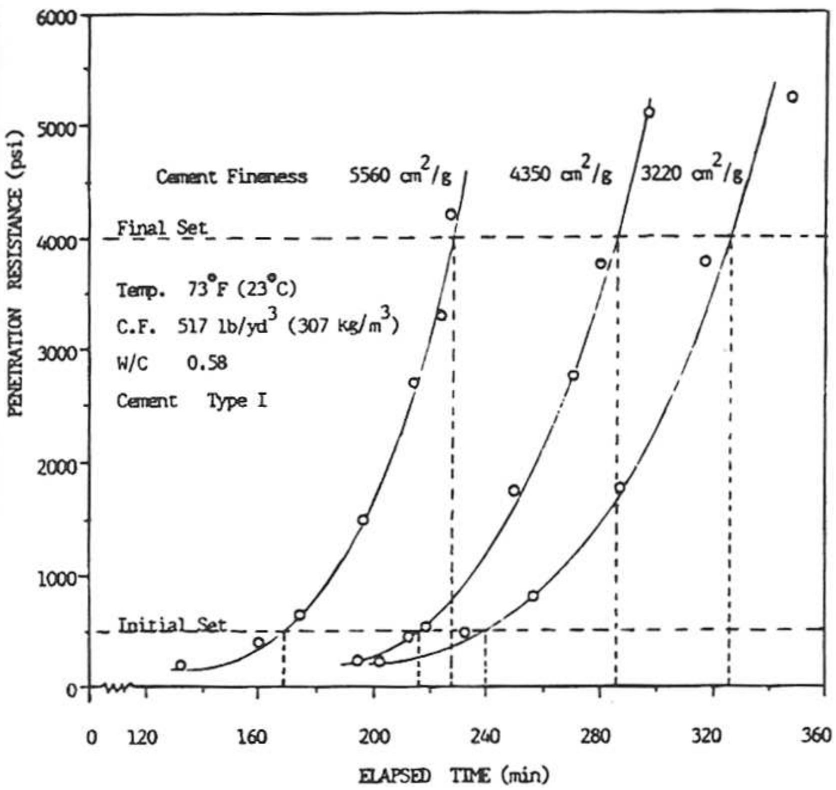


Figure 2.18: Effect of cement fineness on concrete setting (Dobson 1994)

For a given chemical composition, the rate of reactivity with water is dependent on the fineness of that particular cement (Mehta and Monteiro 2006). Although the cost of grinding and the high heat of hydration may set practical limits, cement companies partially control the rate of setting and strength gain by the fineness of the cement particles (Mehta and Monteiro 2006). A study conducted by Dobson (1994) examined the effect of cement fineness on setting times.

2.3.1.2 Cement Type

In order to standardize the different cements produced, ASTM recognizes five distinct portland cement types. These differ only in their relative amounts of cement compounds and their fineness (Mindess, Young, and Darwin 2003). The effect of cement type can be seen in Figure 2.19. .

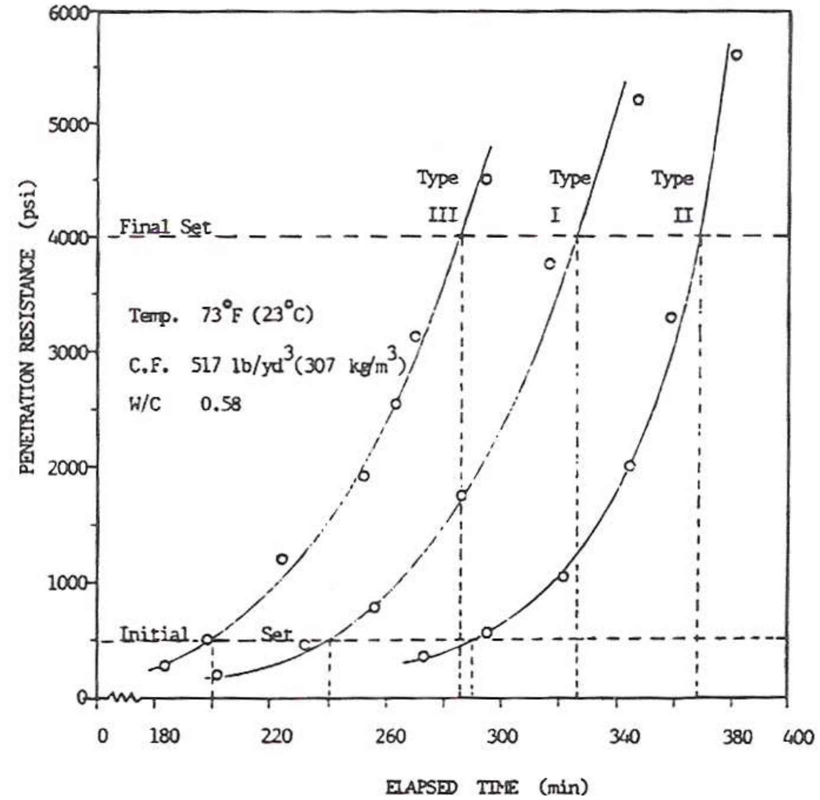


Figure 2.19: Effect of cement type on setting times (Dobson 1994)

Setting times for ASTM Type I, II, and III cements from a single supplier each having individual chemical compositions, particle size distribution, and fineness were examined and are the results are plotted. As is shown in Figure 2.19, the development of penetration resistance is highly dependent on the type of cement used. Setting occurs earliest for the mixture containing a Type III cement, followed by the mixtures containing a Type I and Type II cement, respectively

2.3.1.3 Cement Source

Cement brand also has a definite effect on the properties of concrete, even while using a specific cement type. Dobson (1994) examined the setting times of various Type I cement concrete mixtures that vary only in the source of cement. The results can be seen in Figure 2.20.

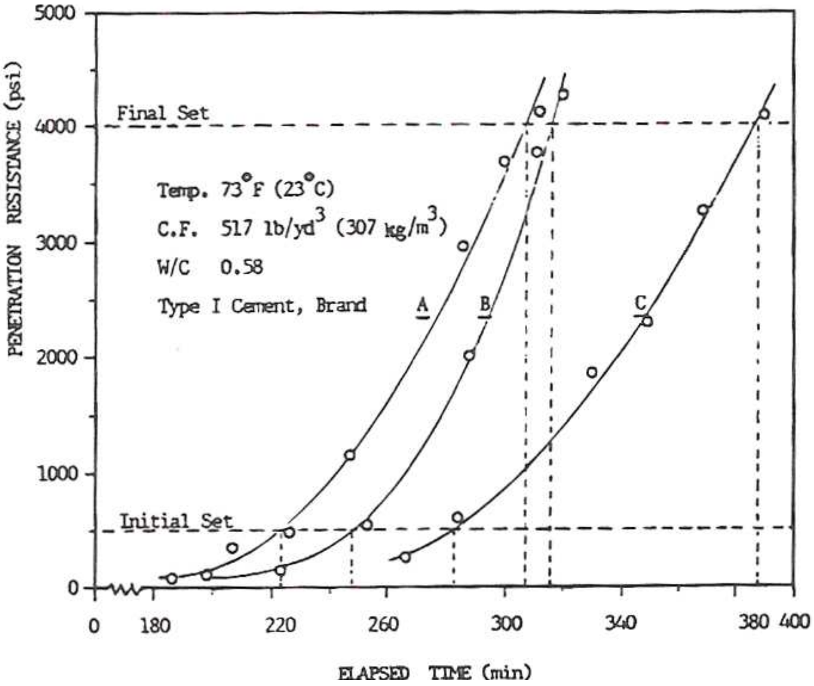


Figure 2.20: Effect of cement brand on concrete setting (Dobson 1994)

Even with all of the above mentioned cement properties being held constant, setting times may still vary based solely on the source of the cement used. As can be seen in Figure 2.20, when two mixtures which were identical except for the cement source used, were tested as per ASTM C 403, concrete setting times varied by as much as an hour.

2.3.2 Concrete Mixture Properties

Specific characteristics of a concrete mixture design also have a measurable impact on setting times measured. The total cement content, ratio of water-cement used, and the presence of chemical admixtures and mineral admixtures, such as GGBF slag and fly ash, all have a substantial effect on the setting behavior of a mixture (Dobson 1994, Bentz et al. 2008, Naik and Singh 1997).

2.3.2.1 Cement content

At a fixed water content, the total cement content in a specific concrete mixture will have a dominating effect on the total amount of hydration products formed in a concrete mixture. Therefore, the total cement content, or the cement factor (mass of cement per cubic meter of concrete), has a significant effect on the rate of hydration, and the rate of concrete setting. This effect was examined by Dobson (1994) and can be seen in Figure 2.21. As can be noted below, the mixture with a higher total cement content reaches initial and final set significantly sooner than that with a lower total cement content, all other things being held constant.

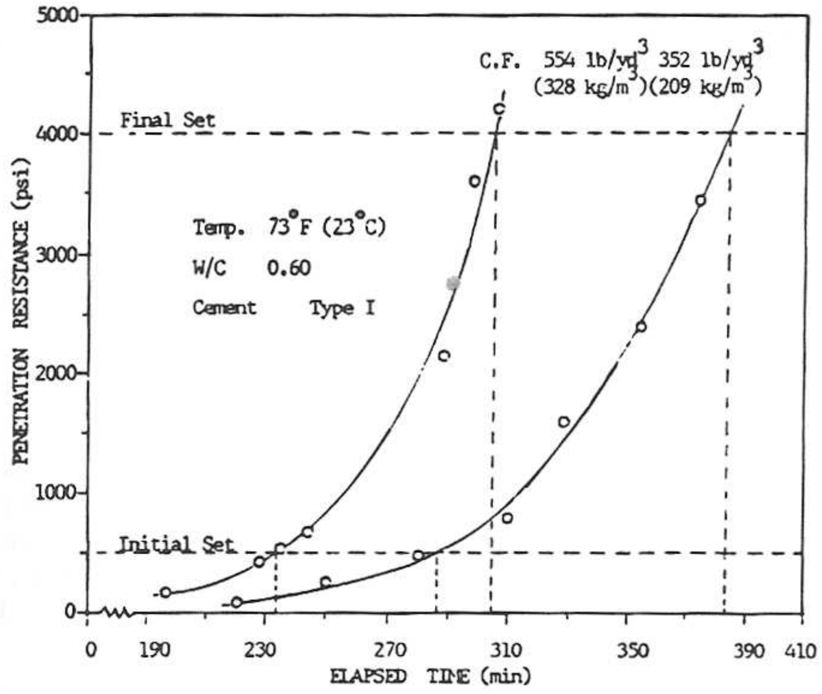


Figure 2.21: Effect of total cement content on time of setting (Dobson 1994)

2.3.2.2 Water-Cement Ratio

Research has noted that the greater the water-cement ratio (w/c) or water-cementitious-materials ratio (w/cm) of a concrete, cement paste or mortar sample, the longer the setting times (Dobson 1994). The effect of w/c on ASTM C 403 setting times can be seen in Figure 2.22. As the w/c increases, so does the time required to reach initial and final set. Bentz et al. (2008) and Struble et al. (2001) noted a similar effect on the setting of cement paste with the use of the vicat penetration test. The testing was completed as per ASTM C 191 (2008) except that instead of “normal” consistency, the paste was mixed with a w/c similar to that of concrete mixtures. Both found a similar delay in setting as w/c increases. Results from Srtuble et al. (2001) can be seen in Figure

2.23. Points of initial and final set are represented by a penetration depth of 25 and 40 mm, respectively.

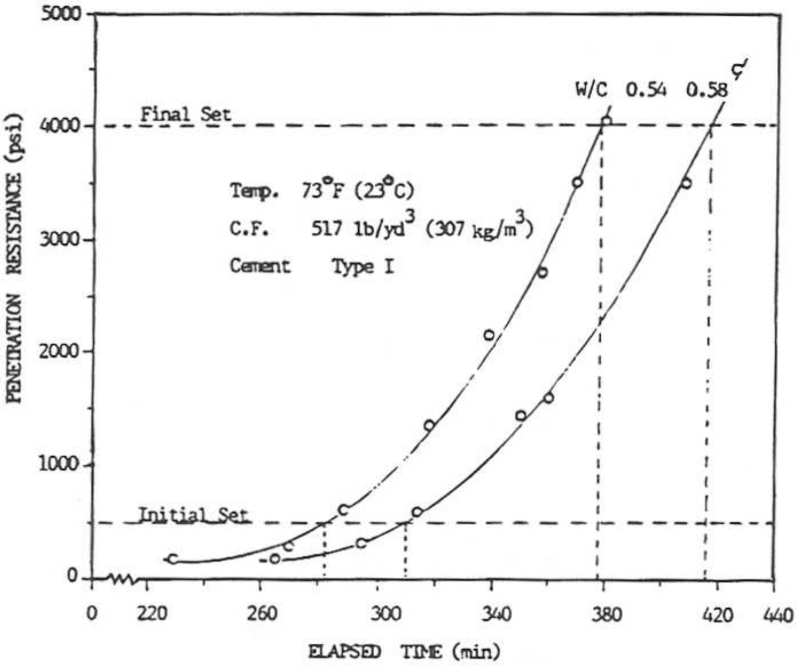


Figure 2.22: Effect of water-cement ratio (w/c) on ASTM C-403 setting times (Dobson 1994)

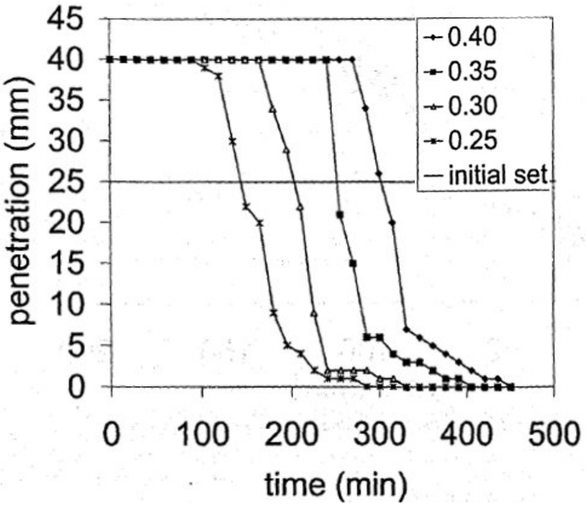


Figure 2.23: Effect of water-cement ratio (w/c) on cement setting (Struble et al. 2001)

Bentz et al. (2008) also noted that, although the w/c had a measurable impact on setting, it had very little to no impact on the heat release of a sample during the setting window (the first 12-hours of testing). Both the heat release and the rate of heat release (heat flow) can be seen in Figure 2.24 for cements of various w/c.

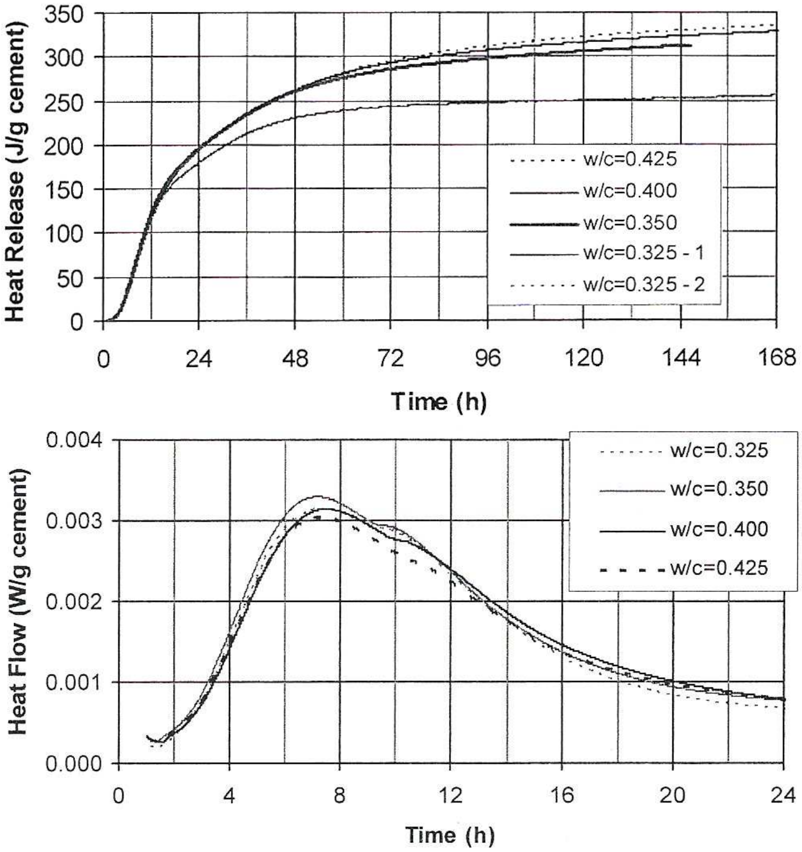


Figure 2.24: Heat released and rate of heat released for cement mixtures of varying w/c (Bentz et al. 2008)

As is evident in Figure 2.24, there is very little difference in results from various w/c mixtures for the first 12-hours of testing. It was determined that, if thermal data were to be used to define setting, the critical degree of hydration would have to be a function of w/c. They theorized that mixtures with higher w/c require a higher degree of hydration for setting to occur due to an increase in distance between cement particles that must be filled with hydration products.

2.3.2.3 Chemical Admixtures

ASTM C 494 (2008) admixtures Types B, C, D, E, and G all have either accelerating or retarding properties. These admixtures are used to accelerate or retard the setting of concrete for placement in specific conditions (i.e. hot or cold weather) or for specific applications where other than typical setting times are needed.

2.3.2.4 Supplementary Cementing Materials

The effect of GGBF Slag on the setting time of concrete is fairly well documented. Sivasundaram and Malhotra (1992) found that, although initial values for mixes that contain 50 to 60 percent GGBF Slag remained fairly constant when compared to traditional concrete, final setting times were greater. This longer lag between initial and final setting times was also noted by Saki et al. (1992). Initial and final setting times were measured for concrete containing various amounts of several sources of GGBF slag and the results were plotted. As can be seen in Figure 2.25, as the percentage of GGBF slag was increased, initial set remained relatively constant, while final set increased considerably (see Figure 2.25).

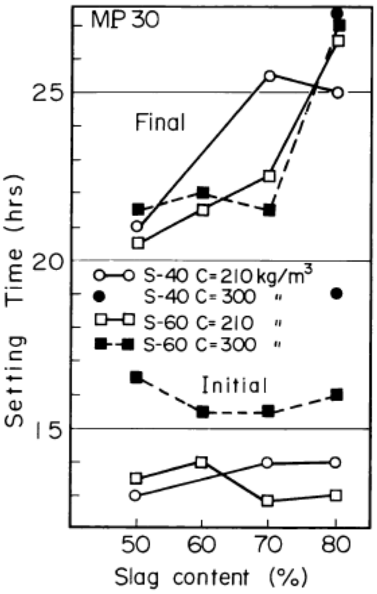


Figure 2.25 Effect of slag content on setting time of concrete (Saki et al. 1992)

Fly ash is generally thought to increase the setting time of a concrete mixture due to its dependency on the formation of calcium hydroxide and its slower rate of hydration. Although Class F fly ash usually delays set, Class C fly ash has been known to both increase and decrease setting times of a mixture (Naik and Singh et al. 1997). Naik and Singh et al. (1997), in a study using four separate sources of fly ash, found that Class C fly ash increased the setting times of a concrete mixture up to about 60 percent replacement, above which setting was accelerated. It should be noted that a constant w/cm was not used for these mixtures, therefore, interference due to these variations are expected. Regardless of this fact, fly ash’s effect on setting can be considered highly dependent on both the source of the fly ash and the amount used. See Figure 2.26 and Figure 2.27 for initial and final set times respectively for mixtures with varying amounts of the four sources of Class C fly ash. It was suggested that delayed set was caused by the effect of dilution while accelerated set was thought to be caused by flash set (due to a severe sulfate imbalance at high percent replacements).

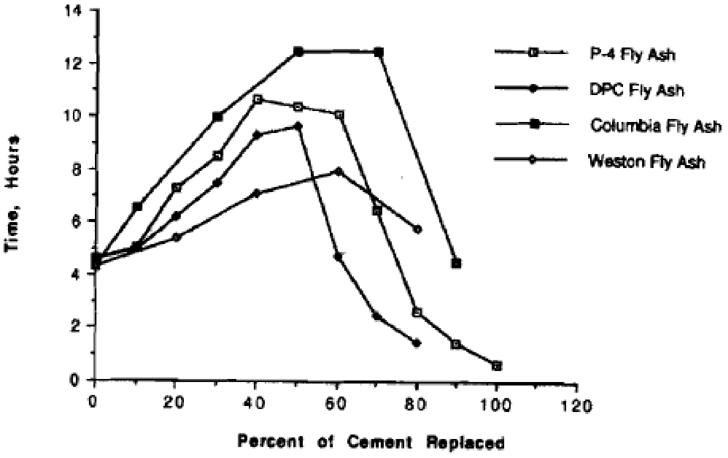


Figure 2.26: Initial set for concrete with varying amounts of Class C fly ash (Naik and Singh et al. 1997)

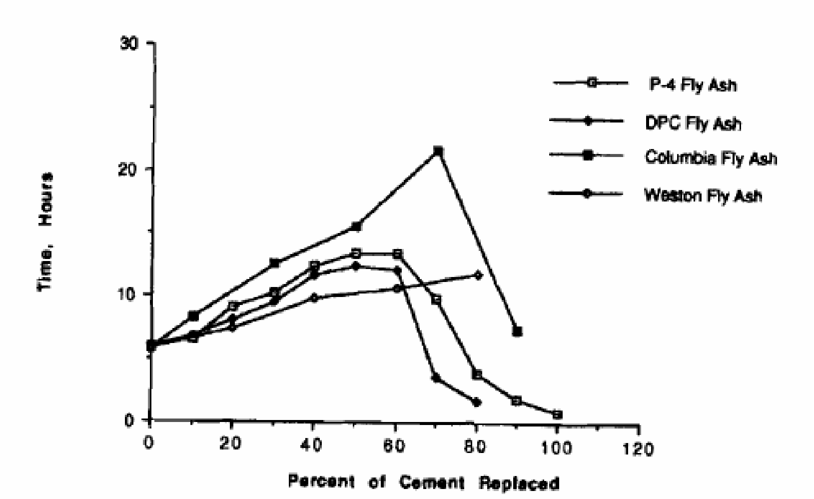


Figure 2.27: Final set for concrete with varying amounts of Class C fly ash (Naik and Singh et al. 1997)

2.3.3 Testing Conditions

The hydration of portland cement is a temperature dependent reaction, therefore the mechanical properties that are measured as indicators of concrete set are highly temperature dependent as well. Tuthill and Cordon (1956) noted this in the development of the Proctor penetration setting test. It was noted that, with the decrease in temperature from 37° C to 10° C, the time of initial set increased by as much as 400 percent. Dobson (1994) documented this trend as well. Figure 2.28 shows penetration resistance readings for concrete at temperatures ranging from 10° C to 32° C. The general increase in setting times as temperature decreases can be seen in Figure 2.29. A similar trend was observed by Eren et al. (1995) for concretes containing 30 to 50 percent Class F fly ash and 50 percent GGBF Slag.

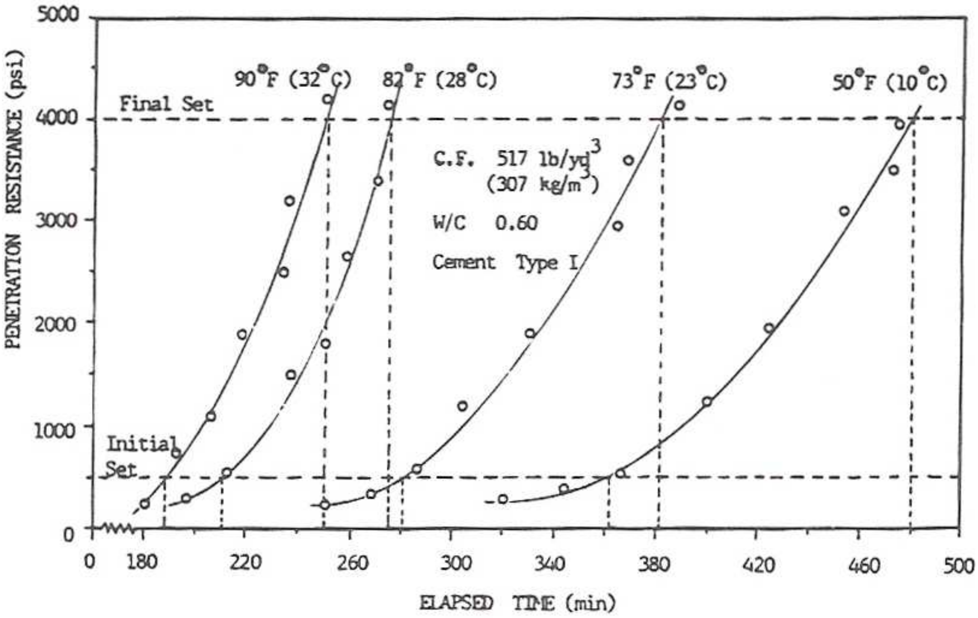


Figure 2.28: Effect of temperature on penetration resistance (Dobson 1994)

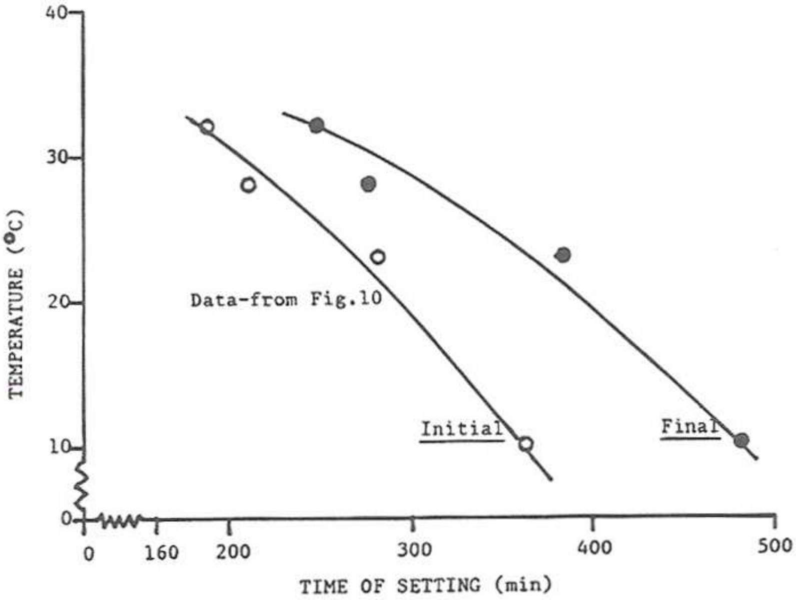


Figure 2.29: Effect of temperature on ASTM C 403 setting times (Dobson 1994)

2.4 The Maturity Method

The maturity method is a technique for estimating concrete behavior and hydration development based on the measured temperature history of the concrete sample. The effects of concrete age and temperature are combined in an incremental analysis which allows a user to accurately predict in-place strength development (Carino 2004). Although a majority of past research has been focused on the application of this method to predict concrete strength, it has been shown to be useful for predicting the effect of temperature on other properties as well. In this section, common maturity functions will be presented, and the application of the maturity method to concrete properties, specifically setting, will be discussed.

2.4.1 Maturity Functions

McIntosh (1949) was the first to publish a method that accounted for time and temperature in the prediction of concrete strength development. It was proposed that the "rate of hardening at any moment is directly proportional to the amount the curing temperature exceeds the [datum] temperature." He suggested the use of the product of time and temperature above a datum temperature, or the temperature below which strength gain will not occur, as a means of quantifying temperature effects.

This product of time and temperature was also used in research by Nurse (1949) and Saul (1951) and eventually led to the Nurse-Saul maturity function. With this function, a maturity index known as the Temperature-Time-Factor (TTF) is computed. This index represents the cumulative amount of temperature and time a concrete sample has accumulated. This function is defined in ASTM C 1074 (2004) as follows:

$$M(t) = \Sigma\,(T_a - T_o)\ \Delta t \qquad \text{Equation 2.10}$$

where:

$M(t)$ = the temperature-time factor at age t (°C-hour),

Δt = a time interval (hours),

T_a = average concrete temperature during time interval, Δt (°C), and

T_o = datum temperature (°C).

This linear-rate function is relatively simple but cannot be adjusted to account for the temperature sensitivity of individual concrete mixtures. The only user-defined variables in this function are the time interval and the datum temperature. ASTM C 1074 (2004) recommends using one half hour or less for the time interval for the first 48 hours, after which longer intervals are permitted. Although various datum temperature values have been proposed, ASTM C 1074 (2004) recommends the use of 0° C for Type I cement mixtures without admixtures at temperatures ranging from 0° C to 40° C (32° F to 104° F). Values for other mixtures can be obtained experimentally by following the procedure outlined in the Annex A of ASTM C 1074 (2004).

In 1977, Freiesleben, Hansen, and Pedersen proposed the use of a maturity function based on the Arrhenius equation. Although slightly more rigorous, this function has proved to be more representative of the time-temperature effects on the strength development of concrete (Carino 2004). It quantifies the effects of temperature on the strength development of concrete cured at arbitrary temperatures in terms of an "equivalent age", or the time that concrete must cure at a reference temperature to result in an equivalent maturity. The Arrhenius method maturity function, also known as the FHP function, is defined in ASTM C 1074 (2004) as follows:

$$\mathrm{t_e} = \sum e^{-\frac{\mathrm{E}}{\mathrm{R}}\left(\frac{1}{T_c}-\frac{1}{T_r}\right)}\Delta\mathrm{t} \qquad \text{Equation 2.11}$$

where:

t_e = equivalent age at specified temperature T_s (hours),

E = activation energy (J/mol),

T_c = average temperature of concrete during time interval Δt (K),

T_r = specified temperature (K),

Δt = time interval, (hours), and

R = universal gas constant.

Values recommended for the time step are identical to those for the Nurse-Saul function. The reference temperature, T_r, has traditionally been taken as 20° C (68° F) but it is permitted to use 23° C (73° F) as well. The activation energy determines the overall effect of temperature within the maturity function (Carino 2004). ASTM C 1074 (2004) recommends the use of 40,000 to 45,000 J/mol for concrete containing Type I cement without admixtures. Activation energy values are highly dependent on mixture proportions and may be determined experimentally for a higher degree of accuracy.

2.4.2 Application of the Maturity Functions

The Freiesleben, Hansen, and Petersen (FHP) maturity function is generally considered the most accurate maturity formulation (Carino 2004). Based on the Arrhenius equation, the FHP function uses the concept of activation energy (E) to define the sensitivity of a particular concrete mixture to changes in temperature. This value becomes more critical the further the curing temperature varies from the reference temperature. An accurate approximation of E is essential for maturity applications in which semi-adiabatic and adiabatic calorimetry is used due to the high temperatures seen during the hydration process (Schindler 2004a).

A number of methods have been suggested for the calculation of E for a particular concrete mixture. One of the most common methods is as per ASTM C 1074 (2004), the *Standard Practice for Estimating Concrete Strength by the Maturity Method*. This specification, based on research by Tank and Carino (1991), outlines a process for calculating E based on the strength of mortar cubes cured at three different isothermal temperatures. Although this method is commonly used for strength prediction, Schindler (2004a) found that these values are unreliable when maturity is to be used to predict the progress of hydration. A method similar to ASTM C 1074 but based on isothermal calorimetry data instead of strength was determined to be a better approximation of E for calculations during the hydration process (Schindler 2004a; Poole et al. 2007).

Schindler (2004a) also proposed a method of estimating E based on the total content of the cement compounds in a given mixture, the fineness of the cement, and presence of supplementary cementing materials. This formulation is as expressed in Equation 2.12.

$$E = 22{,}100 \cdot f_E \cdot p_{C_3A}{}^{0.30} \cdot p_{C_4AF}{}^{0.25} \cdot \text{Blaine}^{0.35} \qquad \text{Equation 2.12}$$

where:

E = activation energy, J/mol,

p_{C_3A} = weight ratio of C_3A in terms of the total cement content,

p_{C_4AF} = weight ratio of C_4AF in terms of the total cement content,

Blaine = Blaine value, specific surface area of cement, m^2/kg, and

f_E = activation energy modification factor for SCM’s, which may be expresses as in Equation 2.13.

$$f_E = 1 - 1.05 \cdot p_{FA} \cdot \left(1 - \frac{p_{FACaO}}{0.40}\right) + 0.40 \cdot p_{SLAG} \qquad \text{Equation 2.13}$$

where:

f_E = activation energy modification factor for SCM's,

p_{FA} = weight ratio replacement of fly ash,

p_{FACaO} = weight ratio of CaO content contained in fly ash in terms of the fly ash content, and

p_{SLAG} = weight ratio replacement of GGBF slag.

Pinto and Hover (1999) demonstrated the use of penetration data obtained as per ASTM C 403 (2008) at different temperatures to calculate the apparent activation energy of a concrete mixture, eliminating the need for costly isothermal devices. Wade et al. (2007) validated these results with a wide variety of concrete mixtures. Garcia et al. (2008) reached similar conclusions when a similar method was applied to the Vicat penetration resistance of various cement pastes.

Once the activation energy of a given mixture is determined and temperatures of a sample are recorded, Equation 2.11 can be used to calculate the equivalent age of a concrete mixture. This equivalent age can be used to account for the effects of temperature on concrete properties and aid in the estimation of in-place strength prediction for form removal, post-tensioning, and other construction practices. In the laboratory it can be used to ensure that samples of differing sizes (which result in differing temperature profiles due to heat of hydration) are properly compared. By using equivalent age instead of actual age as a basis for comparison, inaccurate correlations due to differing temperatures can be avoided (Carino 2004). The effect of comparing concrete strength based on maturity instead of actual concrete age can be seen in Figure 2.30. As can be seen, concrete compressive strength varies substantially at a given *concrete age* for mixtures cured at differing temperatures, but at a given *equivalent age* strength is relatively constant across the temperature range.

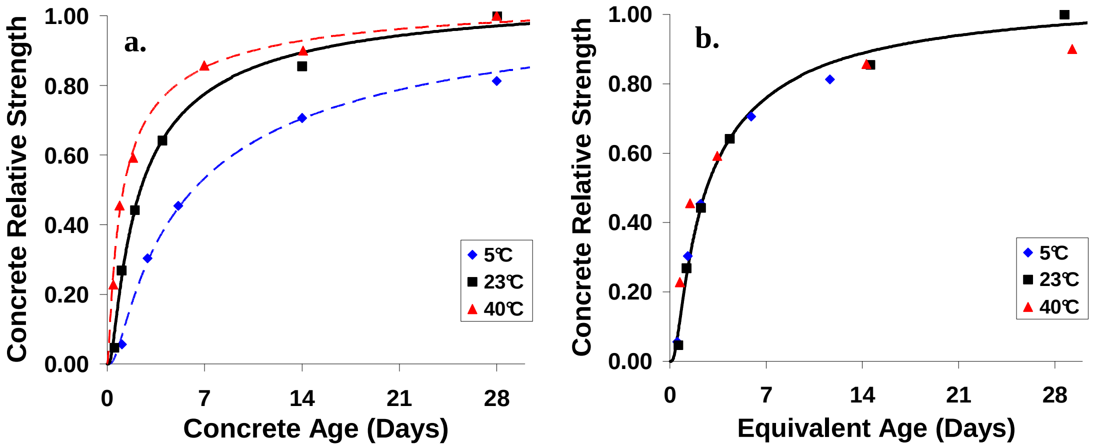


Figure 2.30: Concrete compressive strength versus concrete age and concrete equivalent age

2.4.3 Maturity and Setting

Although maturity concepts have been used primarily to aid in the prediction of concrete strength, maturity methods have been use for other applications as well. Weiss (2002) suggested that a maturity-based definition of concrete setting times would be more appropriate than concrete age due to the dependency of setting on curing temperature.

Pinto and Hover (1999) successfully applied the maturity method to concrete setting as defined by ASTM C 403. An example of the maturity approach as applied to penetration data can be seen Figure 2.31.

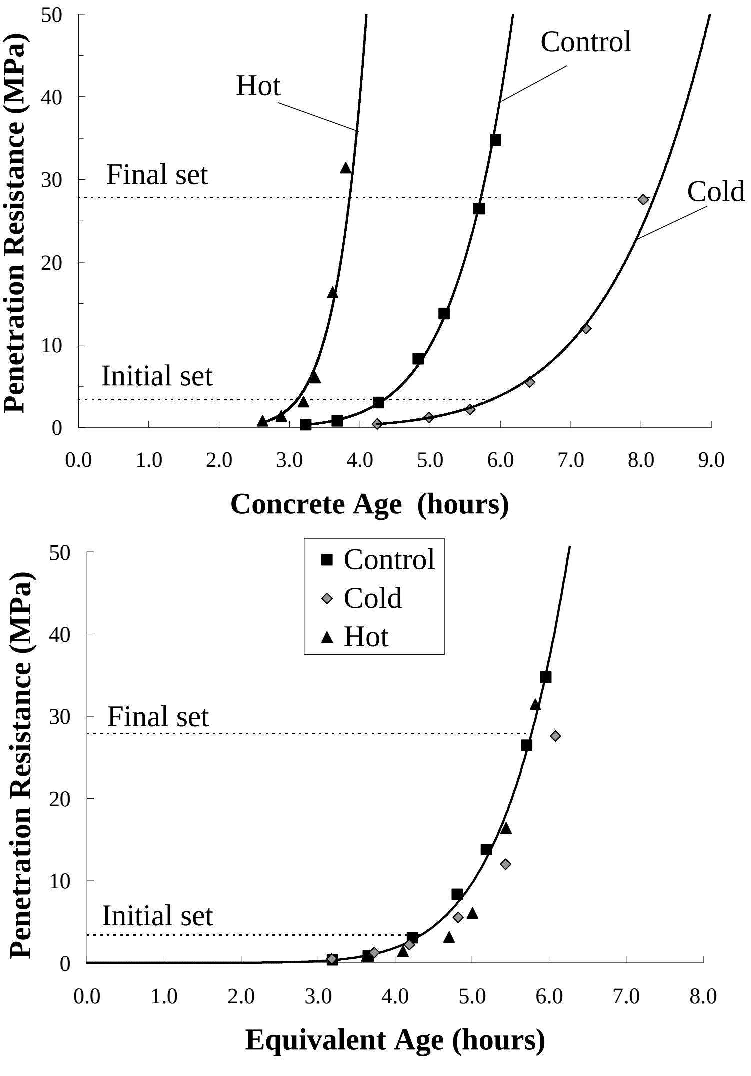


Figure 2.31: FHP maturity function applied to ASTM C 403 penetration resistance readings (adapted from Wade et al. 2007)

It was found that, given the apparent activation energy of a mixture, the maturity method was able to greatly reduce the variability in setting times caused by temperature differences (Pinto and Hover 1999). These observations were based on a single Type I cement concrete mixture with a w/c of 0.33. Wade et al. (2007) reached similar conclusions for a wide variety of concrete mixtures. Garcia et al. (2008), in a study modeled after Pinto and Hover (1999), successfully applied the maturity approach to Vicat penetrations of cement pastes.

Schindler (2004b) used the FHP maturity method in the formulation of a definition of concrete setting. Setting was defined as the maturity (or equivalent age) in which the concrete reached a pre-defined degree of hydration. By using the maturity method, setting was accurately approximated with a variety of mixtures under both laboratory and field conditions.

2.5 Summary

In this chapter, the historical and technical information required to understand current methods of quantifying setting and to evaluate thermal methods to determine setting was presented. Major findings presented in this chapter are summarized below.

Major findings concerning the background and mechanisms of concrete setting are as follows:

- Concrete setting, defined as the increase in rigidity due to the hydration of cement, can be linked to the hydration of tricalcium silicate and tricalcium aluminate. These reactions lead to the formation of calcium silicate hydrate and ettringite, and setting is believed to occur when a specific amount of these products is formed.

- The progress of the hydration reactions of cement compounds can be quantified through the measurement of heat of hydration. Similarly, the progress of the hydration reactions within cement paste or concrete (a.k.a. degree of hydration) can be quantified through the measurement of cement paste or concrete heat development.
- Concrete setting is believed to occur at a pre-defined degree of hydration, dependent on the w/c of the concrete mixture.

Major findings concerning the determination of concrete setting times are as follows:

- Setting times are defined in terms of concrete physical properties. Initial set is generally defined as the time at which concrete loses workability and final set is defined the time at which a significant strength gain begins.
- Concrete setting has traditionally been quantified through the measurement of the penetration resistance of a sieved-mortar sample. Although not a direct measure of setting, this test has been shown to be a good indicator of the setting process and links between shear strength development and penetration resistance have been developed.
- Current penetration resistance-based setting methods have been criticized due to the need for constant human monitoring and the lack of correlation between setting results of sieved-mortar samples and in-place concrete.

- Recent work has explored the use of thermal methods to quantify concrete setting. These methods vary both in thermal devices used for testing and in analysis technique applied.
- Thermal devices that have been used for the measurement of concrete set by thermal methods range from minimally-insulated devices to semi-adiabatic calorimeters that meet the requirements of RILEM TC 119 TEC 1 (1997).
- Concrete setting has been defined in terms of the slope and curvature of thermal temperature profiles, in terms of a fraction of the maximum temperature rise measured during thermal testing, and in terms of degree of hydration determined through analysis of thermal data.

Factors that affect the rate of setting of concrete were also examined in this chapter. Major findings concerning these factors are as follows:

- The effect of specific properties of portland cement used within a concrete mixture were examined. Cement fineness, cement source, and ASTM C 150 (2008) cement type were all found to have a significant effect on setting times obtained as per ASTM C 403 (2008).
- Properties of concrete mixtures were examined, and the cement content, water-cement ratio, use of supplementary cementing materials and the presence of chemical admixtures were all found to have a significant effect on setting times obtained as per ASTM C 403 (2008).
- It was determined that the water-cement ratio of a concrete mixture has a significant impact on setting times obtained as per ASTM C 403 (2008) yet has

little effect on thermal testing results. As a result of this effect, it is believed that mixtures with higher w/c require a higher degree of hydration for setting to occur due to an increase in distance between cement particles.

- Concrete hydration and setting times were determined to be highly dependent on curing temperature. As the temperature of concrete increases, so does its rate of hydration, yielding shorter setting times for concrete that is cured at high temperatures and longer setting times for concrete cured at lower temperatures.
- The equivalent age maturity method was shown to accurately account for the effects of temperature on concrete setting. It was suggested that a maturity-based definition of concrete setting be used to allow for accurate comparisons between setting methods and to allow better correlations between setting result and in-place concrete.

Chapter 3

ASTM Round Robin Study

3.1 Background

Due to the increased interest in the use of thermal methods to predict concrete setting, a draft ASTM standard entitled the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method* (see Appendix A) was proposed. On November 19, 2008, a round robin study was conducted by ASTM Committee C09.21.01 regarding this specification as well as traditional penetration resistance-based methods for determining setting times of concrete. In this study, testing was completed by 12 independent laboratories (one of which included the author) in an attempt to calculate the precision and bias of ASTM C 403 (2008) and to evaluate the use of the thermal methods, as per the draft specification, to determine concrete setting times.

3.2 Research Objectives

The *primary* objectives of this study were as follows:

a) Prepare a precision and bias statement for ASTM C 403 (2008), *Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance,*

b) Evaluate the previously proposed ASTM specification on the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method*, and

c) If possible, prepare a precision and bias statement for the previously proposed ASTM specification on the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method.*

3.3 Experimental Plan

3.3.1 Round Robin Design

In order to prepare a meaningful precision statement and to evaluate the proposed specification, the following tests were conducted by 12 independent laboratories, designated in this thesis by group numbers. Three concrete mixtures with different target setting times ranging from 5 to 10 hours were examined. Setting times for the three mixtures were obtained using both ASTM C 403 (2008) and various versions of the proposed temperature methods. All sampling and testing were done independently, the only requirement being that all applicable specifications be followed. Testing was performed in duplicate or triplicate to ensure necessary information for a meaningful precision statement as required by ASTM C 802 (1996).

Concrete was delivered to the laboratory in a ready mix truck roughly 20 - 25 minutes after batching each mixture. Upon concrete arrival, slump, air content, unit weight, and temperature were recorded and quality control cylinders were made for each mixture as per ASTM C143 (2008), ASTM C231 (2008), ASTM C138 (2008), ASTM C 1064 (2008), ASTM C39 (2008), respectively. Concrete was obtained by each participating group, and setting samples for both ASTM C 403 (2008) and the thermal testing methods were prepared. Specimens for ASTM C 403 (2008) were prepared by wet-sieving mortar by using a vibrating table or sieving screens, and filling the provided container in a single lift. The mortar sample was then consolidated by either rodding, tapping, or by use of a vibrating table as permitted in ASTM C 403 (2008). Group 3 and Group 10 recorded the temperature of one ASTM C 403 (2008) mortar sample from each mixture for analytical purposes. Preparation of the specimen for thermal testing varied depending on the group and temperature-monitoring device. In general, concrete or mortar samples were filled in one lift with minimum consolidation (cylinders were gently tapped on the floor), capped, and placed directly in their respective devices.

ASTM C 403 (2008) testing was initiated when a test specimen reached an initial bearing pressure of approximately 1.03 MPa (150 psi). Each group conducted the test individually, as per ASTM C 403 (2008) with noted deviations. Mortar sample containers smaller than those specified in Section 6.1 were used by all groups excluding Groups 1 and 4 (see Section 3.3.4 for details on containers used). Analysis of the data was completed by each group and verified with the use of a spreadsheet and logarithmic curve fitting of the thermal data.

For temperature method analysis, samples were placed in thermal testing devices and they remained undisturbed for the duration of the test. Temperatures were recorded at varying intervals, but no less than one sample per minute.

3.3.2 Testing Conditions

The concrete used for this study was batched off site and delivered by a ready mix truck to the Heidelberg Technology Center in Atlanta, Georgia. All testing was conducted in an enclosed room unless otherwise stated. For a graph of room temperatures recorded refer to Figure 3.1. Three separate mixtures were developed in order to obtain concrete samples with independent setting times and to evaluate the effect of different cementitious materials on the setting testing methods. For the concrete mixture proportions used in this round robin study see Table 3.1.

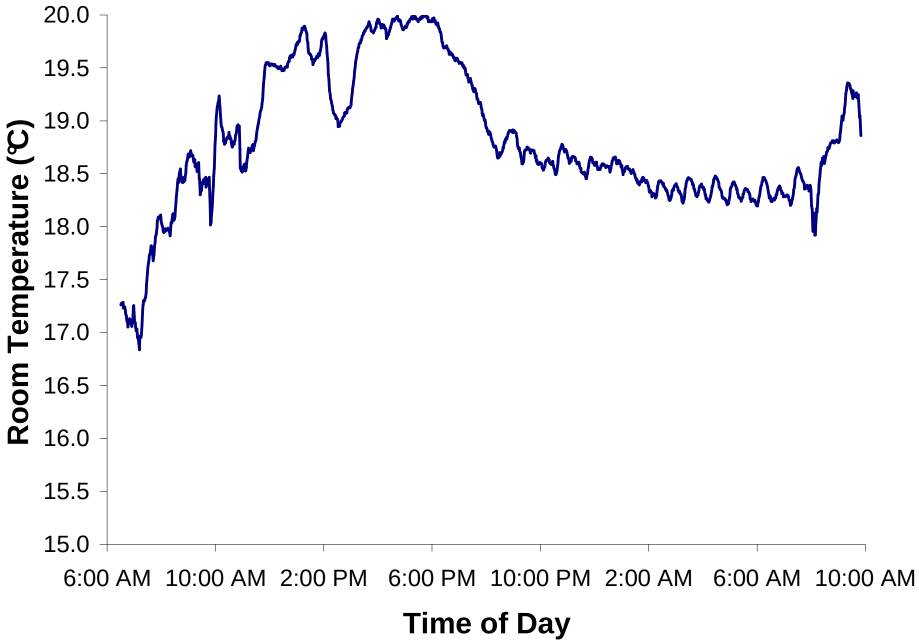


Figure 3.1: Laboratory ambient temperature during testing

Table 3.1: Concrete mixture proportions

Constituent	Mixture Identification		
	1	2	3
Water (kg/m^3)	173	178	178
Type I Portland Cement (kg/m^3)	-	362	279
Type III Portland Cement (kg/m^3)	362	-	-
Class F Fly Ash (kg/m^3)	-	-	71
Coarse Aggregate No. 57 Stone (kg/m^3)	1083	1083	1055
Fine Aggregate Natural Sand (kg/m^3)	310	306	311
Fine Aggregate-Washed Grey Sand (kg/m^3)	482	474	484
Water-reducing Admixture/ Retarder (mL/m^3)	-	471	182
Mid-range Water-reducing admixture (mL/m^3)	471	-	-
Water-cementitious Materials Ratio (w/cm)	0.48	0.49	0.51

3.3.3 Raw Materials

Concrete used in the study was batched and delivered by Thomas Concrete. The coarse aggregate used was a no. 57 graded stone from the Vulcan Materials Norcross, Georgia quarry. The fine aggregate was a blend of natural Atlanta sand and washed grey (WG), a manufactured sand. Both ASTM C 150 (2007) Type I and III portland cement were utilized in this study, as well as a Class F fly ash. Two different chemical admixtures were added to the test mixtures. Sika ViscoCrete 2100 (V-2100), which meets the requirements of ASTM C 494 (2008) Types A and F admixtures, was used in mixture one as a water-reducing admixture. Sika Plastiment, which meets ASTM C 494 (2008) requirements for Type B and D admixtures, was used in mixtures two and three, both as a water-reducing admixture and as a retarding admixture.

3.3.4 Testing Equipment

For ASTM C 403 (2008) testing, a loading apparatus, or penetrometer, and a set of penetration needles were provided by each participating laboratory. 2.3-L (referred to as 5-pound) cylindrical metal ink cans (or slip cover cans) were provided to participants as mortar sample containers for each mixture tested. The containers were 15.2 cm (6 in.) in diameter by 12.7 cm (5 in.) tall. It should be noted that this container does not meet the requirements of Section 6.1 of ASTM C 403 (2008), which states that the height of the container must be at least 15.2 cm. Pictures of the containers can be seen in Figure 3.2.

Figure 3.2: Setting containers used for testing

These containers were used by all groups excluding Groups 1 and 4. One gallon paint cans were used by Group 1 and 3.8-L (1-gal.) plastic paint cans were used by Group 4. These cans were 16.8 cm (6.6 in.) in diameter by 19.1 cm (7.5 in.) tall. In order to investigate the effect of sample size on time of set by ASTM C 403 (2008), additional containers were used by Groups 3, 5, and 10. Group 3 used an additional 4.5-L (referred to as 10-pound) metal ink can (19.1 cm in diameter by 15.6 cm tall) for each mixture, and Groups 5 and 10 used an additional 3.8-L (1-gal.) plastic sealed bucket for each mixture. Temperature of a larger and a smaller mortar sample used for testing was recorded by both Groups 3 and 10. A multi-channel maturity meter that recorded temperature every thirty minutes was used by Group 3, and temperature probes that recorded temperature every 30 minutes were used by Group10 (See Figure 3.3).

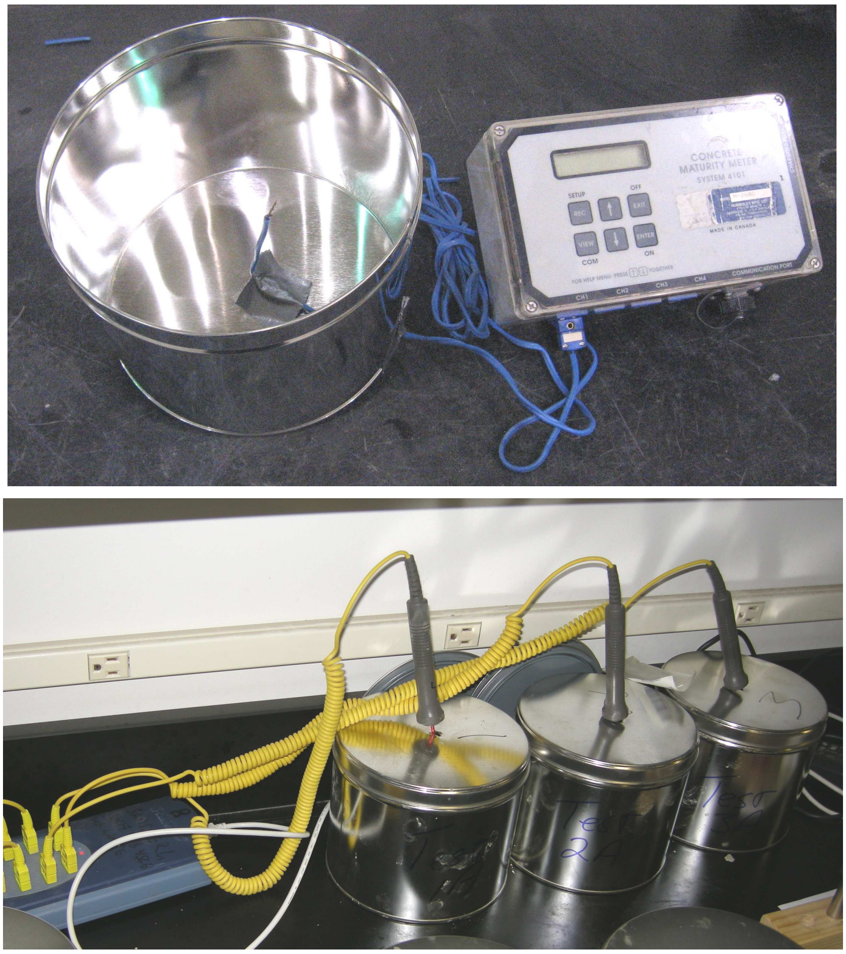


Figure 3.3: Temperature measuring devices for Group 3 (top) and Group 10 (bottom)

In order to evaluate the proposed specification for *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method*, a wide variety of thermal devices were used for testing. For the purpose of analysis and comparison of data, these devices, or insulating chambers, used for the proposed temperature method were grouped into three general categories. The categories are as follows:

I) Extremely-well insulated device with individual closed insulatory cells for specimen.

II) Moderately-insulated single chamber device with multiple open-topped cavities for specimen.

III) Device constructed as per the proposed specification for thermal set time.

A semi-adiabatic calorimeter is defined by RILEM TC 119 TCE 1 (1997) as a calorimeter in which the maximum heat losses are less than 100 J/(h·K). It should be noted that only Category I devices may be classified as semi-adiabatic by this definition. Category II and III devices may not be classified as semi-adiabatic due to the fact that they are open-top units, therefore losses cannot be accurately determined or accounted for. Examples of the temperature histories produced from the three device types can be seen in Figure 3.4. Data markers shown in graphs presented in this thesis are solely for the purpose of distinguishing between data series and do not represent actual data points. Temperature data points collected before samples were placed into their respective devices are left off of all graphs. The time delay in the temperature plot is included in order to account for time between concrete batching and the start of temperature logging.

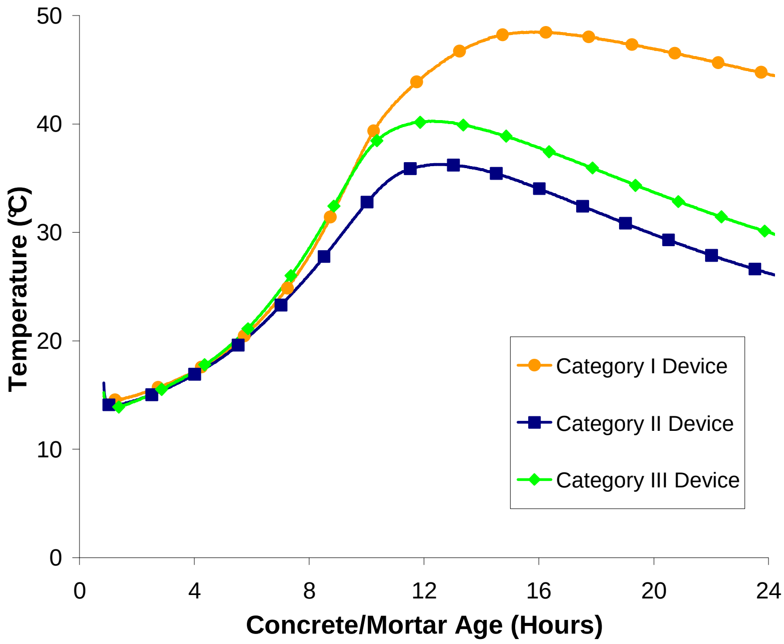
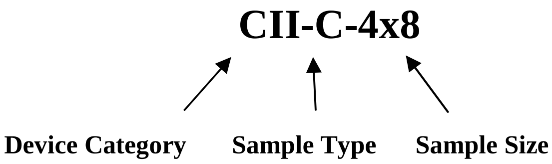


Figure 3.4: Sample temperature profiles for Category I, II and III devices for Mixture 1 (Category I and II device Group 3, Category III device Group 12

The effect of both sample size and sample type on the temperature history of a sample were also evaluated in this study. Samples of both concrete and mortar were tested as well as sample sizes ranging from 7.6-cm (3-in.) diameter by 15.2-cm (6-in.) tall cylinders to 15.2-cm (6-in.) diameter by 15.2-cm (6-in.) tall cylinders. The following notation will be used when presenting and discussing thermal method specimens.

- CI = Category I Device
- CII = Category II Device
- CIII = Category III Device
- C = Concrete Sample

- M = Mortar Sample
- 3 x 6 = 7.6-cm (3-in.) diameter by 15.2-cm (6-in.) tall cylinders
- 4 x 8 = 10.2-cm (4-in.) diameter by 20.3-cm (8-in.) tall cylinders
- 6 x 6 = 15.2-cm (6-in.) diameter by 15.2-cm (6-in.) tall cylinders



It should be noted that many of the specimens and devices used in this study do not meet the proposed ASTM specification for determining time of set by temperature methods, especially with respect to cavity/sample size, amount of insulation, and thermocouple wire type. All devices were included in the analysis to evaluate the impact that various device and sample properties have on the intended test method. For a summary of devices used by each group see Table 3.2. For pictures of all devices used for thermal testing see Appendix B.

Table 3.2: Devices used by each group

Group No.	Device				
	CI-C-4x8	**CII-C-4x8**	**CII-C-3x6**	**CII-M-3x6**	**CIII-C-6x6**
1					
2					
3	x	x	x	x	
4		x			x
5		x	x	x	x
6		x	x	x	x
7		x	x	x	
8		x			x
9	x	x	x	x	x
10		x	x	x	x
11					
12					x

3.4 Thermal Data Analysis

Collected thermal data were analyzed using two previously proposed methods. Sandberg and Liberman (2007) successfully made use of the "Derivatives" Method and the "Fractions" Method for finding time of set from a concrete sample's semi-adiabatic temperature profile. (It should be noted that the devices used by Sandberg and Liberman [2007] cannot be considered semi-adiabatic by the definition provided by RILEM TC 119 – TCE [1997].) In the *Derivatives Method*, final set, (also known as secondary set) was defined as the point of maximum slope of the temperature profile, or the maximum first derivative of the temperature versus concrete age plot. Initial set (also known as *primary* set) was defined as the maximum curvature of the temperature profile, or the maximum second derivative of the temperature versus concrete age plot.

For the *Fractions Method,* set times were defined as a percentage of the total temperature rise of the sample. Although a method was proposed to calculate a "refined fraction" based on the cement type and w/c used for each mixture, default values proposed by Sandberg and Liberman (2007) of 21% for initial, or *primary* set, and 42% for final, or secondary set, were used for analysis in this study. Set times obtained using thermal methods may not necessarily compare directly to the initial and final set times obtained using ASTM C 403 (2008), therefore, all thermal set times will be referred to as "primary set" and "secondary set" for the remainder of this thesis.

Two different methods were used to compute the slope and curvature of the temperature profile for the *Derivatives Method*. The first method was defined in the proposed ASTM specification entitled the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method.* Plots of the first and second derivative were generated by use of a direct slope method using a 200-point (200-minute) smoothing interval. Points of maximum curvature and slope were then located and determined to be *primary set* and *secondary set* respectively. For compatibility with the rest of this thesis, this method will be referred to as the "*Direct-Slope Derivatives Method*" for the remainder of this chapter. Example plots of the first and second derivative of the temperature versus concrete age are shown in Figure 3.5 and Figure 3.6.

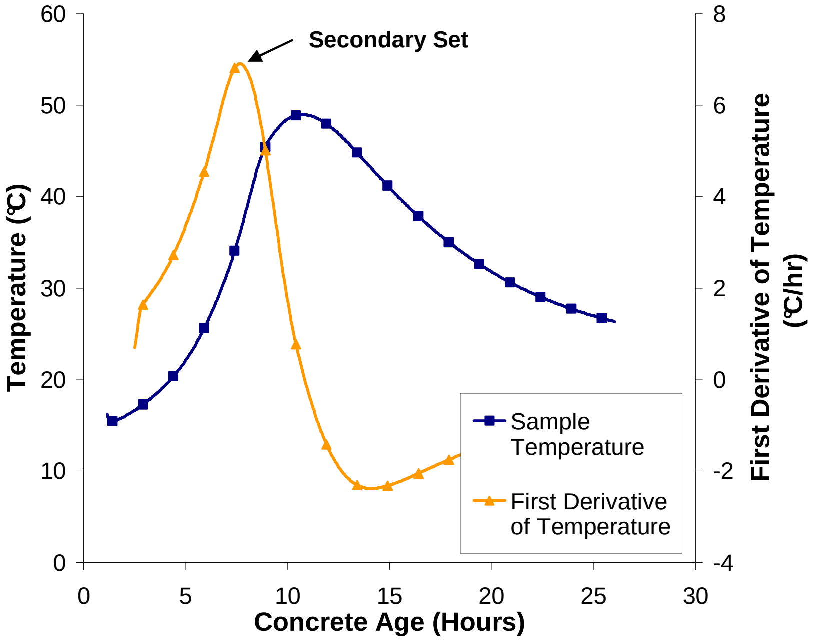


Figure 3.5: Sample temperature and first derivative plots for a given sample (Mixture 1, CII-M-3x6, Group 10)

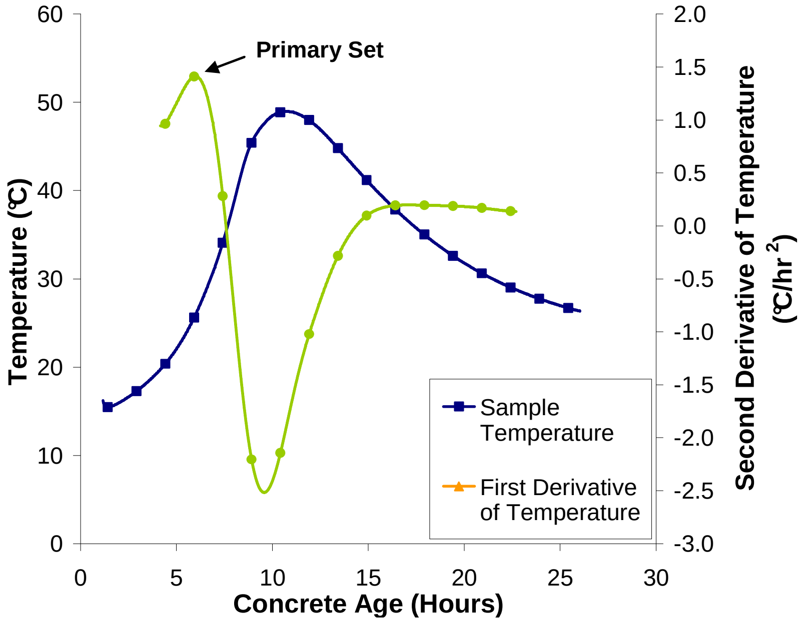


Figure 3.6: Sample temperature and second derivative plots for a given sample (Mixture 1, CII-M-3x6, Group 10)

It should be noted that due to the large smoothing interval, there is no meaningful first derivative data until the 101st minute of testing, and there is no meaningful second derivative data until the 201st minute of testing. These data are therefore left out of all plots and analysis. For mixtures in which *primary set* occurs near or before the 201st minute of testing, the *primary set* times acquired by this method are not reliable. See the second derivative plot in Figure 3.7 for an example of a data set in which the maximum second derivative, or *primary* set, is too early to be accurately determined using the previously described method. The maximum second derivative appears to be at minute 201 (the start of the second derivative plot) even though the curvature, or second derivative, of the temperature plot may have already reached a maximum and started to decrease. For this reason all *primary set* times calculated using the 200 point smoothing interval direct slope method that occur before 5 hours (200 minutes plus the approximate time between batching the concrete and the start of testing) should be considered unobtainable, and were not included in this analysis. Options for avoiding this effect in future research include the use of a shorter logging interval for thermal testing or the use of a shorter smoothing interval for analysis.

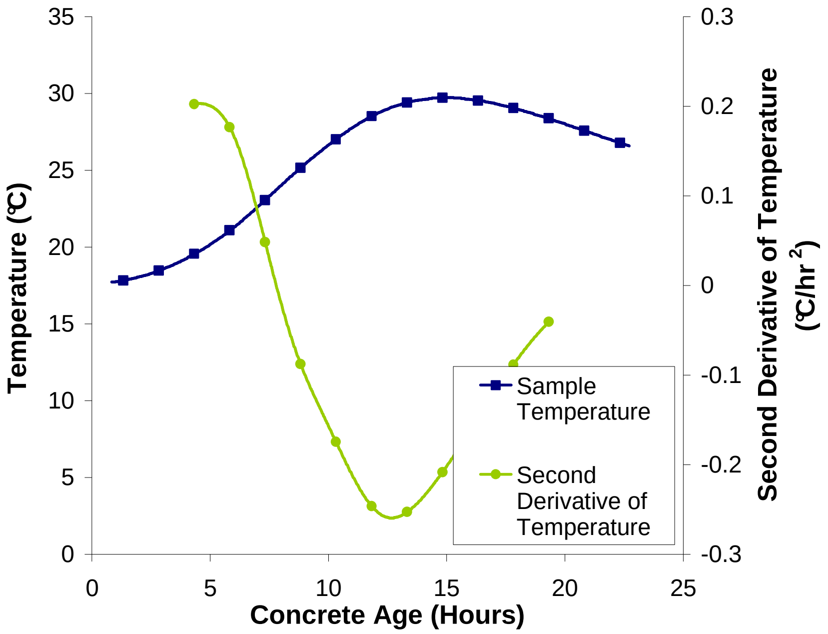


Figure 3.7: Sample temperature and second derivative of temperature

(Mixture 3, CII-C-4x8, Group 7)

Maximum slope and curvature points for the *Derivatives Method* were also obtained by a closed-form solution to a fourth-order polynomial fit to the collected temperature data. Maximum curvature, or *primary* set, was determined as the point at which the third derivative of the temperature polynomial was zero, yielding a maximum second derivative. Likewise, the maximum first derivative, or secondary set, was found by locating the first point at which the second derivative of the temperature polynomial was zero. For compatibility with the rest of this thesis, this method will be referred to as the "*Polynomial Derivatives Method*" for the remainder of this chapter. See Figure 3.8 and Figure 3.9 for example graphs of the collected temperature data points, the best-fit polynomial, and the closed-form first and second derivatives.

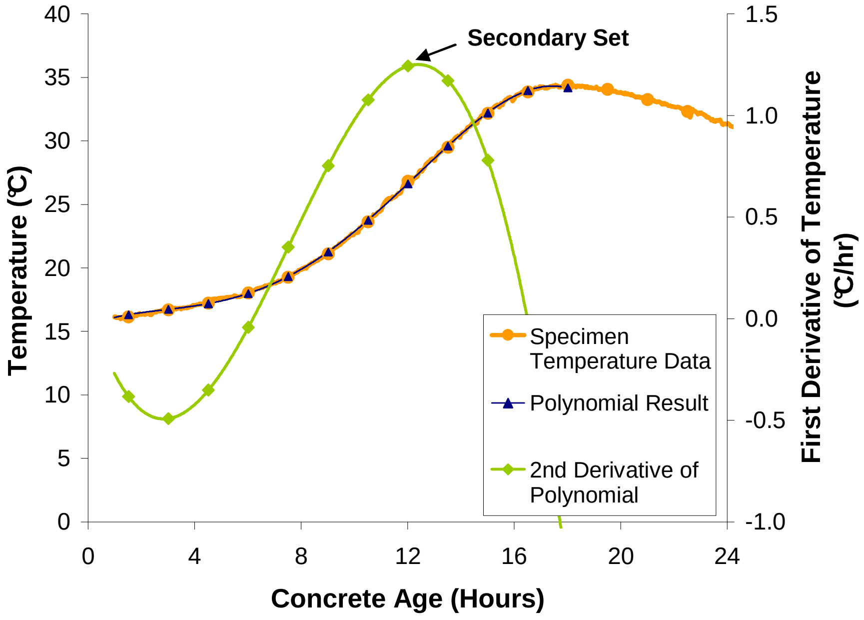


Figure 3.8: Sample temperature, polynomial, and first derivative plots for a given sample (Mixture 2, CIII-C-6x6, Group 5)

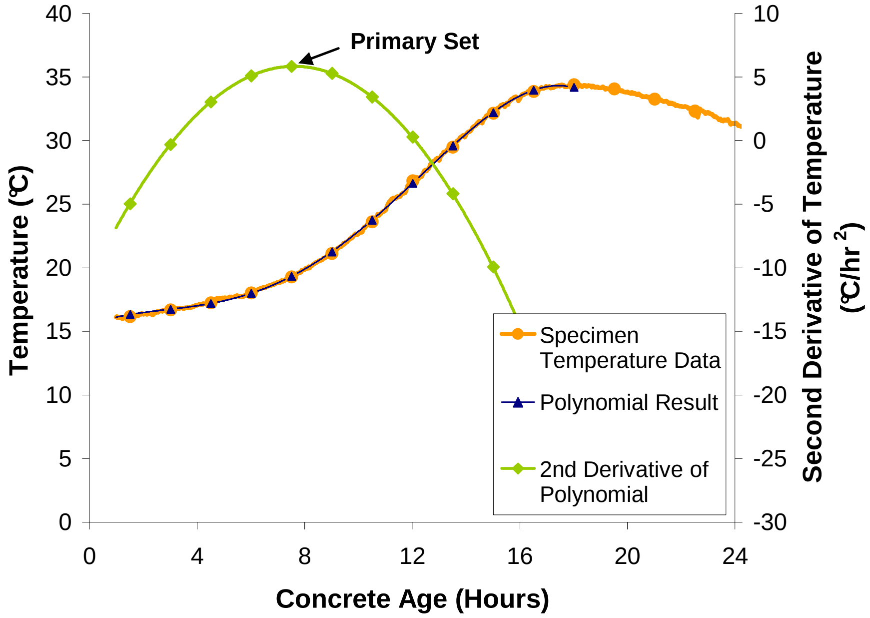


Figure 3.9: Sample temperature, polynomial, and second derivative plots for a given sample (Mixture 2, CIII-C-6x6, Group 5)

Due to the nature of a fourth-order polynomial, the polynomial trend line does not always represent a good fit of the actual temperature data, especially at very early ages. (See Figure 3.10) When this occurs, the maximum curvature of the polynomial does not occur during the setting interval. (In many such cases the maximum closed-form second derivative occurs at a negative time.) Therefore, for the data sets where this is the case, no *primary set* times were determined.

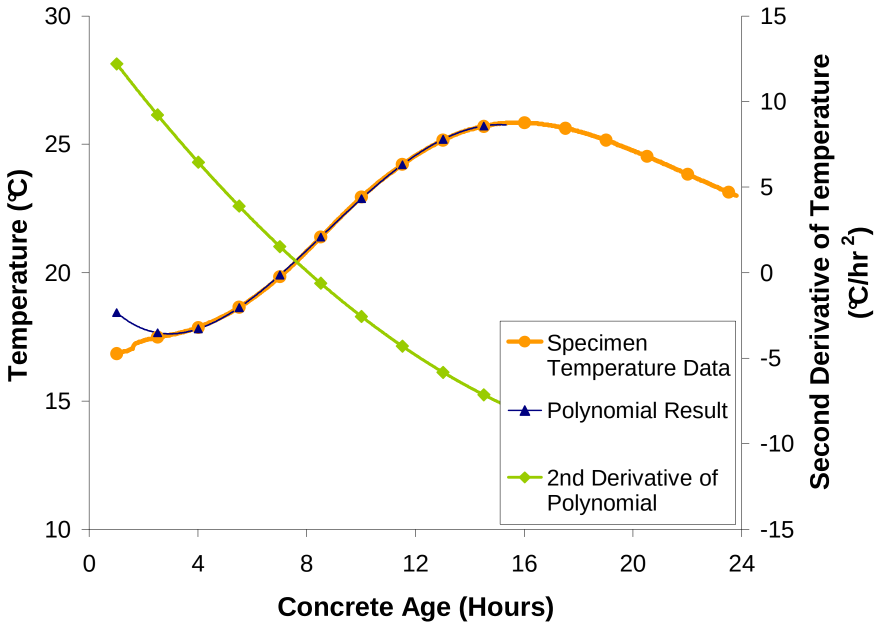


Figure 3.10: Sample temperature, fit polynomial, and derivative plot for a given sample (Mixture 3, CII-C-4x8, Group 10)

Thermal set times were also computed using the *Fractions Method*. As previously discussed, *primary set* for this study was defined as 21% of the total temperature rise of the concrete sample and *secondary set* was defined as 42% of the total temperature rise of the sample. For compatibility with the rest of this thesis, this method will be referred to as the "*Direct Fractions Method*" for the remainder of this chapter. The approach used in this study is illustrated in Figure 3.11. In order to account for equilibrium within the device, the minimum temperature recorded in the first hour of data was used as the "baseline" temperature for each data set. The 21% and 42% fractions could then be calculated as the fraction of the difference in the maximum temperature recorded and the baseline.

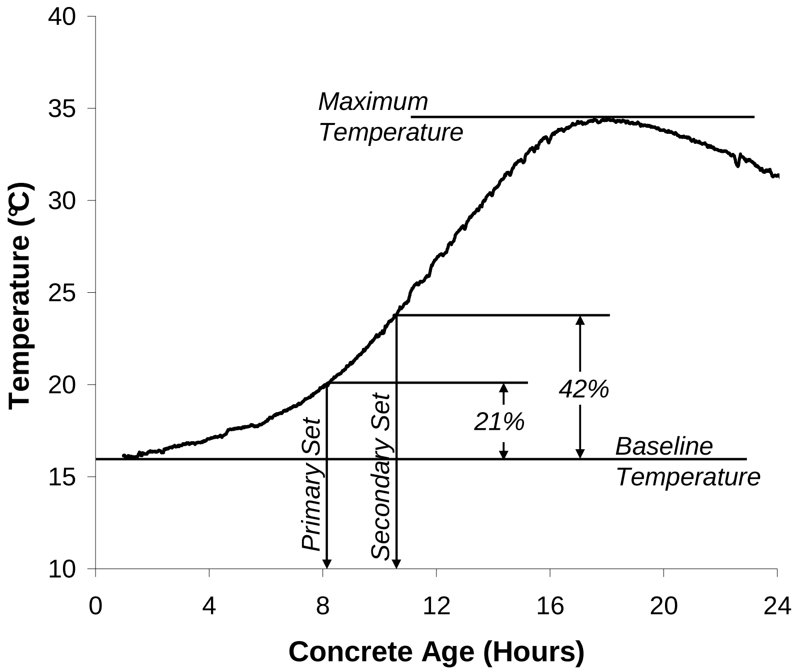


Figure 3.11: *Primary* and *secondary set* by *Direct Fractions Method* (Mixture 2, CIII-C-4x8, Group 5) Adapted from Sandberg and Liberman (2007)

3.5 Results

Results of testing from both ASTM C 403 (2008) and the proposed thermal methods to estimate setting times are presented in this section. A brief summary of the concrete properties for the mixtures used in the study can be seen in Table 3.3.

Table 3.3: Concrete Properties

Concrete Property	Mixture Identification		
	1	**2**	**3**
Batch time	7:24 AM	9:25 AM	10:14 AM
Sample time	8:05 AM	9:50 AM	10:40 AM
Slump (cm)	14.0	13.0	18.0
Unit weight (kg/m^3)	2381	2371	2368
Air content (%)	1.6	1.6	1.6
Mixture temperature at time of sampling (℃)	9	13	15
28-day compressive strength (MPa)	51.1	55.3	59.6

3.5.1 Setting Results by Penetration Resistance

3.5.1.1 Presentation of Results

Results from the ASTM C 403 (2008) testing are presented in this section. Typical penetration resistance versus concrete age plots for all three mixtures are presented in Figure 3.12. Tabulated results from ASTM C 403 (2008) testing for all participants and all mixtures are presented in Table 3.4 and Table 3.5. All setting samples are included in the summary data presented in Table 3.4 and Table 3.5. Results collected from mortar sample containers other than the provided 15.2 cm (6 in.) in diameter by 12.7 cm (5 in.) tall ink can are noted.

Table 3.4: Summary of ASTM C 403 (2008) initial set times

Group Identification	Replicate	Initial Set Times (hours:minutes)		
		Mixture 1	Mixture 2	Mixture 3
1	a[A]	3:52	7:58	5:47
	b[A]	3:54	7:48	5:42
2	a	3:47	7:30	5:52
	b	3:51	7:35	5:45
3	a	3:57	7:42	6:07
	b	3:56	7:40	6:09
	c[B]	4:07	8:13	5:39
4	a[C]	3:52	8:13	6:03
	b[C]	3:59	8:18	6:00
5	a	4:21	8:20	5:54
	b	4:23	8:01	5:55
	c[D]	-	8:11	6:04
6	a	4:01	8:06	5:53
	b	3:59	7:54	5:49
7	a	3:39	7:27	5:42
	b	3:43	7:53	5:38
8	a	3:52	7:47	5:37
	b	3:53	7:46	5:34
9	a	4:03	6:13	6:36
	b	4:04	6:19	6:52
10	a	3:40	7:56	6:01
	b	3:40	7:53	6:02
	c[D]	3:47	7:59	6:07
11	a	3:53	7:23	5:37
12	a	3:53	7:22	5:27
	b	3:53	7:15	5:27

[A] 3.8 L paint can

[B] 10 lb. ink can (20.6 cm x 15.2 cm)

[C] 3.8 L plastic paint can

[D] 7.6 gallon plastic bucket

Table 3.5: Summary of ASTM C 403 (2008) final set times

Group Identification	Replicate	Final Set Times (hours:minutes)		
		Mixture 1	Mixture 2	Mixture 3
1	a[A]	4:59	9:33	7:30
	b[A]	5:01	9:23	7:28
2	a	5:04	9:21	7:32
	b	5:06	9:30	7:33
3	a	5:16	9:36	7:52
	b	5:10	9:08	7:58
	c[B]	5:33	9:48	8:19
4	a[C]	5:14	10:05	7:37
	b[C]	5:19	10:08	7:42
5	a	5:14	10:03	7:38
	b	5:20	12:08	7:39
	c[D]	-	7:51	7:44
6	a	5:07	9:45	7:42
	b	5:03	9:33	7:45
7	a	5:09	9:23	7:32
	b	5:05	9:41	7:45
8	a	4:59	9:32	7:19
	b	5:00	9:30	7:22
9	a	7:21	8:03	8:20
	b	7:24	8:23	8:27
10	a	4:58	9:40	8:04
	b	4:58	9:36	7:52
	c[D]	5:06	9:40	7:53
11	a	5:09	9:32	7:24
12	a	5:09	9:14	7:37
	b	5:11	9:16	7:30

[A] 3.8 L paint can

[B] 10 lb. ink can (20.6 cm x 15.2 cm)

[C] 3.8 L plastic paint can

[D] 7.6 gallon plastic bucket

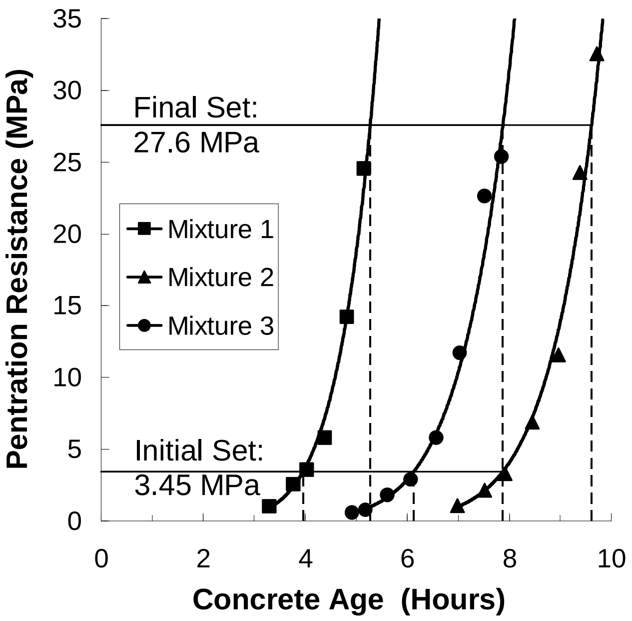


Figure 3.12: ASTM C 403 (2008) penetration resistance versus concrete age for Mixtures 1, 2, and 3 (Group 3)

3.5.1.2 Precision Statement for ASTM C 403

The current precision statement for ASTM C 403 (2008) was based on data obtained from an ASTM round robin study similar to that described in the preceding sections. The concrete examined had setting times that ranged from an average of 169 to 252 minutes for initial set and an average of 240 to 341 minutes for final set. Precision was defined by an acceptable difference, in minutes, between results. Due to the fact that concrete setting may occur much later than in the mixtures examined, it was determined that the precision of mixtures with longer setting times should be examined

A precision statement was prepared for ASTM C 403 (2008) for the setting range observed in this study. ASTM C 403 (2008) contains two sampling procedures, one to prove compliance of a material with performance requirements, and one for all other testing. For proving compliance of a material, three mortar samples are sieved from three separate concrete batches and tested independently, and for all other testing, three samples are sieved from the same batch and tested in triplicate. Due to the fact that multiple batches were not produced for any of the mixtures evaluated in this round robin study, only the precision of the latter of these procedures was able to be determined. This statement, similar to the current statement in ASTM C 403 (2008), should only apply to multiple specimens obtained from a single concrete batch.

All setting results were collected and analyzed as per ASTM C 802 (1996) and a precision statement was proposed as per ASTM C 670 (2003). It should be noted that data from Group 5 and Group 9 was not used in the precision analysis due to questionable setting results. The results from Group 5 were eliminated due to questionable penetration readings and higher than typical variability. The final penetration for 6 out of 8 total samples tested by Group 5 were recorded as exactly 27.6 MPa, which is extremely unlikely, and the regression correlation coefficient for the exponential function fit to penetration data as per ASTM C 403 (2008) for some samples was less than the required 0.98. Also, at least one time of set from Group 5 was determined to be an extreme outlier (differed from the mean of all readings by more than three standard deviations). The results from Group 9 were eliminated due to a noticeably different pattern of change from mixture to mixture as compared to the other participating groups when the average values from each laboratory were plotted (see ASTM C 802 1996 Section 8.2.3). There was also at least one time of set from Group 9 determined to be an extreme outlier.

For analysis it was assumed that the coefficient of variation of the acquired setting times was approximately constant for the setting range tested. Results from the precision analysis for both initial and final set are summarized in Table 3.6 and Table 3.7. The single-operator and multi-operator precision reported only applies to samples taken from a single batch of concrete. The bias of the test cannot be determined because setting time is defined only in terms of this testing method.

Table 3.6: Single-operator precision for initial and final set

Single-Operator Precision

Time of Setting	Single-Operator Coefficient of Variation %[A]	Acceptable Difference Between Two Results, %[A]	Acceptable Range of Three results, %[B]
Initial	1.7	4.8	5.6
Final	1.4	3.9	4.6

[A] These numbers represent, respectively the (1s%) and (d2s%) limits as described in Practice C 670

[B] Calculated as described in the section "*acceptable range of more than two results*" of practice C 670

Table 3.7: Multi-operator precision for initial and final set

Multi-Operator Precision

Time of Setting	Multi-Operator Coefficient of Variation %[A]	Acceptable Difference Between Two Results, %[A]	Acceptable Difference Between Average of Two Results, %[B]
Initial	3.8	10.7	6.2
Final	2.6	7.4	4.2

[A] These numbers represent, respectively the (1s%) and (d2s%) limits as described in Practice C 670

[B] Calculated as described in the section "*multilaboratory precision expressed as a maximum allowable difference between two averages*" of practice C 670

The precision of ASTM C 403, as presented above, is in reasonable agreement with that reported in ASTM C 403 (2008). Although precision values in ASTM C 403

(2008) are presented as an acceptable difference in minutes, when expressed as a percentage of the average setting times, results for the mixtures evaluated are generally within 10 percent of those calculated above.

3.5.2 Thermal Methods to Estimate Setting

Results from temperature methods testing are summarized below. As each mixture had different mixture proportions as well as target set times, the temperature profile varied, as expected, between mixtures. Typical temperature profiles for Mixtures 1, 2, and 3 are presented in Figure 3.13.

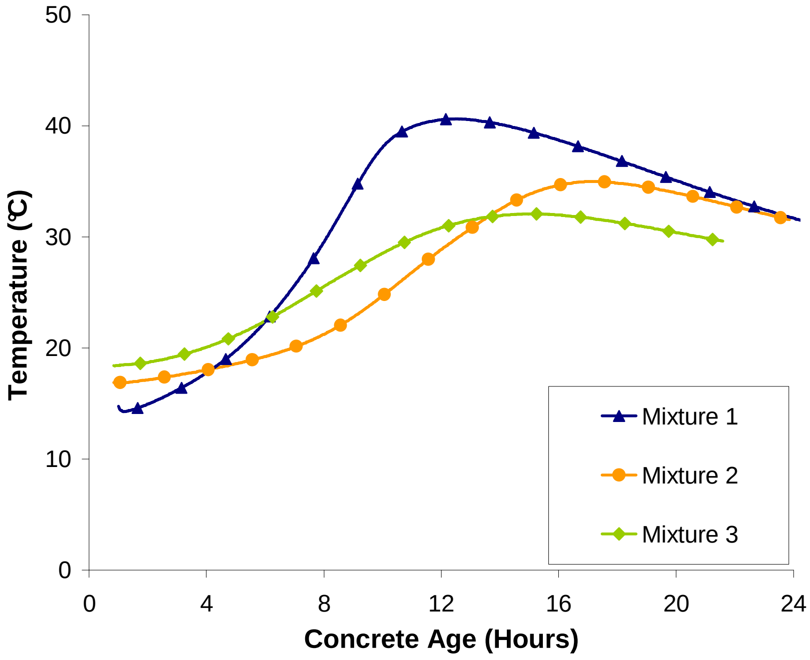


Figure 3.13: Sample temperatures for Mixtures 1, 2, and 3

(CII-C-4x8, Group 4)

Primary and *secondary set* times obtained using thermal methods are summarized in Table 3.8, Table 3.9, and Table 3.10. These tables show average *primary* and *secondary set* values for each device and sample type for all three analysis methods (*Direct-Slope Derivatives Method, Polynomial Derivatives Method,* and the *Direct Fractions Method*). For reasons previously discussed, some *primary set* values were not obtainable using the *Polynomial Derivatives Method*, therefore some values are not reported.

Table 3.8: Average *primary* and *secondary set* times for *Direct-Slope Derivatives Method* by device and sample type

Device & Sample Type	Set Times (hours:minutes)					
	Mixture 1		Mixture 2		Mixture 3	
	Primary	Secondary	Primary	Secondary	Primary	Secondary
I-C-4x8	7:07	8:59	9:01	13:55	5:07	10:15
II-C-4x8	6:02	8:37	8:12	11:01	4:20	7:40
II-C-3x6	4:36	7:56	7:32	9:55	4:22	7:11
II-M-3x6	5:50	7:29	8:31	11:36	5:04	7:55
III-C-6x6	6:16	8:39	8:10	11:25	4:49	7:41

Table 3.9: Average *primary* and *secondary set* Times for *Polynomial Derivatives Method* by device and sample type

Device & Sample Type	Set Times (hours:minutes)					
	Mixture 1		Mixture 2		Mixture 3	
	Primary	Secondary	Primary	Secondary	Primary	Secondary
I-C-4x8	3:50	9:11	7:44	13:58	-	9:40
II-C-4x8	5:24	8:22	7:23	11:51	-	7:58
II-C-3x6	4:57	7:40	6:45	10:42	-	7:02
II-M-3x6	5:11	7:29	7:46	11:39	3:49	8:19
III-C-6x6	5:22	8:15	7:32	11:59	-	7:54

Table 3.10: Average *primary* and *secondary set* times for *Direct Fractions Method* by device and sample type

Device & Sample Type	Set Times (hours:minutes)					
	Mixture 1		Mixture 2		Mixture 3	
	Primary	Secondary	Primary	Secondary	Primary	Secondary
I-C-4x8	6:07	8:06	9:31	12:01	6:17	8:44
II-C-4x8	5:07	7:00	7:37	10:01	5:03	7:06
II-C-3x6	4:24	6:14	6:16	8:49	4:30	6:28
II-M-3x6	5:01	6:34	7:45	9:59	5:07	7:01
III-C-6x6	5:11	7:07	7:39	10:06	5:00	7:03

3.6 Discussion of Results

3.6.1 Setting by Penetration Resistance

3.6.1.1 Precision Statement for ASTM C 403

One objective of this study was to investigate the variability in the standard test method for concrete setting ASTM C 403 (2008). Section 7 of ASTM C 403 (2008) contains requirements for two separate sampling procedures, one in which three mortar samples are sieved from three separate concrete batches, and one in which three samples are sieved from the same batch and tested in triplicate. Therefore, if possible, precision statements for both procedures should be included in the specification. ASTM C 403 (2005) contains a precision statement based on three separate batches of concrete on three separate days. ASTM C 403 (2008) contains only a statement for samples sieved from the same batch and tested in triplicate for concrete with relatively early setting times (final set ranging from 240-341 minutes). Using the data from this study, an additional statement, similar to the statement made in the 2008 version of the specification can be made that is inclusive of concrete with later set times (final set ranging from 307-574 minutes). This statement should be as follows:

Single-Operator Precision- The single-operator coefficients of variation of a single test result are shown in Table 3.11^{A}**.** The results of two properly-conducted tests by the same operator are not expected to differ by more than the percent difference shown in the third column of Table 3.11 ^{A}. The test method calls for reporting three test results. The range (difference between highest and lowest) of the three test results obtained by the same operator on specimens from the same batch of concrete are not expected to exceed the percent difference shown in the fourth column of Table 3.11 ^{B}.

Multi-Operator Precision- The multi-operator coefficients of variation of a single test result are shown in Table 3.12^{A}. The results of two properly-conducted tests by different operators on the same material are not expected to differ by more than the values shown in the third column of Table 3.12 ^{A}. The averages of three test results by two different operators obtained on specimens from a single batch of the same concrete are not expected to differ by more than the values in the fourth column of Table 3.12 ^{B}.

Due to the fact that the precision values calculated in this study are in reasonable agreement with those currently in ASTM C 403 (2008) (when all are converted to a percent difference), it is suggested that a single precision statement should be made for the full range of testing based on the coefficient of variation (as opposed to the standard deviation) of the combination of data from both ASTM round robin studies.

Table 3.11: Single-operator precision for initial and final set

Single-Operator Precision

Time of Setting	Single-Operator Coefficient of Variation %[A]	Acceptable Difference Between Two Results, %[A]	Acceptable Range of Three results, %[B]
Initial	1.7	4.8	5.6
Final	1.4	3.9	4.6

[A] These numbers represent, respectively the (1s%) and (d2s%) limits as described in Practice C 670

[B] Calculated as described in the section "*acceptable range of more than two results*" of practice C 670

Table 3.12: Multi-operator precision for initial and final set

Multi-Operator Precision

Time of Setting	Multi-Operator Coefficient of Variation %[A]	Acceptable Difference Between Two Results, %[A]	Acceptable Difference Between Average of Two Results, %[B]
Initial	3.8	10.7	6.2
Final	2.6	7.4	4.2

[A] These numbers represent, respectively the (1s%) and (d2s%) limits as described in Practice C 670

[B] Calculated as described in the section "*multilaboratory precision expressed as a maximum allowable difference between two averages*" of practice C 670

3.6.1.2 Effect of Sample Size on ASTM C 403 (2008) Results

Variations in the test method permitted in ASTM C 403 (2008) include variations in sample size, sample container type, sample temperature, sieving method, and penetration device. Due to logistical limitations, not all variables that may affect the precision of this test method were assessed by this round robin study. However, since a large majority of the participating groups used a mortar container that is smaller than the specification allows, the effects of sample size on setting time by ASTM C 403 (2008) were briefly examined.

Effects due to temperature have been shown to have a significant effect on concrete properties, including setting times (see Section 2.3.3). As can be seen in Figure 3.14, sample size has a measurable impact on the sample temperature history. Although a larger sample should theoretically generate more heat as it begins to hydrate, the larger setting sample (4440 cm^3) had an overall cooler temperature profile than the smaller sample (2310 cm^3) for all three mixtures. Although this seems counterintuitive, the mortar used in testing was approximately 11 °C cooler than the testing environment at the onset of testing. In the process of reaching equilibrium within the laboratory, a smaller sample gains heat more quickly from its surroundings. This equilibrium effect could explain the elevated temperature of the smaller sample.

Although a temperature difference was observed between samples, the resulting differences in setting time were not statistically significant to a 95% confidence level (p-value for a t-test of 0.087). Therefore, sample size does not have a significant impact on setting times obtained, for the sample sizes and conditions tested; however, further study relating to the effects of sample size on setting times obtained as per ASTM C 403 (2008) is recommended.

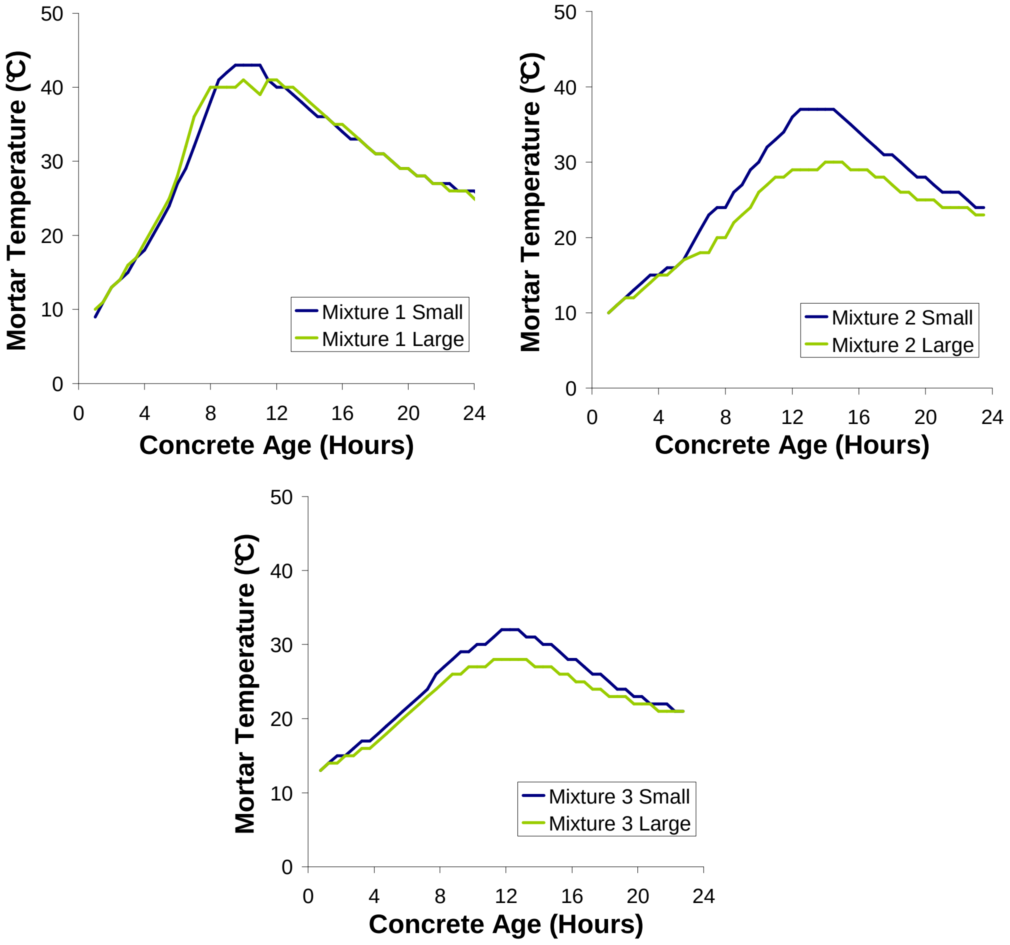


Figure 3.14: Mortar sample temperature profiles for Mixtures 1, 2, and 3 (Group 3)

3.6.2 Thermal Methods to Estimate Setting

The proposed ASTM specification on the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method* proposed the use of a minimally insulated thermal testing device and a *Direct-Slope Derivatives Method* to predict the setting times of concrete. In order to investigate this proposed ASTM specification and define the variability of the test method, the sensitivity of the method to various parameters was briefly investigated. The effects of sample size, sample type, device insulatory properties, and analysis type were examined in the study and results are discussed below.

3.6.2.1 Sensitivity Study

The proposed method for determining concrete setting by thermal testing is difficult to evaluate due to the sensitivity of the specification to certain aspects of testing. For comparison purposes, all setting times evaluated in this sensitivity study were calculated using the *Direct-Slope Derivatives Method* outlined in the proposed specification and the *Direct Fractions Method* discussed previously. A paired t-test was performed on the setting times for each of the variables evaluated. When testing was completed in replicate, values were averaged before analysis. It should be noted that all questionable results due to inconsistencies within the test method were not included in analysis (e.g. *primary setting* points calculated using *Derivatives Method* for mixtures with *primary set* occurring before the 200th minute of testing). The p-values reported represent the probability that the data sets are statistically similar (i.e. the probability that the mean of the difference between paired values is zero). In general, p-values for a t-test that are less than .05 are determined to be significantly different to a confidence level of 95%.

As previously mentioned, sample size can have a significant impact on the temperature profile generated by a sample, which may have an effect on the setting times obtained by thermal methods. The differences in thermal setting times obtained using varying sample size was evaluated by the use of similar Category II devices constructed to hold 10.2-cm (4-in.) diameter by 20.3-cm (8-in.) tall cylinders and 7.6-cm (3-in.)

diameter by 15.2-cm (6-in.) tall cylinders. It should be noted that although the devices were of similar construction, the amount of insulation provided in each was not the same, which could have an impact on the results of this comparison. Examples of devices used for each case can be seen in Figure 3.15.

Figure 3.15: Category II devices for 3x6 and 4x8 cylinders

As can be seen in Figure 3.16, a smaller sample size may yield a higher or lower temperature than a larger sample at a given time depending on the surrounding air temperature and the rate of hydration (represented by the first derivative of temperature). If surrounding temperatures are warmer than a sample (as was the case in this study), a smaller sample will gain heat more quickly than a larger sample, yielding a higher temperature at early ages. As the sample gains heat due to the hydration reaction, the larger sample will gain heat at a faster rate than the smaller sample due to the total amount of cement in the container, yielding a higher final temperature than the smaller sample. The first derivative of temperature, calculated using the direct slope method, is plotted in Figure 3.16 along with temperature for comparison purposes. The maximum first derivative points, which represent *secondary set* obtained by use of the *Direct-Slope Derivatives Method*, differ by as much as 71 minutes in the above example. The paired t-test resulted in a p-value of 5.77 x 10^{-12} for the *Direct-Slope Derivatives Method* and 1.18 x 10^{-12} for the *Direct Fractions Method*; therefore, sample size has a very significant effect on the setting times obtained from thermal data.

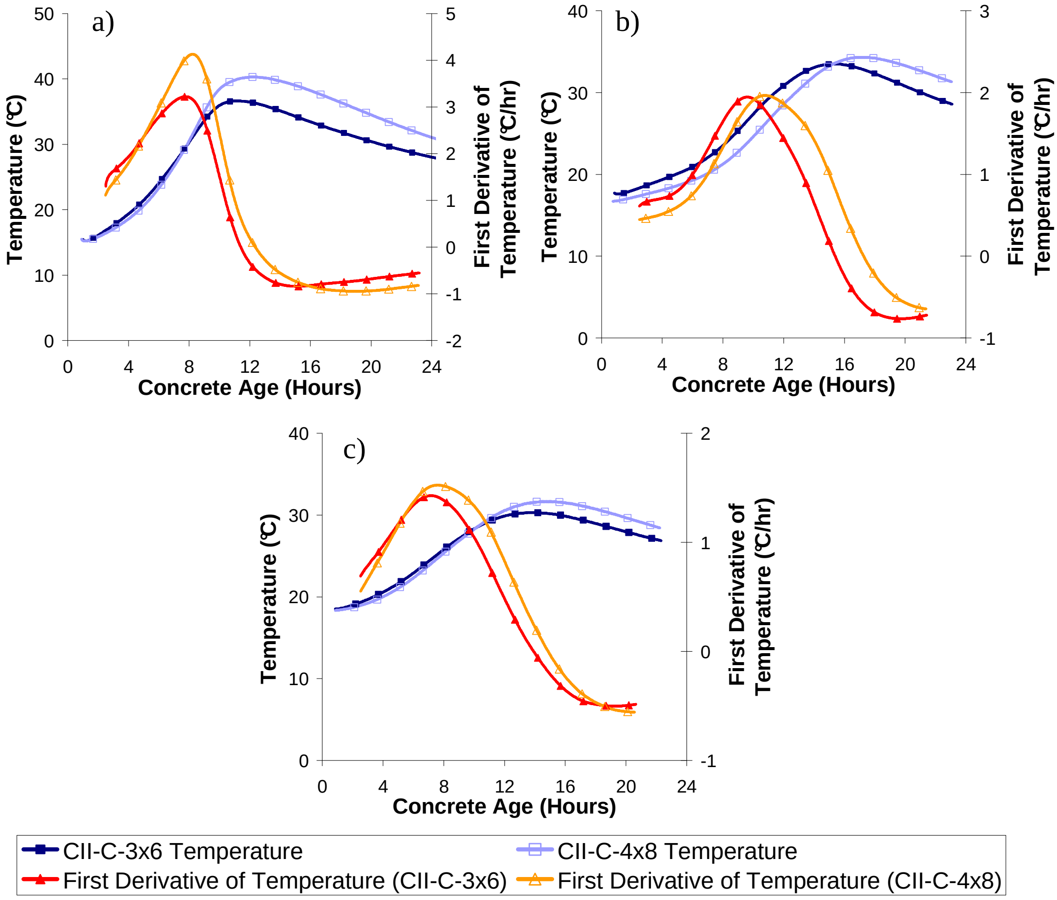


Figure 3.16: Temperature profiles for varying sample sizes for a) Mixture 1, b) Mixture 2 and c) Mixture 3 (Group 9)

Another variable thought to have a significant impact on the temperatures generated by a sample is whether a concrete sample or a sieved-mortar sample is used for testing. The effect of sample type on thermal setting times was analyzed in a similar fashion as sample size. Mortar and concrete were both tested in the same Category II device (3x6 sample size) used for sample size comparisons and the resulting *secondary setting* times were compared. As can be seen in Figure 3.17, the mortar sample generated a total temperature rise 40-70% higher than the temperature rise of the concrete sample, yielding *secondary setting* points that differ by as much as 150 minutes. The p-value for the paired t-test conducted was 3.16×10^{-4} for the *Direct-Slope Derivatives Method* and 4.36×10^{-12} for the *Direct Fractions Method*; therefore, sample type has a significant effect on the setting times obtained by thermal methods.

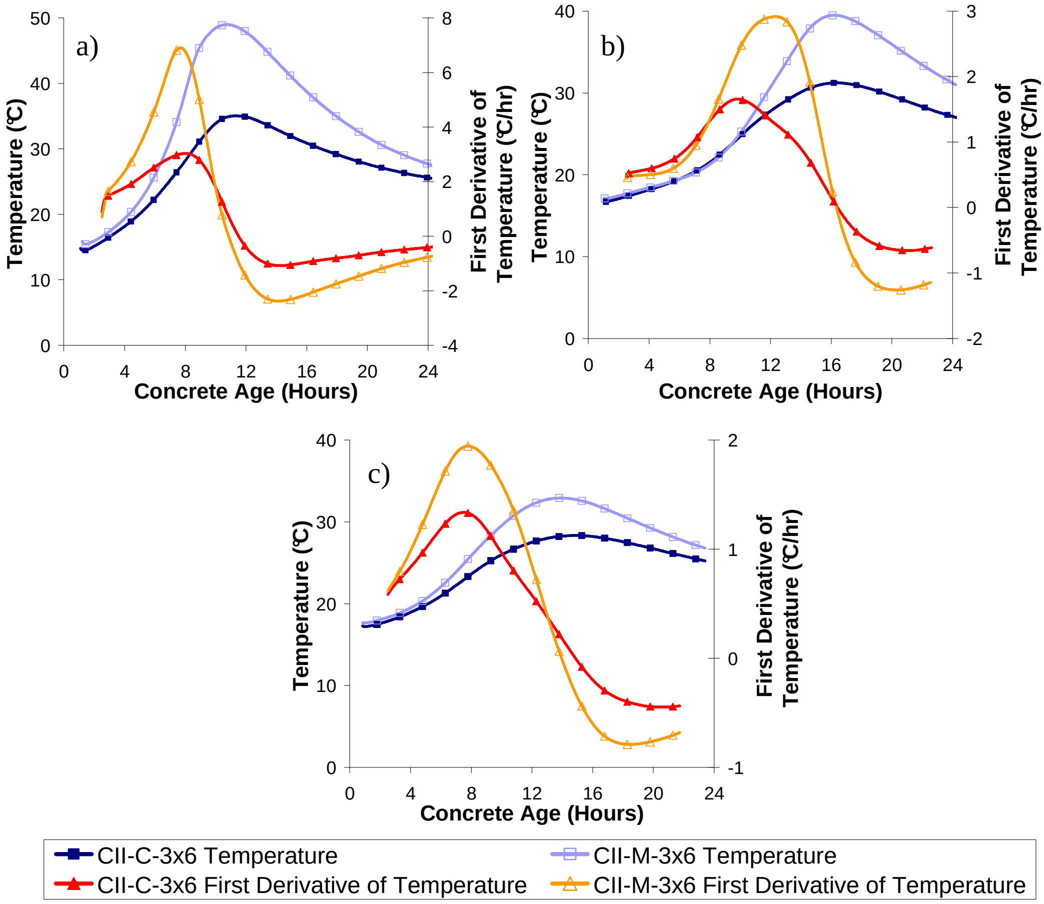


Figure 3.17: Temperature profiles for concrete and mortar samples for a) Mixture 1, b) Mixture 2 and c) Mixture 3 (Group 10)

To compare devices with different insulatory properties, both sample type and sample size must be held constant. Therefore, in order to get an accurate comparison, data from a Category I and Category II device were analyzed using a 4-inch diameter by 8-inch high concrete sample (CI-C-4x8 and CII-C-4x8) as shown in Figure 3.18. The device outlined in the proposed specification for thermal setting (Category III device) was not used in this analysis due to the variability among devices and the fact that there was no other device with a similar sample size for comparison.

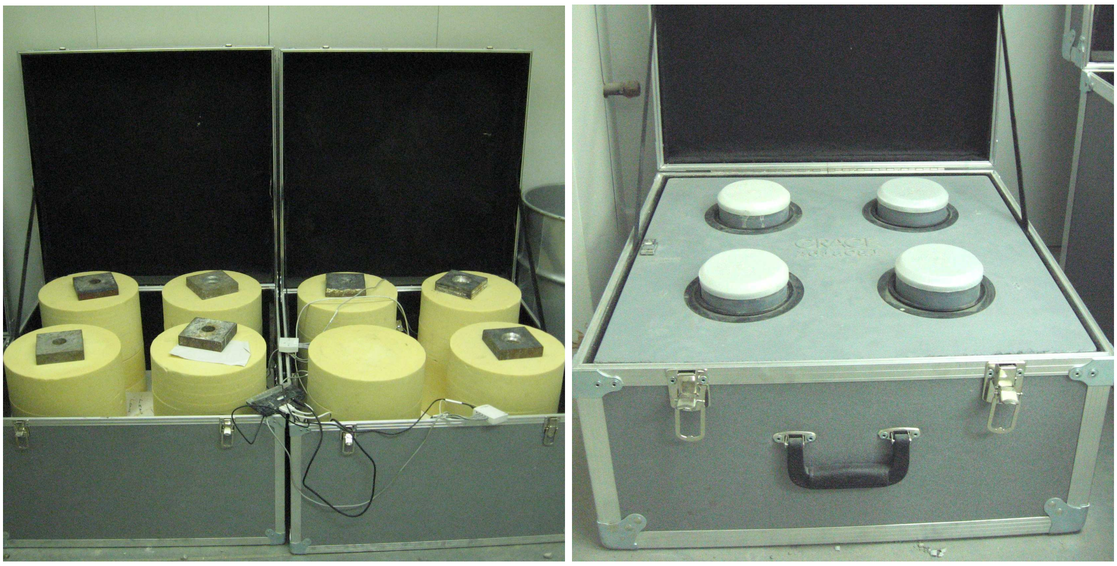


Figure 3.18: Category I and Category II devices with 4 x 8 concrete samples

The insulatory properties of a device have a definite impact on the temperature profile of the sample as can be seen in Figure 3.19. Though the temperatures were similar during the setting window, the slope of the temperature profiles during this interval and the maximum temperatures recorded were significantly different. *Secondary set* times for the *Direct-Slope Derivatives Method* differed by as much as 25% and *secondary setting* times for the *Direct Fractions Method* differed by as much as 20%. When a paired t-test was performed on the setting data, the p-values obtained were 3.50×10^{-4} for the *Direct-Slope Derivatives Method* and 2.09×10^{-7} for the *Direct Fractions Method*. In light of these findings, device insulatory properties have a significant effect on thermal setting times.

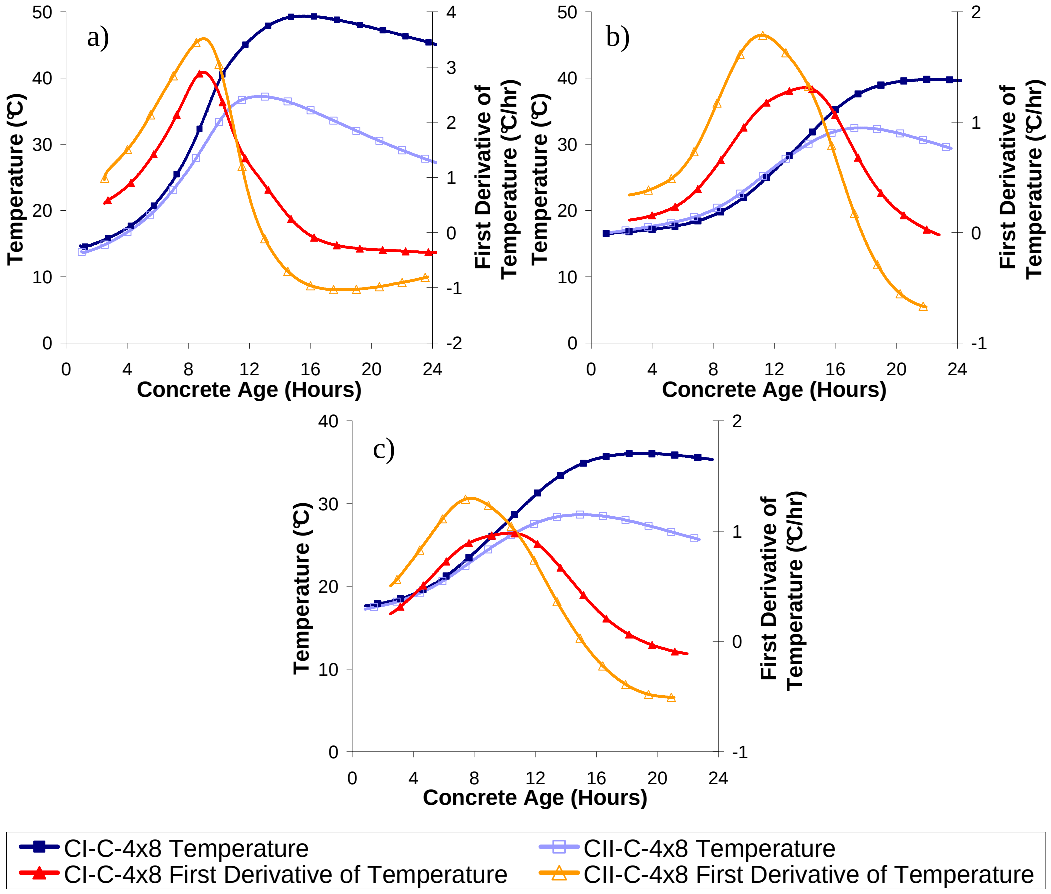


Figure 3.19: Temperature profiles for Category I and II devices for a) Mixture 1, b) Mixture 2 and c) Mixture 3 (Group3)

It should be noted that interference due to ambient temperatures were minimized in this study by performing all tests under room conditions. Effects due to device insulation may be much greater when the surrounding temperature differs substantially from the fresh concrete temperature (as is the case with hot and cold weather concreting). For very well insulated devices (i.e. Category I device), it is possible to approximate the losses due to this thermal gradient to obtain a fully-adiabatic temperature profile. In order to obtain consistent, repeatable results it is recommended that further study be conducted to investigate thermal set methods applied to fully-adiabatic temperature profiles, as opposed to profiles from poorly-insulated devices.

3.6.2.2 Analysis Methods

Analysis procedure can also have an effect on the thermal set times obtained from a sample's temperature history. As previously mentioned, both the *Derivatives Method* and the *Fractions Method* were examined in this study. For the *Derivatives Method*, the data were analyzed using two separate methods of obtaining the first and second derivative. An can be seen in Figure 3.20 the direct slope method and the fourth-order polynomial methods of computing the maximum derivatives of the temperature profile were generally within 20% for all *secondary set* values. The *primary set* values did not compare as well, especially at early setting times. It should also be noted that device category and sample type had a significant impact on the *primary setting* comparison. The few *primary setting* points that exceeded 40% error between methods were either CI-C-4x8 or CII-M-3x6 samples.

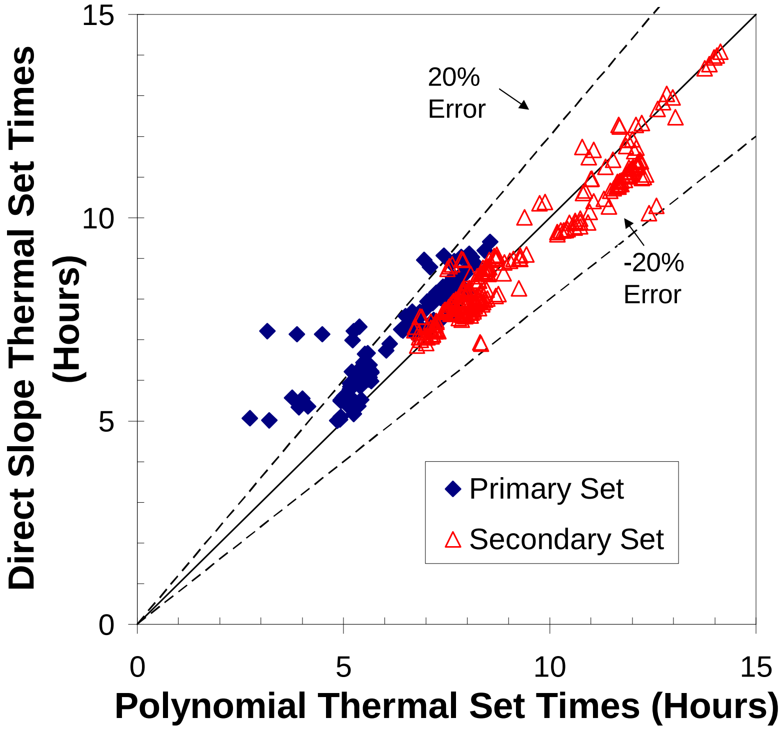


Figure 3.20: *Direct-Slope Derivative Method* set times versus *Polynomial Derivative Method* set times

The *Direct Fractions Method* compared similarly to both methods of computation for the *Derivatives Method* as can be seen in Figure 3.21. *Secondary setting* times calculated using the *Direct Fractions Method* were generally 20% lower than those calculated using either version of the *Derivatives Method*. A majority of the *primary setting* times compared fall within the ±20% error range, but had a much larger scatter than the *secondary setting* data.

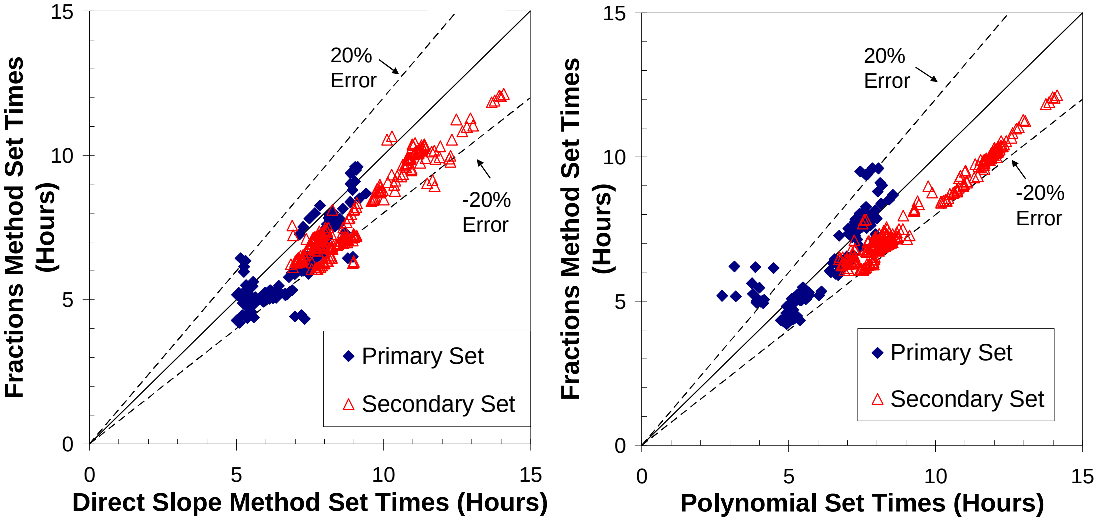


Figure 3.21: *Direct Fractions Method* versus Derivative Method for the *Direct-Slope Derivatives Method* (left) and the *Polynomial Derivatives Method* (right)

One possible reason for the higher variability for *primary set* values is computational weaknesses inherent in all three methods when determining setting times that occur at early ages. As mentioned previously, both methods of calculating setting by the *Derivatives Method* may yield inconsistent results at early setting times due to the large smoothing interval of the direct slope method and the low order of the best-fit polynomial. The *Direct Fractions Method* is also less consistent for early setting times due to the relatively low temperature rise occurring at *primary* set. Due to the shape of the temperature profile, the *Direct Fractions Method* is much more sensitive to a small variation in either the baseline temperature or maximum temperature used for analysis at the 21% default fraction used to define *primary set* than at the higher percentage corresponding to secondary set. This is even more pronounced in mixtures in which there is less of a total temperature rise (e.g. Mixture 3). Therefore, a one to two degree variation in baseline temperature caused by the sample reaching equilibrium within the device can have a significant impact on the *primary setting* times obtained using the *Direct Fractions Method*. This effect paired with the weaknesses of the both versions of the Derivative Method calculations could explain the larger variation among *primary set* results than that of *secondary set* results.

3.6.2.3 Comparison of Setting Results from ASTM C 403 and Thermal Methods

The 3.45 MPa (500 psi) and 27.6 MPa (4000 psi) penetration resistance defined as initial and final set, respectively, by ASTM C 403 (2008) have been described as somewhat arbitrary. It is debated whether they actually represent the vibration limit and complete time of set of a given sample as intended and whether the mortar sample tested is representative of the in-place concrete (Dodson 1994). Similarly, concrete or mortar used for thermal testing may not accurately represent the in-place concrete or the sieved-mortar used in ASTM C 403 (2008). As can be seen in Figure 3.22, a mortar sample may undergo a significantly different temperature history than a concrete or mortar specimen in a thermal device, and will most certainly undergo different conditions than in-place concrete. With this in mind, many would argue that setting times obtained by use of thermal methods should not be compared to ASTM C 403 (2008), but be used as a separate indicator of setting.

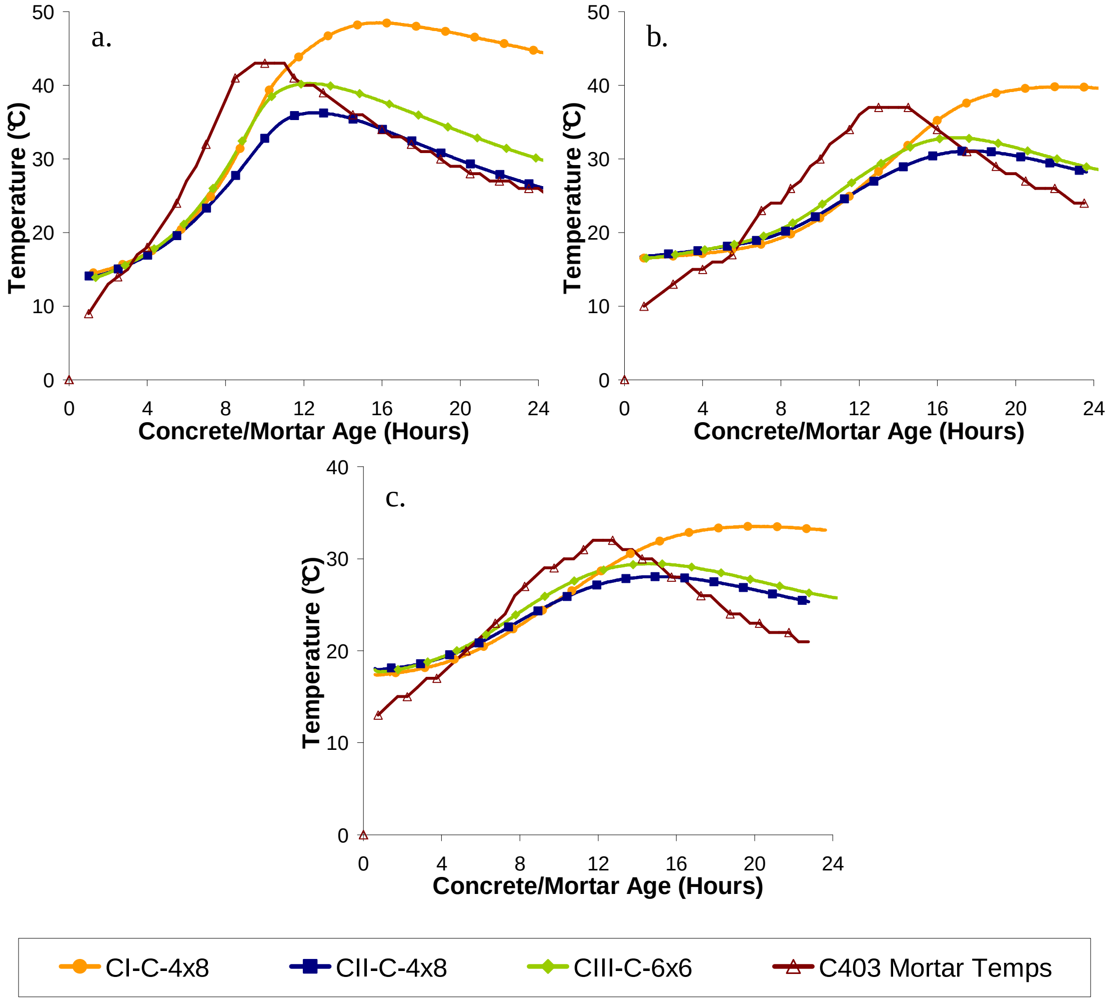


Figure 3.22: Temperature profiles for the three device categories and a (small) ASTM C 403 (2008) mortar sample for a) Mixture 1, b) Mixture 2, and c) Mixture 3

Although ASTM C 403 (2008) may not necessarily represent the exact time of set for in-place concrete, it has been used for many years as an indicator of the time of set of a specific mixture of concrete under controlled conditions. These results have been useful as a means of comparison between different mixture proportions, cement properties, material types, and environmental conditions. In order for a thermal set method to be used for a similar purpose, thermal setting times that represent the hardening characteristics of concrete must be able to be consistently measured. Setting times obtained by the thermal methods discussed above were compared to ASTM C 403 (2008) values in order to determine whether they can be used as a reliable indicator of concrete setting.

In order to be considered a reliable indicator of setting, it was determined that setting times obtained through thermal methods must meet one of three criteria. These criteria are as follows:

a) The accuracy of setting times obtained by thermal methods as compared to those obtained through ASTM C 403 (2008) lies within the variability inherent to ASTM C 403 (2008) (see Figure 3.23a),

b) The accuracy of setting times obtained by thermal methods as compared to those obtained through ASTM C 403 (2008) lies outside of the variability of ASTM C 403 (2008) but within limits that are deemed acceptable for the determination of concrete setting times (see Figure 3.23b),

c) The accuracy of setting times obtained by thermal methods as compared to those obtained thought ASTM C 403 (2008) lies outside of the limits that are deemed acceptable for the determination of concrete setting times, yet is consistent such that setting times obtained by thermal methods may be considered a separate indicator of concrete setting (see Figure 3.23c).

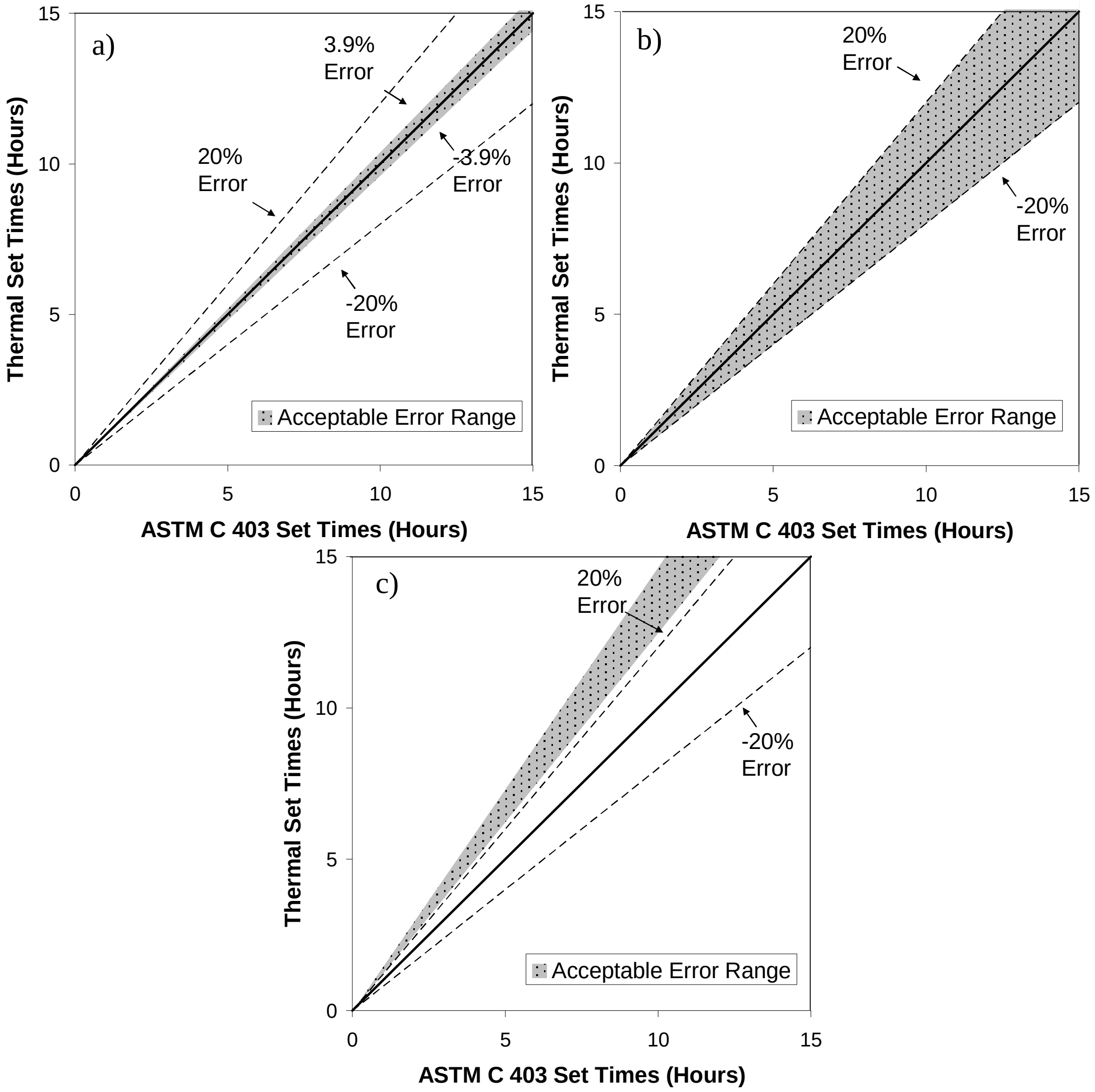


Figure 3.23: Examples of allowable range of error for *secondary set* as compared to ASTM C 403 (2008) results for thermal testing to be considered a reliable indicator of concrete setting for a) criteria a, b) criteria b, and c) criteria c

If the first of these criteria is met, (Criterion a), setting times obtained through thermal methods may be considered as reliable an indicator of concrete setting as ASTM C 403 (2008), and results may be considered comparable. In Figure 3.23a, 3.9 % is shown as the acceptable error range for *secondary set* values. This is the value reported in the single-operator precision statement (see Section 3.5.1.2) for the acceptable difference between two results for final set measured as per ASTM C 403 (2008). In a similar manner, the acceptable error range for *primary set* can be determined from the single-operator precision statement for initial set.

If the second of the above criteria is met (Criterion b), setting times obtained through thermal methods may not be considered as reliable as those obtained through ASTM C 403 (2008), but may still may be adequate indicators of concrete setting, and comparable to penetration resistance-based methods. In the past, the ± 20% error range has been used as an acceptable percent error for the determination of concrete setting times (Edson 2005), therefore the acceptable error range for both *primary set* and *secondary set* as compared to ASTM C 403 (2008) was defined as ± 20%. This range can be seen in Figure 3.23b.

If neither Criterion a or b is met, there is a possibility that setting times obtained through thermal methods may be an independent indicator of setting (i.e. primary set and secondary set are not related to either initial or final set, but may still be considered adequate indicators of concrete setting). For this criterion to be met, primary set and secondary set may compare to ASTM C 403 (2008) with greater than ± 20% error, but must have a reasonable variability (represented by the scatter of data points). An example of this can be seen in Figure 3.23c. Although the range of values lies outside the ± 20% range, a small scatter is required for data to fall within this area. It should be noted that the acceptable error range demonstrated in Figure 3.23c is only one possible configuration for Criterion c; any range outside ± 20% with a reasonable variability would also meet this criterion.

Results from the comparison of setting times obtained through thermal methods and those obtained as per ASTM C 403 (2008) can be seen in Figure 3.24.

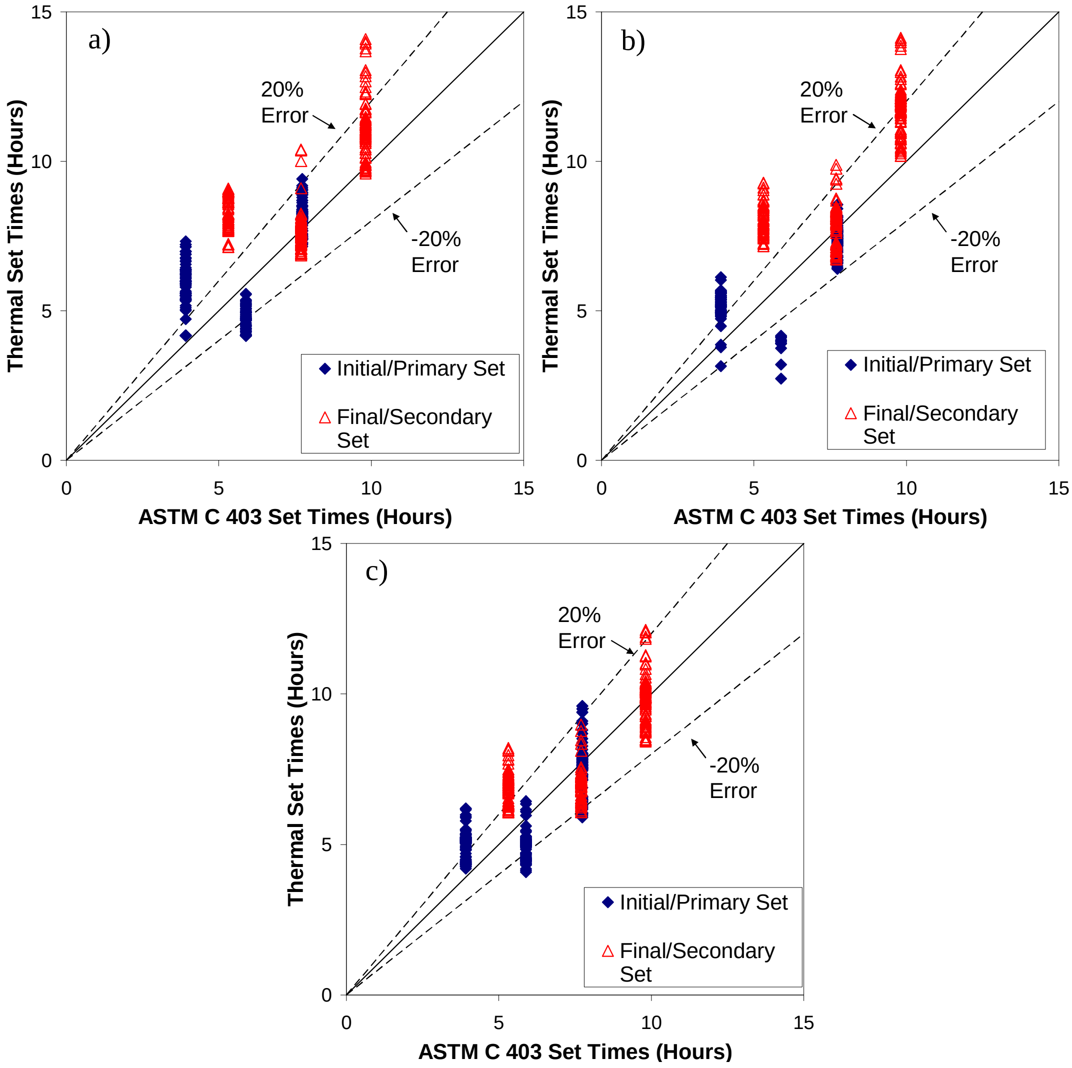


Figure 3.24: Setting times obtained through thermal methods as compared to those obtained through penetration-resistance testing for a) the *Direct-Slope Derivatives Method*, b) the *Polynomial Derivatives Method*, and c) the *Direct Fractions Method*

Of the three analysis methods, the *Direct Fractions Method* appears to yield the most accurate results as compared to ASTM C 403 (2008), but none of the analysis methods meet the criteria outlined for acceptability. Based on these results, the thermal methods for determining time of set as proposed in the draft ASTM specification should not be considered an indicator of the setting time of a concrete mixture.

Due to the effects of device properties on results from thermal testing (see Section 3.6.2.1), average results for each device category were also summarized. These results can be seen in Figure 3.25, Figure 3.26, and Figure 3.27. It should be noted that questionable results due to inconsistencies within the test methods are not included in this summary; therefore, initial/primary set percent difference values are not available in some instances. When this was the case, values are noted with an "X" so they may be differentiated from 0 % error.

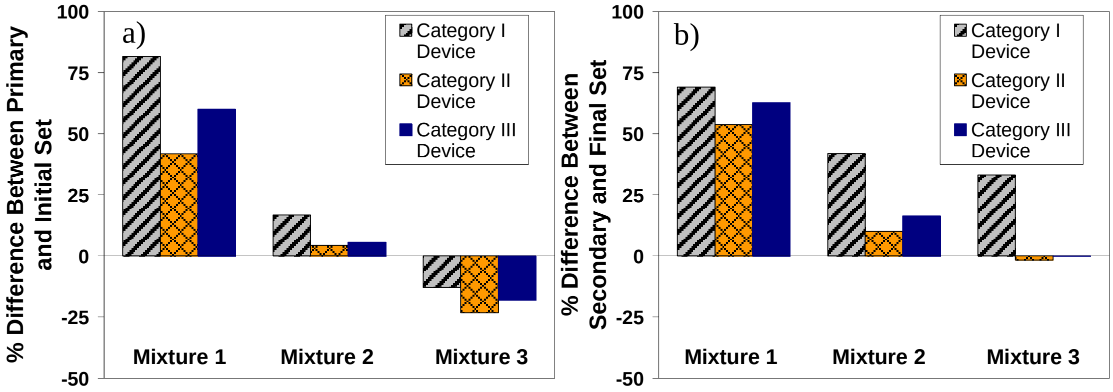


Figure 3.25: Average *Direct-Slope Derivatives Method* versus ASTM C 403 (2008) comparisons for a) initial/*primary set* and b) final/*secondary set*

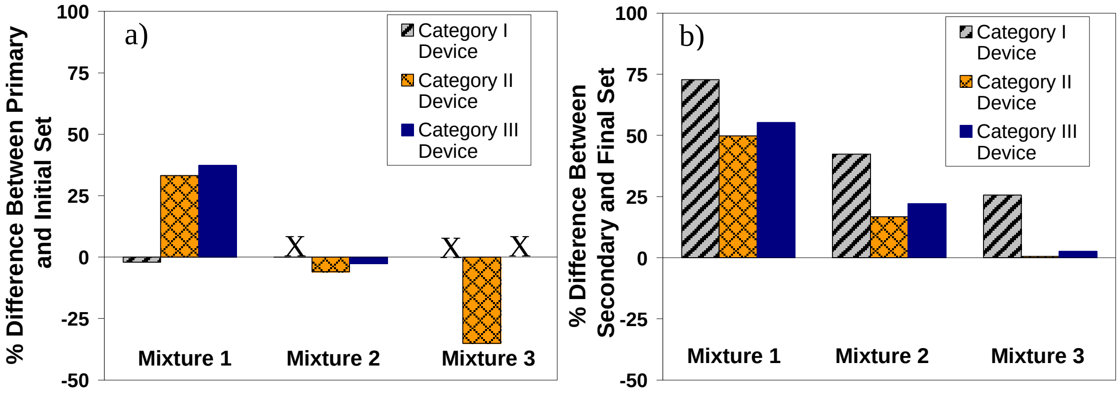


Figure 3.26: Average *Polynomial Derivatives Method* versus ASTM C 403 (2008) comparisons for a) initial/*primary set* and b) final/*secondary set*

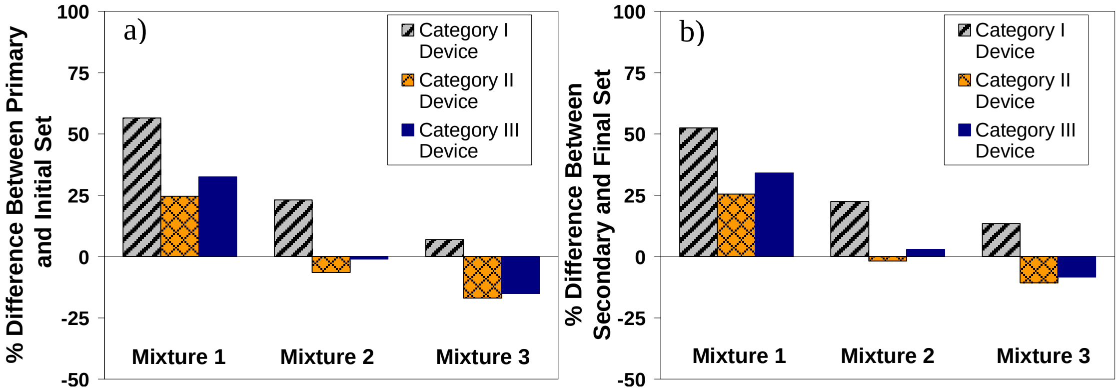


Figure 3.27: Average *Direct Fractions Method* versus ASTM C 403 (2008) comparisons for a) initial/*primary set* and b) final/*secondary set*

As can be seen in Figure 3.25, Figure 3.26, and Figure 3.27, error is, in general, much higher for Category I and Category III devices than for Category II devices for all analysis methods. (This could be due to the fact that both the *Fractions Method* and the *Derivatives Method* were developed by Sandberg and Liberman [2007] with the use of a Category II Device.) It is also evident from Figure 3.25, Figure 3.26, and Figure 3.27

that the average *Direct Fractions Method* results have less overall error when compared to ASTM C 403 (2008) than the average *Direct-Slope Derivatives Method* results and *Polynomial Derivatives Method* results for all device categories.

In order to evaluate the possibility of a slight modification to the draft ASTM specification to limit analysis to a Category II device and the use of the *Direct Fractions Method*, these data were evaluated with the use of the criteria described above. When results are limited to the *Direct Fractions Method* results from Category II Devices, (as is shown in Figure 3.28), results are much closer to the ± 20% error range described in Criterion b above, yet still fall outside of the range defined.

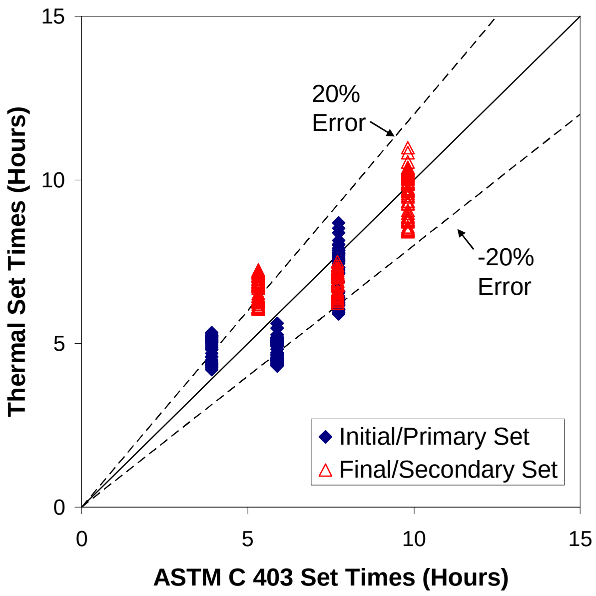


Figure 3.28: Setting times obtained through the *Direct Fractions Method* as compared to those obtained through penetration-resistance testing for Device Category II

As previously mentioned, the mortar and concrete samples used for ASTM C 403 (2008) and thermal testing respectively have significantly different temperature profiles. This difference will vary from mixture to mixture (See Figure 3.22) and could account for some of the inconsistencies in the comparisons. The effects of temperature on a concrete or mortar sample can be accounted for by using of maturity concepts in order to reduce the differences due to these effects. Further study of the application of maturity concepts to setting data is recommended.

3.7 Conclusions and Recommendations

Based on tests performed on three different concrete mixtures by various laboratories, a precision statement for ASTM C 403 was developed for setting times ranging from 307-574 minutes for concrete samples taken from a single batch of concrete.

It was determined that the results of two properly-conducted tests by the same operator are not expected to differ by more than 4.8 % and 3.9 % for initial set and final set, respectively. The range (difference between highest and lowest) of the three test results obtained by the same operator on specimens from the same batch of concrete are not expected to exceed 5.6% and 4.6% for initial set and final set, respectively. Similarly, the results of two properly-conducted tests by different operators on the same material are not expected to differ by more than 10.7% and 7.4% for initial set and final set, respectively. The averages of three test results by two different operators obtained on specimens from a single batch of the same concrete are not expected to differ by more than 6.2% and 4.2% for initial set and final set, respectively.

Further study is recommended to investigate the effects of specification parameters on test method results and the correlation of setting times obtained by ASTM C 403 (2008) to the setting of in-place concrete.

The proposed ASTM *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method* and several additional analysis methods were evaluated using data obtained from an ASTM round robin study involving 12 laboratories and three different concrete mixtures. In order to investigate the sensitivity of the proposed ASTM specification to various testing parameters, a sensitivity study was conducted to examine he effects of sample size, sample type, thermal device properties, and analysis type. Conclusions from this sensitivity study are as follows:

- Sample size was found to have a very significant effect on thermal data and setting times obtained from thermal testing.
- Sample type was found to have a significant effect on thermal data and setting times obtained from thermal testing.
- The insulatory properties of the thermal device used for testing was found to have a significant effect on thermal data and setting times obtained from thermal testing.
- The analytical method used for analysis was found to have an effect on setting times obtained through thermal methods.
- A small variation in recorded temperature due to the process of the concrete specimen reaching equilibrium within the testing device can have a substantial impact on setting times obtained using the Direct Fractions Method.

Thermal setting times obtained through thermal testing with the use of three analysis methods were then compared to those obtained by ASTM C 403 (2008). Conclusions from this comparison are as follows:

- As written, the thermal methods for determining time of set of concrete as proposed in the draft ASTM specification should not be considered an indicator of the setting time of a concrete mixture.
- Of the three analytical methods evaluated, setting times obtained with the use of the *Direct Fractions Method* compared most accurately with those obtained as per ASTM C 403 (2008).
- Of the thermal devices used in this study, results obtained with the use of a moderately-insulated single chamber device with multiple open-topped cavities for specimens (Category II device) yielded the most accurate results when compared to results obtained as per ASTM C 403 (2008).
- Even when analysis was limited to the use of the *Direct Fractions Method* for Category II thermal devices, setting times obtained through thermal methods may not be considered an indicator of the setting time of a concrete mixture.

Recommendations for future study involving the calculation of setting times with the use of thermal methods are as follows:

- Specimen size, specimen type, and device insulatory properties should be further studied and closely controlled in any future specification.

- Due to the effects of equilibrium between concrete specimens and thermal devices on the results obtained through thermal testing, these effects should be closely monitored and, if possible, controlled in future research.
- Due to the fact that only room-temperature concrete was evaluated in this study the effect of testing conditions (especially temperature) and initial temperature of concrete should be further studied.
- The application of maturity concepts and thermodynamic losses for a given thermal device should be investigated as possible solutions to sources of significant error within the proposed methods.

Chapter 4

Laboratory Testing Program

In order to evaluate the use of thermal methods to predict the setting of concrete, a laboratory testing program was undertaken. In this chapter, details of this testing program are discussed. Topics covered include an overview of the testing program, mixture proportions, concrete production, curing methods, testing apparatus, testing procedures, and the properties of the raw materials used.

4.1 Overview of Testing Program

One of the primary objectives of this research was to evaluate the use of thermal methods to quantify the setting process of concrete. In order to accomplish this objective, a testing program was developed in which setting times were predicted by both traditional testing and analysis of data obtained from thermal methods for a variety of concrete mixtures, and the results were compared. As discussed in Chapter 3, the current method of determining concrete setting times is based on the penetration resistance of a sieved mortar sample. ASTM C 403 (2008) is the standard specification and generally accepted method for this testing. In order to establish a valid basis for comparison, the precision and bias of this test method were computed in Chapter 3 and can be found, along with a brief discussion, in Section 3.6.1.1

To evaluate setting times from thermal methods as compared to penetration resistance-based methods, comparisons must be made on the basis of maturity, or equivalent age, as opposed to actual concrete/mortar age. As discussed in Chapter 2 (Section 2.3.3) and Chapter 3 (Section 3.6.2.1), mixture and curing temperature have been shown to have a significant impact on setting, and a significant temperature difference may exist between concrete samples used for thermal testing and the mortar samples used for ASTM C 403 (2008). To avoid inaccurate correlations due to temperature differences, all measurements of setting were adjusted for maturity with the use of the FHP maturity formulation. Defining setting in terms of maturity instead of traditional concrete age may also aid in the accurate prediction of in-place setting in field applications. To convert measured setting times from actual concrete/mortar age to an equivalent age, the temperature of each concrete specimen must be recorded and the mixture's sensitivity to temperature must be determined. (For an overview of the equivalent age maturity method, see Section 2.4.)

Seven individual concrete mixtures were evaluated in this program in order to examine setting across a range of mixture proportions. These mixtures were representative of concrete used for a variety of application, and included the use of several supplementary cementing materials (SCMs). In order to calculate the temperature sensitivity of each mixture and to evaluate setting across a range of environmental conditions, each mixture was mixed and cured at three different temperatures. The mixture and curing conditions used are as follows:

(a) One batch from each mixture was produced and cured at laboratory ambient temperature which varied from 20 to 26°C,

(b) One batch from each mixture was produced and cured at temperatures meant to simulate summer conditions with environment temperatures which varied from 32 to 38°C, and

(c) One batch from each mixture was produced and cured at temperatures meant to simulate winter conditions with environment temperatures which varied from 7 to 13°C.

For convenience, testing was divided into two general phases. Phase I consists of the determination of the temperature sensitivity of each mixture quantified through the calculation of the activation energy (E). This value can then be used along with collected temperatures to account for the effects of temperature on setting through the use of the equivalent age maturity method. E was calculated using both penetration resistance data and isothermal calorimetry data. This allowed for an E value to be determined that best characterized the effect of temperature on setting as well as an E value that best characterized the effect of temperature on the hydration process.

Phase II consists of the measurement of concrete setting and the evaluation of the various thermal methods for determining concrete setting. For each concrete batch produced, setting was measured as per ASTM C 403 (2008) and through semi-adiabatic calorimetry testing. All set times were adjusted for equivalent age based on measured temperatures during testing, and the E value calculated in Phase I and results were compared. Thermal methods for determining concrete setting were each evaluated based on the comparison of thermal-based results to results obtained through ASTM C 403 (2008). A schematic of Phase I and Phase II of testing can be seen in Figure 4.1

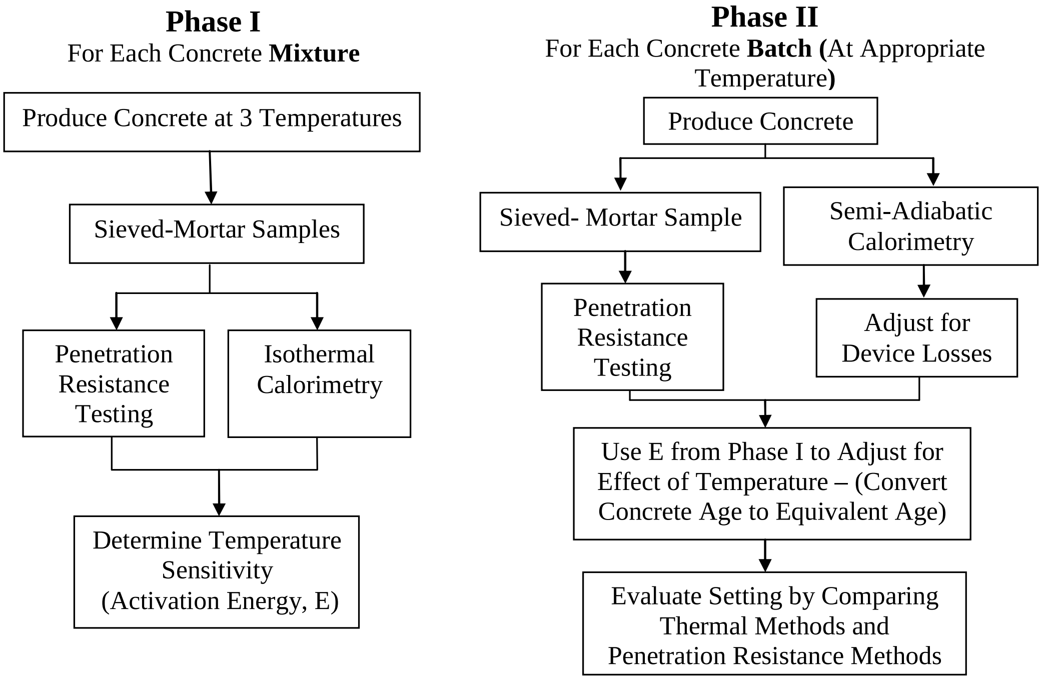


Figure 4.1: Schematic of Phase I and Phase II of testing

4.2 Mixture Proportions

Seven individual concrete mixtures were developed for this testing program to evaluate the effect of w/c, and supplementary cementing materials on methods to determine concrete setting. The notation for these seven mixtures that will be used throughout this thesis are as follows:

- Control - Type I cement with w/cm of 0.44
- 30% C - 30% replacement of Type C fly ash
- 30% F - 30% replacement of Type F fly ash
- 50% Slag - 50% replacement of GGBF slag
- w/c 0.36 - Type I cement with w/cm of 0.36

- w/c 0.40 - Type I cement with w/cm of 0.40
- w/c 0.48 - Type I cement with w/cm of 0.48

A control mixture was established with fresh and hardened properties similar to concrete used in the Alabama concrete industry. In order to evaluate the effects of SCMs and w/c, SCM mixtures and variable water-cement mixtures were kept as close as possible to the control mixture, changing only the variable in consideration as well as admixture amounts to control workability, when appropriate. Small adjustments were made in the aggregate proportions due to variations in material volume, but these were less than 3% by weight, therefore the effects were considered negligible. Mixture proportions for the seven mixtures can be seen in Table 4.1 . All batch weights reflect aggregate at a saturated surface-dry state.

Three batches of each mixture were produced, notated “cold” for the batch that simulates winter conditions, “room” for the batch kept at laboratory ambient temperatures, and “hot” for the batch simulates summer conditions. Select batched were re-mixed for quality control purposes and in order to confirm trends in testing. These batches are denoted with a batch number following the standard batch name. The following notation is used to denote a particular batch of concrete:

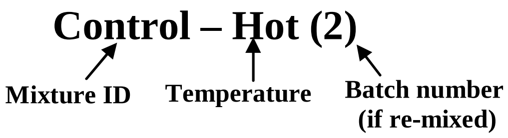


Table 4.1: Mixture Proportions

Constituent	Mixture Identification						
	Control	**30% C**	**30% F**	**50% Slag**	**w/c 0.36**	**w/c 0.40**	**w/c 0.48**
Water (kg/m^3)	162	162	162	162	151	156	176
Type I Cement (kg/m^3)	368	257	257	184	418	390	368
Class F Fly Ash (kg/m^3)	-	-	110	-	-	-	-
Class C Fly Ash (kg/m^3)	-	110		-	-	-	-
GGBF Slag (kg/m^3)	-	-		184	-	-	-
Coarse Aggregate (kg/m^3)	1,225	1,206	1,194	1,206	1,201	1,211	1,190
Fine Aggregate (kg/m^3)	689	689	689	696	694	699	687
ASTM Type A & D Admixture WRDA 64 (mL/m^3)	719	719	719	959	-	-	-
ASTM Type A & F Admixture MIRA 92 (mL/m^3)	-	-	-	-	2,727	638	-
Water-Cement Ratio, w/c	0.44	0.63	0.63	0.88	0.36	0.40	0.48
Water-Cementitious Materials Ratio, w/cm	0.44	0.44	0.44	0.44	0.36	0.40	0.48

4.3 Concrete Production

In order to obtain consistent reproducible results, a batching and mixing procedure was developed for all concrete produced. For every mixture, a 0.10 cubic meter (3.5 ft^3) batch was produced for each testing condition. In addition, for select mixtures, a 0.04 cubic meter (1.5 ft^3) batch was produced in order to obtain mortar samples for isothermal calorimetry testing. The procedure outlined in the sections below was followed for all concrete produced.

4.3.1 Batching of Materials

To achieve the target fresh concrete temperature and curing conditions above an insulated temperature-controlled environmental chamber was utilized for raw material storage, testing procedures, and the curing and hardening of test specimen. See Figure 4.2 for images of the temperature controlled chamber.

Figure 4.2: Insulated temperature controlled environmental chamber

All materials were batched one day before the concrete was mixed. For hot and cold batches, batch weights were approximated and all materials were placed in the temperature controlled chamber in 22.7 liter (5 gal.) buckets in order to condition the materials. This was necessary to replicate hot and cold weather concreting, where materials are stored in stockpiles in ambient conditions. Due to the fact that the concrete was mixed in a room temperature environment, materials were over-heated or cooled in order to obtain target fresh concrete temperatures for cold and hot batches of 10 and 35 °C, respectively. For hot batches, the chamber was set to 43 °C which yielded an average fresh concrete temperature of 35 °C. For cold batches, the chamber was set to 2 °C, which yielded an average fresh concrete temperature of 10 °C. Room temperature batched were batched and stored in the laboratory. Ambient temperature in the laboratory was approximately 23 °C.

Moisture corrections were completed with the use of a hot plate and small digital scale as per ASTM C 566 (1997) just prior to mixing. It should be noted that moisture corrections were not completed in advance for aggregates stored for hot and cold mixtures due to moisture variations while being conditioned. After being removed from the chamber, aggregates were mixed thoroughly and a sample was taken, weighed, heated until dry, and re-weighed. The moisture content of the aggregates were then compared to the absorption capacities of the course and fine aggregate and adjustments were made to the batch weights to reflect the actual moisture condition of the aggregates. All chemical admixtures were batched just before the concrete was mixed.

4.3.2 Mixing procedure

All concrete was mixed as per ASTM C 192 (2007). A 0.34 cubic meter (12 ft^3) mixer was used for all 0.10 cubic meter (3.5 ft^3) batches and a 0.17 cubic meter (6.0 ft^3) mixer was used for all 0.04 cubic meter (1.5 ft^3) batches to ensure proper mixing. The concrete mixers used can be seen in Figure 4.3.

Figure 4.3: 0.34-m^3 and 0.17-m^3 mixers

Before mixing, each mixer was "buttered" with a mixture of fine aggregate, cement, and water in order to evenly coat the sides of the mixer. In general, a mixture of approximately 2/3 fine aggregate and 1/3 cement was added to the mixer along with enough water to allow it to coat the mixer without leaving excess water in the mixer itself. The excess mortar from the butter mixture was then discarded in preparation for the concrete mixture.

All aggregates were added next, alternating between buckets of coarse and fine aggregates, along with approximately 80% of the mixing water. The materials were then allowed to mix for two minutes. A lid was used to cover the mixer during all mixing cycles to help maintain constant moisture at all three temperatures, and to reduce heat loss/gain during the mixing process. After two minutes of mixing, all cement and SCMs were added, along with the rest of the water and any chemical admixtures. The concrete was allowed to mix for three minutes, followed by a rest of three minutes, and a final three minutes of mixing. At this point fresh concrete properties were measured and further testing commenced. For room temperature batches further testing was completed under standard laboratory temperature conditions, while for all hot and cold batches, all further testing was completed in the temperature-controlled environmental chamber.

4.4 Testing Methods

The testing plan developed to evaluate concrete setting was divided into two phases. Phase I consists of the determination of the temperature sensitivity of each mixture considered in the program, and Phase II consists of the determination of concrete setting and the comparison of setting methods in terms of concrete equivalent age. In the following sections, the testing apparatus and procedure for Phase I, Phase II and all general quality control testing is described in detail.

4.4.1 Phase I Testing

In order to adjust setting results for the effects of temperature using FHP maturity function, the activation energy of each mixture must be known. Phase I of testing consists of the determination of this value through both penetration resistance testing and isothermal calorimetry. For each mixture considered in the testing program, batches of concrete were produced and tested at three separate temperatures, and the results were analyzed in order to calculate E. In the following sections, the testing procedure and equipment for all Phase I testing is discussed.

4.4.1.1 Penetration Resistance Testing

All penetration resistance testing was completed as per ASTM C 403 (2008) with one modification to allow measurement of the mortar temperature that will be discussed. Testing apparatus consisted of a standard penetrometer, and set of loading needles, a temperature recording device, and a modified mortar container. An upright penetration resistance loading apparatus, or penetrometer, and standard set of proctor penetration needles were used for all testing, and can be seen in Figure 4.4. It should be noted that two identical penetrometers were used during the testing program. Penetrometer 1 was used for all hot and room temperature batches excluding w/c 0.40 – Hot (2), 30%C – Room (2), w/c 0.36 – Room (2), and w/c 0.40 – Room (3). Penetrometer 2 was used for all other batches. There was no significant difference between results obtained from Penetrometer 1 and Penetrometer 2. 4.5-L (referred to as ten-pound) cylindrical metal ink cans (or slip cover cans) were used as mortar sample containers for each batch. These aluminum containers measured 19.1 cm (7.5 in.) in diameter by 15.6 cm (6.1 in.) tall, and had a tight fitting lid. A rubber band was used around the container under the lid to act as a gasket and prevent moisture loss during testing. This container can be seen in Figure 4.4.

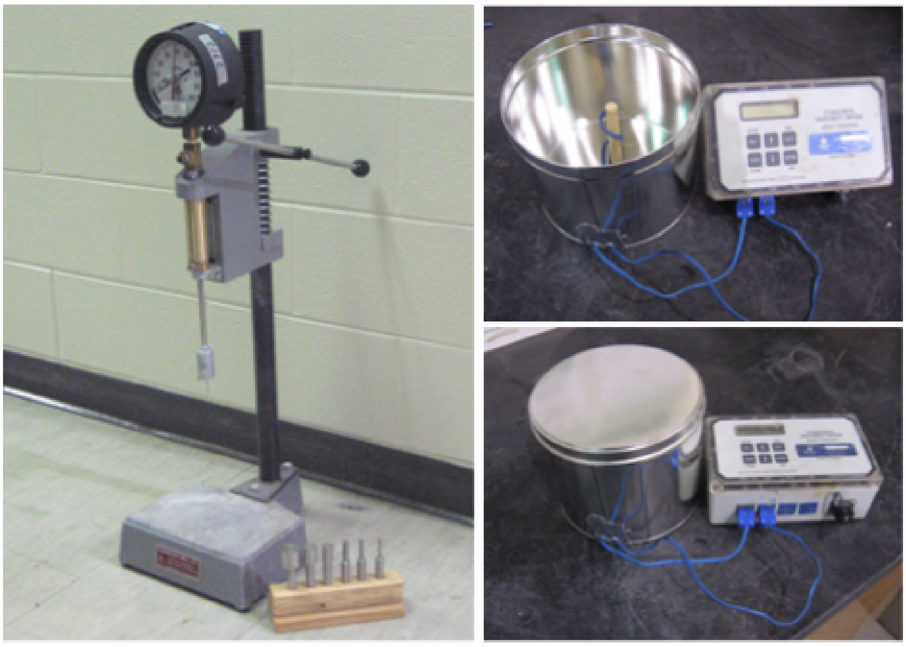


Figure 4.4: Penetrometer 1, mortar sample container, and multi-channel maturity meter for ASTM C 403 (2008) testing

In order to allow for the calculation of the activation energy (E) of each mixture, ASTM C 403 (2008) was to allow the recording of mortar temperatures throughout testing. This was accomplished with the use of a multi-channel maturity meter (see Figure 4.4), which records temperature at thirty-minute increments for the first 24 hours of testing and at one-hour increments thereafter. Two thermocouples were used for testing in each mortar container: one in the center of the specimen, and one in the middle of the specimen, near the surface of the concrete. Thermocouples were attached with the use of a 1.3-cm (0.5 in.) diameter wooden dowel positioned vertically at the center of the sample, so as not to interfere with the penetration readings. A diagram of the modified setting can with thermocouple locations may be seen in Figure 4.5.

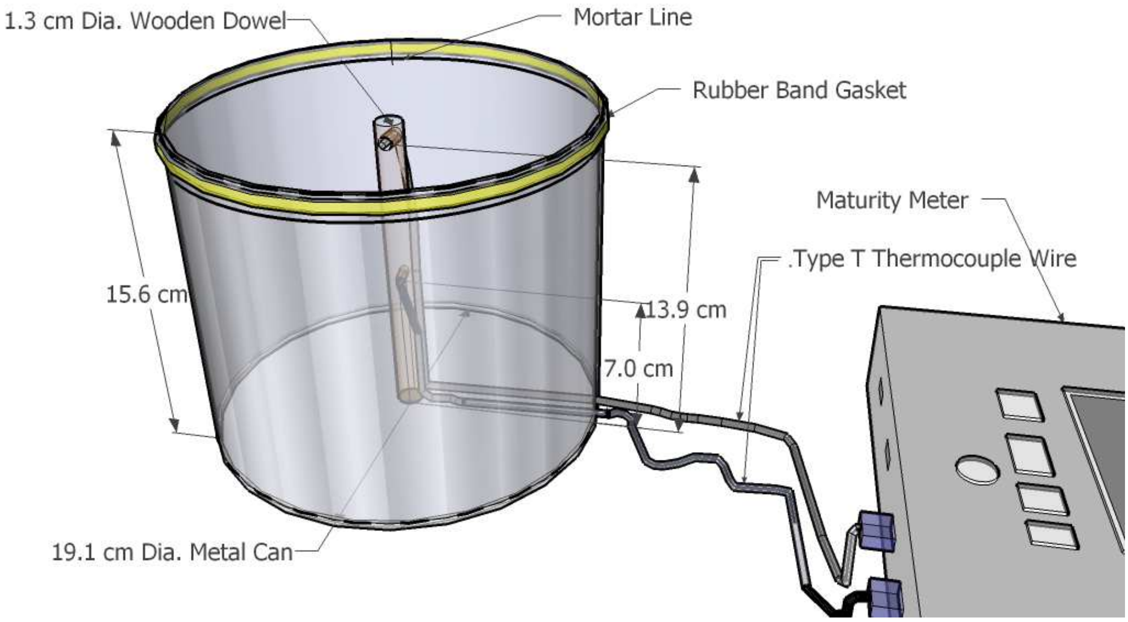


Figure 4.5: Modified sample container (lid not shown) used for penetration resistance tests

Preparation of the sample for penetration resistance testing began immediately after fresh concrete properties were measured and concrete was moved to its respective temperature controlled environment. Concrete was sieved through a 4.75-mm sieve with the use of a vibrating table in order to obtain a mortar sample, and the setting container was filled in one lift. The mortar was then consolidated using the vibrating table and the top of the specimen was leveled. Temperature collection was started as soon as the mortar container was filled with the use of the maturity meter.

Throughout the testing process, the mortar sample and testing apparatus remained in the temperature-controlled environment. Penetration readings were measured at approximately equal time intervals throughout testing. It should be noted that, although

Section 1.1 of ASTM C 403 (2008) states that the test method is intended to apply only to concrete specimen with a slump greater than 0, all concrete specimens were tested as per this specification regardless of fresh concrete properties.

4.4.1.2 Isothermal Calorimetry

Isothermal calorimetry, or conduction calorimetry, was also used to calculate the activation energy to account for the effect of temperature on the process of hydration. In isothermal calorimetry, a sample is allowed to hydrate at a constant temperature and the heat required to maintain that temperature is recorded. The calorimeter unit used is manufactured by W. R. Grace and Company (AdiaCal TC) and can be seen in Figure 4.6. It is an eight-cell calorimeter for testing cement paste or mortar samples. Details concerning this device are not known to the author

Figure 4.6: Isothermal calorimeter

The isothermal calorimeter was prepared for testing at least 24 hours before a batch of concrete was to be produced. The testing temperature was set and the device was allowed to reach equilibrium at the testing temperature. In order to reduce the amount of heat lost or gained due to exposure to laboratory temperatures, isothermal samples were prepared as soon as possible after mixing. Immediately after the fresh concrete properties were measured and concrete was moved to its respective temperature controlled environment, a sample was prepared for testing. Due to the small sample size of the isothermal device (see Figure 4.6), concrete was sieved through a 4.75-mm sieve to obtain a mortar sample to be used for testing. Two to three 125 to 200-g samples of the mortar were then placed in five cm in diameter plastic containers and the samples' weight were determined. The specimens were then placed in their respective cells and the device was started. Samples were left in the device for 36 hours, or until it was determined that significant hydration had ceased.

Due to the late acquisition of the device, isothermal testing was not completed for each 0.10 cubic meter batch from which all other testing was performed. For batches for which this was the case, a separate 0.04 cubic meter batch was produced for this test. Fresh concrete properties and quality control cylinders were tested in order to ensure proper batching and mixing, and isothermal samples were prepared and tested in the same manner as those from the larger batches. For batches in which this was the case see Table 4.2.

Table 4.2: Batches for which a separate 0.04-m3 batch was produced

Mixture	
Control	Room Hot
30% C	Cold Hot
30% F	Cold Room Hot
50% Slag	Cold Room
w/c 0.36	Cold Hot
w/c 0.48	Cold Room Hot

4.4.2 Phase II Testing

In Phase II of testing, setting characteristics of each concrete batch were measured by penetration resistance testing and the temperature released due to hydration measured by semi-adiabatic calorimetry. Measurements were then adjusted for maturity with the use of activation energy values obtained from Phase I, and results were compared. Thermal methods to estimate setting could then be evaluated as compared to set times obtained from the standard penetration resistance test, ASTM C 403 (2008). In the following sections, the testing apparatus and procedure for all Phase II testing will be discussed.

4.4.2.1 Semi-Adiabatic Calorimetry

Setting times as measured by thermal methods were obtained with the use of semi-adiabatic calorimeters to measure the heat of hydration of concrete samples. A semi-adiabatic calorimeter is defined by RILEM TC 119 TCE 1 (1997) as a calorimeter in which the maximum heat losses are less than 100 J/(h·K). Three insulated temperature recording devices were used for testing, two of which (denoted Calorimeter I and Calorimeter II) can be classified as semi-adiabatic by this definition. Calorimeter III could not be classified as semi-adiabatic due to the fact that it is an open- top unit, therefore losses cannot be accurately determined or accounted for. All three calorimeters met the heat loss requirements provided in the previously proposed ASTM specification entitled *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method*. However, the well-insulated devices were preferred in this study over less-insulated devices due to the impact of the testing environment on results. Although calorimeter III was not used for analysis, samples were still tested using this device for comparison purposes. Calorimeter I, Calorimeter II, and Calorimeter III can be seen in Figure 4.7, Figure 4.9, and Figure 4.11, respectively.

Calorimeter I, which can be seen in Figure 4.7, is a calorimeter produced by W. R. Grace and Company (AdiaCal Plus). Two separate devices were used for testing, and all four cells of each device underwent a hot water calibration before testing to determine properties used for heat loss adjustment. As can be seen, Calorimeter I is a multi-sample calorimeter in which each sample is insulated on all sides. The insulation used was selected by the manufacturer and its properties are unknown to the author. The effective R value of each unit was calculated during the calibration data and was determined to be 25 k/W.

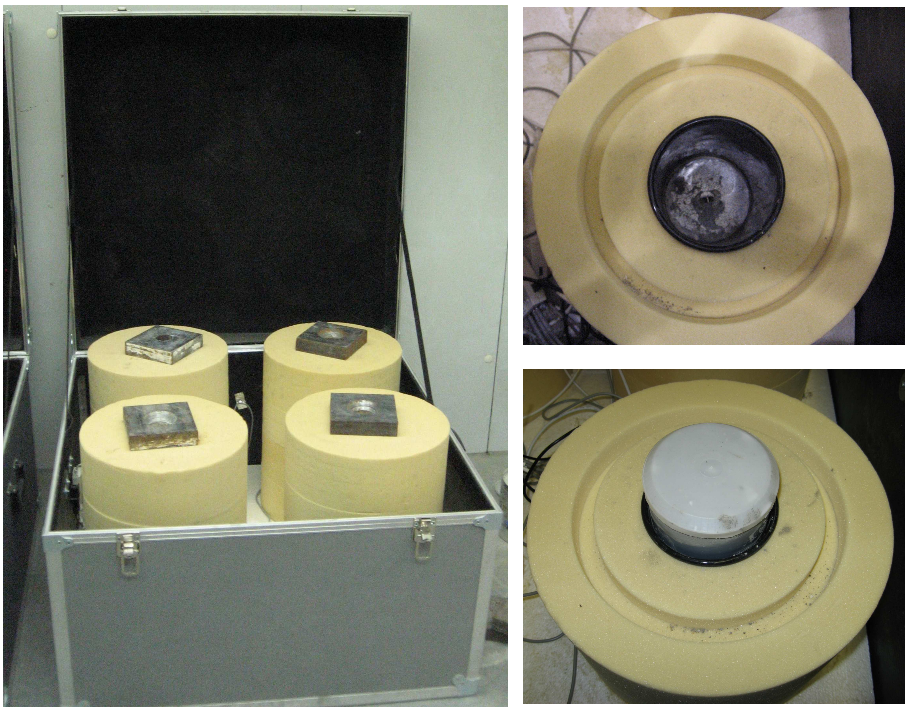


Figure 4.7: Calorimeter I

The device makes use of a thermistor-type temperature sensor attached to a metal plate below each specimen (as can be seen in Figure 4.7 and Figure 4.8). The thermistor and external data logger used has an accuracy of ± 0.5 °C and a sampling rate of one point per minute. Samples are 10 cm (4 in.) in diameter by 20 cm (8 in.) high concrete cylinders. All four cells of both units were used simultaneously for all testing. Modifications to the device include 4.5-kg (10-lb) weights placed on top of each cell during testing, and two ambient temperature sensors attached to the box outside of the insulated cells. For a dimensioned diagram of Calorimeter I see Figure 4.8.

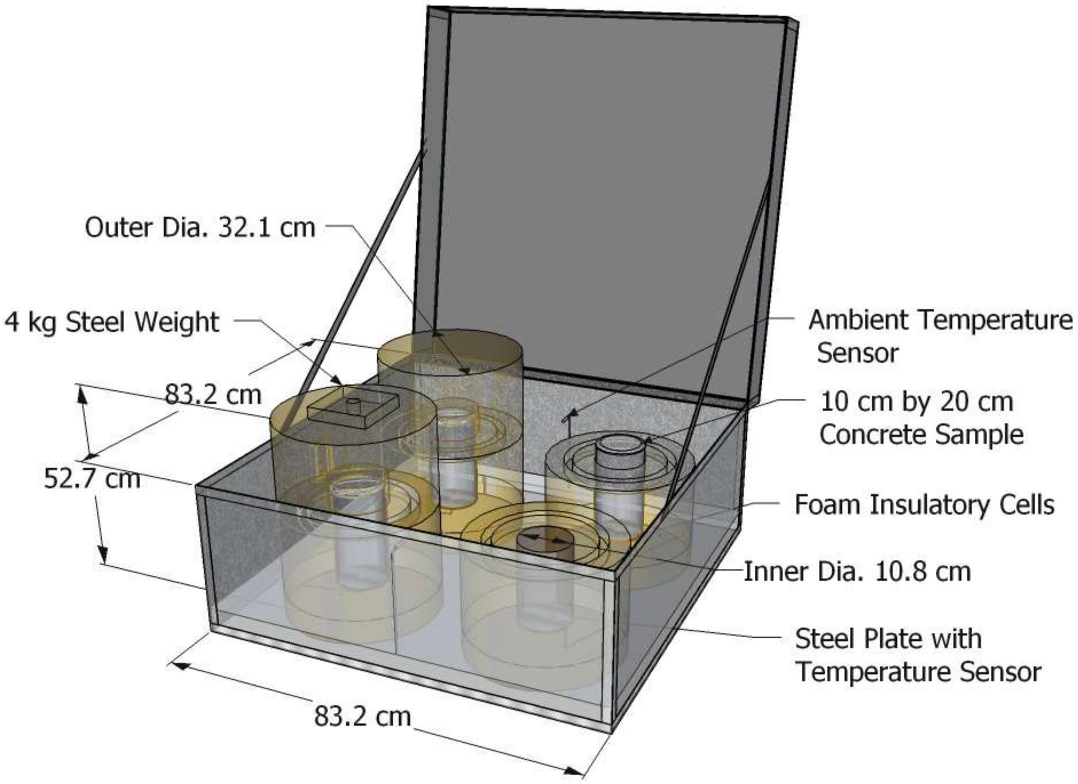


Figure 4.8: Diagram of Calorimeter I

Calorimeter II, which can be seen in Figure 4.9, is a semi-adiabatic hydration drum produced by Digital Site Systems of Pensylvania (device model number is iQdrum 300). Two separate devices were used for testing, therefore each calorimeter was calibrated before testing began. As can be seen in Figure 4.9, and Figure 4.10, Calorimeter II is a single sample device that is well-insulated on all sides with an internal temperature sensor (a sensor is inserted directly into the concrete). The insulation used was selected by the manufacturer and its properties are unknown to the author. The effective R value of the unit was calculated as 9.8 K/W.

Figure 4.9: Calorimeter II

The temperature sensor is a reusable Type-T thermocouple with a 5-cm (2-in.) long plastic tip that can be inserted through the specimen mold cap into the top of a concrete specimen. An ambient temperature sensor and a flux sensor are also included within the device. The Type-T thermocouple used has an accuracy of ± 0.5 °C and temperatures are logged at 15-minute intervals throughout testing. All samples are standard 15 cm (6 in.) in diameter by 30 cm (12 in.) high concrete cylinders. For a dimensioned diagram, see Figure 4.10.

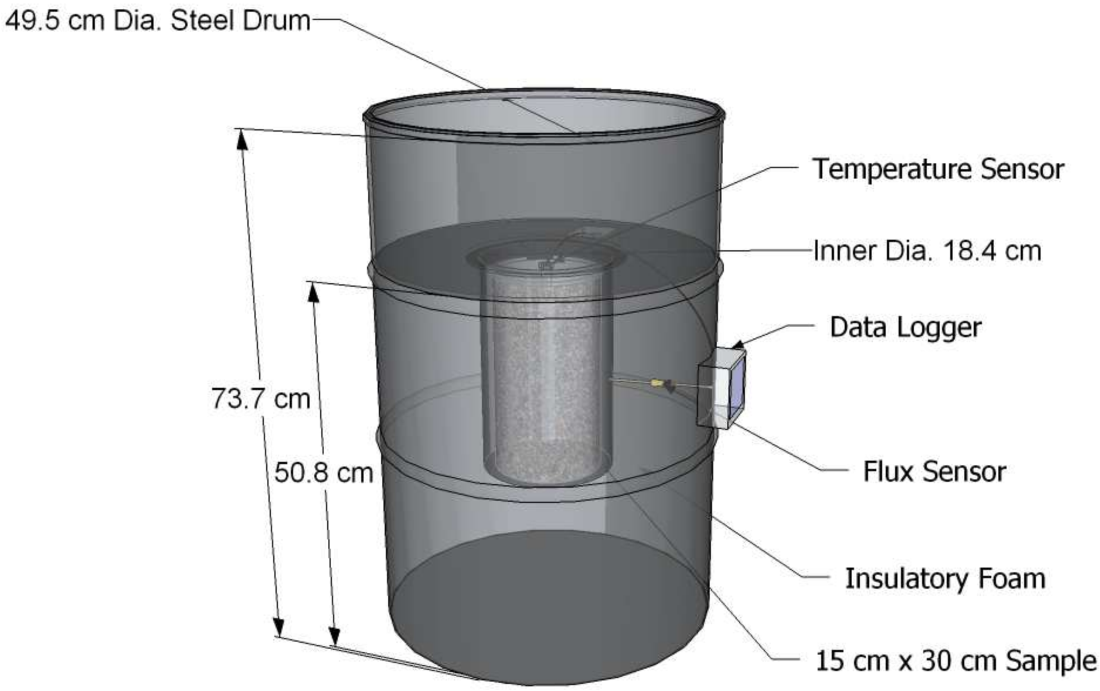


Figure 4.10: Diagram of Calorimeter II

Calorimeter III, which can be seen in Figure 4.11, is also produced by W. R. Grace and Company (AdiaCal 4). This device is a multi-sample calorimeter in which each sample is insulated on the sides, but not the top. Due to the open top construction, the effective R value of the unit could not be computed. The temperature sensor, sampling rate, and sample size are identical to Calorimeter II. Only one cell at a time was used during testing.

Figure 4.11: Calorimeter III

4.4.2.2 Thermal Testing Procedure

For each batch of concrete, nine 20 cm in diameter by 40 cm tall cylinders and one 30 cm in diameter by 60 cm tall we made as per ASTM C192 (2007) and placed in calorimeters for approximately 150 hours. In order to reduce the effects of thermal equilibrium, all calorimeters were placed in the testing environment for a specific mixture at least 24 hours before mixing. This allowed the device to be as close to the fresh concrete temperature of the concrete as possible at the onset of testing. All calorimeters remained in this testing environment at the specified temperature for the duration of testing.

Calorimeters II and III were used solely for thermal testing. For Calorimeter II testing, the weight of the 15-cm by 30-cm cylinder was recorded and the temperature probe was placed into the top of the cylinder as close to the center as possible. The cylinder was then lowered into the device, and the device was closed and started. The device remained undisturbed for the remainder of the test. For Calorimeter III testing, one 10-cm by 20-cm cylinder was weighed and placed directly into the device. The outer lid of the device remained open throughout testing, and the device remained undisturbed.

Calorimeter I was used for both thermal testing and early-age strength determination. Eight 10-cm by 20-cm cylinders were weighed and placed into the eight wells of the device. The top was placed on each well and a 4.5-kg weight was placed on top of each to reduce heat loss. The outer lid of the device was left open throughout testing. Two of these cylinders (one in each device) were left undisturbed throughout testing for determination of setting through thermal methods. The remaining six cylinders were removed at early ages and tested for compressive strength as per ASTM C 39 (2004). For all batches whose final set as per ASTM C 403 (2008) was not greater than 13 hours, cylinders were tested at one and two days. For all batches whose final set was greater than 13 hours, cylinders were tested at two and four days. Based on these requirements, all batches excluding 30% C – Cold, 30% F – Cold, and 50 % Slag – Cold, cylinders were tested at 1 and 2 days.

4.4.2.3 Penetration Resistance Testing

The penetration resistance results from Phase I testing was also used for Phase II. All testing apparatus and procedures are identical to Phase I testing and were discussed in Section 4.4.1.1.

4.4.3 Quality Control Testing

Several fresh and hardened concrete properties were measured for each batch made as a means of quality control. Immediately after mixing each batch of concrete, slump, unit weight, temperature, and air content were measured as per ASTM C 143 (2008), ASTM C 138 (2008), ASTM C 1064 (2008), ASTM C231 (2008), respectively. These tests were completed in the laboratory at laboratory under standard laboratory temperature conditions.

After the fresh concrete properties were measured, fresh concrete was transferred to the temperature controlled conditions previously noted for each respective concrete batch. Three 10 cm in diameter by 20 cm tall quality control cylinders were made for each batch as per ASTM C 192 (2007). It should be noted that all cylinders with a slump greater than 2.5 cm (1 in.) were consolidated by rodding, while cylinders with a slump of 2.5 cm or less were consolidated with the use of a vibrating table. Plastic caps were put on each cylinder to protect against moisture loss and the cylinders were left in their respective environments for 24 hours. After 24 hours, cylinders were removed from the chamber and moved to a curing environment. For the remainder of the curing and hardening process, cylinders were either stored in a moist curing room at 23 °C and 100% relative humidity, or in temperature controlled lime curing tanks (see Figure 4.12). For a summary of quality control cylinder curing conditions see

Table 4.3 and Table 4.4. It should be noted that all testing for batches not included in

Table 4.3 was performed using concrete from the 0.10 cubic meter batch, therefore no 0.04 cubic meter batch was produced.

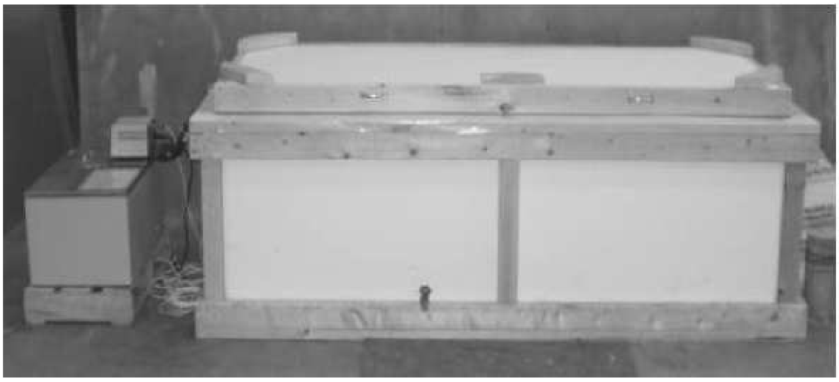


Figure 4.12: Temperature-controlled curing tank

Table 4.3: Curing conditions for quality control cylinders for 0.04-m^3 batches

Mixture		**First 24 Hours**		**1 Day- 28 Days**	
		Location	**Temperature**	**Location**	**Temperature**
Control	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
30% C	Cold	TCC	10 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
30% F	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
50% Slag	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
w/c 0.36	Cold	TCC	10 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
w/c 0.48	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C

TCC = Temperature-Controlled Chamber
TCLT = Temperature-Controlled Lime Tank
Lab = Laboratory
MCR = Moist-Cured Room

Temperature-controlled water tanks were also utilized in order to control the curing temperature of moist-cured quality control cylinders. To control the temperature of the lime-saturated water inside each tank, a heating and cooling water circulator was attached to 1.3-cm copper tubing inside each (see Figure 4.12). The temperature of the curing tank varied depending on the concrete batch tested. For hot, room temperature, and cold batches, the tank was set to 40 °C, 23 °C, and 10 °C respectively.

Table 4.4: Curing conditions for quality control cylinders from 0.10-m^3 batches

Mixture		First 24 Hours		1 Day- 28 Days	
		Location	Temperature	Location	Temperature
Control	Cold	TCC	10 °C	TCLT	5 °C
	Room	Lab	23 °C	TCLT	23 °C
	Hot	TCC	35 °C	TCLT	40 °C
30% C	Cold	TCC	10 °C	TCLT	5 °C
	Room	Lab	23 °C	TCLT	23 °C
	Hot	TCC	35 °C	TCLT	40 °C
30% F	Cold	TCC	10 °C	TCLT	5 °C
	Room	Lab	23 °C	TCLT	23 °C
	Hot	TCC	35 °C	TCLT	40 °C
50% Slag	Cold	TCC	10 °C	TCLT	5 °C
	Room	Lab	23 °C	TCLT	23 °C
	Hot	TCC	35 °C	TCLT	40 °C
w/c 0.36	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
w/c 0.40	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C
w/c 0.48	Cold	TCC	10 °C	MCR	23 °C
	Room	Lab	23 °C	MCR	23 °C
	Hot	TCC	35 °C	MCR	23 °C

TCC = Temperature-Controlled Chamber *Lab = Laboratory*
TCLT = Temperature-Controlled Lime Tank *MCR = Moist-Cured Room*

Quality control cylinders were tested in compression as per ASTM C 39 (2004) at 28 days. In addition to these standard 28-day strengths, cylinders from thermal testing

(Calorimeter II) were tested for early-age compressive strength (See Section 4.4.2.2 for details).

4.5 Raw Materials

A single source of each material was used throughout the project in order to minimize variations in testing due to differences in material properties. The source and properties of all cement, SCMs, aggregates, and admixtures are discussed in the following sections.

4.5.1 Cement and Supplementary Cementing Materials

The cement used for all mixtures was a Type I cement manufactured by Lafarge North America in Calera, Alabama. Its chemical composition was determined by X-Ray Fluorescence and chemical compounds were computed as per ASTM C 150 – 08. Additionally, its free lime content and Blaine specific surface area (SSA) were determined. A summary of its chemical composition, specific gravity, and fineness can be seen in Table 4.5.

Several of the mixtures tested contained supplementary cementing materials in order to evaluate the setting methods across a wide range of concrete mixtures. Both Class C and Class F fly ash were utilized as well as Grade 120 ground-granulated blast-furnace slag (GGBF slag). The Class C fly ash used was produced by Holcim (US) Inc. in Quinton, Alabama. The Class F fly ash was obtained from Boral Material Technologies in Stilesboro, Georgia. The Grade 120 GGBF slag was distributed by Holcim from their plant in New Orleans, Louisiana. All fly ash and slag was certified by its respective distributors as meeting all applicable ASTM specifications for concrete production. Additionally, samples of all three materials were sent to independent laboratories for chemical analysis. The results of the analysis as well as specific gravities and Blaine specific surface area can be seen in Table 4.5.

Table 4.5: Properties of cement and supplementary cementing materials

Parameter	Cement	Fly Ash		GGBF Slag
	Type I	Class C	Class F	Grade 120
Aluminum oxide, Al_2O_3 (%)	4.98	18.51	29.10	9.01
Calcium oxide, CaO (%)	63.49	23.01	1.56	35.92
Iron oxide, Fe_2O_3 (%)	3.10	5.71	7.54	0.45
Potassium oxide, K_2O (%)	0.52	-	-	-
Magnesium oxide, MgO (%)	2.87	5.26	0.94	13.24
Sodium oxide, Na_2O (%)	0.09	-	-	-
Equivalent Sodium oxide, Na_2O_{eq} (%)	0.43	2.25	2.04	0.47
Silicon dioxide, SiO_2 (%)	20.56	39.94	53.09	38.75
Sulfur trioxide, SO_3 (%)	2.61	1.56	0.10	1.69
Free CaO (%)	0.94	-	-	-
Loss on ignition, LOI (%)	1.03	0.23	2.19	-0.48
Tricalcium aluminate, C_3A (%)	7.94	-	-	-
Tetracalcium aluminoferrite, C_4AF (%)	9.45	-	-	-
Tricalcium silicate, C_3S (%)	56.75	-	-	-
Dicalcium silicate, C_2S (%)	16.15	-	-	-
Blaine SSA (m^2/kg)	377	380	230	547
Specific gravity	3.15	2.63	2.29	2.93

Values not reported in chemical analysis are denoted by -

4.5.2 Aggregates

The fine aggregate used throughout the testing program was a natural siliceous river sand from the Shorter Sand and Gravel Waugh Pit quarry located in Mt. Meigs, Alabama. Samples were taken twice during the duration of the project and gradation, bulk specific gravity, and absorption capacity were determined as per ASTM C136 (2006) and ASTM C128 (2007), respectively. Average values are summarized in Figure 4.13 and Table 4.6 . As can be seen in Figure 4.13, the fine aggregate gradation does not meet the requirements of ASTM C 33 (2003). Both the 1.18mm (no. 16) and the 600μm (no. 30) sieves retained approximately 2% by weight more aggregate than allowed by ASTM C 33 – 03. Although this is over the upper limit set by ASTM, aggregate gradations have little effect on the setting mechanism, therefore its effect was considered negligible.

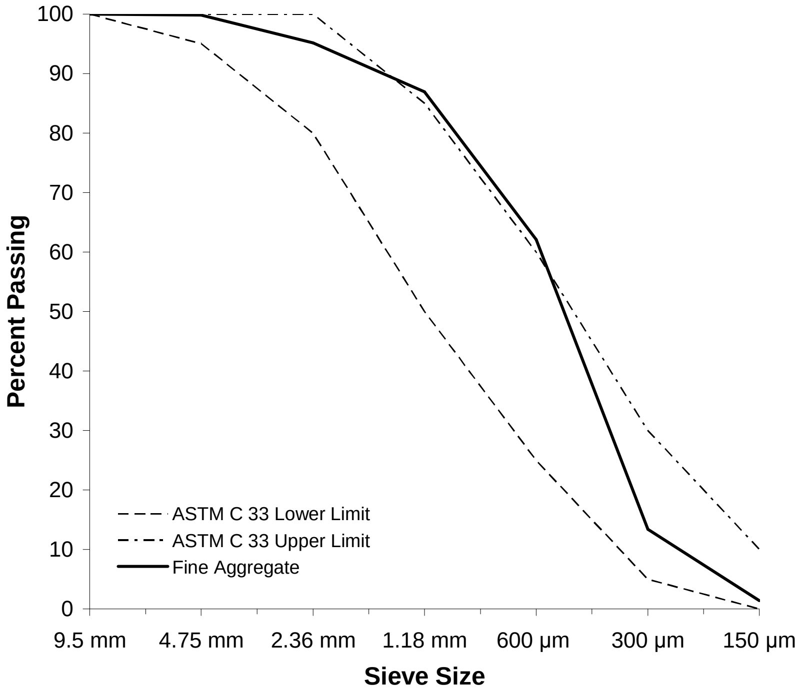


Figure 4.13: Fine aggregate gradation results

Table 4.6: Bulk specific gravity and absorption % of coarse and fine aggregate

Material	Bulk Specific Gravity	Absorption Capacity (%)
Silicious River Sand	2.60	0.90
# 57 Crushed Limestone	2.81	0.57

The coarse aggregate used was a crushed no. 57 graded limestone from APAC Mid-South's Opelika Quarry in Opelika, Alabama. Samples were taken during the duration of the project and gradation, bulk specific gravity, and absorption capacity were determined as per ASTM C136 (2006), and ASTM C127 (2007), respectively. Average values are reported in Figure 4.14 and Table 4.6 . As can be seen in Figure 4.14, the coarse aggregate met the gradation requirements of ASTM C33 (2003).

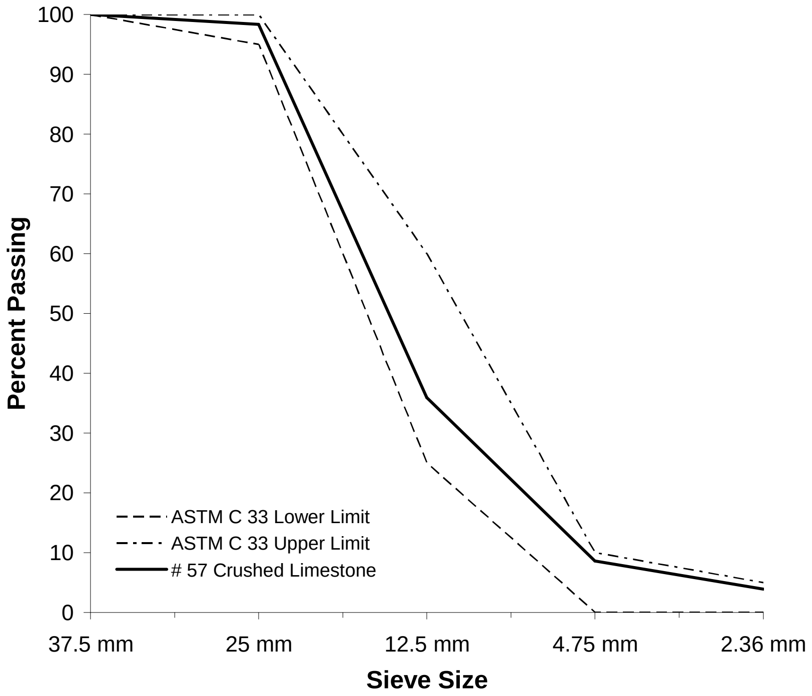


Figure 4.14: No. 57 graded crushed limestone gradation results

4.5.3 Chemical Admixtures

Two chemical admixtures were used in some of the concrete mixtures in order to maintain a desired level of workability. All admixtures were supplied by W. R. Grace and Company and were certified to meet all applicable ASTM specifications. Dosages recommended by the manufacturer were followed for all mixtures. For specific dosages for individual mixtures see Table 4.1

WRDA 64 is classified in accordance with ASTM C494 (2008) as a Type A and Type D chemical admixture, and was used in select mixtures as a water reducing and retarding admixture. MIRA 92, which meets the requirements of ASTM (2008) as a Type A and Type F admixture, was used in select mixtures as a mid-range as well as a high-range water reducing admixture. No air-entraining admixture was used in any of the mixtures due to its temperature dependent behavior.

Chapter 5

Presentation of Results

In this chapter, selected results from the testing as described in Chapter 4 are presented. Fresh concrete properties and general quality control tests results are given followed by results from both Phase I and Phase II of testing. Analysis and discussion of these results can be found in Chapter 6. The notation used in this chapter is consistent with that described in Section 4.2.

5.1 Fresh Concrete and Quality Control Test Results

As discussed in Chapter 4, standard fresh concrete properties were measured for each batch of concrete produced. The results of these fresh concrete tests for all 0.1-m^3 and 0.04-m^3 batches are summarized in Figure 5.1, Figure 5.2, Figure 5.4, Figure 5.5, Figure 5.6, and Figure 5.7.

As can be seen in Figure 5.1, and Figure 5.2, slump values ranging from 0 mm to 220 mm were measured for the concrete batches produced. As the temperature of concrete increases, the slump decreases (Daniel 2006), therefore, since admixture dosages were kept constant for all batches of a mixture, variations in slump due to temperature were expected between batches from the same mixture. The maximum variation measured between batches of a specific mixture was 170 mm. This was deemed acceptable for the purpose of this study. A slump limit of the greater of ± 25-mm or 10% was set for two batches of the same mixture produced at the same temperature for quality control purposes. All batches that were produced as both a 0.04-m^3 and a 0.10-m^3 batch met this requirement.

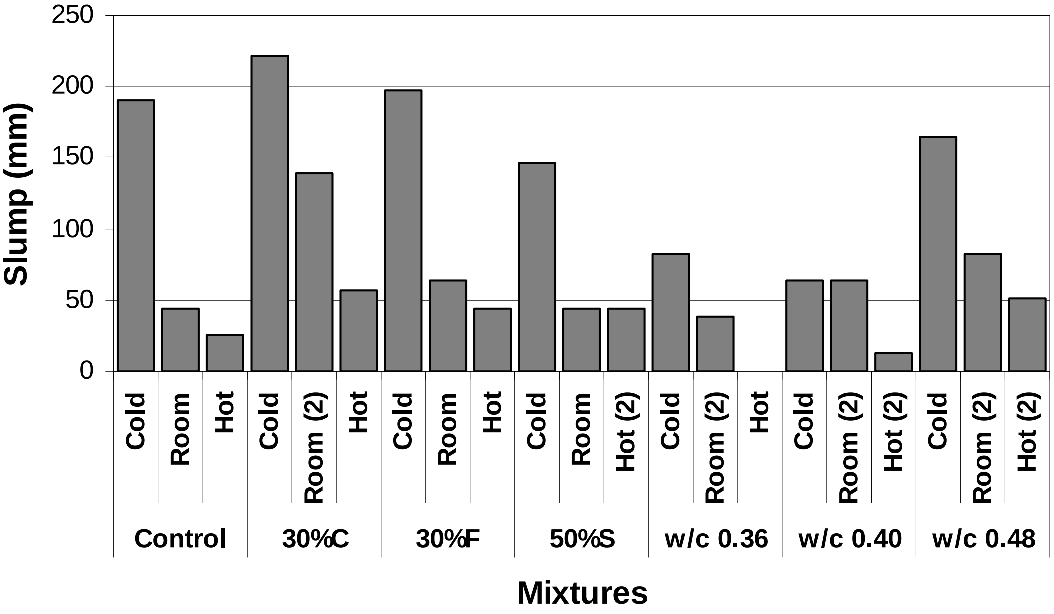


Figure 5.1: Slump for 0.1-m^3 batches

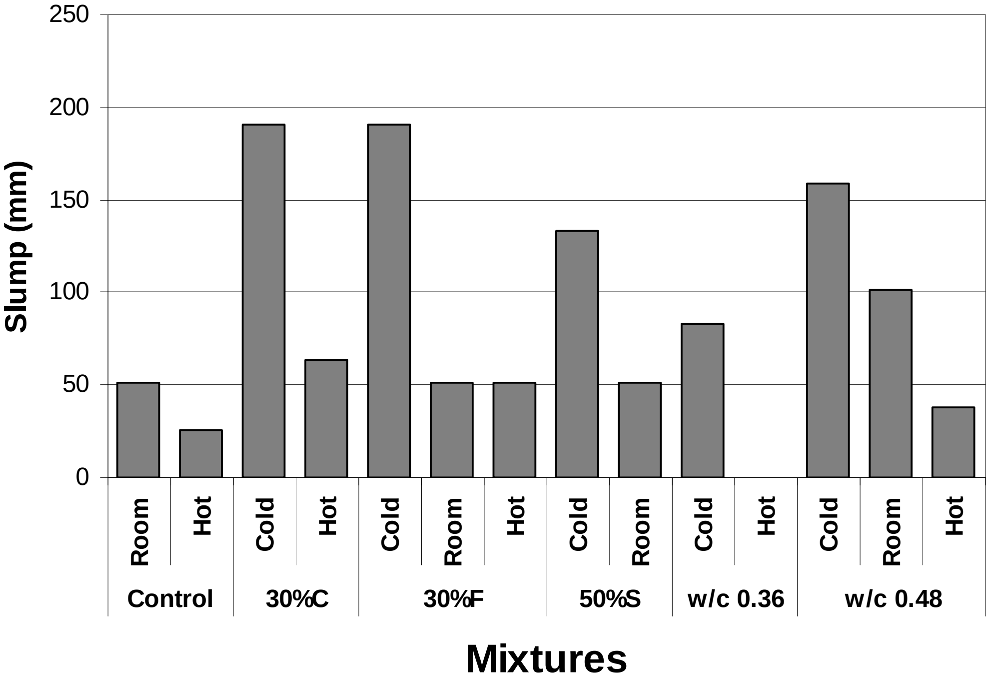


Figure 5.2: Slump for 0.04-m^3 batches

A target slump range of 0 to 200 mm was defined for all mixtures for quality control purposes, and special provisions were taken for any concrete batch with a slump value less than 25 mm or greater than 200 mm. Cylinders for mixtures with slump values of 25 mm or less were consolidated by vibration instead of rodding, but were otherwise tested as normal. For mixtures with high slump values (greater than 200 mm), compressive strength cylinders were visually inspected for signs of segregation. As can be seen in Figure 5.3, 30 % C – Cold showed no visible signs of segregation, therefore its constancy was deemed acceptable.

Figure 5.3: 30 % C-Cold Strength specimen which was visually inspected for signs of segregation

Air content and unit weight, as measured for each batch tested, can be seen in Figure 5.4, Figure 5.5, Figure 5.6, and Figure 5.7. The total air content varied by no more than 1.5 percent for all batches of a specific mixture. This range was deemed acceptable for the purpose of this study. Unit weight for all batches of a specific mixture varied by no more than 50 kg/m^3, which was deemed acceptable for the purpose of this study. Although the unit weight of the w/c 0.48 – Hot (2) batch appears higher than one would expect when compared the other batches for that particular mixture, the slump-value and compressive strength for this batch are within acceptable ranges, therefore, the propertied were deemed acceptable for the purpose of this study.

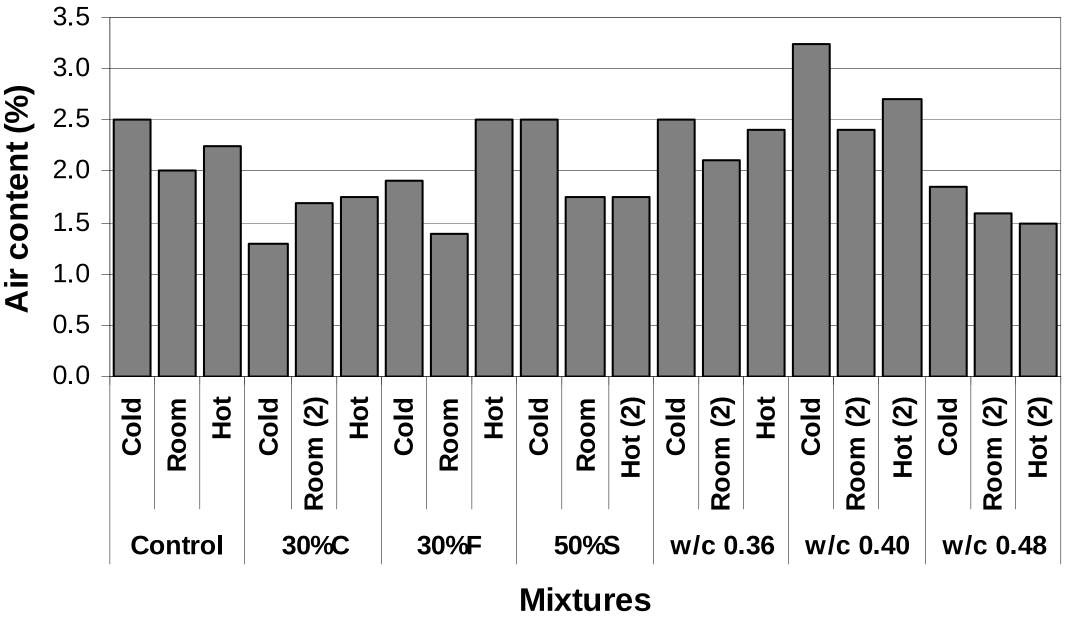


Figure 5.4: Air Content for 0.1-m^3 batches

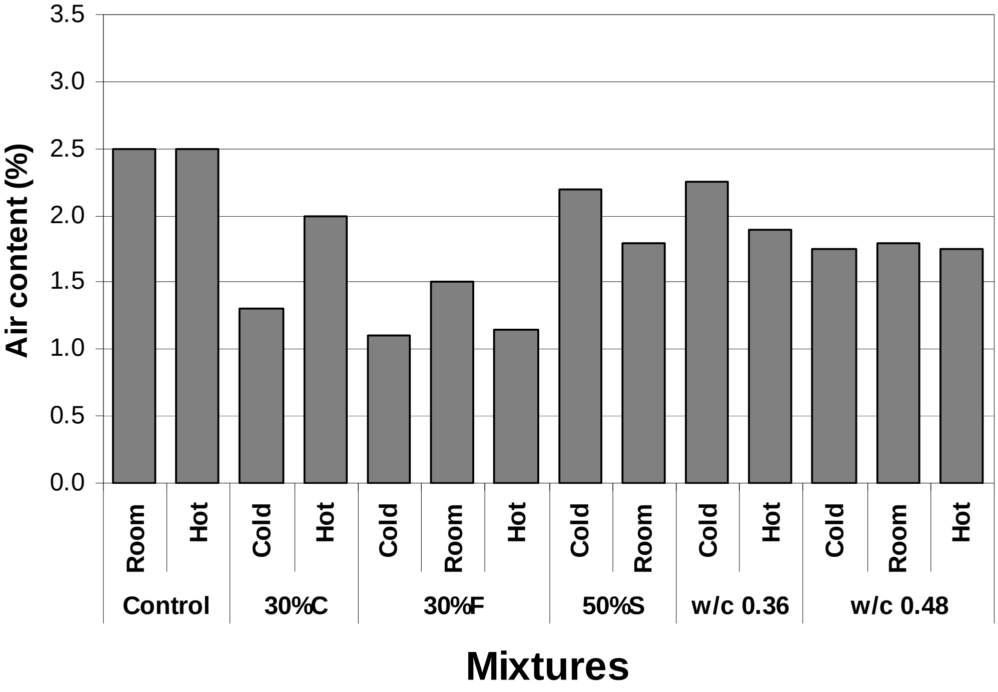


Figure 5.5: Air Content for 0.04-m^3 batches

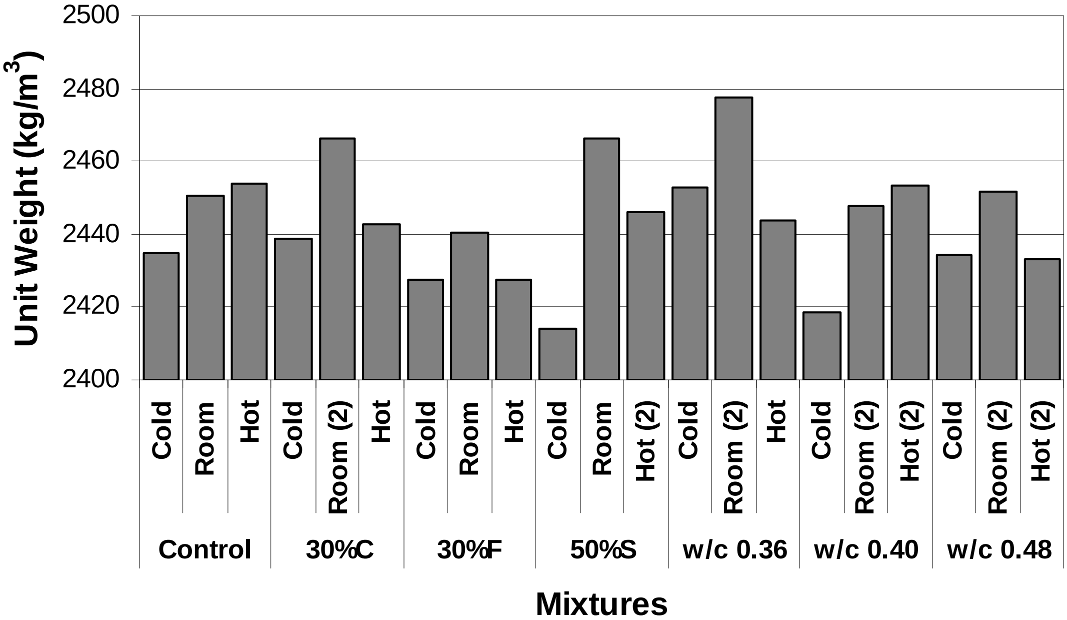


Figure 5.6: Unit Weight for 0.10-m^3 batches

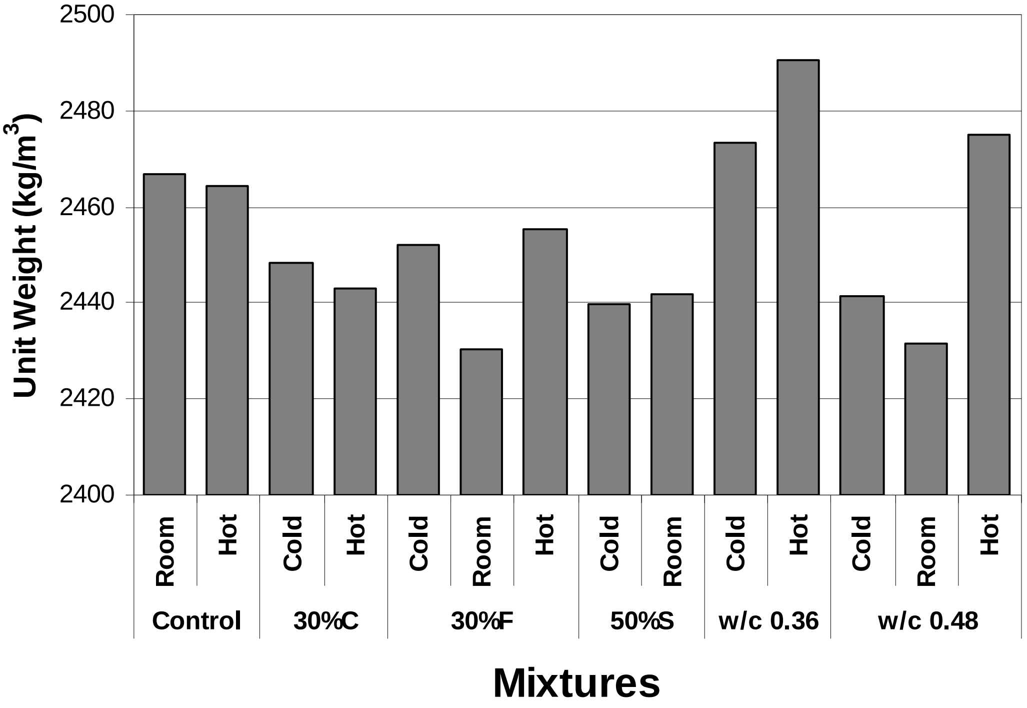


Figure 5.7: Unit Weight for 0.04-m^3 batches

Hardened concrete properties were also measured for each batch as a means of quality control. As can be seen in Figure 5.8 and Figure 5.9, all concrete batches tested had a 28-day compressive strength of no less than 30 MPa. It should be noted that curing methods and temperatures for concrete strength specimens varied between batches of concrete. For a summary of curing conditions for all 28-day compressive strength cylinders see Table 4.3 and Table 4.4. The 28-day concrete strengths measured for each concrete batch were deemed acceptable for the purposes of this study.

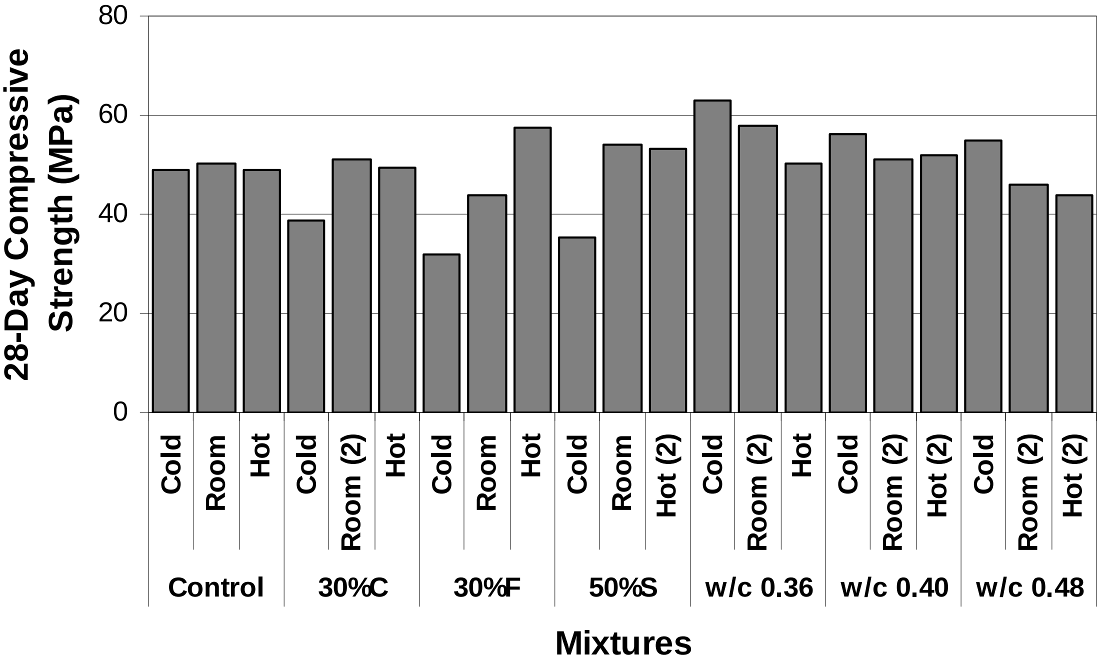


Figure 5.8: 28-Day compressive strength for 0.10-m^3 batches

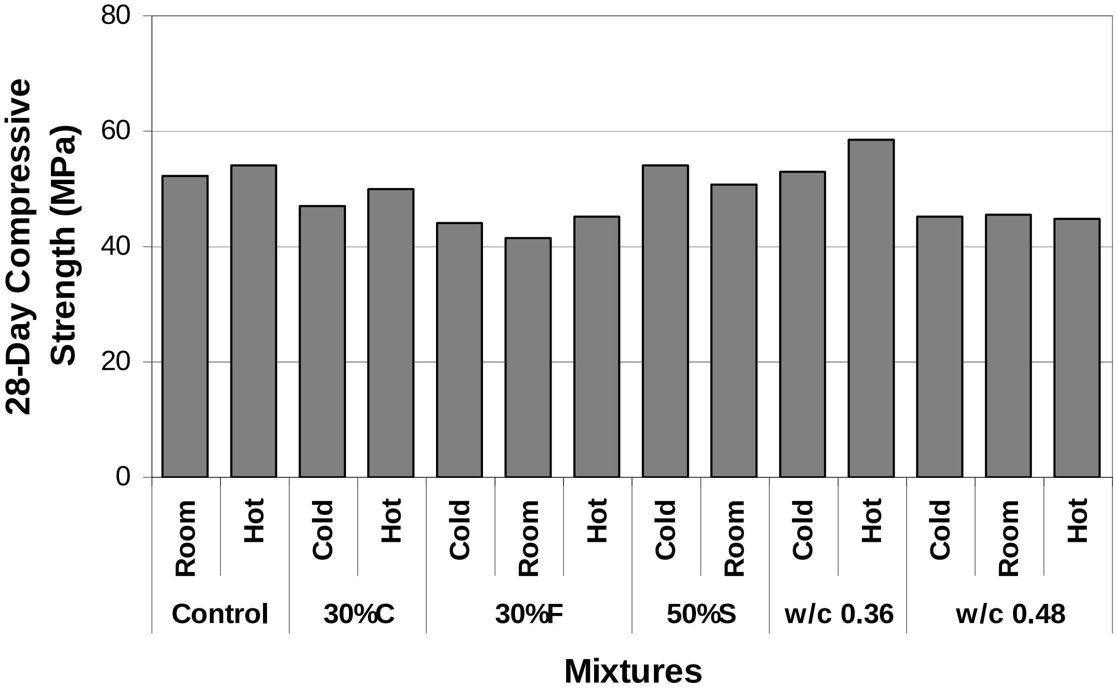


Figure 5.9: 28-Day compressive strength for 0.04-m^3 batches

Early-age compressive strength results were also obtained with the use of cylinders from semi-adiabatic calorimeter testing. In order to demonstrate the progression of strength with concrete for samples hydrating at various temperatures, strength data were plotted versus time for select mixtures. Results for selected mixtures can be seen in Figure 5.10.

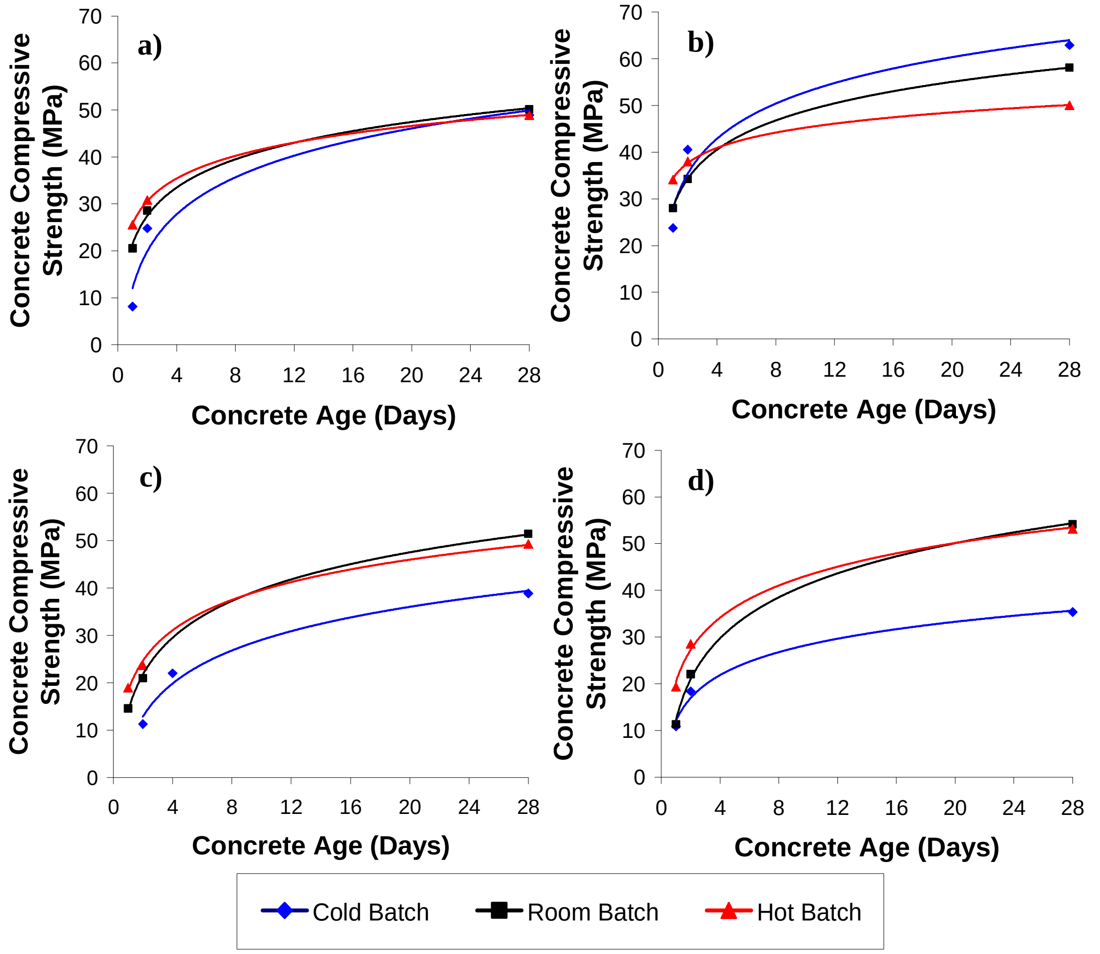


Figure 5.10: Compressive strength versus age plots for a) Control, b) w/c 0.36, c) 30 % C, and d) 50% Slag

It should be noted that the early-age compressive strength specimens were cured within the semi-adiabatic calorimeters while the 28-day specimens were cured as noted in Tables 4.2 and 4.3. Concrete compressive strength data obtained as per ASTM C 39 (2004) as well as strength versus time plots for all concrete batches tested is presented in Appendix C.

5.2 Phase I Test Results

In Phase I of testing, the temperature sensitivity of each mixture was determined by the calculation of the activation energy, E, that best characterizes the effect of temperature on the mixture's setting and hydration behavior. Results from both isothermal calorimetry and penetration resistance testing can be seen in the following sections. Due to similarities between mixture results, only the results of selected mixtures will be graphically presented in the following section. In order to demonstrate results for a variety of materials and w/cm, the following mixtures will be presented: Control, w/c 0.36, 30 %C, and 50% Slag. Further details concerning these methods and analytical procedure for the calculation of E are covered in detail in Chapter 6.

5.2.1 Isothermal Calorimeter Results

Results from isothermal calorimetry testing for select mixtures can be seen in Figure 5.12. Each mortar sample was tested for 72 hours, or until additional heat evolution was deemed insignificant. Data were normalized per gram of cement (g_{cem}) tested in order to allow comparison of varying sample sizes. Results from all three batches of concrete (Hot, Room, and Cold) are plotted on the same graph for comparison purposes. It should be noted that data markers shown in all graphs in this chapter are, unless otherwise noted, for distinguishing between data series only, and do not represent actual data points. Actual data points were collected at one-minute intervals.

Temperature effects on a mixture's isothermal temperature profile can be seen in Figure 5.11 and Figure 5.12. Due to the fact that the specimens are held at a constant temperature throughout testing, the variation in the rate of heat evolution that can be seen at various temperatures is caused solely by the variation in the rate of hydration due to the various testing temperatures.

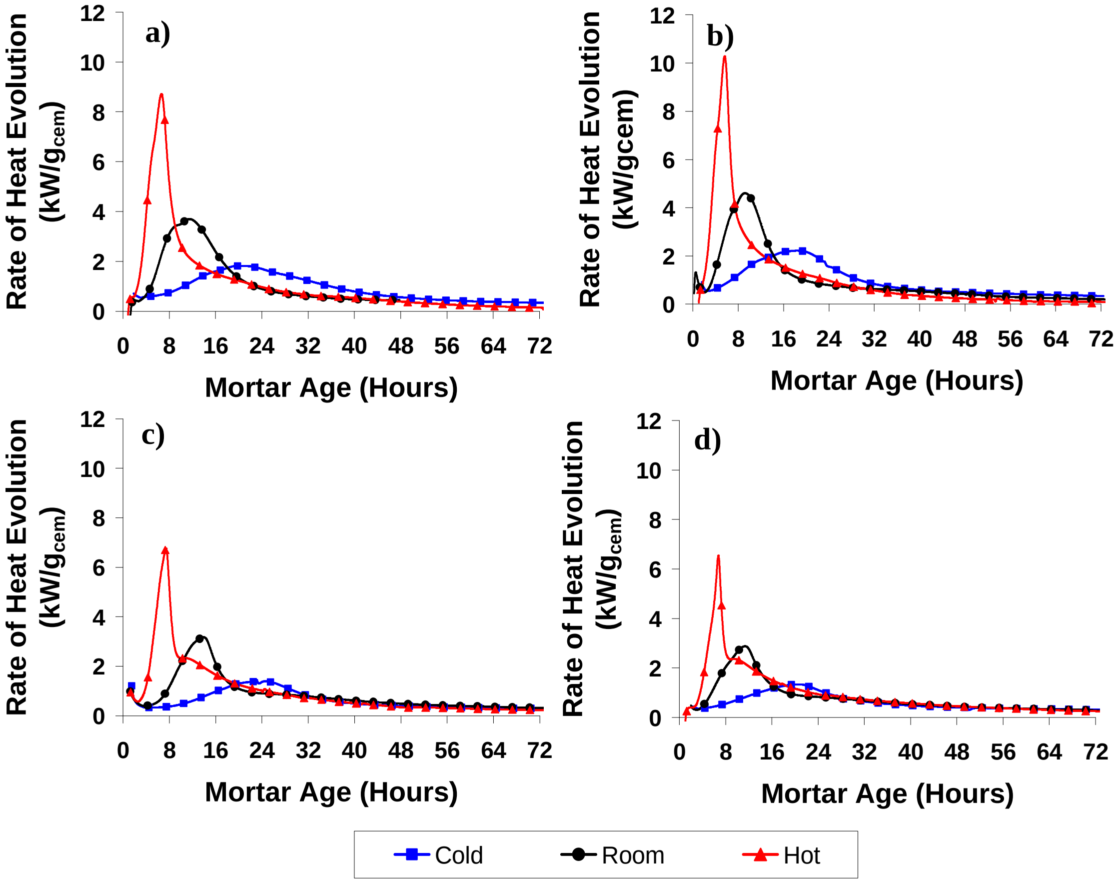


Figure 5.11: Rate of heat evolution from Isothermal Calorimetry for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

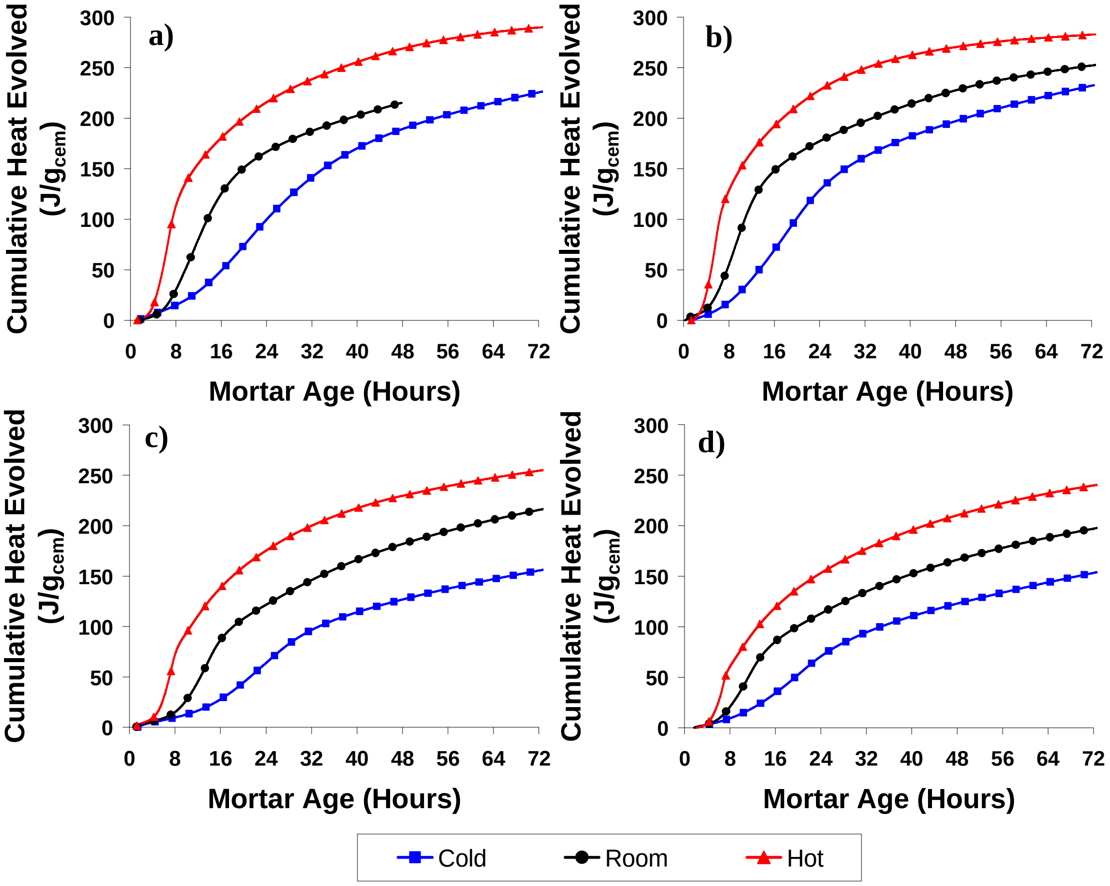


Figure 5.12: Cumulative heat evolution from Isothermal Calorimetry for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

The cumulative heat evolution of a hydrating sample has been directly linked to its degree of hydration (see Section 2.1.3.1). It should be noted that, although the batches produced at higher temperatures seem to reach a higher final degree of hydration (indicated by larger amounts of heat evolved in Figure 5.12), if left in the device for an acceptable amount of time, all three samples should reach approximately the same degree of heat evolution (Powers and Brownyard 1948). As can be seen in Figure 5.11, the primary difference in the specimens produced at different temperatures is not the final degree of heat evolution, but the rate of heat evolution (see Section 2.1.3.2).

It should also be noted that the isothermal cumulative heat evolved and rate of heat evolution is dependent upon not only the curing temperature but the mixture proportions of a sample. As can be seen in Figure 5.11, the rate of heat evolution of each mixture at the hot and cold temperatures as compared to the room temperature varies for each mixture. This is also evident in Figure 5.12. It should be noted that there is a much greater distance between curves in Figure 5.12c than there is in Figure 5.12d, which suggests that the isothermal response of the 30 % C mixture is more sensitive to temperature effects than the 50% slag mixture. As is evident in Figure 5.11 and Figure 5.12, both mixture proportions and addition of SCMs have an effect on the temperature sensitivity of the mixture in question; therefore E values were calculated independently for each mixture.

5.2.2 Penetration Resistance Testing

In addition to isothermal methods, E was also calculated with the use of penetration resistance data collected at various temperatures for each concrete mixture. (Details regarding the analysis of penetration resistance data to obtain E can be found in Chapter 6.) Results from ASTM C 403 (2008) for select mixtures can be seen in Figure 5.13. Penetration resistance was plotted versus concrete age, and a regression analysis with a power function was performed as per ASTM C 403 (2008). Calculated initial and final set points can be seen in Figure 5.13. All three batches of concrete (Hot, Room, and

Cold) are plotted on the same graph for comparison purposes. Data markers shown represent actual penetration resistance readings while the regression analysis is represented by a solid line. For a summary of initial and final set times obtained for all batches tested as per ASTM C 403 (2008) see Table 5.1.

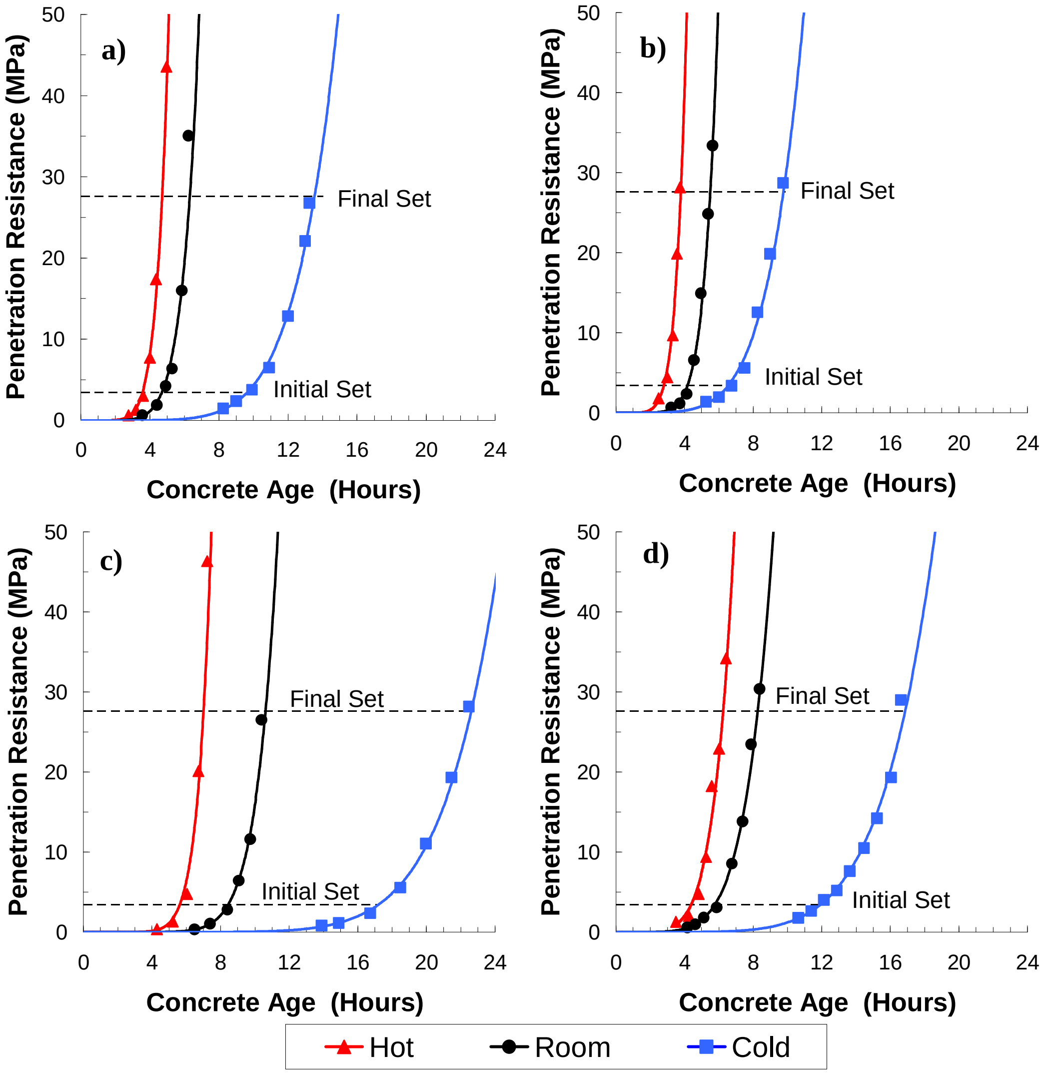


Figure 5.13: Penetration resistance plots as per ASTM C 403 (2008) for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

Table 5.1: Initial and final set times as per ASTM C 403 (2008)

Mixture		**Initial Set (Hours)**	**Final Set (Hours)**
Control	Cold	9.35	14.02
	Room	4.70	6.31
	Hot	3.57	4.71
30% C	Cold	17.22	22.61
	Room	8.43	10.51
	Hot	5.63	7.01
30% F	Cold	11.43	15.72
	Room	6.72	9.30
	Hot	4.21	5.35
50% Slag	Cold	11.87	16.96
	Room	5.78	8.28
	Hot	4.74	6.07
w/c 0.36	Cold	6.48	9.90
	Room	4.18	5.50
	Hot	2.80	3.79
w/c 0.40	Cold	6.82	10.34
	Room	3.95	5.21
	Hot	2.68	3.47
w/c 0.48	Cold	7.89	9.74
	Room	3.94	5.55
	Hot	3.05	4.05

The effect of both temperature and mixture proportions on setting can be observed in Figure 5.13 and Table 5.1. As can be seen in Figure 5.13 and Table 5.1, and as discussed in Chapter 2, the rate of setting is highly dependent on both the composition of a concrete mixture and the curing temperature. Hot batches reach initial and final set much faster than room temperature and cold batches, as do mixtures with a low w/cm ratio. Also, a replacement of a portion of cement with either fly ash or GGBF slag appears to delay the setting process further. For setting results for all batches see Appendix D.

It should also be noted that the setting times observed for room temperature and hot batches are much closer together than those of the cold batches. This can be explained by examining the measured temperature of each mortar sample during testing. As discussed in Section 2.3.3, the rate of hydration and setting of concrete is highly dependent on curing temperature. Therefore, since the measured temperatures of mortar samples from the hot and room temperature batches are much closer together than those of the room temperature and cold mortar batches (see Figure 5.15), it should follow that the differences in setting times observed between a hot and room temperature batch are significantly less than the difference noted between room temperature and cold batches.

In order to calculate activation energy from penetration resistance data, the temperature history of the individual mortar samples must also be known. As discussed in Chapter 4, two temperature probes were used for each mortar specimen in order to examine the effect of ambient temperature and heat loss/gain on mortar temperature. Raw data from both temperature probes are in Figure 5.14.

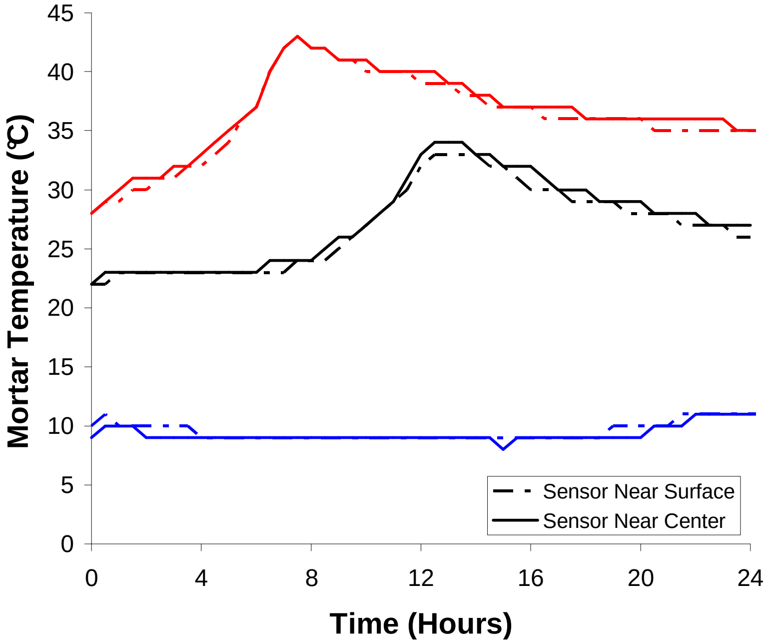


Figure 5.14: ASTM c 403 (2008) mortar temperature results from two sensor locations for all three batch temperatures (30 %C)

As can be seen in Figure 5.14, the location of the temperature probe had little effect on the temperature history recorded at all three temperatures. In general, the temperature difference was less than 1 °C. It should be noted that this difference is similar to the accuracy of the thermocouple used to measure these temperatures. It was determined that, for the purpose of this project, the effect of the sensor location was negligible, and data from the near surface probe was used for all analysis.

Sample mortar temperature histories can be seen in Figure 5.15. Mortar temperatures for each batch of concrete tested are presented in Appendix D. To account for the change in mortar temperature during the sieving process, a linear extrapolation was added to each data set from the first temperature recorded to the initial batch temperature. As can be seen in Figure 5.15, this temperature change was often significant for hot batches. This rapid loss of heat is thought to be due to the low relative humidity in the temperature-controlled chamber yielding high heat loss due to evaporation during the sieving of mortar specimens.

As noted previously, the measured temperature histories are much more similar for samples obtained from hot and room temperature batches than for those from room temperature and cold batches. This is thought to be due to the effect of ambient temperature on the samples. A hot sample will generate heat more quickly than it can be lost to the surrounding temperature of the air. A cold sample hydrates more slowly, therefore it generates much less, thus only a slight temperature change, if any, occurs. The rapid heat generation in a hot sample results in a higher sample temperature, therefore more heat generation and faster setting. A cold setting sample may experience a small heat peak due to hydration, but will generally lose heat faster than it can produce it resulting in a temperature profile that resembles the ambient temperature during testing. For examples of this effect, see the recorded mortar temperatures presented in Figure 5.15.

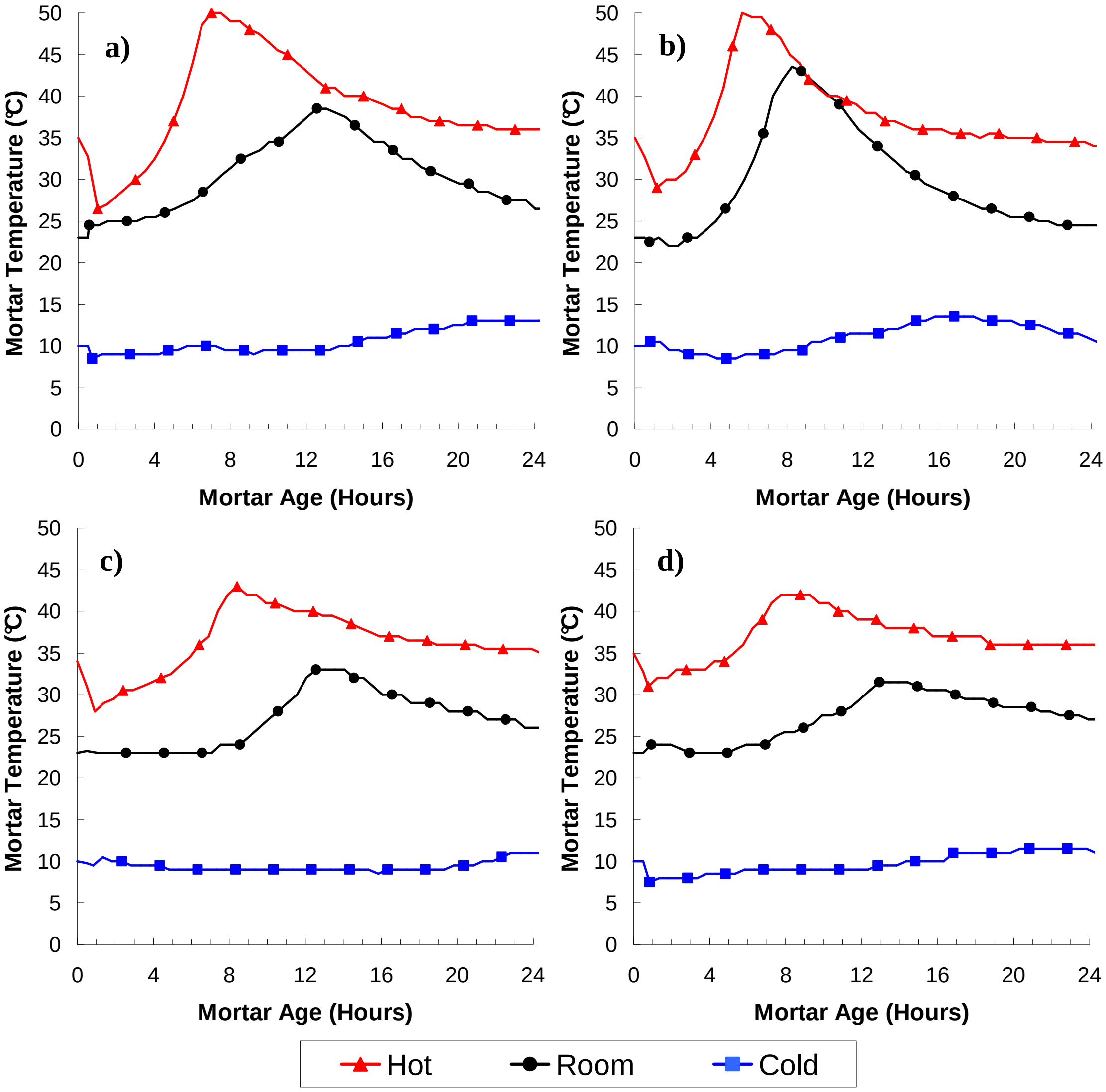


Figure 5.15: Mortar temperature plots from penetration resistance mortar containers for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

5.3 Phase II Test Results

Phase II of testing consisted of the determination of setting times for each batch of concrete by both penetration resistance and thermal methods, and the evaluation of thermal methods as compared to ASTM C 403 (2008). In this section, results from all Phase II testing are presented. Only select mixtures will be shown in figures included in the following sub sections. To demonstrate the range of results due to the varying of w/cm and addition of SCMs, results for the following mixtures will be presented along with the control mixture: w/c 0.36, 30 %C and 50% Slag.

5.3.1 Penetration Resistance Setting Results

Penetration resistance data included in Phase I of testing were used for Phase II of the testing program as well. For sample results see Figure 5.13 and Table 5.1.

5.3.2 Semi-Adiabatic Calorimetry Data

Results from semi-adiabatic calorimeter tests can be seen in Figure 5.16 (Calorimeter I), and Figure 5.17 (Calorimeter II). Results from all three batches of concrete (Hot, Room, and Cold) are plotted on the same graph for comparison purposes. Each concrete sample was tested for approximately 144 hours, or until the temperature profile was determined to have reached ambient conditions. It should be noted that, due to device malfunction, Calorimeter II results are not available for 50% Slag – Hot and 30 %F – Hot. Semi-adiabatic calorimetry test results for all batches of concrete tested as part of this testing program can be seen in Appendix E.

Important properties to note from the Figure 5.16 and Figure 5.17 include the maximum temperatures recorded and the rate of hydration during the acceleration phase, and the effects of both mixture properties and temperature on semi-adiabatic calorimetry temperature profiles. As can be seen in Figure 5.16 and Figure 5.17, mixtures with a lower w/cm achieve higher temperatures than those with a higher w/cm, and reach these peak temperatures earlier. The presense of SCMs appears to lower both the maximum temperature reached and the maximum rate of hydration of a specimen. It should be noted that the effect of mixture proportions are not as evident in the data examined in this research program due to the use of various chemical admixtures in several of the mixtures examined in order to obtain acceptable levels of workability. The range of behavior observed may have been greater if no admixtures were used.

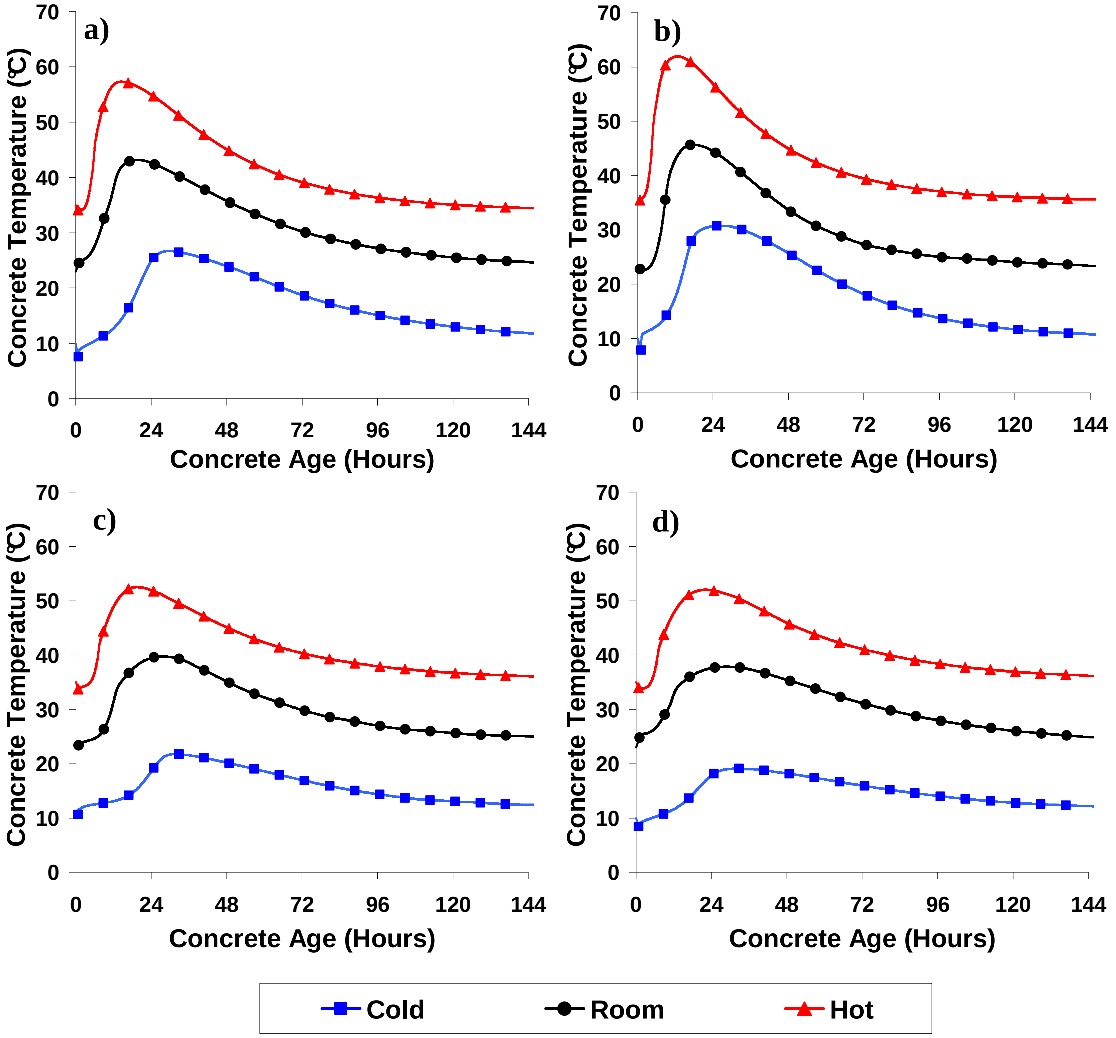


Figure 5.16: Results from Calorimeter I for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

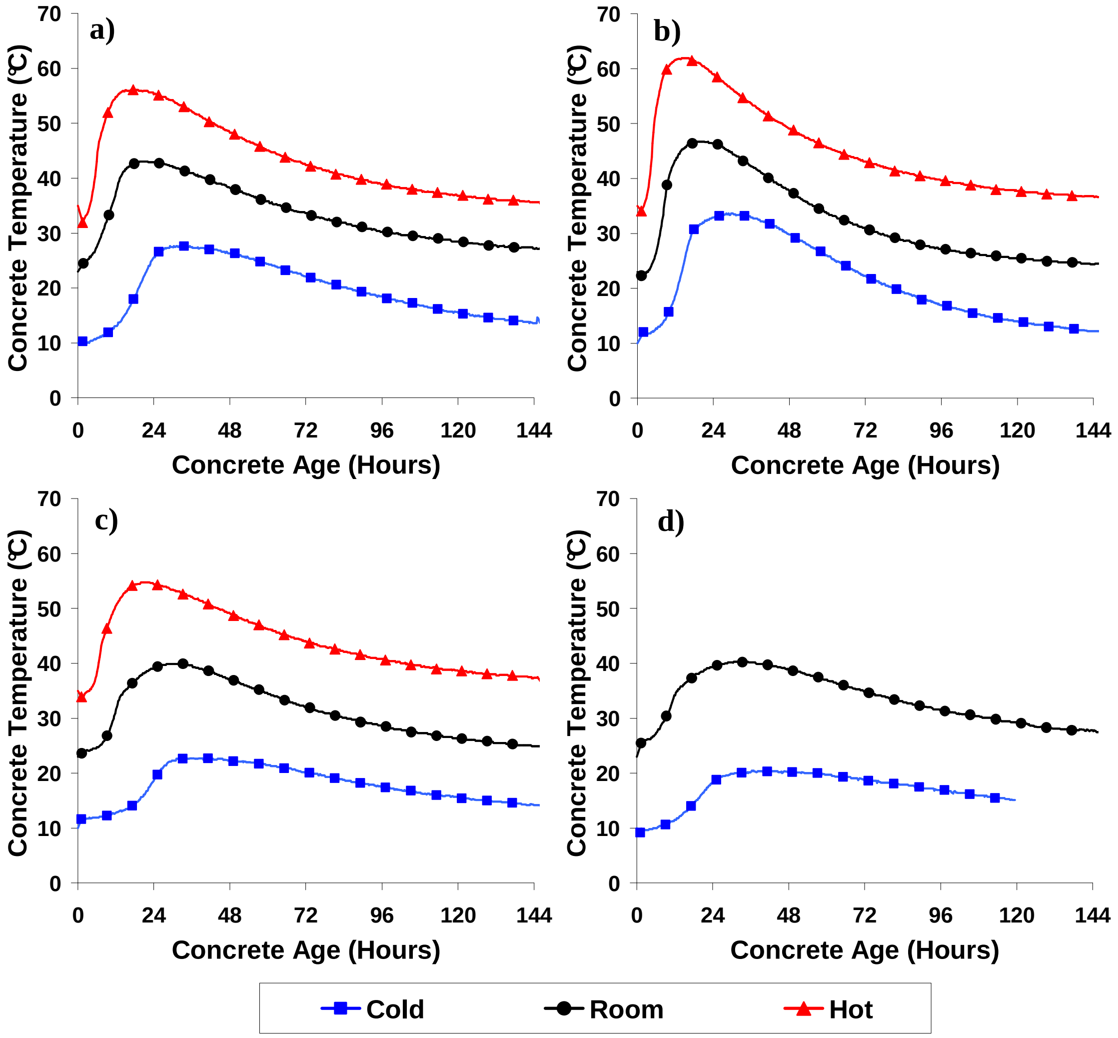


Figure 5.17: Results from Calorimeter II for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

The effect of curing temperature on the temperature profiles is also evident from Figure 5.16, and Figure 5.17. Batches that start out at a higher fresh concrete temperature reach a peak temperature much faster than those at lower temperatures and generally have a greater temperature rise. While the former of these observations is due to the temperature dependent nature of the reactions involved in cement hydration, the latter has more to do with time dependent losses during the hydration process. More information on these losses will be presented in Chapter 6.

It should also be noted that there is very little difference in the data obtained from Calorimeter I and Calorimeter II for a given concrete mixture. Both the general shape and the maximum temperatures recorded are similar for each batch tested, even though the devices vary both in insulation properties and in concrete sample size. The differences in the two devices are discussed in more detail in the following section and in Chapter 6.

5.3.3 Differences in Semi-Adiabatic Calorimeter I and II

Unlike the devices discussed in Chapter 3, true semi-adiabatic devices that meet the loss requirement given RILEM TC 119 – TCE (1999) and can be adjusted for losses, have minimal differences in thermal response. Once thermal data have been adjusted for both losses and maturity (See Section 6.2.1) overall temperature profiles are (for the purposes of this analysis) identical. The main differences noted during testing between results from Calorimeter I and Calorimeter II involved both the sampling rate and the location of the concrete temperature probe.

Due to the lower sampling rate, (1 record every 15 minutes for Calorimeter II as compared to one record every minute for Calorimeter I) results from Calorimeter II had a greater variation from point to point. Although unnoticeable when viewing the raw data, this difference in sampling rate became problematic during analysis, especially when determining the rate of temperature gain.

The effect of the location of the temperature sensor within the device was the other main difference in the data obtained from Calorimeter I and Calorimeter II. Calorimeter II was outfitted with temperature probe that was inserted within the concrete specimen during testing, while Calorimeter I made use of a probe attached to a metal plate below the specimen (see Chapter 4). Even though calorimeters were placed in their respective testing environments well before testing, due to losses or gains in temperature that occurred during sample preparation, the initial concrete temperature upon the start of testing did not always match that of the testing device.

For Calorimeter II this was not a substantial problem. Although there were still small equilibrium effects due to the sample and device being at different temperatures, the temperature recorded by the device were representative of the actual concrete temperature. For Calorimeter I this was not necessarily the case. Since its temperature probe was not inserted directly into the concrete, when there was a temperature difference between the device and sample temperature, there was also a slight error in concrete temperature recorded. This is because this probe will initially record temperatures somewhere between concrete and device temperature until a state of thermal equilibrium is reached between the concrete and the wall of the calorimeter. Examples of this effect can be seen in Figure 5.18. A significant temperature differences can be noted between results obtained from Calorimeter I and Calorimeter II for both the hot and cold batches for the mixtures presented. For more details on this effect, see Section 6.2.3.5.

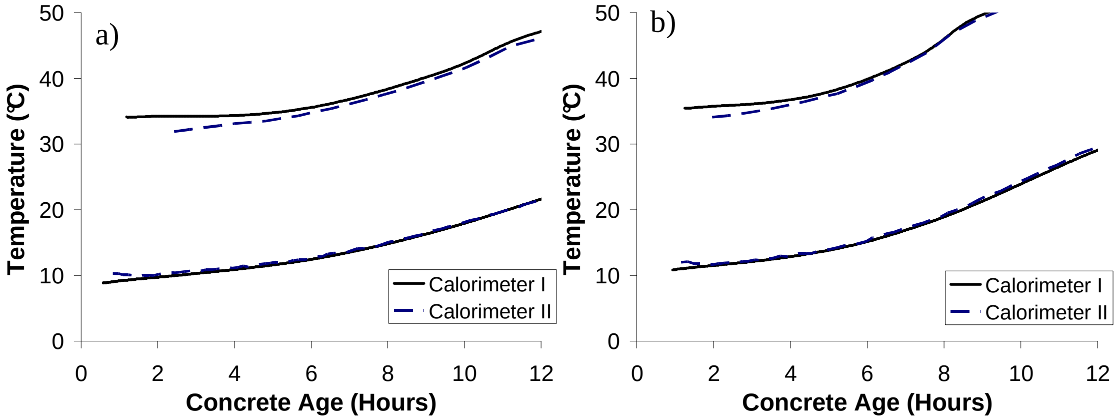


Figure 5.18: Temperature date from Calorimeter I and II for a) Control (cold and Hot), and b) w/c 0.36 (Cold and Hot) and

5.4 Summary

In this section, results from the seven concrete mixtures examined in this study were discussed. Quality control testing was reported, and it was determined that all batches tested consisted of good quality concrete. Sample results from isothermal calorimetry and penetration resistance testing were also presented in preparation for determination of temperature sensitivity, and trends were noted. Results were also given for semi-adiabatic calorimetry tests, and significant trends were discussed. Major findings presented in this chapter are as follows:

- All batches tested met all quality control guidelines and were deemed acceptable for the purpose of this testing program.
- Both curing temperature and mixture proportions have a significant impact on the rate of penetration resistance development, the rate of isothermal heat development, and the rate of semi-adiabatic temperature rise of a given specimen.

- The shape of temperature histories measured from ASTM C 403 (2008) mortar samples varies with temperature. In general, Hot and Room batch samples have a similar temperature profile shape, where Cold batch samples show little, if any, temperature rise during testing.
- The location of the temperature sensor within semi-adiabatic calorimeters has a significant effect on initial temperature data points collected and may be a significant source of error for thermal testing.

Chapter 6

Analysis and Discussion of Results

Results from both Phase I and Phase II of testing are presented in Chapter 5. In this chapter, the analysis of these results in order to obtain setting times and the evaluation of these setting measurements as compared to ASTM C 403 (2008) can be found. The process of determining the temperature sensitivity of each mixture is presented, as well as various methods for determining setting of concrete through thermal methods. Various thermal methods to determine setting times are evaluated and discussed and conclusions are drawn as to their effectiveness. Finally, the sensitivity of these thermal methods to various activation energy values of each mixture is examined.

6.1 Phase I – Determination of Temperature Sensitivity

The use of maturity as the basis of comparison for temperature dependent concrete properties is important when comparing results to avoid inaccurate correlations (Carino 2004). The need to account for the effect of temperature on behavior becomes important as the temperature of a specimen is further away from the reference temperature (23 °C). Therefore, in comparing setting results obtained through methods with vastly different temperature histories, it is very important to account for the effects of temperature.

The FHP maturity function (see Equation 2.11) is widely considered the most accurate maturity formulation for the prediction of temperature dependent concrete properties (Carino 2004). In light of this, the FHP maturity formulation was used for all maturity calculations in this project. While other formulations, such as the Nurse-Saul maturity function, assume a linear relationship between concrete age and maturity, the FHP function treats this relationship as a nonlinear function that is dependent on properties of a given mixture. To accurately approximate the effects of temperature using this function, an accurate determination of the temperature sensitivity of a given mixture, defined by a mixture's activation energy, E, is necessary. Schindler (2004a) proposed the use of the development of degree of hydration, determined through isothermal calorimetry, to accurately determine E for a concrete mixture. Pinto and Hover (1999) demonstrated the use of penetration resistance data obtained as per ASTM C 403 (2008) to calculate the apparent activation energy of a concrete mixture. For this project, a method similar to that proposed by Pinto and Hover (1999) was used and the results were validated with the use of isothermal calorimetry. Details concerning both methods of calculating E will be discussed in the following sections.

6.1.1 Isothermal Calorimetry Based Approach

Schindler (2004a) proposed a method of calculating of calculating activation energy through isothermal calorimetry. He demonstrated a link between the hydration time parameter (τ), in the degree of hydration formulation used by Freiesleben, Hansen, and Petersen (1984), and the rate conversion factor used to derive the FHP function. It was shown that when this theory is applied to isothermal results at varying temperatures, results can be used to define the activation energy of a mixture. This procedure was applied to isothermal results from each mixture in the testing program to obtain E.

Three isothermal tests were performed on a mortar specimen sieved from each concrete sample, one at 23 °C, one at 5 °C, and one at 35 °C. The maximum heat evolution recorded (usually from the Hot batch) was assumed as a degree of hydration of 100%, and all other batches were adjusted accordingly. For each temperature, the Freiesleben, Hansen, and Petersen degree of hydration formula presented in Chapter 2 (Equation 2.8) was fit to the isothermal data by a least-squares regression analysis. Schindler (2004a) determined that the parameters α_{ult} (ultimate degree of hydration) and β (the hydration shape parameter) could be considered independent of temperature effects. Therefore, α_{ult} and β were treated as constants for a given mixture, and only τ (the hydration time parameter) was varied between mixture temperatures. All batches considered had regression correlation coefficients of at least 0.98. For examples of hydration curves fit to isothermal data see Figure 6.1. Data markers shown do not represent all data points measured (one point per hour was plotted due to the small time step used for measurement). Solid lines represent the best-fit curve obtained from regression analysis.

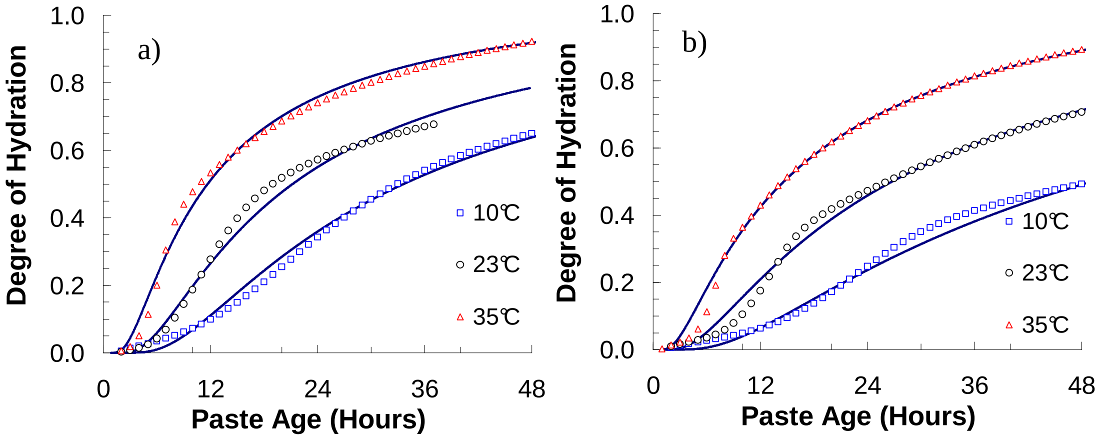


Figure 6.1: Exponential hydration curve fit to isothermal calorimeter data for a) Control and b) 30%C mixtures

In his methodology, Schindler (2004a) established a link between τ as calculated above, and the rate constant, k used to form Arrhenius plots in ASTM C 1074 (2004), the *Standard Practice for Estimating Concrete Strength by the Maturity Method*. Once τ was determined, an Arrhenius plot was developed for each mixture, and activation energies were calculated as the negative slope of the best-fit line to the Arrhenius data multiplied by the universal gas constant, R (ASTM C 1074 2004; Carino 2004). A good linear fit (regression correlation coefficient of at least 0.98) for a mixture demonstrates an E that is independent of temperature as shown by Schindler (2004a), and was used as a quality control measure. All mixtures considered had regression correlation coefficients of at least 0.98 excluding 30%F, which had a correlation coefficient of 0.97. Examples of Arrhenius plots for select mixtures are shown in Figure 6.2. For a summary of E values as calculated by isothermal calorimetry see Table 6.1. All E values were determined to the nearest 100 J/mol.

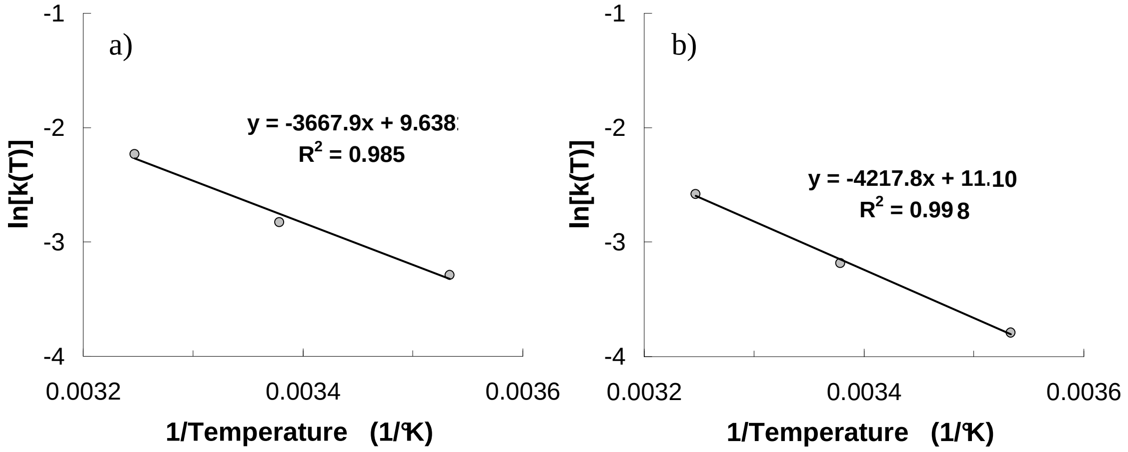


Figure 6.2: Arrhenius Plots for a) Control and b) 30%C mixtures

Table 6.1: Activation energies as calculated by isothermal calorimetry

Mixture	**Isothermal Calorimetry Activation Energy, E (J/mol)**
Control	30,500
30% C	35,000
30% F	35,300
50% Slag	30,400
w/c 0.36	33,600
w/c 0.40	28,300
w/c 0.48	33,100

6.1.2 Penetration Resistance Based Approach

Pinto and Hover (1999) successfully applied the maturity method to setting results obtained through ASTM C 403 (1996). In their approach, E was estimated with the use of setting times obtained by testing batches of the same mixture at various temperatures. Using the assumption that the rate constant, k, as defined by ASTM C 1074 is inversely proportional to setting times, Arrhenius plots were constructed without the use of mortar strength or isothermal calorimeter data. Similarly, penetration resistance data were used by Wade et al. (2007) to calculate E needed for setting maturity calculations. In the approach used by Wade et al. (2007), mortar temperatures were recorded during the setting process, and an activation energy was calculated such that the error between initial and final setting times, in terms of equivalent age, for batches at three different temperatures, were minimized.

An approach similar to the latter of these was used to calculate activation energies for each mixture in this testing program. Using the recorded mortar temperatures presented in Chapter 5, all ASTM C 403 (2008) measurements of mortar age were converted into an equivalent age for mortar cured at 23 °C using the FHP maturity function (Equation 2.11) and an assumed activation energy values of 40,000 J/mol. Activation energy values could then be solved for by minimizing the square of the error between initial and final setting times, in terms of equivalent age, from the three batches of concrete produced (Cold, Room, and Hot). Figure 6.3 demonstrates the application of this method to calculate the “best-fit” activation energy for a given mixture. Data points shown represent actual penetration resistance readings while the regression analysis for each temperature is represented by a solid line. Activation energies calculated using penetration resistance data can be seen in Table 6.2.

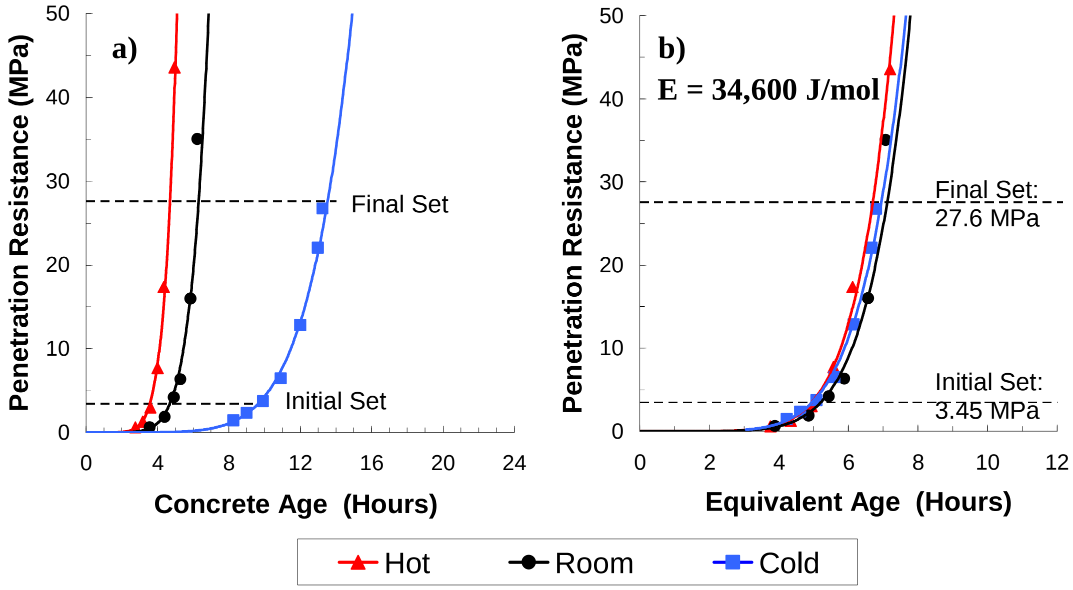


Figure 6.3: Penetration resistance versus a concrete age and b. equivalent age for the Control mixture

Table 6.2: Activation energies as calculated by penetration resistance

Mixture	Penetration Resistance Activation Energy, E (J/mol)
Control	34,600
30% C	36,700
30% F	30,600
50% Slag	30,500
w/c 0.36	28,500
w/c 0.40	33,900
w/c 0.48	31,000

6.1.3 Discussion of Phase I Results

Results obtained through isothermal calorimetry compared relatively well to those obtained through manipulation of ASTM C 403 (2008) data. Table 6.3 shows E values calculated with both approaches as well as the percent difference.

Table 6.3: Activation energy as calculated by both isothermal calorimetry and penetration resistance

Mixture	Isothermal Calorimetry Activation Energy, E (J/mol)	Penetration Resistance Activation Energy, E (J/mol)	Percent Difference (%)
Control	30,500	34,600	12.6
30% C	35,000	36,700	4.7
30% F	35,300	30,600	14.3
50% Slag	30,400	30,500	0.3
w/c 0.36	33,600	28,500	16.4
w/c 0.40	28,300	33,900	18.0
w/c 0.48	33,100	31,000	6.6

It should be noted that the activation energy values above, obtained with the use of early-age data, are smaller than those traditionally used for strength prediction. (ASTM C 1074 recommends the use of 40,000 J/mol to 45,000 J/mol for concrete made with a standard Type I cement.) This was also the case for setting activation energies presented by Schindler (2004b) and Wade et al. (2007).

One of the objectives of this research was to develop a repeatable testing method for determining concrete set times through thermal methods. Due to the elevated temperatures experienced during semi-adiabatic calorimetry, the use of the equivalent age maturity method with a mixture-specific E value was considered essential. For this testing program, activation energies calculated using penetration resistance data were used for all maturity calculations due to the similarity of these values as compared to those observed from isothermal hydration tests. Due to the fact that there was some difference between the values obtained with the use of the two aforementioned E-value calculation methods, a sensitivity study was completed (Section 6.3) in order to examine the effect that activation energy has on the proposed thermal methods.

6.2 Phase II – Analysis of Thermal Results

As discussed in Chapter 2, several analytical methods have been proposed for the prediction of concrete setting with the use of thermal data. Analysis of thermal data can generally be divided into two categories: 1) setting is defined as a function of the slope and/or curvature of the temperature data collected, or 2) setting is defined in terms of a specific adiabatic temperature rise which may be considered a measure of a sample's degree of hydration. The former of these methods will be referred to in this chapter as the *Derivatives Method* and the latter the *Fractions method*.

When these past analytical methods are examined, variations in thermal data (due to the variety of thermal devices used) and analysis technique are evident. In order to evaluate the use of thermal methods to predict the setting of concrete, the variations in determining the temperature profile of a given mixture must be understood and calculations methods must be standardized. Therefore, all analyses for this project were performed on data collected with a semi-adiabatic calorimeter that was adjusted for device specific losses. Also, in order to approximate the effects of temperature on the setting process, the maturity method was applied to all data. Several variations of both the *Derivatives Method* and the *Fractions Method* were evaluated in the analysis of thermal data in an attempt to fully evaluate the use of these methods to estimate concrete setting. Although methods for the prediction of setting proposed by Sandberg and Liberman (2007) and Schindler (2004b) were utilized for analysis, these methods were modified slightly as discussed later.

In the following section, several analysis procedures used to predict concrete setting will be presented. Modifications to raw thermal data in order to address concerns uncovered during the analysis process will be discussed as well as the application of the maturity method to both thermal and penetration resistance data. The background and methodology developed for each analytical method used will then be presented and a discussion of the results will follow.

6.2.1 Modification of Semi-Adiabatic Data to Account for Device Losses

In the 2008 ASTM round robin study for the proposed specification entitled the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method,* several sample and device properties were identified as having a substantial impact on setting times obtained through thermal-based methods. As discussed in Section 3.6.2.1, these properties include sample size, sample type, and device insulation properties. In light of the fact that thermal testing, associated with this research program, was limited to concrete samples, the effect of sample type was neglected. As only mixtures at room temperature were evaluated in the round robin study, it was hypothesized that effects of the surrounding temperature may also have some impact on results obtained when devices with varying insulatory properties are used.

By definition, fully-adiabatic temperature profiles are independent of sample size and ambient temperatures due to the fact that no heat is transferred to or from the test specimen. In an adiabatic system, while more heat is generated by a larger sample, a larger sample requires a proportionally larger amount of heat to raise the temperature of the sample itself. This can be shown with the use of fundamental thermodynamic relationships. The temperature rise of a sample due to the addition or subtraction of heat to or from a system can be expressed as follows (Lucke 1912):

$$\Delta T = \frac{Q}{C_p \cdot m} \qquad \text{Equation 6.1}$$

where:

ΔT = temperature rise of the sample, °C,

Q = heat gained or lost, (J),

C_p = specific heat of the sample (J/g/°C), and

m = sample mass, g.

For an adiabatic system, the heat gain (Q) as a concrete specimen hydrates can be calculated from the heat of hydration generated by the cementiuos materials in the concrete mixture. Due to the fact that no heat is gained or lost from the adiabatic system during testing, all heat gain is from the hydration reaction alone. With the use of an expression similar to that derived by Schindler and Folliard (2005), Q can be calculated from heat of hydration as follows:

$$Q = H \cdot P_c \cdot m \qquad \text{Equation 6.2}$$

where:

Q = heat gained due to hydration of cementitious material, (J),

H = heat of hydration of cementitious materials, (J/g),

P_c = weight ratio of cement in terms of total sample weight, and

m = sample mass, g.

The total temperature rise of a concrete specimen can then be calculated with Equation 6.3.

$$\Delta T = \frac{H_u \cdot p_c}{C_p} \qquad \text{Equation 6.3}$$

where:

ΔT = temperature rise of the sample, °C,

H_u = total heat of hydration of cementitious materials, (J/g),

p_c = weight ratio of cement in terms of total sample weight, and

C_p = specific heat of the sample (J/g/°C).

Equation 6.3 is similar to that presented by Gajda (2007) for the approximation of adiabatic temperature rise of concrete specimens. As can be seen above, due to the fact that a fully-adiabatic response is obtained without the loss of heat during hydration, it is completely independent of sample size. While a fully-adiabatic response is difficult to obtain directly, it is possible to adjust a semi-adiabatic response for losses that occur during testing to obtain a "nearly" adiabatic response. By limiting analysis to semi-adiabatic data that have been adjusted for device losses, inconstancies due to device size and ambient temperature can be largely eliminated. Discrepancies due to differences in device properties can also be minimized if each device is independently calibrated and all data are adjusted to reflect its respective device's properties.

To minimize the effects of device dependent heat loss and ambient temperature on thermal set analysis, each temperature profile recorded was adjusted for the losses associated with its respective device. To "add back" these losses, insulatory properties of each device must be known, therefore each device was pre-calibrated using a hot-water calibration to determine an effective R value (R_e) for each specimen cell.

R_e was calculated for Calorimeter II based on data from a flux sensor located within the device. A cylinder of known weight was filled with hot water (50 °C) and the sample was tested for seven days. Heat transfer equations derived for the case of a continuous cylindrical object (an assumption was made that all heat loss is from the sides of the device, not the top) were applied to determine the thermal conductivity of the device and R_e was calculated from this value. Due to the fact that Calorimeter I was not equipped with a thermal flux sensor, R_e was calculated for each cell indirectly. A concrete specimen was placed in each cell of Calorimeter I as well as in Calorimeter II and tested for seven days. Loss calculations were then performed for the sample from Calorimeter II using the R_e value previously calculated. The effective R value for each cell of Calorimeter I could then be solved for by minimizing the square of the error between results from the two devices.

Temperature profiles from Calorimeter I that have been adjusted to account for thermal losses can be seen in Figure 6.4. Results from all three batches of concrete (Hot, Room, and Cold) are plotted on the same graph for comparison purposes.

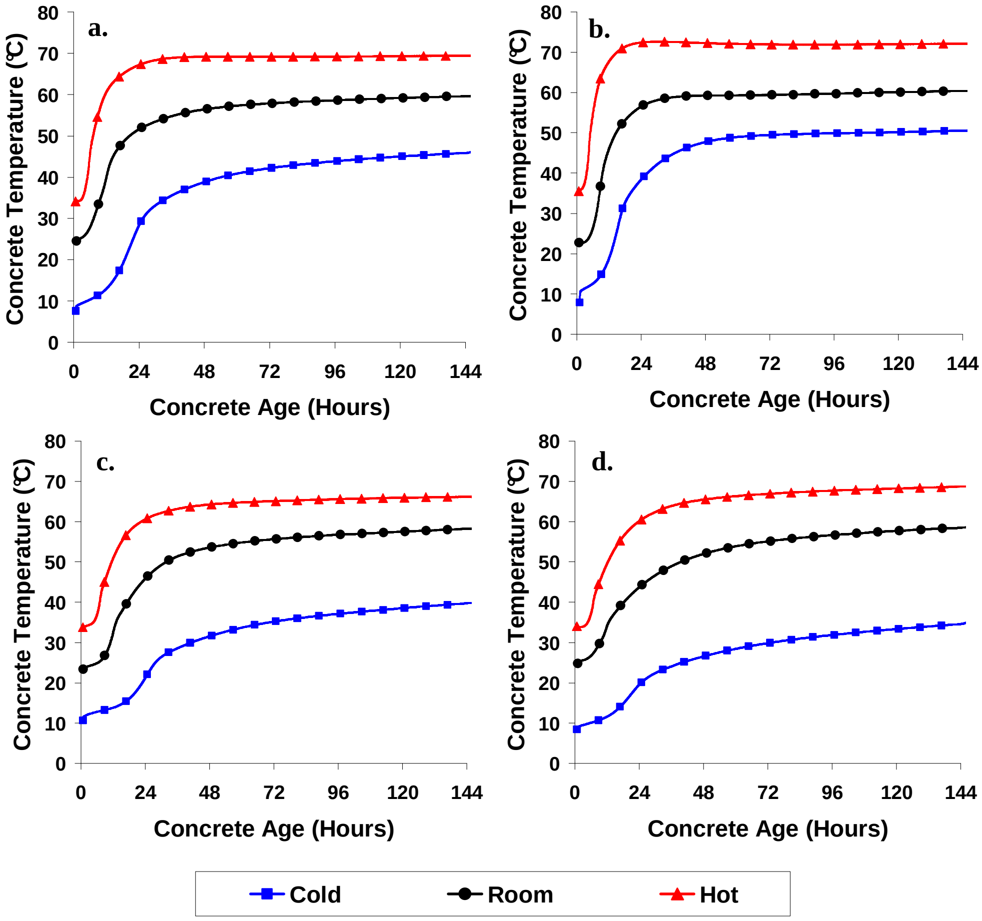


Figure 6.4: Calorimeter I data adjusted for device losses for a. Control, b. w/c 0.36, c. 30 % C, and d. 50 % Slag mixtures

Although these temperature profiles are adjusted for thermal losses, it should be noted that, due to the temperature dependent nature of concrete reactions, this is not a fully-adiabatic response. Figure 6.5 shows a semi-adiabatic response, semi-adiabatic response that has been adjusted for losses (referred to as "nearly-adiabatic"), and the fully-adiabatic response for a mixture. As can be seen, the difference between the fully-adiabatic response and the nearly-adiabatic are relatively small, and are almost non-existent during the setting window (which is 4.5 to 6.5 hours for this mixture). The difference between these two curves was considered negligible and all further analysis was performed on the nearly-adiabatic response for each concrete sample.

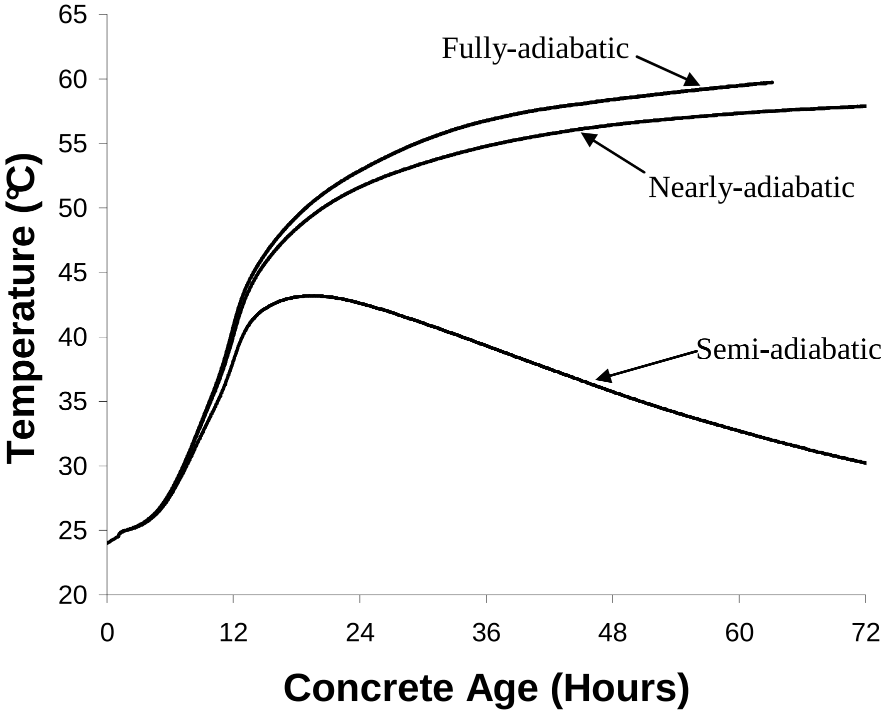


Figure 6.5: Semi-adiabadic, nearly-adiabatic and fully-adiabatic response for the Control – Room mixture

To confirm the independence of sample size and device properties on the nearly-adiabatic response, a paired t-test identical to that used in Chapter 3, was performed on adjusted semi-adiabatic results from Calorimeter I and Calorimeter II. Matched pairs for the t-test were obtained through *Direct-Slope Derivatives Method* analysis (See Section 6.2.3.2) of data from each calorimeter, which have differing sample size and insulatory properties. Results from the t-test suggested that the difference between the data was not statically significant (two-tail p-value of 0.854). Although this does not prove independence of the properties in question, the high p-value indicates that any variation in data is due to statistical variation, not sample size or device properties. In comparison, similar analysis from thermal devices in Chapter 3 yielded a p-value of 5.77×10^{-12} (See Section 3.5.2.1) Based on this test, and the fully-adiabatic equations above, effects due to sample size and properties of Calorimeter I and II were considered negligible.

6.2.2 Application of the FHP Maturity Method

The maturity method has been successfully used for the prediction of temperature dependent concrete properties for some time. Although rarely applied to the setting mechanism, it has been shown to provide an accurate approximation of the effect of temperature on concrete and cement paste setting (Pinto and Hover 1999; Wade et al. 2007; Garcia et al. 2007; Schindler 2004b). One of the problems often cited with the penetration resistance test method of concrete setting (ASTM c 403) is the lack of correlation with in-place concrete setting (Christensen 2006). The use of a maturity-based definition of concrete setting could improve this correlation by accounting for the thermal effects caused by a difference in temperature between setting samples and in-place concrete.

In addition to prediction of in-place properties, the maturity method is useful when comparing concrete properties of specimens with differing temperature profiles. As can be seen in Figure 6.6, mortar samples used in ASTM C 403 (2008) and concrete samples used for thermal analysis (Calorimeter I) may experience vastly different temperature profiles during testing. Unless corrected for this difference in temperature, comparisons may be meaningless due to the effects of temperature on concrete setting times.

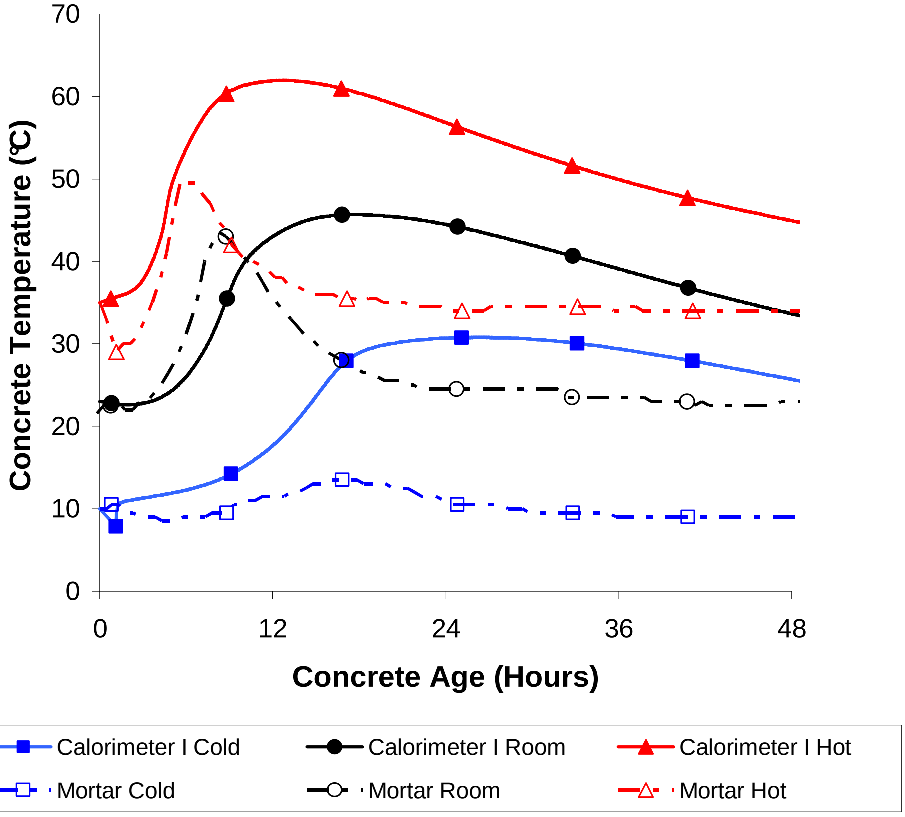


Figure 6.6: Temperature profiles from Calorimeter I and ASTM C 403 (2008) mortar sample for w/c 0.36

Similarly, specimens cured at differing temperatures such as exists between a laboratory and field conditions may undergo significant variation in temperature and therefore behave much differently. By adjusting for these effects with the use of the maturity method, there is a possibility that the lack of correlation between in-place concrete behavior and laboratory setting times may be avoided.

To approximate the effects of temperature on setting, all penetration resistance data were adjusted for maturity before being used as a basis for comparison for concrete setting. With the use of the FHP maturity function (see Section 2.4.1), E values calculated in Phase I of testing, and temperature data recorded from the ASTM C 403 (2008) mortar specimens, all measurements of concrete age were converted to an equivalent age for concrete cured at 23 °C. For an example of this adjustment see Figure 6.3. Penetration resistance plotted in terms of maturity instead of traditional concrete age for select mixtures can be seen in Figure 6.7. For penetration resistance results for all batched see Appendix D All three batches of concrete (Hot, Room, and Cold) are plotted on the same graph for comparison purposes. Data markers shown represent actual penetration resistance readings while the regression analysis is represented by a solid line. A summary of setting times in terms of concrete age and maturity can be seen in Table 6.4.

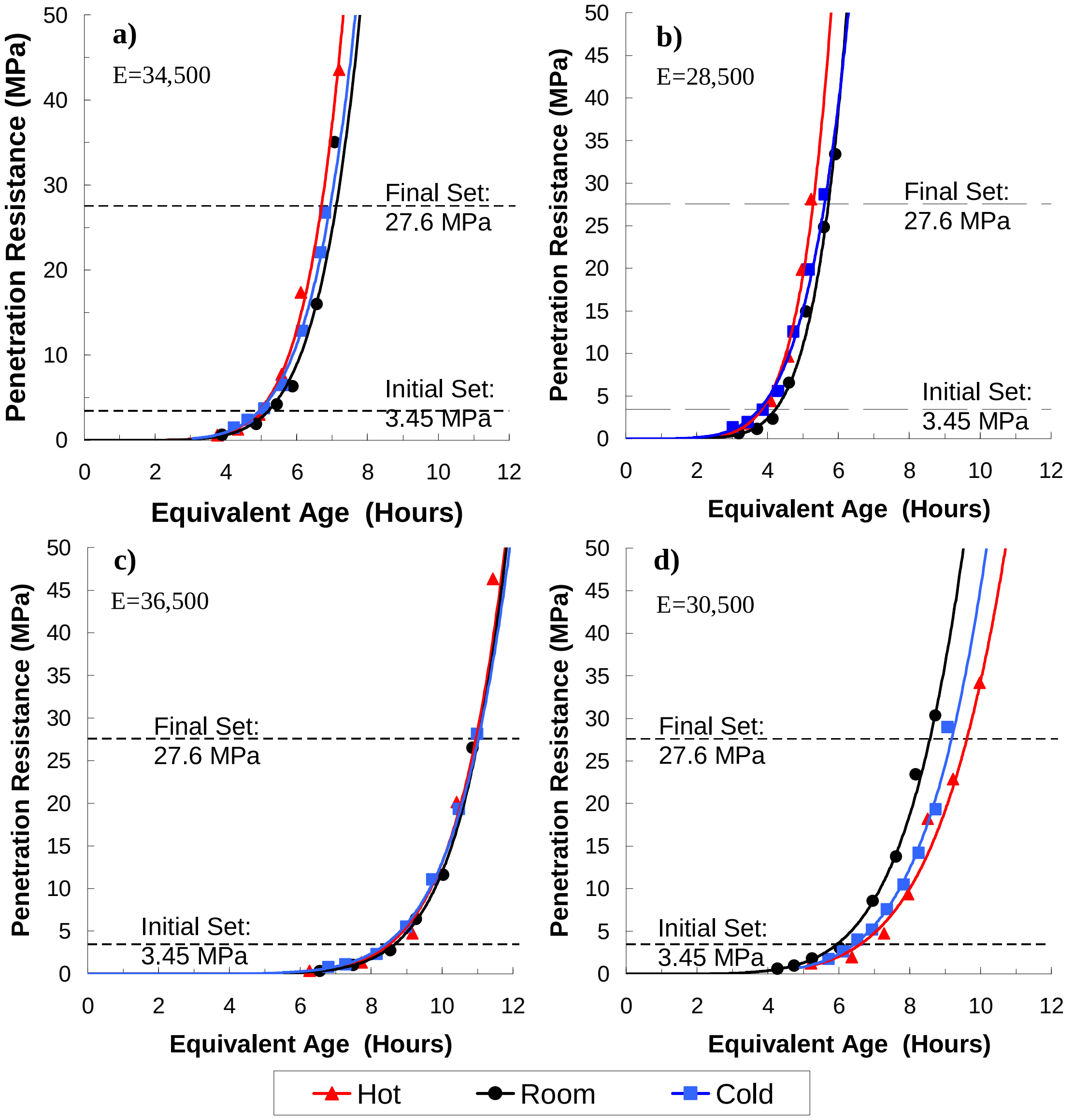


Figure 6.7: Penetration resistance versus equivalent age plots for a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

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Table 6.4 ASTM C 403 (2008) initial and final setting times in terms of concrete age and maturity

Mixture	Batch	Initial Set Concrete Age (Hours)	Final Set Concrete Age (Hours)	Initial Set Equivalent Age (Hours)	Final Set Equivalent Age (Hours)	Activation Energy, E (J/mol)
Control	Cold	9.35	14.02	4.80	7.21	34,600
	Room	4.70	6.31	5.18	7.15	
	Hot	3.57	4.71	4.90	6.74	
30% C	Cold	17.22	22.61	8.39	11.06	36,700
	Room	8.43	10.51	8.58	11.01	
	Hot	5.63	7.01	8.43	11.00	
30% F	Cold	11.43	15.72	6.46	8.92	30,600
	Room	6.72	9.30	6.86	9.78	
	Hot	4.21	5.35	6.25	8.22	
50% Slag	Cold	11.87	16.96	6.38	9.26	30,500
	Room	5.78	8.28	5.92	8.61	
	Hot	4.74	6.07	7.13	9.35	
w/c 0.36	Cold	6.48	9.90	3.72	5.71	28,500
	Room	4.18	5.50	4.20	5.75	
	Hot	2.80	3.79	3.83	5.32	
w/c 0.40	Cold	6.82	10.34	3.53	5.41	33,900
	Room	3.95	5.21	3.77	5.06	
	Hot	2.68	3.47	3.87	5.29	
w/c 0.48	Cold	7.89	9.74	4.39	5.44	31,000
	Room	3.94	5.55	3.68	5.41	
	Hot	3.05	4.05	4.19	5.79	

Similar to penetration resistance data, the equivalent age maturity method was applied to all thermal data in order to account for the effects of temperature on concrete setting. E values from Phase I was used to convert all concrete age measurements to an equivalent age for a concrete sample cured at 23 °C. It should be noted that, although temperature profiles were adjusted to include the effects of device losses, maturity calculations were performed based on the original data collected in order to reflect the actual temperature profile the sample experienced. Figure 6.8 demonstrates the effect of applying the maturity method to thermal data. The temperature data were normalized to reflect a temperature rise from the batch temperature, rather than actual specimen temperature, and all three batches (Cold, Room, and Hot) are plotted versus both concrete age and equivalent age.

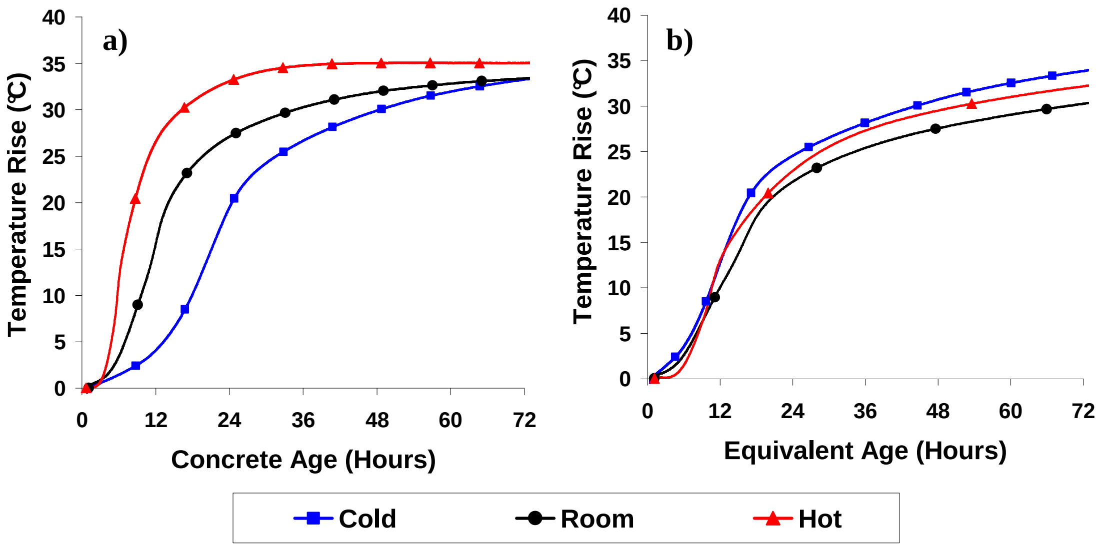

Figure 6.8: Calorimeter I temperature profiles plotted versus a) concrete age and b) equivalent age for the Control mixture tested at Cold, Room, and Hot conditions

Temperature profiles for select mixtures that have been adjusted for device losses and maturity and normalized to reflect a temperature rise for select mixtures can be seen in Figure 6.9. Results from all three batches of concrete (Hot, Room, and Cold) are plotted on the same graph for comparison purposes.

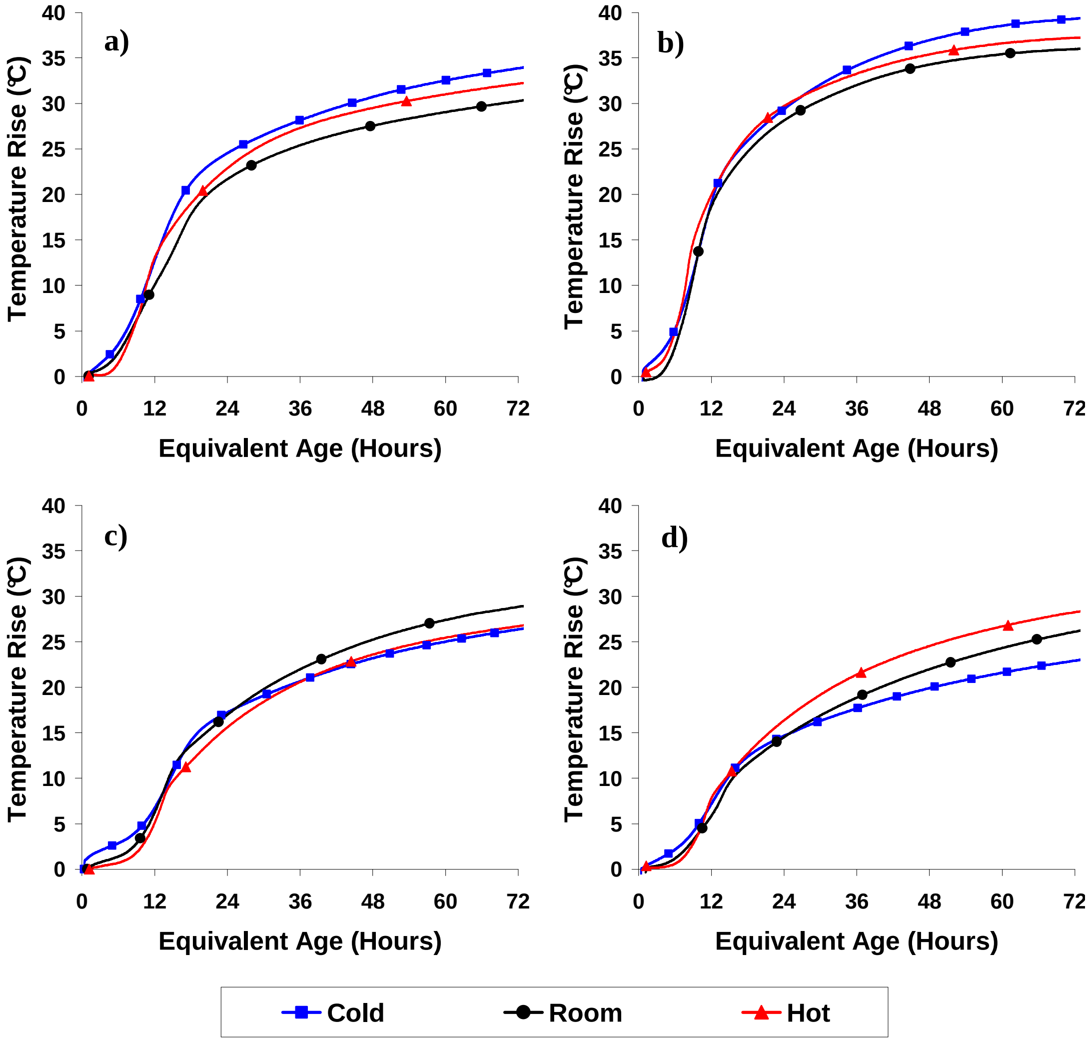

Figure 6.9: Calorimeter I data adjusted for device losses and maturity for

a) Control, b) w/c 0.36 c) 30 % C, and d) 50 % Slag mixtures

6.2.3 Analysis of Thermal Results to Obtain Concrete Setting Times

To fully assess the use of thermal methods to measure set times of concrete, a variety of analytical methods were evaluated in the course of this research program. In the following sections, several methods of analysis, based on both the *Derivatives Method* and the *Fractions Method*, will be presented. Each of the analytical variations of the *Derivatives Method* and the *Fractions Method* examined is presented in Figure 6.10. All thermal data referred to in the following sections have been adjusted for device losses and maturity. Similarly, all ASTM C 403 (3008) data were adjusted for maturity.

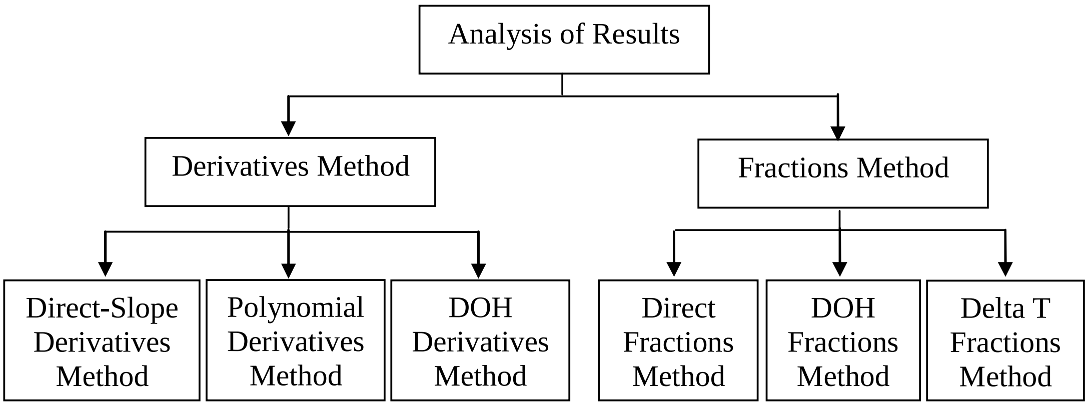

Figure 6.10: Analysis methods evaluated for determining setting of concrete through thermal methods

6.2.3.1 *Derivatives Method* – Background and Methodology

The rate of heat evolution of a cement paste sample has been used to quantify the setting of cement paste, mortar, and concrete for some time (Weiss 2002). In general, final set has been associated with the maximum rate of heat evolution of a sample, and initial set has been associated with the point of maximum curvature of the heat evolution versus time plot. In 2007, Sandberg and Liberman made use if two methods of determining setting based on thermal data originally proposed by Bruce Christensen (although unpublished). One of these, referred to as the *Derivatives Method*, defined setting in terms of the rate and curvature of thermal data collected with the use of an open top insulated thermal device. It should be noted that this device would not be considered semi-adiabatic by the definition provided by RILEM TC 119 TCE 1 (1997).

An ASTM specification was proposed based on this method, and examined in a round robin study in 2008, which is discussed in Chapter 3. It was found that the proposed thermal method for determining setting times of concrete is not a dependable indicator of concrete setting. In addition to the testing properties identified and discussed in Section 6.2, difficulties were encountered in obtaining consistent first and second derivative results, and these were identified as a possible sources of error during the round robin testing.

Calculation of Derivatives

To fully evaluate the *Derivatives Method*, three different approaches to determine the derivatives of the temperature profile were attempted. These methods are as follows:

(a) The *Direct-Slope Derivatives Method*,

(b) The *Polynomial Derivatives Method*, and

(c) The *DOH Derivatives Method*

In the *Direct-Slope Derivatives Method,* slope and curvature were calculated directly from the raw data by calculating the average slope over a large smoothing interval. In the *Polynomial Derivatives Method*, a high-order polynomial was fit to the data from which a closed-form solution could be derived to calculate the derivatives. Similarly, in the *DOH Derivatives Method*, the hydration equation presented in Section 2.1.3.3 (see Equation 2.8) was fit to the temperature data from which a closed-form solution for the derivatives could be calculated. Examples of these three methods are presented in Figure 6.11.

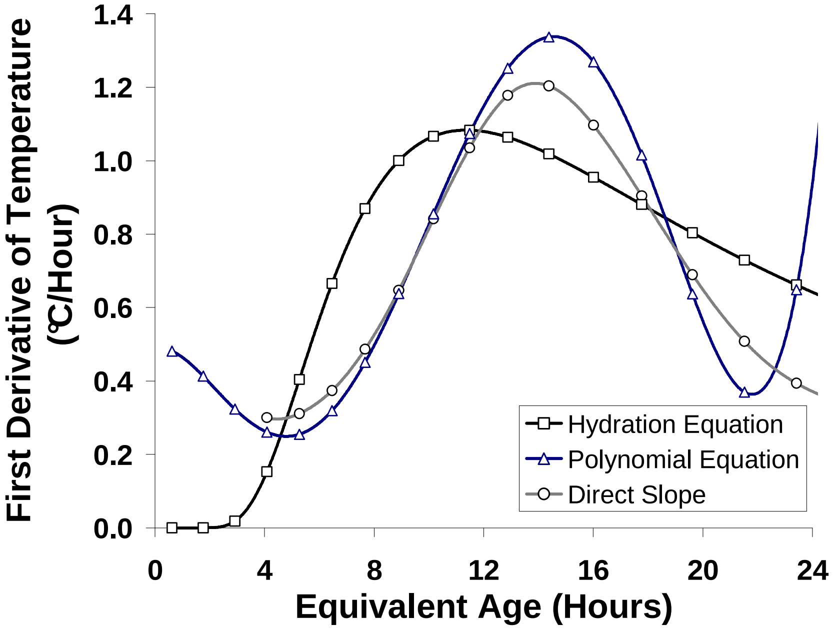

Figure 6.11: First derivative of temperature as calculated by the *Direct-Slope Derivatives Method*, the *Polynomial Derivatives Method*, and the *DOH Derivatives Method* for 30 % C - Cold

As can be seen, the three methods of calculating derivatives of the temperature versus time plot can yield different results. Due to computational issues inherent to each method, it is difficult to determine which method more closely represents the true slope of the thermal data collected. As is demonstrated in the above figure, the *Direct-Slope Derivatives Method* calculation and *Polynomial Derivatives Method* equation are usually more comparable to each other than the *DOH Derivatives Method* equation, but there are still measurable differences between all three. Rather than focus the evaluation of these methods on the accuracy of the derivative itself, each method was evaluated by the consistency with which it can be used to predict setting.

From the analysis of the data obtained through the ASTM round robin (Section 3.4) and preliminary analysis of data from this project, it was determined that the second derivative of the temperature profile was difficult to accurately obtain regardless of the method used. Fluctuations in data that occur while the thermal devices reach equilibrium with the samples, and difficulty with fitting curves to early-age data let to inconsistent results with all three methods. For this reason, setting for the *Derivatives Method* for this research program was defined only in terms of the slope, or first derivative, of the temperature profile.

Determining Initial and Final Set with the Derivatives Method

Although Sandberg and Liberman (2007) defined setting in terms of the maximum slope and curvature of the hydration peak (temperature profile), these definitions were based on the analysis of thermal data from open-top calorimeters that are not considered semi-adiabatic by RILEM TC 119 TCE 1 (1997) standards. A plot of the first derivative of the temperature profile for Control – Hot can be seen in Figure 6.12. As can be seen, initial set from ASTM C 403 (2008) corresponds not to the maximum slope, but to a point that is approximately 30% of the maximum slope. Similarly, final set from ASTM C 403 can be equated to approximately 60% of the maximum slope for this mixture.

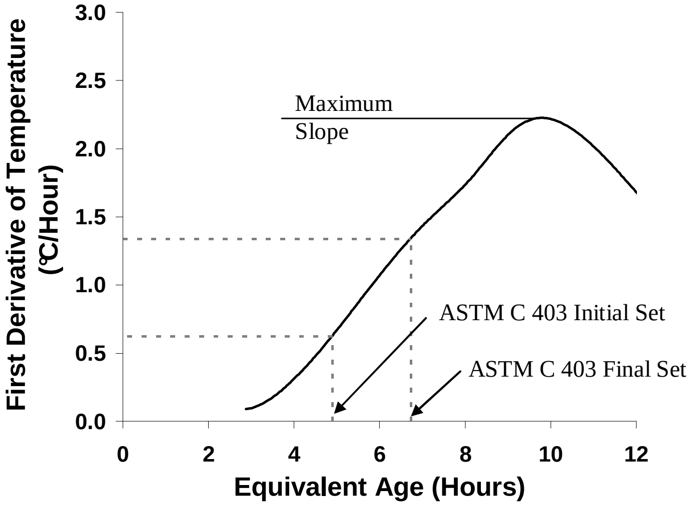

Figure 6.12: First derivative of temperature and ASTM C 403 (2008) setting times for Control - Hot

For all *Derivatives Method* analysis, initial and final set were defined as the equivalent age at which some ratio of the maximum slope of the temperature profile is reached. This ratio, notated K_{Di} for initial set and K_{Df} for final set, was calibrated based on thermal data collected, then used to predict setting for each individual batch.

The point on a temperature profile at which concrete sets may not be constant for all mixtures. Bentz et al. (2008) noted the dependency of the critical degree of hydration of cement on its w/c (i.e. mixtures with a higher w/c required a higher degree of hydration to produce setting). Similarly, K_{Di} and K_{Df} as described above may be dependent on w/cm. To examine this possibility, both constant ratios and ratios that are a function of the w/cm were examined during testing for all derivative-based analysis methods.

***Derivatives Method* Methodology**

The *Direct-Slope Derivatives Method*, *Polynomial Derivatives Method* and the *DOH Derivatives Method* were each evaluated individually. The general methodology can be divided into three stages, and a schematic of these are shown in Figure 6.13. First, for each method, thermal data from both semi-adiabatic calorimeters were adjusted for device losses and maturity, and derivatives were calculated as per the analysis method under consideration. *K* values (both constant and w/c-dependent) were calculated for each batch (Cold, Room, and Hot for each mixture) based on the maturity-adjusted penetration resistance data obtained as per ASTM C 403 (2008) and summarized to obtain the average overall response for the method. Lastly, these values were used to predict setting of each batch, and each analysis method could then be evaluated by comparing setting times to those obtained by the penetration resistance method.

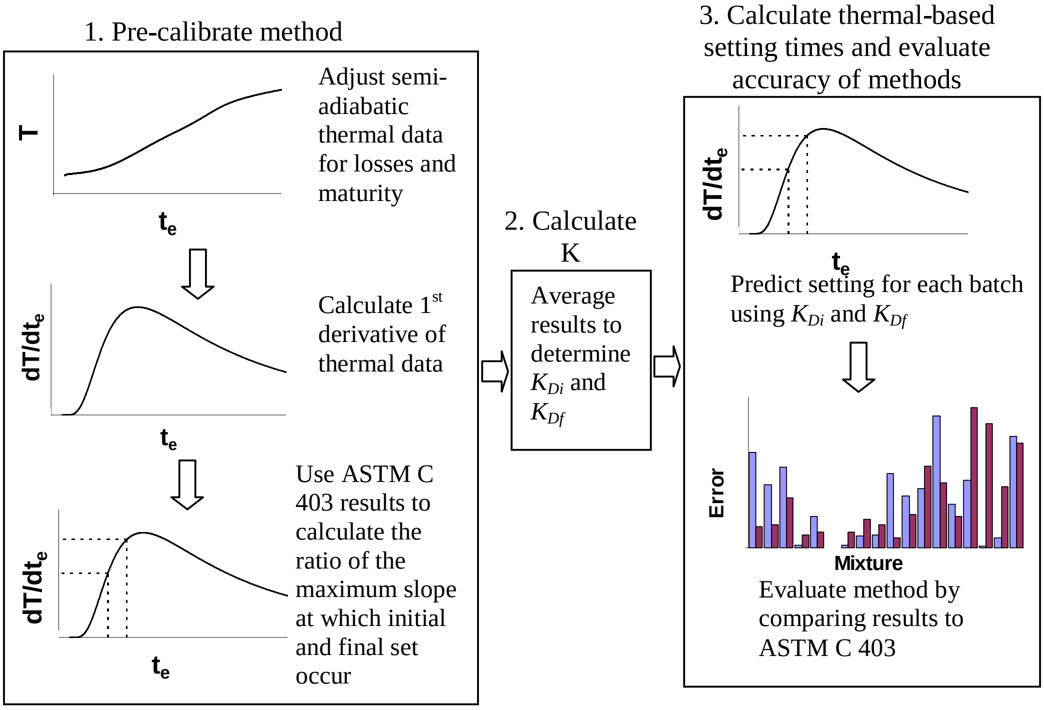

Figure 6.13: Schematic of methodology used to evaluate the *Derivatives Method*

6.2.3.2 *Derivatives Method* - Limits of Acceptability

To evaluate each method, limits of acceptability must be defined to determine if the proposed methods can be considered an acceptable means of indication concrete setting. Criteria defined in Chapter 3 for the comparison of setting times obtained by thermal methods to setting times obtained as per ASTM C 403 (2008) were used to define these limits These criteria are as follows:

a) The accuracy of setting times obtained by thermal methods as compared to those obtained through ASTM C 403 (2008) lies within the variability inherent to ASTM C 403 (2008) (see Figure 6.14a),

b) The accuracy of setting times obtained by thermal methods as compared to those obtained through ASTM C 403 (2008) lies outside of the variability of ASTM C 403 (2008) but within limits that are deemed acceptable for the determination of concrete setting times (see Figure 6.14b),

c) The accuracy of setting times obtained by thermal methods as compared to those obtained thought ASTM C 403 (2008) lies outside of the limits that are deemed acceptable for the determination of concrete setting times, yet is consistent such that setting times obtained by thermal methods may be considered a separate indicator of concrete setting (see Figure 6.14c).

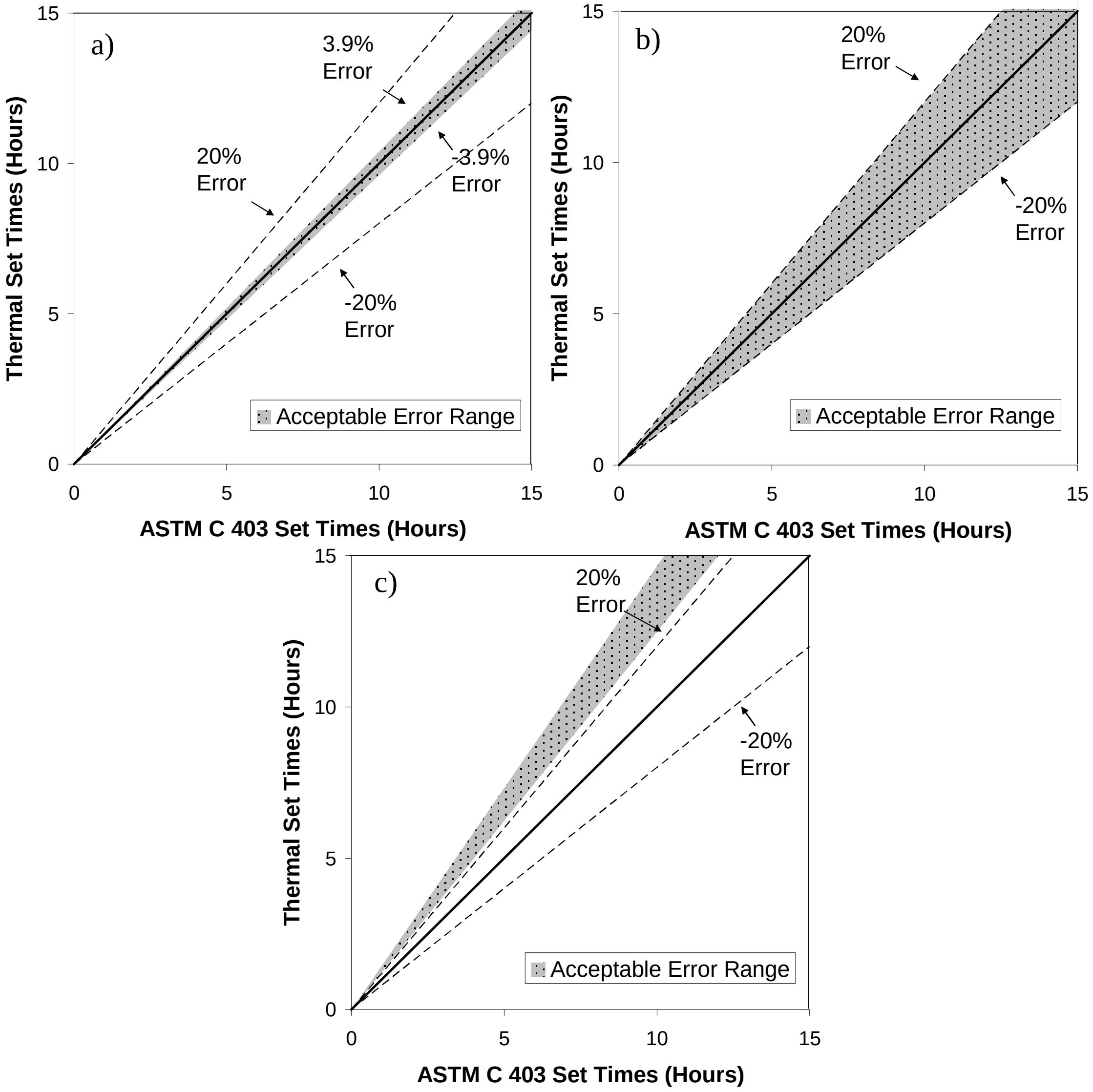

Figure 6.14: Examples of allowable range of error for final set as compared to ASTM C 403 (2008) results for setting times obtained through thermal testing to be considered a reliable indicator of concrete setting for a) criteria a, b) criteria b, and c) criteria c

If the first of these criteria is met, (Criterion a), setting times obtained through thermal methods may be considered as reliable an indicator of concrete setting as ASTM C 403 (2008), and results may be considered comparable. In Figure 6.14a, 3.9% is shown as the acceptable error range for final set values. This is the value reported in the single-operator precision statement (see Section 3.5.1.2) for the acceptable difference between two results for final set measured as per ASTM C 403 (2008). In a similar manner, the acceptable error range for initial set can be determined from the single-operator precision statement for initial set.

If the second of the above criteria is met (Criterion b), setting times obtained through thermal methods may not be considered as reliable as those obtained through ASTM C 403 (2008), but may still may be adequate indicators of concrete setting, and comparable to penetration resistance-based methods. In the past, the ± 20% error range has been used as an acceptable percent error for the determination of concrete setting times (Edson 2005), therefore the acceptable error range for both initial set and final set as compared to ASTM C 403 (2008) was defined as ± 20%. This range can be seen in Figure 6.14b.

If neither Criterion a or b is met, there is a possibility that setting times obtained through thermal methods may be an independent indicator of setting (i.e. primary set and secondary set are not related to either initial or final set, but may still be considered adequate indicators of concrete setting). For this criterion to be met, primary set and secondary set may compare with greater than ± 20% error, but must have a reasonable variability (represented by the scatter of data points). An example of this can be seen in Figure 6.14c. Although the range of values lies outside the ± 20% range, a small scatter is required for data to fall within this area. It should be noted that the acceptable error range demonstrated in Figure 6.14c is only one possible configuration for Criterion c; any range outside ± 20% with a reasonable variability would also meet this criterion.

6.2.3.3 *Derivatives Method* – Analytical Procedure and Results

In the following section, analytical procedures for a variety of derivative-based methods are presented. Details concerning the analysis technique are presented as well as results and a brief discussion.

Direct-Slope Derivatives Method

For the *Direct-Slope Derivatives Method*, derivatives were calculated directly from thermal data. Just as in the proposed ASTM specification entitled the *Standard Test Method for Determination of Setting Time of Concrete by the Temperature Method*, the first derivative of data was calculated by calculating the slope of the data over a large smoothing interval.

Although the specification made use a 200-point (200-minute) smoothing interval, this constant interval yielded problems for mixtures with early setting times. As mentioned in Section 3.4, when a large smoothing interval is used to obtain derivatives, no meaningful data can be obtained before half of the interval is reached. (Data are smoothed for one half of the smoothing interval before and after each data point so for a 200 minutes interval, no meaningful first derivative data can be obtained before the 100th minute of testing.) Since initial set often occurred around or before the 100th minute of testing, a shorter smoothing interval was needed. Also, data from Calorimeter II were collected at a much larger sampling interval than in the proposed specification. A smoothing interval of 200 points for data from Calorimeter II would yield no meaningful derivative results before the 25th hour of testing. To alleviate these issues, a smoothing interval equal to the number of points collected at initial set for each batch considered was used. This led to an average smoothing interval for Calorimeter I and Calorimeter II of approximately 250 points and 15 points, respectively.

After calculating derivatives, ratios of the slope of the thermal data at setting to the maximum slope were determined for each specimen tested. A summary of these values for each batch tested can be seen in Table 6.5. These K values (from Calorimeter I and Calorimeter II) were averaged to obtain K_{Di} and K_{Df}. A constant K_{Di} and K_{Df} of 0.46 and 0.73, respectively and a w/cm-dependent K_{Di} and K_{Df} of 1.1 times the w/cm and 1.7 times the w/cm, respectively was calculated for the *Direct-Slope Derivatives Method.*

Table 6.5: *Direct-Slope Derivatives Method K* values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	Batch	Constant K Value				w/cm-Dependent K Value			
		k_{Di}		k_{Df}		k_{Di}/(w/cm)		k_{Df}/(w/cm)	
		CI	CII	CI	CII	CI	CII	CI	CII
30%C	**Cold**	0.47	0.35	0.81	0.55	1.08	0.80	1.83	1.26
	Room	0.56	0.45	0.94	0.78	1.28	1.03	2.13	1.78
	Hot	0.50	0.38	0.96	0.84	1.13	0.87	2.18	1.91
30%F	**Cold**	0.53	0.47	0.83	0.78	1.20	1.07	1.89	1.76
	Room	0.66	0.58	0.87	0.80	1.51	1.31	1.97	1.81
	Hot	0.51	-	0.88	-	1.15	-	2.01	-
50%S	**Cold**	0.50	0.38	0.84	0.69	1.15	0.86	1.91	1.57
	Room	0.43	0.40	0.72	0.62	0.98	0.90	1.63	1.41
	Hot	0.36	-	0.96	-	0.83	-	2.18	-
Control	**Cold**	0.42	0.44	0.69	0.56	0.96	0.99	1.57	1.27
	Room	0.57	0.53	0.86	0.88	1.29	1.22	1.96	2.01
	Hot	0.28	0.40	0.60	0.64	0.63	0.91	1.37	1.46
w/c 0.36	**Cold**	0.31	0.32	0.55	0.56	0.86	0.88	1.53	1.55
	Room	0.33	0.39	0.60	0.58	0.91	1.08	1.67	1.61
	Hot	0.26	0.36	0.54	0.64	0.71	1.01	1.51	1.76
w/c 0.40	**Cold**	0.40	0.34	0.64	0.61	1.00	0.85	1.61	1.51
	Room	0.43	0.48	0.67	0.69	1.45	1.65	1.67	1.72
	Hot	0.33	0.59	0.63	0.75	1.34	1.56	1.56	1.87
w/c 0.48	**Cold**	0.59	0.62	0.71	0.66	1.23	1.29	1.49	1.37
	Room	0.58	0.66	0.85	0.89	1.21	1.37	1.77	1.85
	Hot	0.54	0.62	0.77	0.88	1.12	1.30	1.61	1.83
Average		0.46		0.73		1.10		1.71	

Setting was then calculated for each mixture using these values. Results from the *Direct-Slope Derivatives Method* were then compared to those obtained by penetration-resistance testing. Setting, in terms of equivalent age, for both the *Derivatives Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.15. All data points that fall between the dashed lines represent mixtures in which setting times obtained by the two methods were within an error of ± 20%.

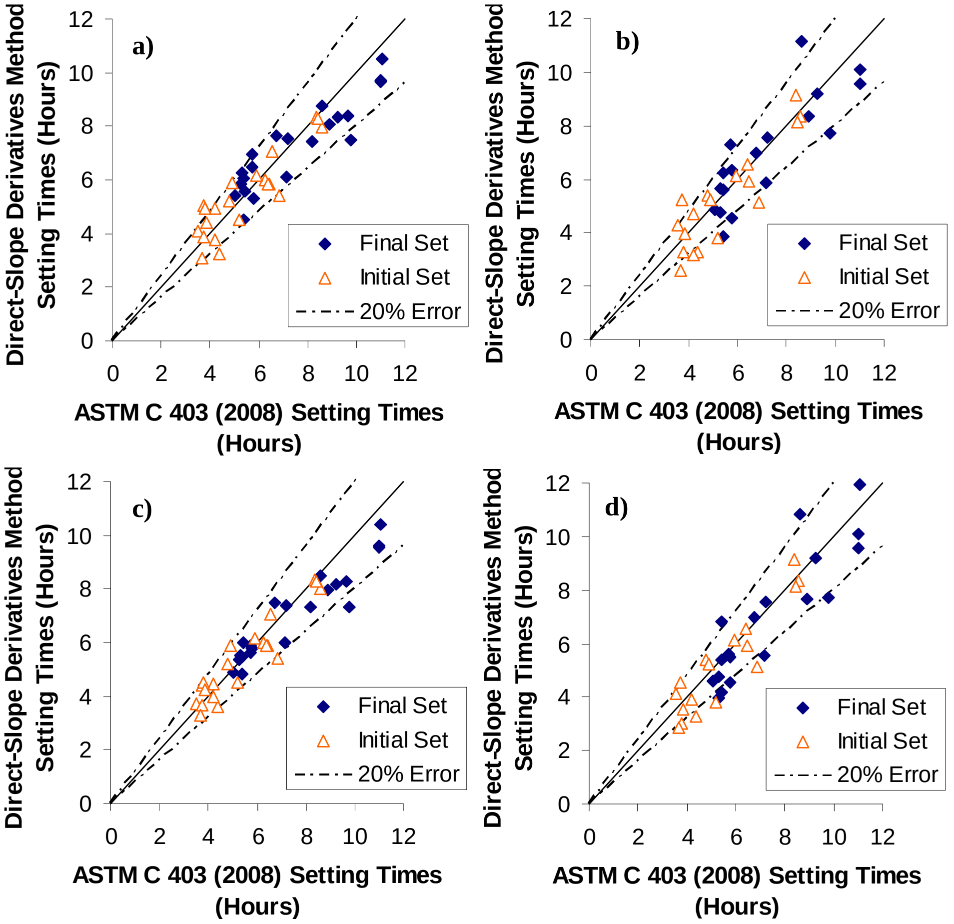

Figure 6.15: Plot of initial and final set as calculated by penetration resistance and the *Direct-Slope Derivatives Method* for a) constant K values for Calorimeter I, b) constant K values for Calorimeter II, c) w/cm-dependent K for Calorimeter I, and d) w/cm-dependent K for Calorimeter II

As is evident in Figure 6.15, the setting times calculated by the *Direct-Slope Derivatives Method* compared with less error to those calculated by ASTM C 403 (2008) for concrete specimens tested in Calorimeter I than specimens tested in Calorimeter II. One possible reason for this is the relatively small smoothing window used for data from Calorimeter II due to a larger sampling interval. Results from the w/cm-dependent *K* values case also appear to be more accurate than those from the constant *K* values case. A more detailed evaluation of the method can be seen in Figure 6.16 for data from Calorimeter I only. In this figure, the absolute error of *Direct-Slope Derivatives Method* setting times as compared to setting times obtained as per ASTM C 403 (2008) can be seen for each batch of concrete tested for both constant *K* values and w/cm-dependent *K* values.

As can be seen in Figure 6.16, results from the use of w/c-dependent *K* values are more accurate overall. It should be noted that mixtures with a w/cm near the average w/cm for all mixtures (0.43) had relatively little change between the two cases. Mixtures with a w/cm farther from the average experienced a much greater change between the case of constant *K* values and the w/cm-dependent *K* values. Neither the constant *K* value nor the w/cm-dependent *K* value case meet any of the criteria outlined above, therefore, the *Direct-Slope Derivatives Method* should not be considered an adequate indicator of concrete setting.

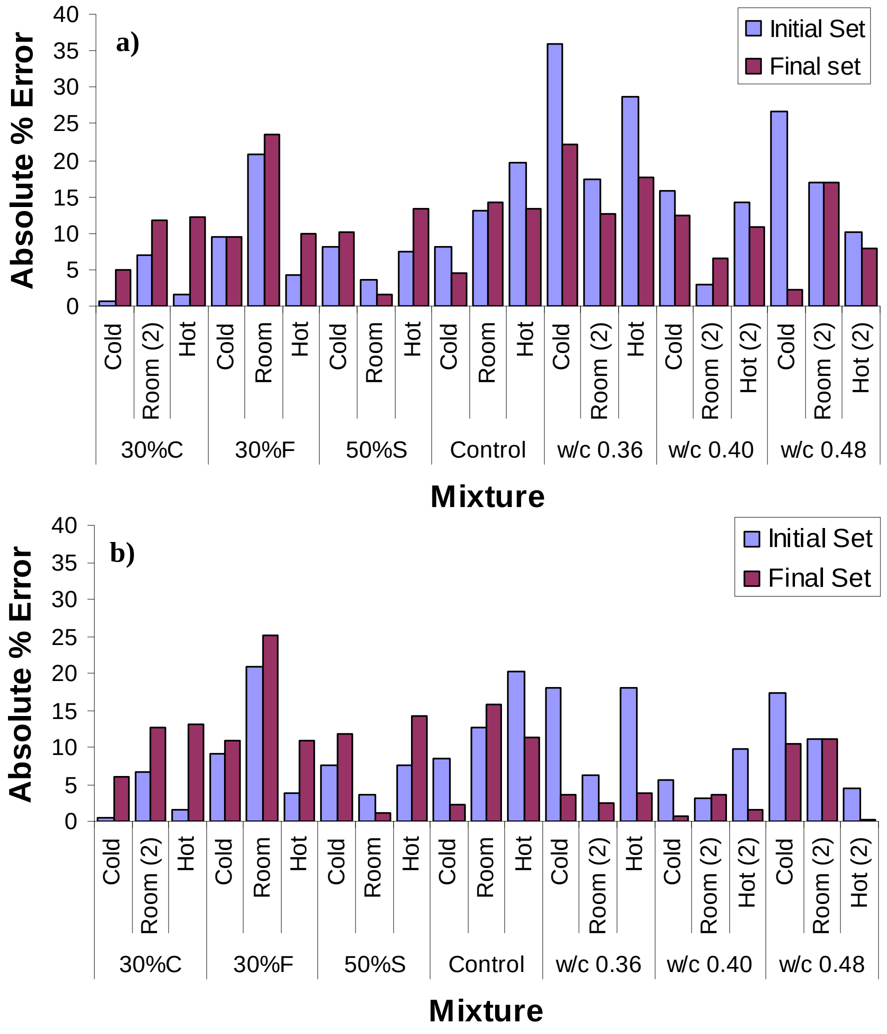

Figure 6.16: Absolute error of initial and final set as calculated by the *Direct-Slope Derivatives Method* as compared to ASTM C 403 (2008) for a) constant *K* values, and b) w/c-dependent *K* values for Calorimeter I

Polynomial Derivatives Method

The first derivative of the temperature profile for the *Polynomial Derivatives Method* was calculated in a similar fashion as *Derivatives Method* calculations in Chapter

3. A high-order polynomial function was fit to the first 24 hours of temperature data with the use of a regression analysis in a commercial spreadsheet program. A closed-form derivative could then be calculated from this polynomial for analysis.

In Section 3.4, it was suggested that a fourth-order polynomial used in analysis did not have adequate degrees of freedom to accurately model the very-early ages of the temperature data for some mixtures. In order to attempt to minimize this effect, a sixth order polynomial was used for all analysis. Although the order of the polynomial used may appear excessive, the intention of its use was to evaluate the possibility of the use of a polynomial function with sufficient degrees of freedom to accurately and consistently predict setting. If successful, further analysis as to the effects of a lower-order polynomial function would be merited.

Just as for the *Direct-Slope Derivatives Method*, K_{Di} and K_{Df} values were calculated by averaging results from all batches tested. These averages, as well as values calculated from Calorimeter I (CI) and Calorimeter II (CII) for each batch tested, can be seen in Table 6.6. Constant K_{Di} and K_{Df} values of 0.51 and 0.79 respectively were calculated for the method. Similarly, w/cm-dependent K_{Di} and K_{Df} values were calculated as 1.2 times the w/cm and 1.9 times the w/cm respectively. These values were then used to calculate *Polynomial Derivatives Method* setting times and the results were compared to ASTM C 403 (2008) penetration resistance results. Setting, in terms of equivalent age, for both the *Derivatives Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.17. All data points that fall between the dashed lines represent mixtures in which setting times obtained by the two methods are within an error of ± 20%.

Table 6.6: *Polynomial Derivatives Method K* values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	**Batch**	**Constant K Value**				**w/cm-Dependent K Value**			
		k_{Di}		**k_{Df}**		**k_{Di}/(w/cm)**		**k_{Df}/(w/cm)**	
		CI	**CII**	**CI**	**CII**	**CI**	**CII**	**CI**	**CII**
30%C	**Cold**	0.41	0.40	0.75	0.73	0.94	0.91	1.71	1.67
	Room	0.60	0.59	0.90	0.92	1.36	1.34	2.05	2.09
	Hot	0.32	0.52	0.53	0.83	0.73	1.18	1.21	1.88
30%F	**Cold**	0.52	0.13	0.84	0.20	1.19	0.28	1.90	0.44
	Room	0.46	0.72	0.74	0.82	1.05	1.63	1.69	1.87
	Hot	0.57	-	0.91	-	1.29	-	2.08	-
50%S	**Cold**	0.51	0.51	0.85	0.83	1.15	1.17	1.94	1.89
	Room	0.29	0.37	0.71	0.77	0.66	0.85	1.62	1.75
	Hot	0.50	-	0.94	-	1.14	-	2.13	-
Control	**Cold**	0.39	0.38	0.70	0.71	0.89	0.87	1.60	1.61
	Room	0.50	0.59	0.68	0.80	1.14	1.33	1.55	1.82
	Hot	0.40	0.48	0.73	0.77	0.92	1.08	1.66	1.76
w/c 0.36	**Cold**	0.45	0.33	0.82	0.79	1.24	0.93	2.27	2.19
	Room	0.52	0.57	0.77	0.85	1.43	1.58	2.13	2.36
	Hot	0.66	0.41	0.88	0.89	1.85	1.14	2.43	2.47
w/c 0.40	**Cold**	0.41	0.37	0.65	0.66	1.03	0.92	1.63	1.64
	Room	0.57	0.47	0.84	0.82	1.61	1.69	2.09	2.04
	Hot	0.73	0.53	0.94	0.94	2.00	1.83	2.34	2.35
w/c 0.48	**Cold**	0.63	0.60	0.75	0.77	1.30	1.24	1.56	1.60
	Room	0.64	0.67	0.90	0.91	1.34	1.40	1.87	1.90
	Hot	0.80	0.73	0.96	0.97	1.66	1.53	2.01	2.02
Average		0.51		0.79		1.22		1.87	

As can be seen in Figure 6.17, with the exception of one data point (30 % F – Room), results do not vary significantly between data obtained from Calorimeter I and Calorimeter II, as the polynomial fit is less dependent on the sampling rate of the calorimeter. Also, it should be noted that very little difference can be seen between data points resulting from the use of w/cm-dependent *K* values and constant *K* values. Neither the constant *K* value case or the w/cm-dependent *K* value case for either Calorimeter I or Calorimeter II meet any of the criteria outlined in Section 6.2.3.2, therefore, the *Polynomial Derivatives Method* should not be considered an adequate indicator of concrete setting. For a summary of the absolute percent error between setting times obtained with the *Polynomial Derivatives Method* and penetration resistance, for both the constant *K* value case and the w/c-dependent *K* values case for each batch tested see Figure 6.18. Data in Figure 6.18 were obtained from Calorimeter I testing, but are representative of results from both calorimeters.

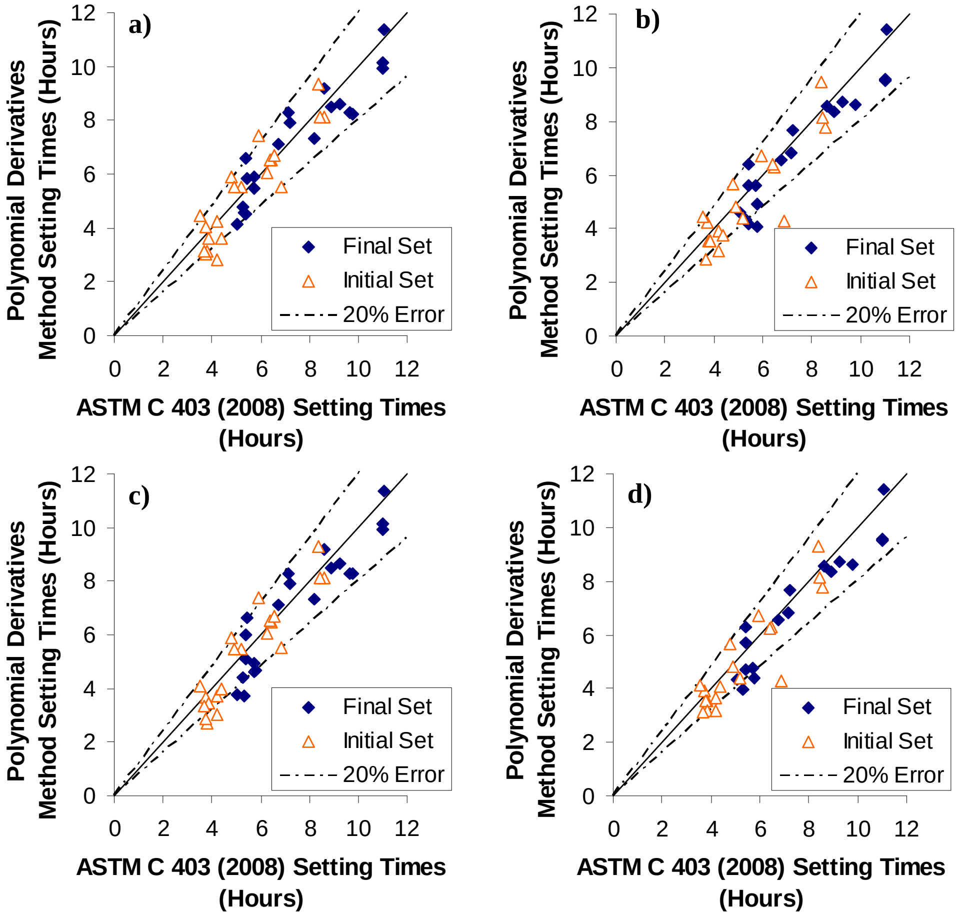

Figure 6.17: Plot of initial and final set as calculated by penetration resistance and the *Polynomial Derivatives Method* for a) constant *K* values for Calorimeter I, b) constant *K* values for Calorimeter II, c) w/cm-dependent *K* values for Calorimeter I, and d) w/cm-dependent *K* values for Calorimeter II.

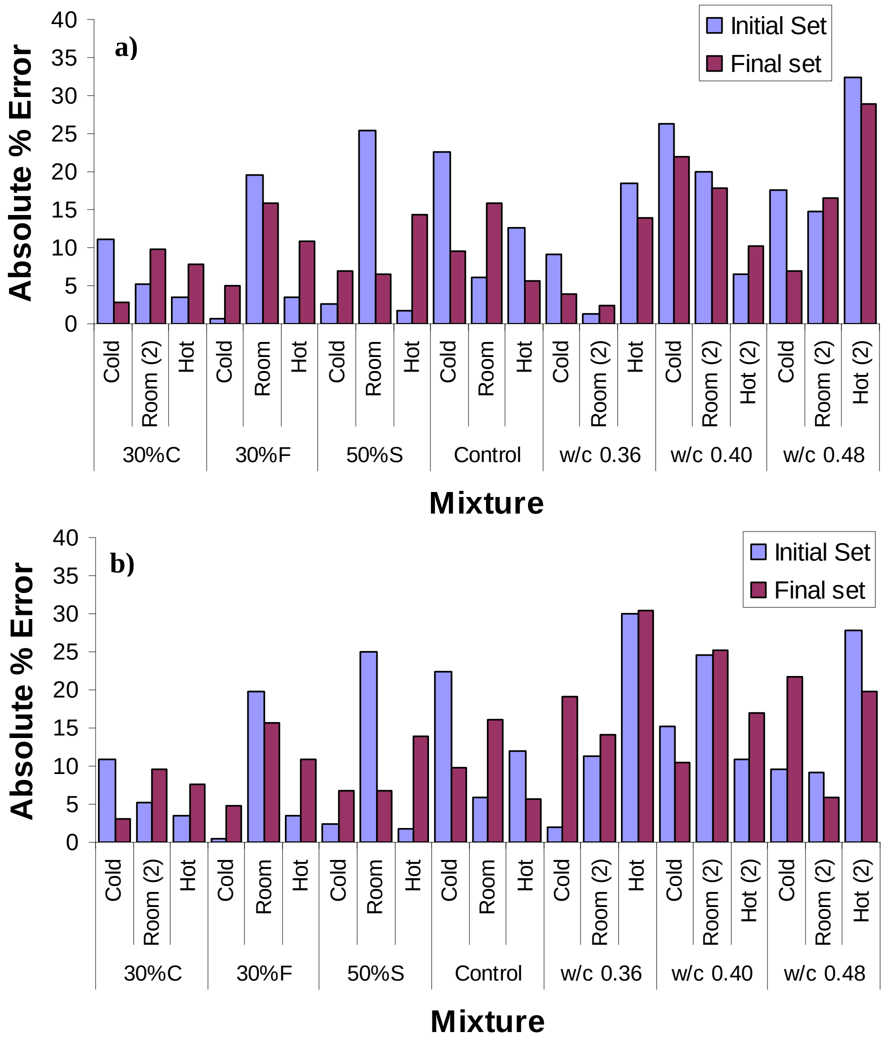

Figure 6.18: Absolute error of initial and final set as calculated by the *Polynomial Derivatives Method* as compared to ASTM C 403 (2008) for a) constant *K* values, and b) w/c-dependent *K* values for Calorimeter I

Although higher than average absolute error is noted for the constant *K* values case for mixtures with a w/cm significantly different than the average w/cm of all the mixtures (0.43), this same trend can be seen when w/cm-dependent *K* values are used (see Figure 6.18). This may suggest that a polynomial fit “smoothes out” some of the effects of w/cm on the slope of the temperature profile. As can be seen in Figure 6.19, the fit for the w/cm 0.36 mixture does not follow the contours of the curve as well as the Control mixture, which has a w/cm of 0.44.

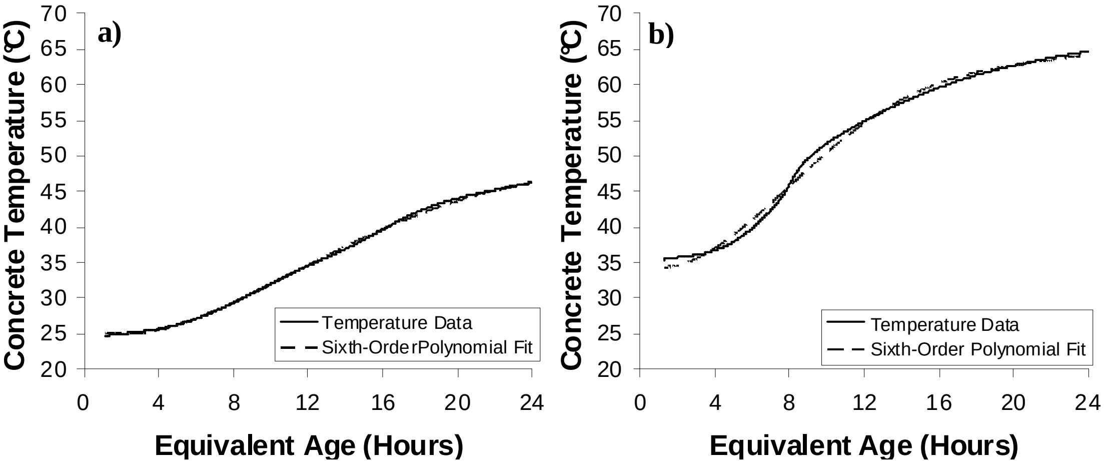

Figure 6.19: Nearly-adiabatic data and sixth-order polynomial fit to temperature data for a) Control - Room, and b) w/c 0.36 - Hot

It should be noted that, although an extremely high-order polynomial function was used in order to minimize error at early ages, the trend of some data sets were not able to be accurately captured by the function. As is evident from Figure 6.19, certain distinct trends in the slope of the temperature data were not fully represented in the polynomial fit for some temperature profiles. A typical polynomial fit, representative of a majority of the batches tested, can be seen in Figure 6.19a, where Figure 6.19b is an example of a mixture in which the polynomial does not give as accurate of a fit. Although the difference in the two curves in Figure 6.19b may seem small, there is a measurable difference in the point of maximum slope of the raw temperature data and that of the polynomial fit (approximately 10 percent). Similarly, polynomial functions fit to data from two different calorimeters may not necessarily yield consistent results (as was the case with the 30 % F – Room point mentioned earlier). Due to the fact that this "smoothing" effect does not occur in all batches tested, it affects both the accuracy and consistency with which setting can be predicted.

DOH Derivatives Method

Just as in the *Polynomial Derivatives Method*, in the *DOH Derivatives Method*, a function was fit to the first 24 hours of the temperature data and a closed-form derivative was calculated from this function. Rather than use a polynomial for the regression function, the hydration curve discussed in Chapter 2 (Equation 2.8) was selected. The "s-shape" of this function, developed by Freiesleben, Hansen, and Petersen (1984) makes it ideal for use with portland cement concrete hydration. A least-square regression analysis was performed to best-fit the hydration curve to the temperature data.

Constant K_{Di} and K_{Df} values of 0.71 and 0.97 respectively and w/cm-dependent K_{Di} and K_{Df} values of 1.7 times the w/cm and 2.3 times the w/cm, respectively, were calculated for the *DOH Derivatives Method*. A summary of the K values calculated for each batch tested for Calorimeter I (CI) and Calorimeter II (CII) can be seen in Table 6.7. It should be noted that a K_{Df} of 2.3 times the w/cm for a mixture with a w/cm of 0.48 would result in a K_{DF} of greater than 1.0, with which setting can not be calculated. Therefore, a limit for the K_{DF} value was set as 1.0.

Table 6.7: *DOH Derivatives Method K* values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	Batch	Constant K Value				w/cm-Dependent K Value			
		k_{Di}		k_{Df}		k_{Di}/(w/cm)		k_{Df}/(w/cm)	
		CI	CII	CI	CII	CI	CII	CI	CII
30%C	**Cold**	0.88	0.83	1.00	0.99	2.00	1.89	2.27	2.26
	Room	0.85	0.91	0.99	1.00	1.94	2.07	2.26	2.27
	Hot	0.78	0.83	0.98	0.99	1.76	1.89	2.22	2.25
30%F	**Cold**	0.85	0.85	1.00	1.00	1.94	1.93	2.27	2.27
	Room	0.81	0.83	1.00	1.00	1.85	1.89	2.27	2.27
	Hot	0.77	-	0.99	-	1.76	-	2.25	-
50%S	**Cold**	0.91	0.86	1.00	1.00	2.06	1.94	2.27	2.27
	Room	0.69	0.77	0.97	0.99	1.56	1.76	2.20	2.25
	Hot	0.62	-	0.97	-	1.40	-	2.21	-
Control	**Cold**	0.61	0.56	0.98	0.96	1.39	1.26	2.24	2.19
	Room	0.62	0.72	0.94	0.97	1.40	1.64	2.13	2.21
	Hot	0.50	0.72	0.88	0.97	1.14	1.65	2.01	2.21
w/c 0.36	**Cold**	0.41	0.34	0.92	0.89	1.13	0.94	2.56	2.48
	Room	0.49	0.68	0.89	0.95	1.35	1.90	2.47	2.63
	Hot	0.55	0.57	0.92	0.96	1.51	1.58	2.57	2.67
w/c 0.40	**Cold**	0.47	0.43	0.93	0.90	1.18	1.07	2.32	2.24
	Room	0.60	0.68	0.91	0.96	1.79	2.08	2.29	2.39
	Hot	0.70	0.80	0.97	1.00	2.20	2.32	2.42	2.50
w/c 0.48	**Cold**	0.80	0.76	0.95	0.95	1.66	1.58	1.99	1.97
	Room	0.71	0.83	0.99	1.00	1.49	1.73	2.06	2.08
	Hot	0.88	0.93	1.00	1.00	1.83	1.94	2.08	2.08
Average		0.71		0.97		1.69		2.27	

Setting, in terms of equivalent age, for both the *DOH Derivatives Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.20. All data points that fall between the dashed lines represent mixtures in which setting times obtained by the two methods were within an error of ± 20%. Some differences were noted in results obtained from Calorimeter I and Calorimeter II as is evident by the general shift downward of the points in Figure 6.20. These differences were not as distinct as those noted for the *Direct-Slope Derivatives Method*, but may indicate a slight reliance of the *DOH Derivatives Method* on the sampling rate of the thermal device.

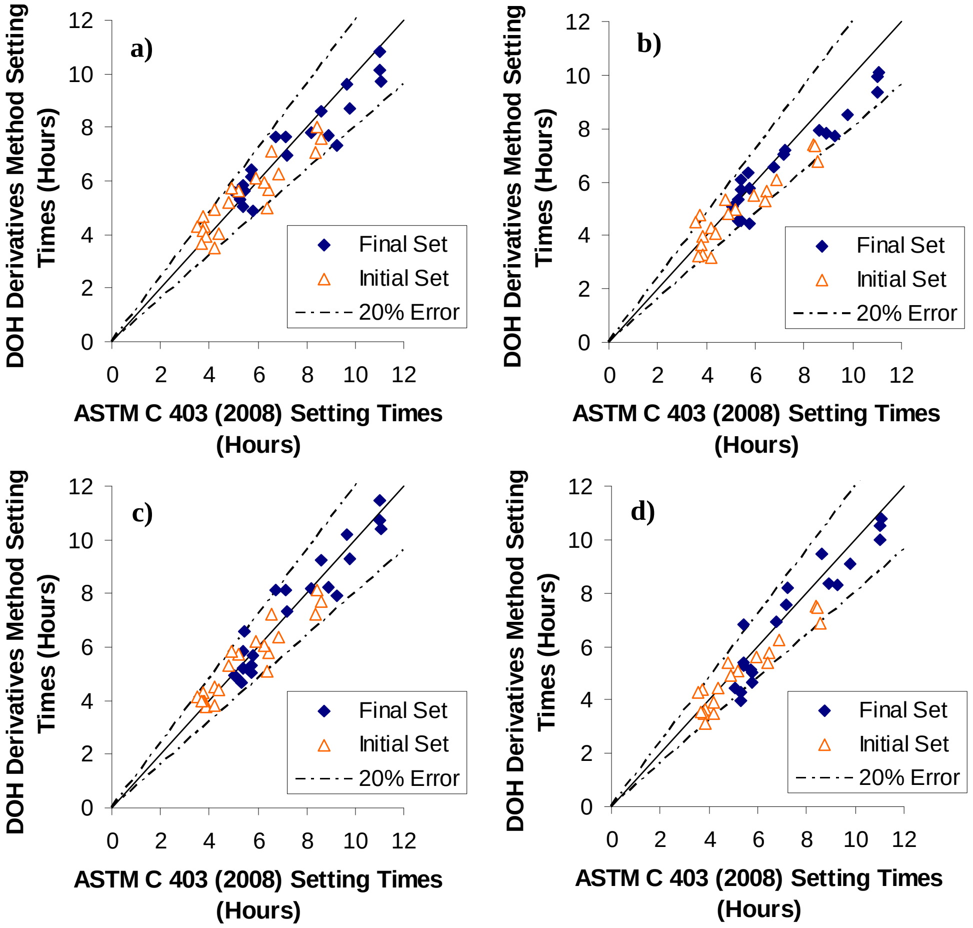

Figure 6.20: Plot of initial and final set as calculated by penetration resistance and the *DOH Derivatives Method* for a) constant *K* values for Calorimeter I, b) constant *K* values for Calorimeter II, c) w/cm-dependent *K* values for Calorimeter I, and d) w/cm-dependent *K* values for Calorimeter II.

It should be noted that, similar to the function used in the *Polynomial Derivatives Method*, the hydration function, when fit to data is not always representative of the temperature data itself. As can be seen in Figure 6.21, there is noticeable error between the raw temperature rise and the temperature rise of the fit hydration curve.

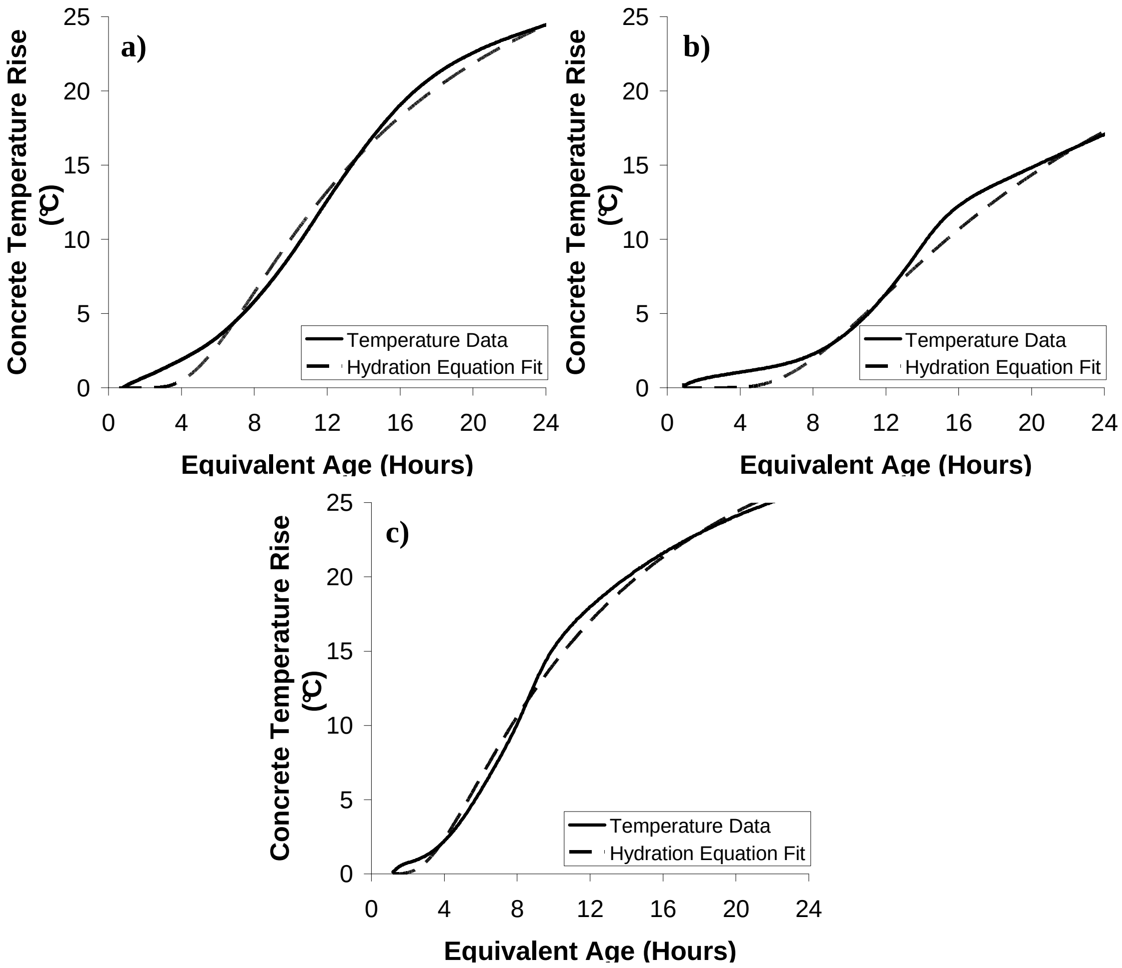

Figure 6.21: Best-fit temperatures with the DOH function versus measured temperature for a) Control – Cold, b) 30%C – Room (2), and c) w/c 0.48 – Hot (2)

Temperatures in Figure 6.21 have been converted to temperature rise by subtracting the mixtures initial temperature in order to aid in comparisons between batches of differing testing temperatures. One notable difference in the error introduced by the polynomial function versus that introduced by the hydration curve is the consistency with which it occurs. While the polynomial fit may or may not fit well to the data, a trend can be observed in error of the hydration equation fit. As can be seen in Figure 6.21, the error that is introduced by the hydration equation is relatively consistent across both a range of mixture proportions and mixture temperatures. Although this may lead to a lack of accuracy in the calculation of the derivatives themselves, setting was still able to be measured with reasonable accuracy and consistency.

For a summary of the absolute percent error between setting times obtained through the *DOH Derivatives Method* and penetration resistance, for both the constant *K* value case and the w/c-dependent *K* value case for each batch tested see Figure 6.22. Data in Figure 6.22 were obtained from Calorimeter I testing. Calorimeter II results were similar, but yielded slightly greater error. Initial set predicted by the w/cm-dependent case of the D*OH Derivatives Method* for data from Calorimeter II, compare to ASTM C 403 (2008) results within the ± 20% error range defined by Criterion b as outlined in Section 6.2.3.2. Although the w/cm-dependent results from Calorimeter I are extremely close to meeting Criterion b, final set for both the Control – Hot and the w/cm 0.48 – Cold (2) batches compare to ASTM C 403 (2008) results with greater than 20 % error. Since none of the criteria outlined in Section 6.2.3.2 were met for final set, the D*OH Derivatives Method* may be considered an adequate indicator of initial set, but should not be considered an indicator of final set.

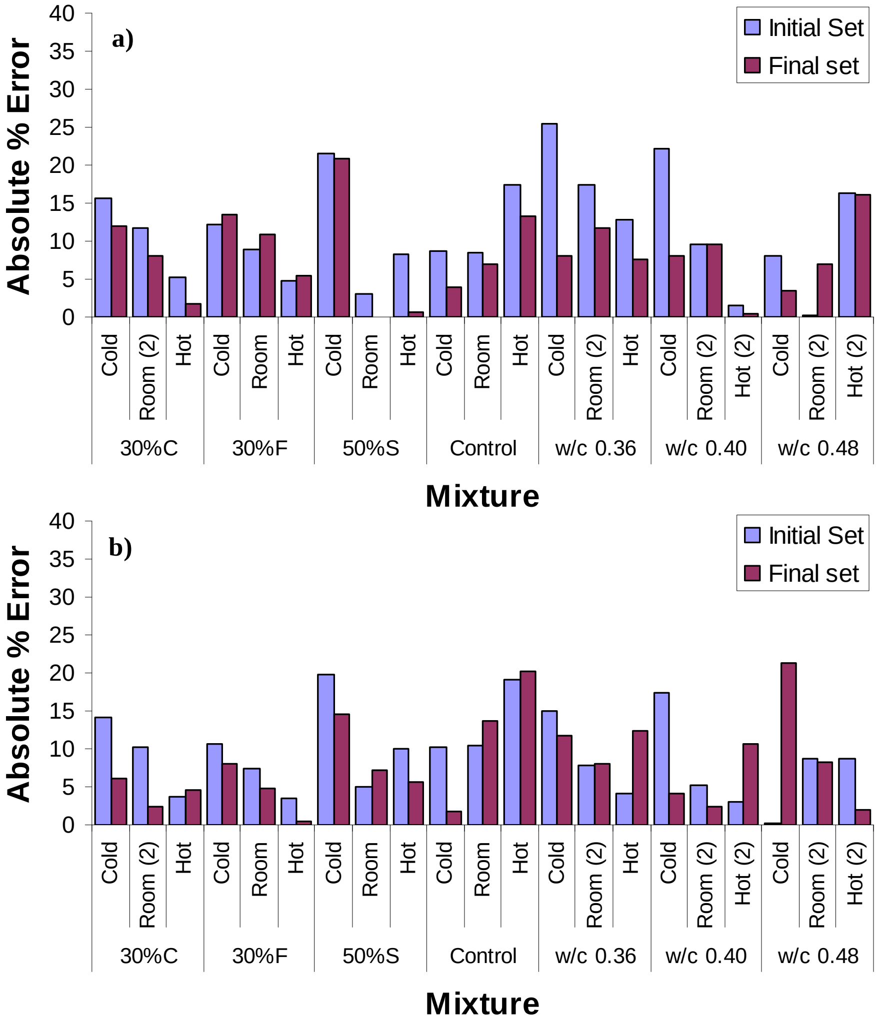

Figure 6.22: Absolute error of initial and final set as calculated by the *DOH Derivatives Method* as compared to ASTM C 403 (2008) for a) constant *K* values, and b) w/c-dependent *K* values for Calorimeter I

6.2.3.4 *Derivatives Method* – Discussion

When evaluating methods of determining setting of concrete, it is important to consider both the accuracy with which set can be predicted, and the application of the method both in the field and the laboratory. In this section, methods are compared, and the various advantages and disadvantages of each discussed. Recommendations concerning the *Derivatives Method* in general are also presented.

Comparison Between Various Derivatives Methods

The absolute error for each method was averaged for initial and final set times and tabulated for both Calorimeter I and Calorimeter II and can be seen in Table 6.8. Based on the three versions of the *Derivatives Method* evaluated, setting times were measured most accurately (as compared to penetration resistance methods) for the *DOH Derivatives Method*. Results from different calorimeters are kept separate in this discussion due to the varying influence that calorimeter type has on the different analysis methods. In general, data from Calorimeter I will be used for all comparisons, although data from both calorimeters can be seen in Table 6.8.

Table 6.8: Average absolute error of initial and final set values for *Derivatives Methods* as compared to ASTM C 403 (2008)

Analysis Type	**Average Absolute Error (%)**			
	Calorimeter I		Calorimeter II	
	Constant K value	w/cm-Dependent K value	Constant K value	w/cm-Dependent K value
Direct-Slope	12.2	8.8	13.6	13.1
Polynomial	11.8	12.6	11.8	12.1
DOH	9.7	8.7	10.8	10.4

The *DOH Derivatives Method* yielded the most accurate results when compared to ASTM C 403 (2008) for both the constant and w/cm-dependent cases, although the *Direct-Slope Derivatives Method* yielded a similar average absolute error for w/cm-dependent *K* values. For both the *Direct-Slope Derivatives Method* and the *DOH*

Derivatives Method, the w/cm-dependent *K* value case was more accurate than the constant *K* –value. The polynomial-based method had the highest overall absolute error for the w/cm-dependent case, and was the only method in which error increased for the w/cm-dependent case.

When evaluating the feasibility of using thermal methods to indicate setting of concrete, both the relative error of the method and logistical considerations must be examined. The Direct-Slope Method measures setting with a similar error as the *DOH Derivatives Method* when compared to ASTM C 403 (2008), however, the *DOH Derivatives Method* is the most accurate. Additionally, the *Direct-Slope Derivatives Method* has several logistical advantages over other methods. Since the method does not require a best-fit curve to calculate derivatives, the 24-hour testing period required for the other proposed methods is not needed to determine setting times. Setting can be determined as soon as the maximum derivative can be calculated. Also, the derivatives calculated for this method are more representative of the true slope of the temperature profile than some methods.

Disadvantages of the *Direct-Slope Derivatives Method* include its reliance on a high sampling rate, and the fact that a relatively large number of points are required in order to calculate the first derivative. As was mentioned previously, the variation in results noted between setting times obtained from the analysis of Calorimeter I data and Calorimeter II data is attributed to the different sampling rate of the two devices. Data points from Calorimeter II were sampled at one point every 15 minutes, which led to a smoothing interval of 1/15th of that used for analysis of Calorimeter II data. This large sampling interval led to less accurate derivative calculations and a higher variability in results for Calorimeter II. Although having a high sampling rate is advantageous for a number of reasons, this method's dependence on this fact puts it at a disadvantage when compared to other methods. Due to the smoothing interval required by this method, the maximum first derivative can not be established until a few hours after final set has occurred. Although as proposed in this project, this can be considered an advantage over the other methods (which require at least 24 hours of data to calculate setting), when using some of the methods for real-time measurement of setting.

Of the three *Derivatives Method*s considered, the *Polynomial Derivatives Method* had the highest error when compared to penetration resistance-based setting times. There are a few logistical disadvantages of the method as well. First, the high-order polynomial function used to calculate the derivatives sometimes could not accurately fit to temperature data yielding higher than normal variability and high errors associated with w/cm-dependent *K* values. The unpredictability of this function, as well as the need to use a high-order polynomial regression analysis (witch would be cumbersome to carry out without the use of a commercial spreadsheet product) to calculate results puts this method at a disadvantage when compared to other methods. Also, since the method needs 24 hours of data to calculate setting would render this method impractical for most field applications. The one significant advantage of this method is that results are not highly dependent on the sampling rate of the thermal device used.

The *DOH Derivatives Method* was determined to have the highest accuracy of the three *Derivatives Method*s evaluated. Additional advantages for the method include the lack of dependence on device sampling rate, and the fact that the use of the hydration equation to quantify concrete hydration is a fairly established practice (see Section 2.1.3).

Also, although as proposed the *DOH Derivatives Method* requires around 24 hours of data to calculate setting, this method can be modified in order to allow for a real-time calculation of final. Since final set for the *DOH Derivatives Method* occurs at or near the maximum first derivative of the temperature profile, when modified, setting can be calculated as soon as final set occurs. Since final set occurs at approximately 70-80 % of the maximum derivative as calculated by the polynomial equation, final set would be long past before it could be calculated by a similarly modified *Polynomial Derivatives Method*. No such modification is possible for the *Direct-Slope Derivatives Method* for reasons mentioned previously. A modification to the *DOH Derivatives Method* will be presented in the following section.

There are also a few disadvantages to the *DOH Derivatives Method* worth noting. Due to the fact that early-age data are not fit well using the hydration equation, the derivative calculated is not representative of the true slope of the temperature profile measured. This does not affect the variability or accuracy with which setting can be calculated, but it may lead to incompatibility with true derivative-based methods to estimate concrete setting. Also, although the relatively high K values calculated for the *DOH Derivatives Method* is adventitious for the possibility of real-time measurement of setting, it has its disadvantages as well. For the case of a w/cm-dependent K value, K_{Df} was determined to be 2.2 times the w/cm. For mixtures with a w/cm that is greater than 0.45, the method proposed would yield a ratio of the slope at setting to the maximum slope of greater than 1.0. This effect may limit the effectiveness of the w/c-dependent *DOH Derivatives Method*, as proposed, to mixtures with a w/cm of less than 0.45.

Modified DOH Derivatives Method

The real-time calculation of thermal setting could be very useful for field applications. Although the *DOH Derivatives Method* as proposed does not allow for the immediate determination of initial set, modifications were proposed to allow for calculation of final set as, or even possible before, it occurs by continually fitting the hydration curve to data until the maximum derivative is reached.

To investigate the possibility of repeatedly calculating a derivative as testing progresses, the hydration function shown in Equation 2.8 was fit to temperature data at 45-minute intervals. The closed-form derivative of each hydration equation was then calculated and plotted to produce the real-time calculation of the maximum rate of hydration with the *DOH Derivatives Method.*

As can be seen in Figure 6.23, the maximum calculated first derivative of the hydration equation continues to increase as the sample hydrates. Once the maximum slope of the temperature data are reached, successive best-fit hydration curve yield continually decreasing maximum first derivatives (all occurring at approximately the same time). The point at which the maximum rate begins to decrease can be considered the point of maximum slope of the temperature profile. This allows a user to find the set time relatively soon after the maximum is reached, which, due to elevated temperatures in the calorimeter, may occur before in-place final set. Setting was calculated by this modified version of the *DOH Derivatives Method* for the batch investigated and a similar error as the original method was noted (12.7 % error when compared to ASTM C 403 as compared to 11.9 % in the original *DOH Derivatives Method*).

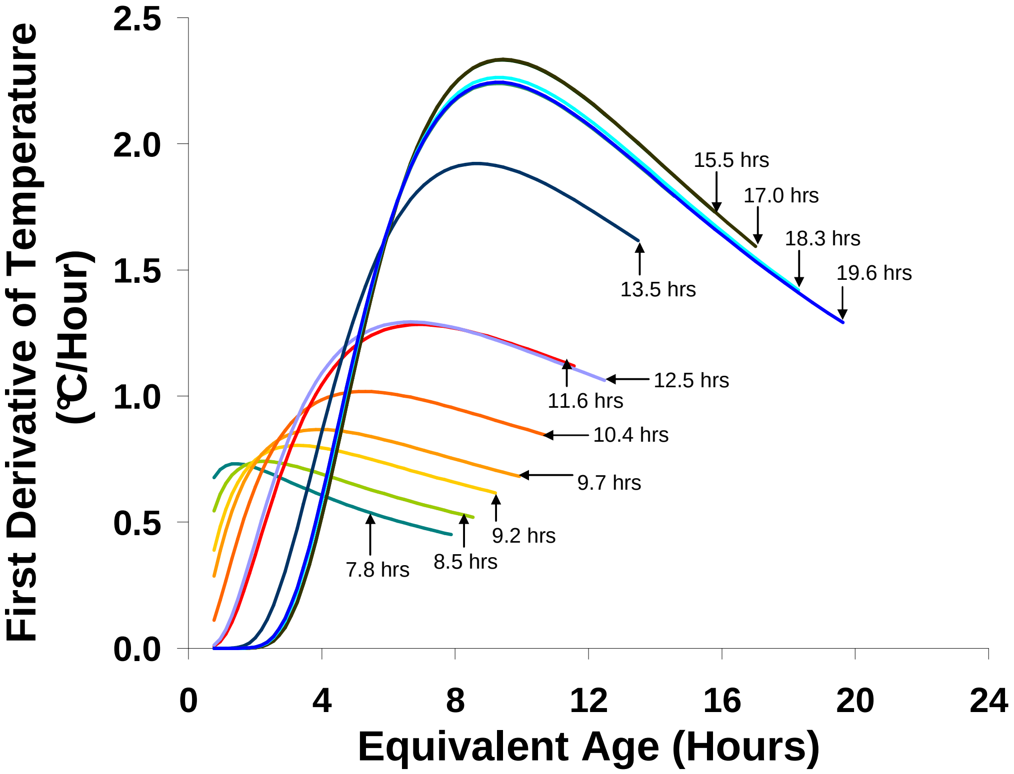

Figure 6.23: First derivative of the temperature profile as calculated by the hydration equation fit to data at 45-minute increments for 30%C – Cold

Recommendations for the use of the Derivatives Method

Recommendations specifically for calculation of setting by the *Derivatives Method* and future work are as follows:

- w/cm-dependent *K* values should be used in order to minimize error due to mixture proportions,
- the *DOH Derivatives Method* should be used over other methods evaluated in this thesis to minimize error and allow for the possibility of real-time calculation of final set, and

- calorimeter sampling rate should be limited to no less than one point per minute.

6.2.3.5 *Fractions Method* – Background and Methodology

In 1980, Byfors proposed the use of a degree of hydration based formulation to define the start of significant strength gain, or final set, of a concrete mixture. Since then, several researchers have acted on the assumption that setting occurs at a pre-defined degree of hydration (Schindler 2004b, Edson 2007). In theory, once concrete has hydrated to these pre-defined degree of hydration values, enough hydration products have formed to lead to the loss of workability (initial set) and start of strength gain (final set) respectively. Due to its ease of use, temperature-based methods for defining and measuring degree of hydration have been the method of choice for many researchers. For more information on the use of thermal methods to measure the degree of hydration see Section 2.1.3.

Recently, two general methods have emerged from this research for the prediction of setting from temperature profiles. In 2007, Sandberg and Liberman proposed the use of thermal data obtained from open-topped insulated devices to measure setting. Along with the *Derivatives Method* discussed earlier, the *Fractions Method* was proposed. In the *Fractions Method,* initial and final set are defined as specific fractions of the maximum temperature measured during testing. Further analysis of the specific testing and methodology used (see ASTM round robin testing in Chapter 3) yielded results that were dependent on the type of thermal device used, sample properties, and ambient temperatures. Schindler (2004b) proposed a method in which the degree of hydration of each sample is calculated using thermal data and setting is defined in terms of a critical degree of hydration. A w/cm-dependent critical degree of hydration was defined for both initial and final set.

Variations of both of these methods were evaluated in this testing program. An approach similar to Sandberg and Liberman's (2007) *Fractions Method* modified to be used with data that has been adjusted for device losses and maturity as discussed in Section 6.2.1 was attempted and will be called the *Direct-Fractions Method*. The method proposed by Schindler (2004b) was also slightly modified and evaluated in this study and will be referred to as the *DOH Fractions Method*. In addition to these two methods, a third, in which setting is defined as occurring after a pre-defined temperature rise was attempted. This method will be identified as the *Delta T Fractions Method*.

Equilibrium Effect on Initial Temperatures

It should be noted that, regardless of the version of the *Fractions Method* used, both initial and final set occur at a very low degree of hydration (or temperature rise). As is shown in Table 6.9, approximately 2 °C adiabatic temperature rise occurs at initial set and around a 4 °C adiabatic temperature rise occurs at final set. Due to these relatively small values being used to define setting, the *Fractions Method* is sensitive to small fluctuations in the measurement of temperature caused by the testing apparatus.

Table 6.9: Adiabatic Temperature Rise corresponding to initial and final set for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	Batch	Delta T			
		Initial Set		Final Set	
		CI	CII	CI	CII
30%C	Cold	2.8	2.4	4.8	4.3
	Room	2.5	2.5	4.9	4.7
	Hot	1.6	2.4	3.8	4.9
30%F	Cold	2.3	1.9	4.3	3.9
	Room	1.7	2.1	3.9	4.1
	Hot	1.5	-	3.9	-
50%S	Cold	2.3	1.8	4.2	4.0
	Room	1.5	1.5	3.2	3.3
	Hot	0.8	-	3.6	-
Control	Cold	2.7	1.9	5.0	4.2
	Room	1.9	1.7	3.8	3.5
	Hot	0.6	1.7	2.4	3.8
w/c 0.36	Cold	1.8	1.3	3.9	3.1
	Room	1.1	1.7	3.1	3.8
	Hot	1.1	1.7	3.0	3.8
w/c 0.40	Cold	2.3	1.1	4.2	3.0
	Room	1.5	1.6	3.2	3.5
	Hot	1.2	2.0	3.3	4.8
w/c 0.48	Cold	2.5	2.0	3.7	3.1
	Room	2.0	2.1	4.7	4.9
	Hot	2.3	2.4	5.0	5.6
Average		1.9		4.0	

One example of a fluctuation due to setup is introduced while a sample just inserted in the calorimeter is reaching equilibrium with the testing environment. An example of this effect can be seen in Figure 6.24. In this example, concrete mixed at three temperatures (Hot, Room, and Cold) was tested in a room temperature calorimeter. The thermal temperature probe was inserted directly into the concrete at the beginning of testing. As can be seen, when a concrete sample is placed in a semi-adiabatic device that is at a substantially different temperature than the sample itself, there is a one to two hour period in which the device and sample must reach an equilibrium temperature. This equilibrium period is represented in Figure 6.24 as a temperature *drop* for the Hot batch and a temperature *rise* for the Cold batch. This can lead to difficulty determining an initial temperature for a concrete sample from which to measure adiabatic temperature rise. Although care was taken to avoid such effects as much as possible, fluctuations of just one to two degrees Celsius can lead to substantial error in the prediction of setting times.

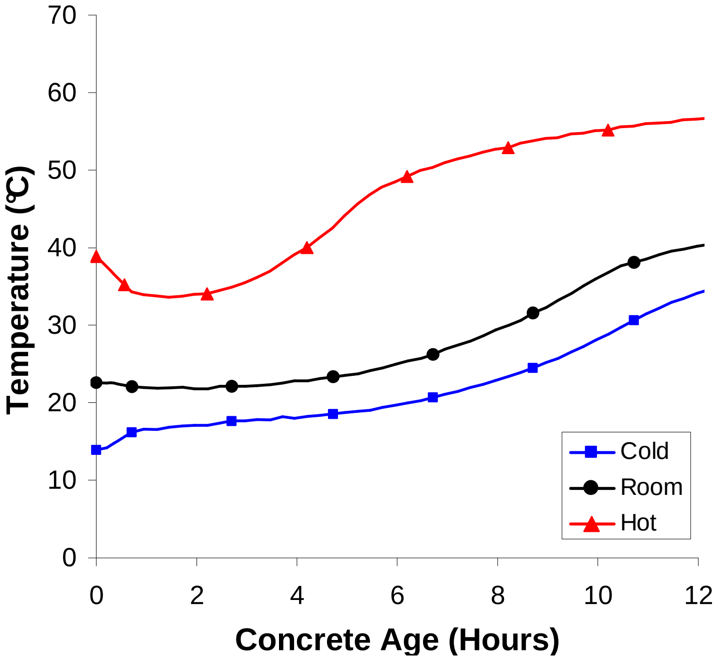

Figure 6.24 : Example of equilibrium effect during semi-adiabatic testing for Calorimeter II

One unexpected cause of error introduced by the testing setup was the location of the thermal probe within the testing device. Calorimeter I had a thermal probe located *under* the concrete specimens attached to a metal plate, whereas Calorimeter II had a probe that was inserted directly into the concrete. To investigate the effect of probe location, a test run was completed post-testing in which a concrete specimen was batched and mixed as per the testing procedure outlined in Chapter 4 for a Hot batch, and tested in Calorimeter I for 24 hours with both the original thermal sensor below the specimen and an additional sensor inserted into the center of the concrete specimen. Results from this test run can be seen in Figure 6.25.

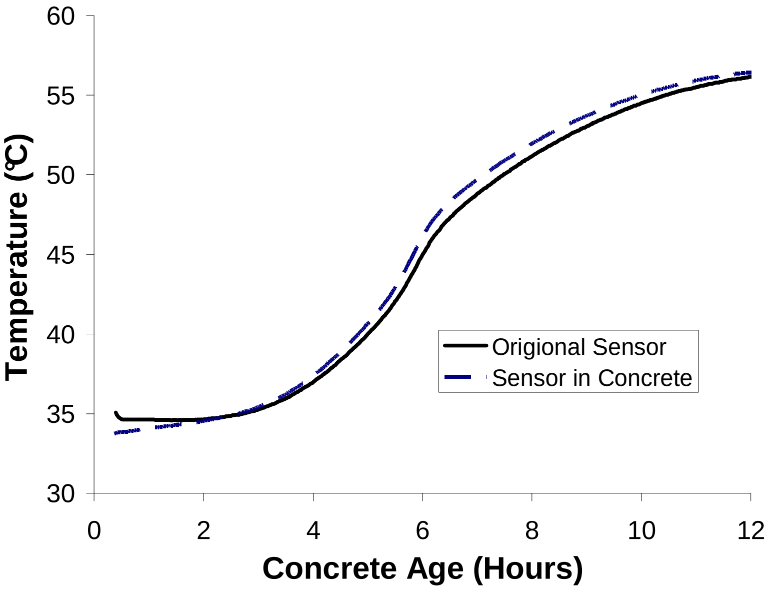

Figure 6.25: The effect of sensor location on measured temperatures for Calorimeter I (Hot)

As can be seen, the sensor in the concrete provides temperature values that start approximately 1.5 °C cooler than the sensor under the specimen. Therefore, since the probe for Calorimeter I was not in contact with the concrete itself, there was a time period (approximately 1-1.5 hours) in which the thermal data recorded by the probe were not representative of the temperature of the concrete. After 1.5 hours, the sensor within the concrete gradually reaches the temperature of the other sensor, and then remains approximately 1 °C warmer until approximately 12 hours.

This effect can be explained by the fact that, although the batch temperature after mixing was 35 °C, heat was lost during sampling, therefore the sample was cooler than the testing environment (which is kept at 35 °C for hot batches) at the onset of testing. After approximately 1.5 hours, the sensor under the sample has reached the temperature of the concrete specimen, but is slightly slower to register the heat gain of the specimen; therefore the in-concrete sensor registers a slightly higher temperature. Due to the fact that a cold concrete specimen is usually slightly warmer than the device at the beginning of testing, the effect of the temperature sensor being located under the specimen would be the opposite of the effect of a hot batch. While setting times for hot batches tend to be over-predicted by using false thermal data, setting times for cold batches would be under-predicted.

A similar trend was noticed when results from the Calorimeter I and Calorimeter II were compared for a given mixture. As can be seen in Figure 6.26, there is a measurable difference in the very early-age mixture temperatures recorded by the two calorimeters for the w/c 0.36 Hot and w/c 0.36 Cold batches. The room temperature batch seemed unaffected as the target concrete temperature is very similar to the concrete laboratory temperature.

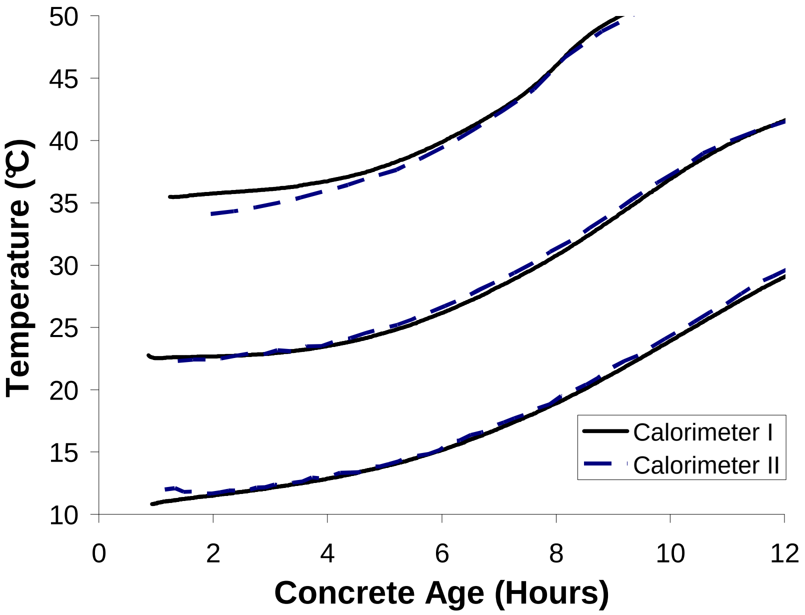

Figure 6.26 Temperature Date from Calorimeter I and II for w/c 0.36 (Cold, Room, and Hot)

This effect may appear insignificant, but can lead to significant error when setting is defined by a small change in recorded temperature. In the test case (Figure 6.26), an error of approximately 19% and 10% would be introduced if one defined setting times as the time elapsed before the sensor of Calorimeter I registered a temperature rise of 2 °C and 4 °C, respectively. Although it was assumed that this effect occurred for all Hot and cold batches, it is difficult to know whether it was present for all mixtures, and to what extent, therefore no adjustment to the collected data could be made prior to analysis. For this reason, data from Calorimeter II, although collected using a much higher sampling interval, were found to me more reliable for some analysis methods.

Defining Fractions Method Initial and Final Set Times

For the *Fractions Method*s, setting was defined three different ways. Both initial and final set were defined as the equivalent age at which either some fraction of the maximum temperature (K_{Fi} and K_{Ff}) is reached, a specific degree of hydration (α_{cr}) is reached, or a specific rise in adiabatic temperature (ΔT) has occurred. In all three of these cases, both a w/cm independent and a w/cm-dependent formulation was developed, just as was done for the *Derivatives Method*. Details of the definitions of setting for the *Direct-Fractions Method*, the *DOH Fractions Method* and the *Delta T Fractions Method* will be evaluated in detail in the following sections.

Methodology

The *Direct-Fractions Method, DOH Fractions Method,* and *Delta T Fractions Method* were each evaluated individually. The general methodology followed for all *Fractions Method* analyses can be divided into three general stages: 1) the pre-calibration of the method, 2) the calculation of set indicators, and the calculation and 3) evaluation of the *Fractions Method* set times. A schematic of the analysis methodology for the *Fractions Method* can be seen in Figure 6.27.

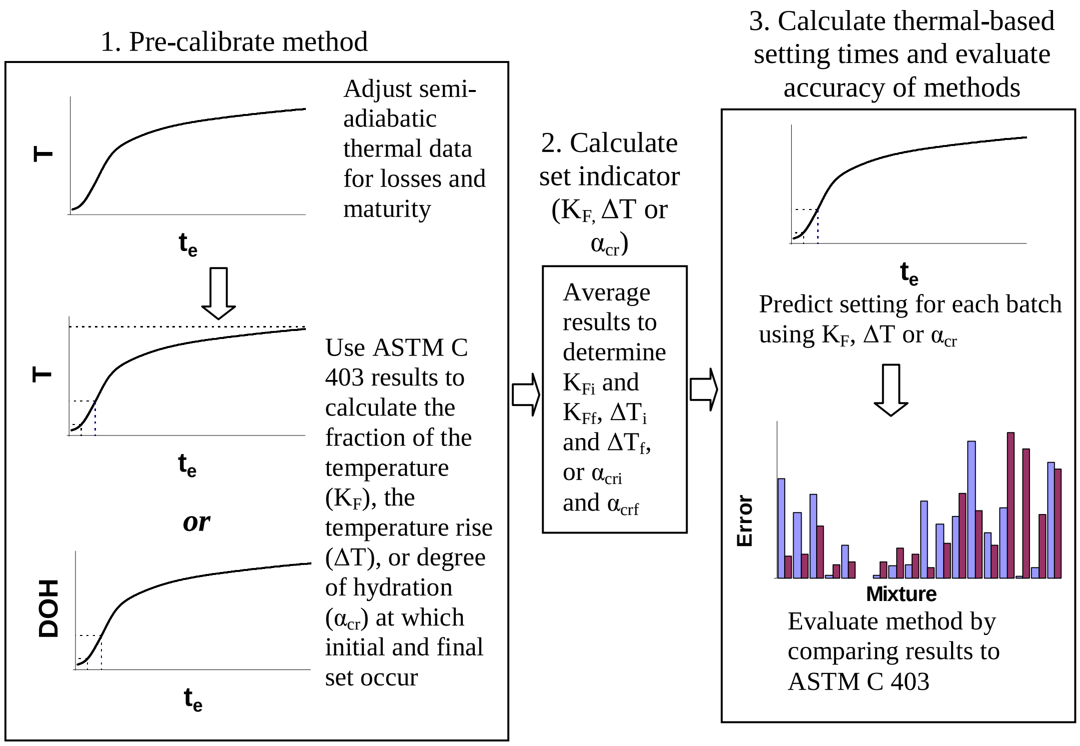

Figure 6.27: Schematic showing analytical methodology used to evaluate the *Fractions Method*

6.2.3.6 *Fractions Method* – Limits of Acceptability

Limits of acceptability identical to those defined for the *Derivatives Method* were used to evaluate the various versions of the *Fractions Method*. For details, see Section 6.2.3.2.

6.2.3.7 *Fractions Method* – Analytical Procedure

In the following section, analytical procedures for a variety of degree of hydration-based methods are presented. Details concerning the analysis method are presented as well as results and a brief discussion.

Direct-Fractions Method

For the *Direct-Fractions Method*, initial and final set times were defined as the equivalent age at which a temperature rise equal to a certain percentage of the maximum nearly-adiabatic temperature rise is measured. Just as in the method proposed by Sandberg and Liberman (2007), a baseline was set, and fractions were measured as the ratio of the temperature rise from this baseline to the maximum temperature measurement. In this case, all analysis was completed on nearly-adiabatic temperature data that has been adjusted for maturity rather than raw temperature data. A graphical representation of the *Direct-Fractions Method* can be seen in Figure 6.28. The variable K_{Fi} was used to notate the fraction corresponding to initial set and likewise, K_{Ff} corresponds to final set.

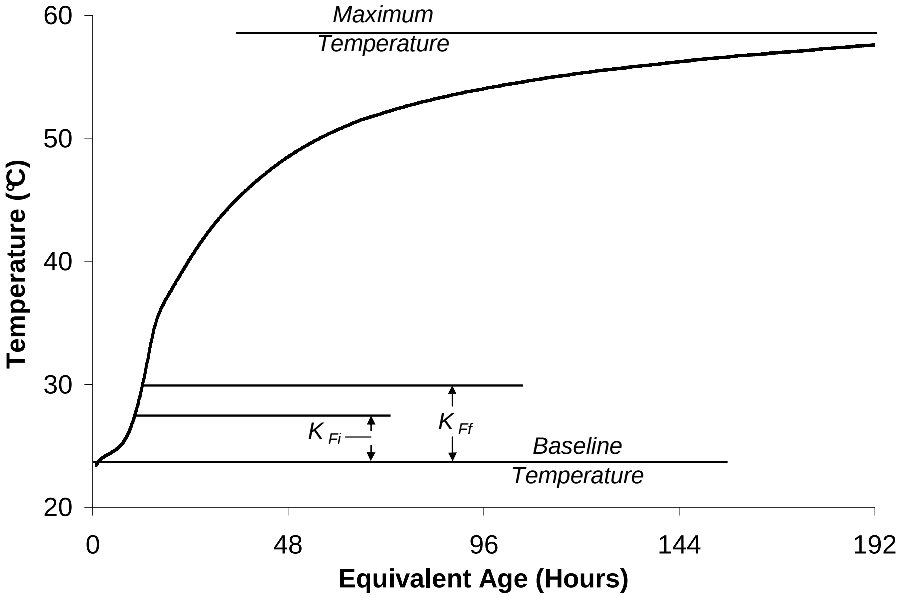

Figure 6.28: Initial set and final set as defined by the *Direct-Fractions Method* (adapted from Sandberg and Liberman 2007)

The baseline temperature used for analysis, as seen in the above figure, was taken as the minimum temperature measured. Although care was taken to ensure that all mixtures had an initial temperature of 10 °C, 23°C, or 35 °C, due to heat loss during sampling and equilibrium effects within the device, this was not always the case. The minimum temperature recorded for a given batch was taken to be the baseline, even if it was not necessarily the first temperature recorded.

Both constant K values and w/cm-dependent K values were used during analysis to attempt to account for the increase in cement particle spacing for mixtures with a higher w/cm. A summary of K values calculated for each batch for Calorimeter I and Calorimeter II is presented in Table 6.10. For the constant K value case, average K_{Fi} and K_{Ff} were calculated as 0.06 and 0.12 respectively. For the w/cm-dependent case, K_{Fi} and K_{Ff} were calculated as 0.13 times the w/cm, and 0.27 times the w/cm respectively.

It should be noted that these values are significantly lower than the 0.21 and 0.42 default fraction values proposed by Sandberg and Liberman (2007) and used in the analysis of the ASTM round robin data (See Chapter 3). When using the nearly-adiabatic temperature profile instead of semi-adiabatic or non-adiabatic temperatures, it would logically follow that the fractions that are associated with setting would be significantly different. Although the fractions themselves are less than one third of the default values originally proposed, the actual temperature rise that occurs before setting is similar to that seen in past work. (In fact, due to very early age thermal losses, temperature rise values measured in this research were slightly *greater* than those seen in past work). The main source of the differences in *K* values is the much higher *final temperature* recorded when device losses are taken into effect.

Table 6.10: *Direct Fractions Method K* values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	**Batch**	**Constant K Value**				**w/cm Dependent K Value**			
		k_{Fi}		**k_{Ff}**		**k_{Fi}/(w/cm)**		**k_{Ff}/(w/cm)**	
		CI	**CII**	**CI**	**CII**	**CI**	**CII**	**CI**	**CII**
30%C	**Cold**	0.10	0.08	0.17	0.15	0.22	0.19	0.38	0.34
	Room	0.07	0.08	0.14	0.14	0.16	0.17	0.31	0.33
	Hot	0.05	0.06	0.11	0.13	0.11	0.15	0.26	0.30
30%F	**Cold**	0.09	0.08	0.17	0.16	0.20	0.18	0.38	0.36
	Room	0.06	0.08	0.14	0.16	0.14	0.18	0.32	0.36
	Hot	0.05	-	0.13	-	0.11	-	0.29	-
50%S	**Cold**	0.09	0.07	0.16	0.15	0.20	0.16	0.37	0.34
	Room	0.04	0.05	0.09	0.11	0.10	0.11	0.21	0.25
	Hot	0.02	-	0.10	-	0.05	-	0.23	-
Control	**Cold**	0.07	0.05	0.13	0.12	0.16	0.12	0.30	0.27
	Room	0.05	0.06	0.11	0.12	0.12	0.13	0.24	0.27
	Hot	0.02	0.05	0.07	0.10	0.04	0.10	0.15	0.23
w/c 0.36	**Cold**	0.05	0.03	0.10	0.08	0.12	0.09	0.27	0.22
	Room	0.03	0.04	0.08	0.10	0.08	0.12	0.23	0.28
	Hot	0.03	0.04	0.08	0.09	0.08	0.11	0.22	0.26
w/c 0.40	**Cold**	0.06	0.03	0.11	0.08	0.16	0.08	0.28	0.20
	Room	0.04	0.04	0.08	0.09	0.10	0.11	0.21	0.22
	Hot	0.03	0.06	0.09	0.13	0.08	0.14	0.22	0.33
w/c 0.48	**Cold**	0.07	0.06	0.11	0.09	0.15	0.12	0.22	0.19
	Room	0.05	0.06	0.12	0.14	0.11	0.12	0.26	0.28
	Hot	0.07	0.06	0.14	0.14	0.14	0.13	0.30	0.30
Average		0.06		0.12		0.13		0.27	

Setting was then calculated for each mixture using these values. Results from the *Direct-Fractions Method* were then compared to those obtained by penetration resistance testing. Setting, in terms of equivalent age, for both the *Fractions Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.29. All data points that fall between the dashed lines represent mixtures in which setting times obtained by the two methods were within an error of ± 20%.

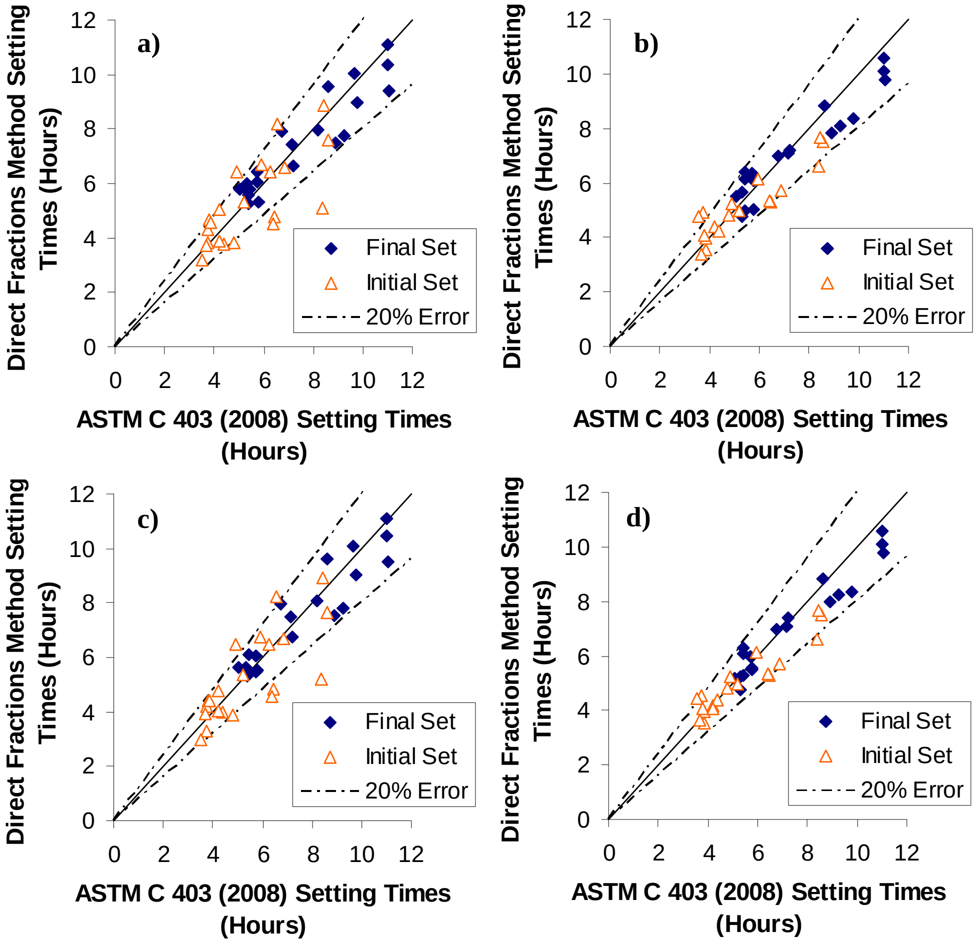

Figure 6.29: Plot of initial and final set as calculated by penetration resistance and the *Direct-Fractions Method* for a) constant *K* values for Calorimeter I, b) constant *K* values for Calorimeter II, c) w/cm-dependent *K* values for Calorimeter I, and d) w/cm-dependent *K* values for Calorimeter II.

As can be seen in Figure 6.29, results were generally more accurate for specimens tested with Calorimeter II than those tested with Calorimeter I as is evident by the tighter scatter of data points in Figure 6.29b and Figure 6.29d. All final set and most initial set data points fall within the ± 20% error lines for data from Calorimeter II for both the constant *K* values case and the w/cm-dependent *K* values case, but overall scatter appears to be less for set times calculated using w/cm-dependent *K* values. A more detailed summary of results from Calorimeter II can be seen in Figure 6.30. It should be noted that, due to device malfunction, data are not available for 30% F – Hot and 50 % Slag – Hot from Calorimeter II. Each Batch in which no data are available is indicated with an X on the horizontal axis to distinguish it from a batch with 0 % absolute error.

As shown Figure 6.30, the *Direct-Fractions Method* performs reasonably well for all batches tested. The w/cm-dependent *K* values yielded more consistent results across the range of mixtures, and lower overall error than the constant *K* values (average absolute percent error of 9.0 as compared to 10.9 for initial set, and 6.7 as compared to 8.8 for final set). A maximum error of 16 percent for final set and 26 percent for initial set was measured when the *Direct-Fractions Method* with w/cm-dependent *K* values was compared to ASTM C 403 (2008). (It should be noted that these values do not include data from Calorimeter I). Based solely on data from Calorimeter I, the w/cm-dependent *Direct-Fractions Method* may be considered an adequate indicator of final set, but should not be considered an indicator of initial set.

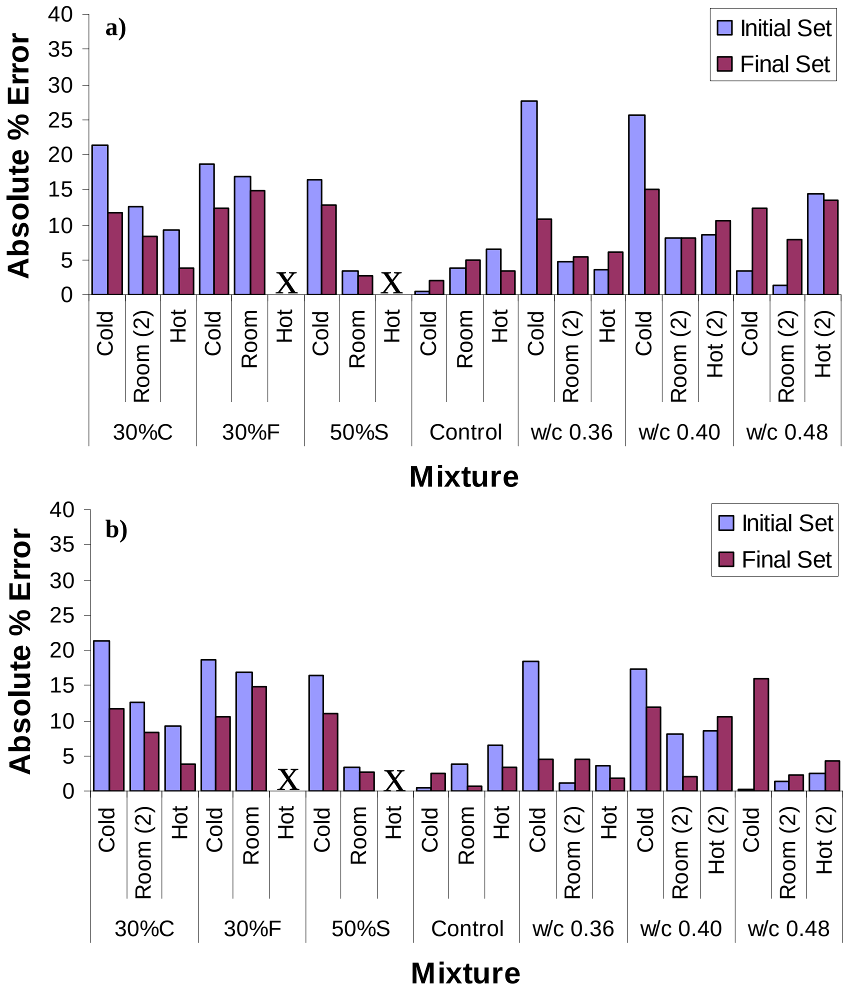

Figure 6.30: Absolute error of initial and final set as calculated by the *Direct-Fractions Method* compared to ASTM C 403 (2008) for a) constant *K* values, and b) w/c-dependent *K* values

DOH Fractions Method

As presented in Chapter 2, Byfors (1980) introduced the idea that the start of significant strength gain of concrete (or final set) occurs at a specific degree of hydration (α). A degree of hydration defined in terms of a w/c-dependent value ranging between

0.40 and 0.46 times the w/c was suggested (see Equation 2.9). Schindler (2004b) took this a step further and proposed a method of calculating initial and final setting points to correspond to those obtained through penetration resistance testing based on Byfors' equation. Multipliers (k_s) to the w/cm of 0.14 and 0.26 were proposed to calculate the critical degree of hydration corresponding to initial and final set, respectively. Edson (2007) carried out a similar analysis and found only slightly lower k_s values of 0.12 and 0.22 for initial and final set, respectively.

For *DOH Fractions Method* Analysis, initial and final set times were defined as the equivalent age at which a certain degree of hydration (α_{cri} and α_{crf}) is reached. Degree of hydration was calculated with the use of a formulation similar to Equation 2.3. Nearly-adiabatic temperature data were divided by an estimation of the total temperature rise that would be seen for a fully-adiabatic completely hydrated sample. This value was obtained through analysis of the cement compounds (see Section 2.1.3.1). Equations 2.4 and 2.5 were used to calculate the total heat of hydration available to the cementitious material at full hydration. Equation 6.3 was then used to calculate the corresponding adiabatic temperature rise. For a summary of the values calculated, see Table 6.11.

Table 6.11: Maximum adiabatic temperature rise available based on cementitious material composition

Mixture ID	Adiabatic Temperature Rise at α = 1.0 (℃)
Control	65.2
30% C	62.5
30% F	47.2
50% Slag	63.8
w/c 0.36	74.3
w/c 0.40	69.2
w/c 0.48	64.5

Both w/cm-dependent and constant α_{cr} values were calculated by averaging the degree of hydration data for initial and final set for all batches. α_{cri} was calculated as 0.030 or 0.068 times the w/cm. α_{crf} was calculated as 0.063 or 0.15 times the w/cm. The values calculated for each batch along with the average for each case can be seen in Table 6.12 for Calorimeter I and Calorimeter II. It should be noted that these values are significantly lower than values reported by both Schindler (2004b) and Edson (2007).

Table 6.12: *DOH Fractions Method α_{cr}* values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	Batch	Constant α_{cr}				w/cm Dependent α_{cr}			
		α_{cri}		α_{crf}		α_{cri}/(w/cm)		α_{crf}/(w/cm)	
		CI	CII	CI	CII	CI	CII	CI	CII
30%C	Cold	0.045	0.039	0.076	0.069	0.103	0.087	0.173	0.157
	Room	0.041	0.040	0.078	0.076	0.092	0.091	0.176	0.171
	Hot	0.026	0.039	0.061	0.079	0.058	0.088	0.137	0.179
30%F	Cold	0.048	0.040	0.092	0.082	0.110	0.091	0.207	0.187
	Room	0.036	0.044	0.083	0.087	0.082	0.100	0.189	0.198
	Hot	0.031	-	0.082	-	0.070	-	0.185	-
50%S	Cold	0.035	0.029	0.067	0.062	0.080	0.065	0.151	0.141
	Room	0.023	0.024	0.051	0.052	0.053	0.055	0.115	0.118
	Hot	0.013	-	0.056	-	0.029	-	0.127	-
Control	Cold	0.041	0.030	0.077	0.065	0.093	0.067	0.174	0.148
	Room	0.029	0.027	0.058	0.054	0.065	0.061	0.131	0.122
	Hot	0.009	0.026	0.036	0.058	0.021	0.059	0.082	0.132
w/c 0.36	Cold	0.025	0.017	0.053	0.043	0.068	0.048	0.146	0.117
	Room	0.015	0.022	0.042	0.051	0.042	0.061	0.116	0.140
	Hot	0.016	0.023	0.041	0.051	0.043	0.062	0.111	0.140
w/c 0.40	Cold	0.034	0.017	0.060	0.043	0.084	0.041	0.150	0.108
	Room	0.022	0.024	0.047	0.050	0.054	0.059	0.117	0.125
	Hot	0.018	0.029	0.047	0.070	0.044	0.073	0.118	0.174
w/c 0.48	Cold	0.040	0.032	0.058	0.049	0.083	0.067	0.121	0.102
	Room	0.032	0.032	0.073	0.076	0.067	0.068	0.153	0.158
	Hot	0.036	0.037	0.079	0.087	0.076	0.077	0.164	0.182
Average		0.030		0.063		0.068		0.146	

One possible explanation for this discrepancy is the method with which the previously published data were obtained. Both Schindler (2004) and Edson (2007) made use of the hydration curve presented in Chapter 2 (see Equation 2.8) to represent the degree of hydration of a sample. Thermal data were fit to this curve and hydration parameters were obtained for each batch tested, from which the degree of hydration corresponding to setting was calculated. When a similar method was applied to data from this project, it was noted that, although the hydration curve represented the shape of the entire temperature profile very well, there was greater than expected error at early ages (around the setting window). As can be seen in Figure 6.31, when plotted over the 4 days (equivalent age), the function has very food fit to the degree of hydration data. When data for the same batch are plotted over 12 hours (equivalent age), there is significant error around the equivalent age at which setting occurs. For the data shown in Figure 6.31, at final set (5.4 hours) the data yields a α_{cr} value of 0.15 times the w/cm for the raw degree of hydration data and 0.20 times the w/cm for the hydration curve.

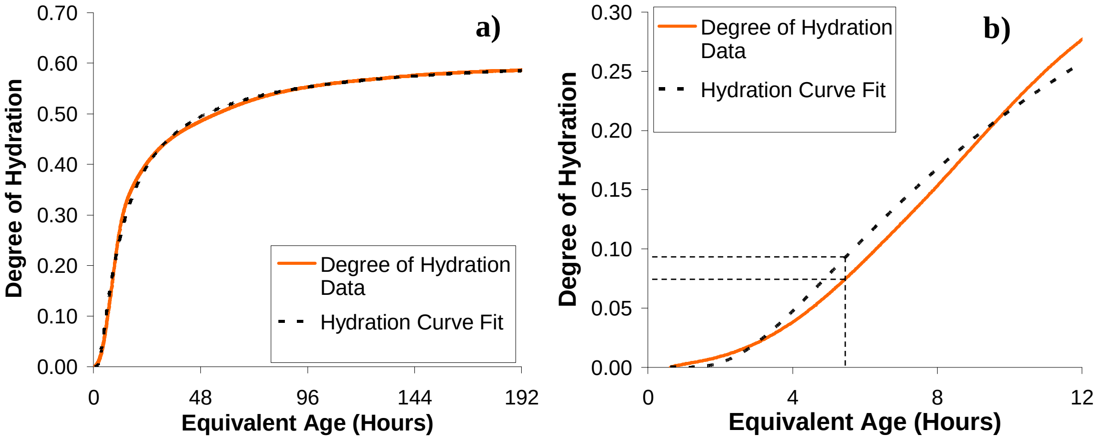

Figure 6.31: Degree of hydration as obtained from raw temperature data and a fit of the hydration equation

While it was not possible to evaluate the fit of the hydration curve to past data, it is possible that a similar trend exists. Regardless of the source of the difference in values obtained and published data, the major objective of this study is to evaluate the use of thermal methods to determined set. Although the values used for α_{cr} may vary due to differing degree of hydration calculation methods or the use of the hydration curve, the use of the method to measure setting can still be evaluated. If the method is shown to reliably measure setting, future research can be performed to determine the values of α_{cr} that should be used.

Just as in past methods, setting was calculated for each mixture using the values obtained and results were compared to those obtained by penetration resistance test results. Setting, in terms of equivalent age, for both the *Fractions Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.32.

As noted for the *Direct-Fractions Method*, results from Calorimeter II have a smaller scatter and generally less error than those from Calorimeter I. All final set values computed (from both calorimeters) compared to ASTM C 403 (2008) values with less than 20 % error from both the constant α_{cr} case and the w/cm-dependent α_{cr} case. Initial set had slightly more variability, but in general yielded results that compared to ASTM C 403 (2008) results with less than 30 % error.

A more detailed summary of results for both the constant and w/cm-dependent α_{cr} cases from Calorimeter II can be seen in Figure 6.33 It should be noted that, due to device malfunction, data are not available for 30% F – Hot and 50% Slag – Hot from Calorimeter II. Each batch for which no data are available is indicated with an X on the horizontal axis to distinguish them from 0% absolute error.

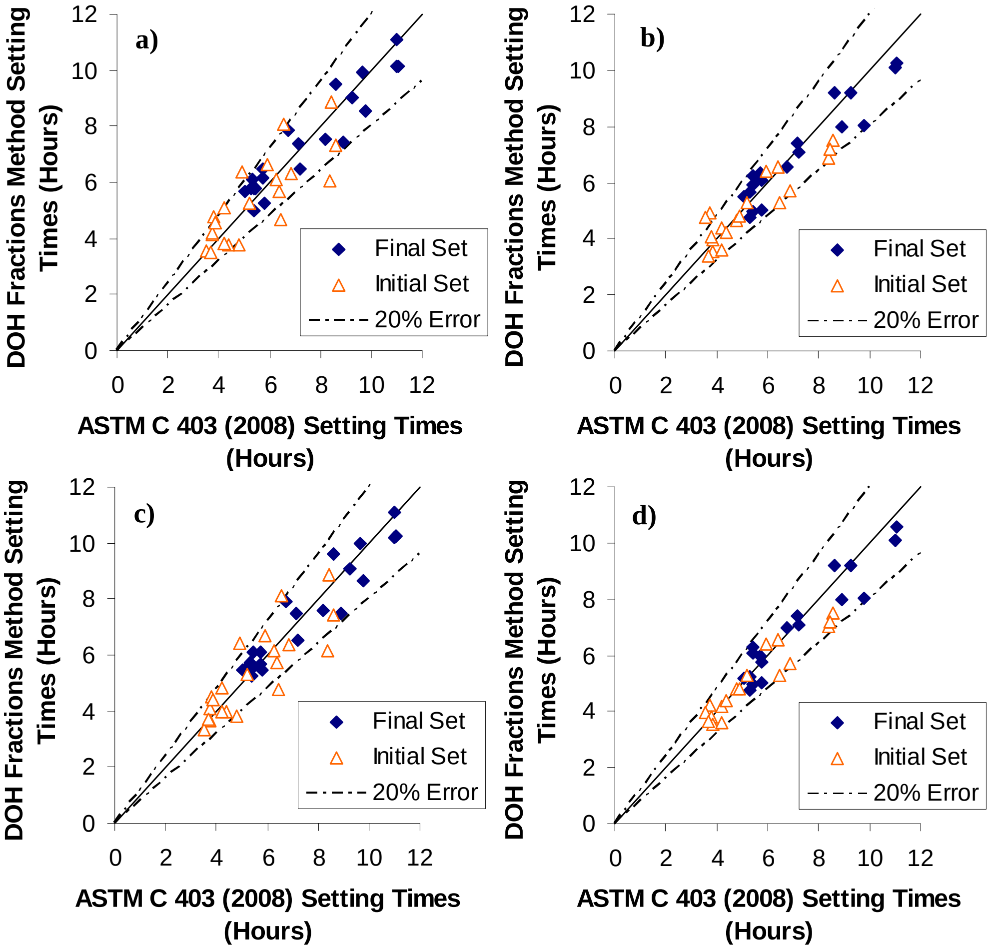

Figure 6.32: Plot of initial and final set as calculated by penetration resistance and the *DOH Fractions Method* for a) constant α_{cr} values for Calorimeter I, b) constant α_{cr} values for Calorimeter II, c) w/cm-dependent α_{cr} values for Calorimeter I, and d) w/cm-dependent α_{cr} values for Calorimeter II.

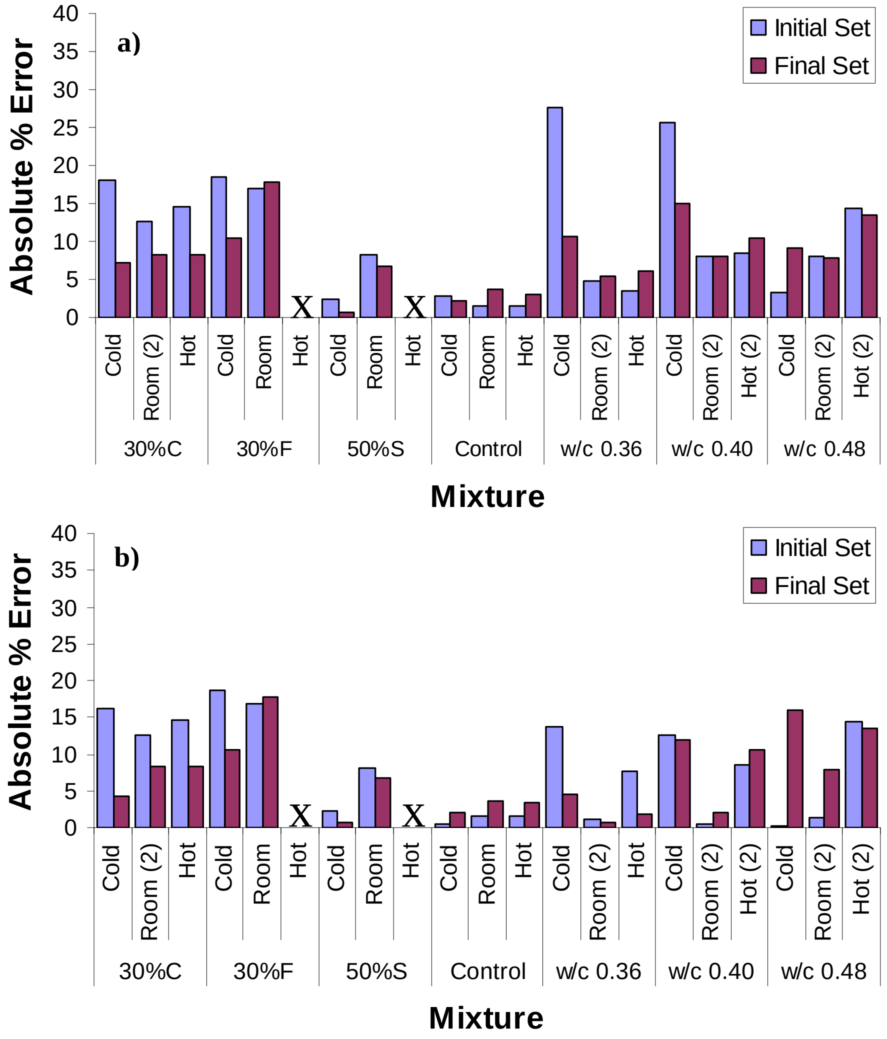

Figure 6.33: Absolute error of initial and final set as calculated by the *DOH Fractions Method* compared to ASTM C 403 (2008) for a) constant α_{cr} values, and b) w/c-dependent α_{cr} values

As can be seen in Figure 6.33, setting times obtained through the *DOH Fractions Method* compare relatively well to those obtained as per ASTM C 403 (2008). The absolute error for the w/cm-dependent case is below 20 percent for initial and final set for all batches tested and, excluding initial set for w/c 0.36 – Cold and w/c 0.40 – Cold, for the constant α_{cr} value case. In general, the w/cm-dependent case predicted values with less overall error than the constant α_{cr} values case, but results were very similar (average absolute percent error of 8.3 as compared to 10.6 for initial set and 7.1 as compared to 8.1 for final set). Based on these results and the criteria defined in Section 6.2.3.2, the w/cm-dependent *DOH Fractions Method* may be considered an adequate indicator of concrete setting for initial and final set.

Delta T Fractions Method

The general theory of both the *Direct-Fractions Method* and the *DOH Fractions Method* is that the heat released by hydration products is an indicator of the relative amount of hydration products formed. Setting is then defined in terms of a percentage of the total heat (or hydration products). Due to the fact that hydration products release a defined amount of heat as they hydrate, a similar theory can be applied and setting can be defined in terms of a pre-defined temperature rise (as opposed to a fraction of the total temperature rise, or degree of hydration). For this method, once a defined amount of hydration products have formed, concrete can be considered set, regardless of the amount of products formed at complete hydration.

For the *Delta T Fractions Method*, setting is defined as the equivalent age at which a predefined temperature rise (ΔT) is reached. Again, both constant and w/cm-dependent cases were evaluated for this method. ΔT_i and ΔT_f were calculated based on an average of all batches tested. Results are summarized in Table 6.13. ΔT_i was calculated as 1.9 °C, or 4.3 times the w/cm. ΔT_f was calculated as 4.0 °C or 9.2 times the w/cm.

Table 6.13: *Delta T Fractions Method* ΔT values calculated for Calorimeter I (CI) and Calorimeter II (CII)

Mixture	Batch	Constant ΔT				w/cm Dependent ΔT			
		ΔT_i		ΔT_f		ΔT_i/(w/cm)		ΔT_f/(w/cm)	
		CI	CII	CI	CII	CI	CII	CI	CII
30%C	**Cold**	2.84	2.41	4.77	4.34	6.42	5.46	10.81	9.82
	Room	2.53	2.51	4.86	4.73	5.74	5.69	11.00	10.70
	Hot	1.60	2.43	3.79	4.93	3.63	5.50	8.57	11.16
30%F	**Cold**	2.29	1.89	4.32	3.89	5.18	4.28	9.79	8.81
	Room	1.71	2.09	3.93	4.12	3.87	4.74	8.90	9.34
	Hot	1.46	-	3.87	-	3.31	-	8.75	-
50%S	**Cold**	2.25	1.85	4.25	3.96	5.09	4.17	9.61	8.97
	Room	1.50	1.54	3.23	3.33	3.39	3.49	7.31	7.53
	Hot	0.80	-	3.58	-	1.82	-	8.09	-
Control	**Cold**	2.66	1.92	5.01	4.23	6.03	4.35	11.34	9.59
	Room	1.87	1.74	3.77	3.50	4.23	3.94	8.53	7.93
	Hot	0.61	1.69	2.35	3.78	1.39	3.82	5.33	8.57
w/c 0.36	**Cold**	1.83	1.29	3.93	3.15	5.01	3.53	10.78	8.63
	Room	1.12	1.66	3.14	3.78	3.08	4.54	8.62	10.38
	Hot	1.15	1.67	3.00	3.78	3.15	4.58	8.23	10.37
w/c 0.40	**Cold**	2.34	1.14	4.17	2.98	5.82	2.83	10.38	7.43
	Room	1.50	1.64	3.25	3.47	3.73	4.09	8.07	8.64
	Hot	1.22	2.03	3.27	4.83	3.03	5.06	8.13	12.01
w/c 0.48	**Cold**	2.55	2.05	3.70	3.13	5.32	4.28	7.73	6.53
	Room	2.04	2.08	4.70	4.85	4.26	4.34	9.81	10.14
	Hot	2.33	2.37	5.03	5.57	4.87	4.94	10.52	11.65
Average		1.85		3.96		4.30		9.21	

Initial and final setting times were then measured using these values. Setting, in terms of equivalent age, for both the *Fractions Method* and ASTM C 403 (2008) for data from Calorimeter I and Calorimeter II can be seen in Figure 6.34. As can be seen in Figure 6.34, results are very similar to those of the other *Fractions Method*s. Again Calorimeter II results are more consistent than Calorimeter I and the w/cm-dependent formulation yields slightly more accurate results. All data points from Calorimeter II lie between the two 20% error lines. For a more detailed summary of the error see Figure 6.35. Again, results from 30% F – Hot and 50% Slag – Hot are not available for

Calorimeter II, therefore are indicated with an X on the horizontal axis to distinguish them from 0% absolute error.

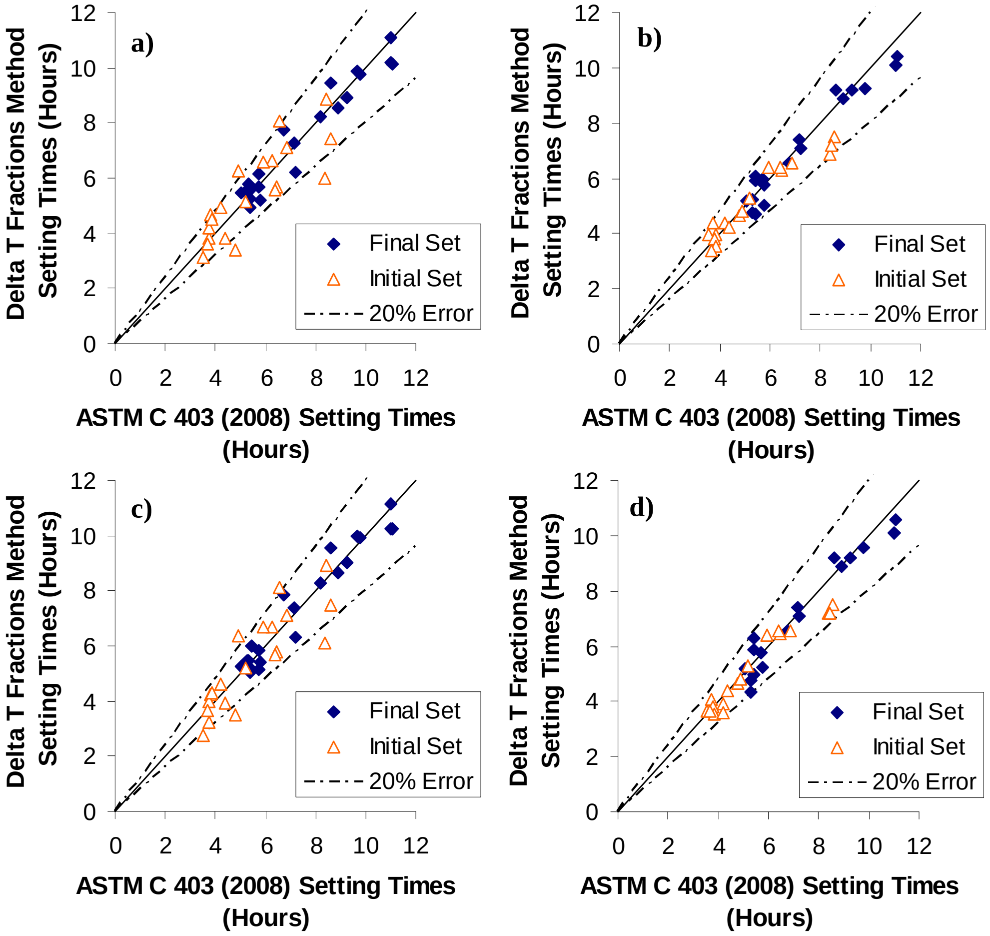

Figure 6.34: Plot of initial and final set as calculated by penetration resistance and the *Delta T Fractions Method* for a) constant *ΔT* values for Calorimeter I, b) constant *ΔT* values for Calorimeter II, c) w/cm-dependent *ΔT* values for Calorimeter I, and d) w/cm-dependent *ΔT* values for Calorimeter II.

As can be seen in Figure 6.34, results are very similar to those of the other *Fractions Method*s. Again Calorimeter II results are more consistent than Calorimeter I and the w/cm-dependent formulation yields slightly more accurate results. All data points from Calorimeter II lie between the two 20% error lines. For a more detailed summary of the error see Figure 6.35. Again, results from 30% F – Hot and 50% Slag – Hot are not available for Calorimeter II, therefore will be notated with an X on the horizontal axis to distinguish them from 0% absolute error.

Results from the *Delta T Fractions Method* compare with those from ASTM C 403 (2008) slightly better than the other *Fractions Method*. The average absolute percent error for w/cm-dependent case from Calorimeter II was calculated as 6.36 as compared to 7.83 for the *Direct-Fractions Method* and 7.69 for the *DOH Fractions Method*. Again it should be noted that the w/cm-dependent case yielded slightly more consistent results than the constant case, but much less so for this method than other methods. The absolute error falls below 20 percent for initial and final set for all batches tested and is below 15 percent for most batches for the w/cm-dependent case. Therefore, based on the criteria defined in Section 6.2.3.2, both the constant and the w/cm-dependent cases of the *Delta T Fractions Method* may be considered an adequate indicator of concrete setting for initial and final set.

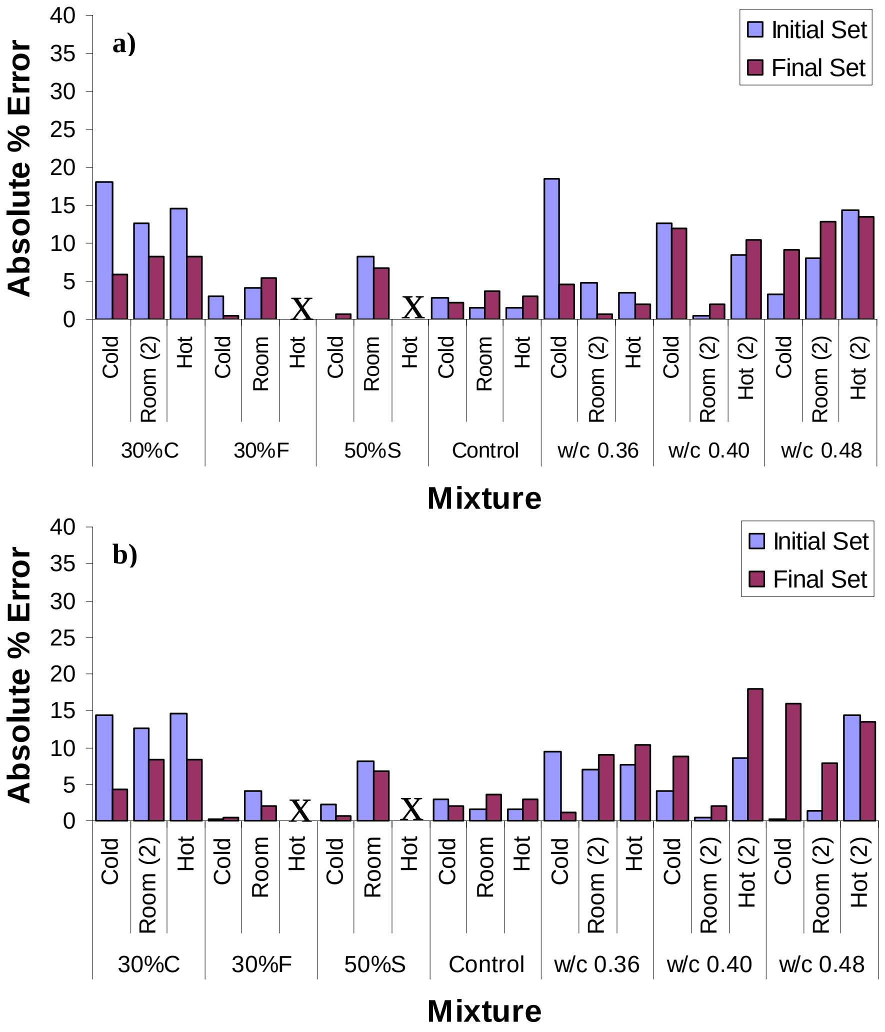

Figure 6.35: Absolute error of initial and final set as calculated by the *Delta T Fractions Method* compared to ASTM C 403 (2008) for a) constant *ΔT* values, and b) w/c-dependent *ΔT* values

Pre-Calibrated Fractions Method

In an effort to increase the accuracy of the *Fractions Method*, an attempt was made to pre-calibrate the method to mixture-specific properties. In practice, a test batch would be mixed and tested in the laboratory before actually mixing concrete and setting could be defined and calculated based on these results. This process was evaluated by using the room temperature batch of each mixture as a basis for computing setting for the hot and cold batches. Only the *Delta T Fractions Method* was used and all analysis was performed on data from Calorimeter II only.

Results are shown in Figure 6.36. Data from 30% F – Hot and 50 % Slag – Hot was not evaluated and each batch is indicated with an X on the horizontal axis to distinguish them from a batch with 0 % absolute error. As can be seen in Figure 6.36, initial and final set times compare to ASTM C 403 (2008) results with slightly more error than those computed using the original *Delta T Fractions Method*. Average absolute percent error is slightly less (5.6 percent for the pre-calibrated analysis as compared to 6.3 for the w/cm-dependent case originally), but the maximum absolute error is significantly greater (21.6 percent for the pre-calibrated analysis as compared to 18 percent for the w/cm-dependent case originally). The fact that there is very little difference in these values leads one to believe that error between *Fractions Method* setting measurement and penetration-based setting measurement is more dependent on mixture temperature than mixture proportions. It should be noted that, due to the fact that a large range of mixture-dependent behaviors were not evaluated in this testing program, (due to the use of set-controlling admixtures) these data may not be representative of all concrete mixtures.

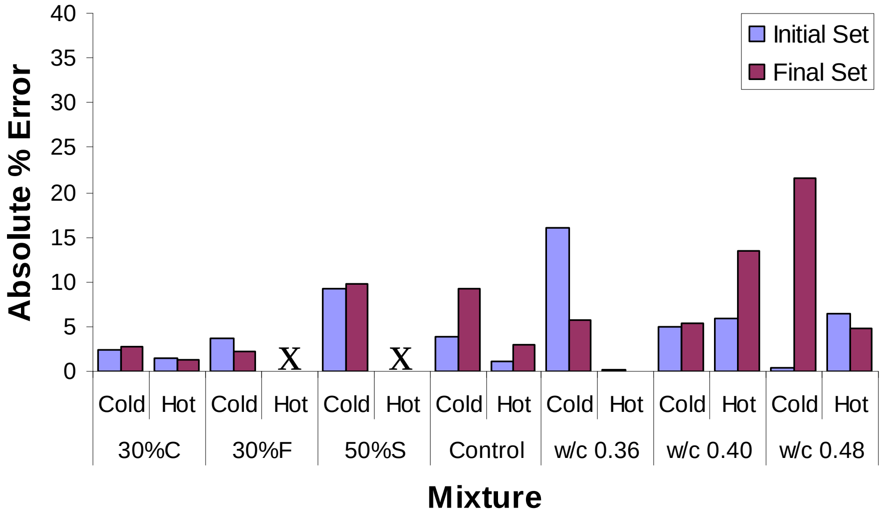

Figure 6.36: Absolute error of initial and final set as calculated by the pre-calibrated *Delta T Fractions Method* and compared to ASTM C 403 (2008)

6.2.3.8 *Fractions Method* – Discussion

In this section, the various *Fractions Method*s to determine set times are compared, the various advantages and disadvantages of each are discussed and recommendations concerning the *Fractions Method* in general are presented.

Comparison Between Fractions Methods

The absolute error for each method was averaged for initial and final set values and tabulated for both Calorimeter I and Calorimeter II and can be seen in Table 6.14. Of the three versions of the *Fractions Method* attempted, setting was measured most accurately (as compared to penetration-resistance methods) for the *Delta T Fractions Method*. Results from different calorimeters are kept separate in this discussion due to the varying influence that calorimeter type has on the different analysis methods. In general, data from Calorimeter II will be used for all comparisons, although data from both Calorimeters are presented in Table 6.14.

Table 6.14: Average absolute error of initial and final set values for *Fractions Method*s as compared to ASTM C 403 (2008)

Analysis Type	**Average Absolute Error (%)**			
	Calorimeter I		Calorimeter II	
	Constant K, α, ΔT value	w/cm-Dependent K, α, ΔT value	Constant K, α, ΔT value	w/cm-Dependent K, α, ΔT value
Direct	12.0	11.1	9.8	7.8
DOH	11.6	9.7	9.4	7.7
Delta T	9.5	9.0	6.6	6.4

The effects of the calorimeter used for testing as well as the effects of the constant and w/cm-dependent cases for each analysis method and analysis type can be seen in Table 6.14. Although results from the *Delta T Fractions Method* for Calorimeter II are slightly more accurate than the other two methods, results from the w/cm-dependent cases for Calorimeter II compare to ASTM C 403 (2008) with reasonable absolute error. The w/cm of the mixtures tested appears to have a much greater effect on results from the *DOH Fractions Method* and the *Direct-Fractions Method* than the Delta T Fractions. Since both the *Direct-Fractions Method* and the DOH Fractions make use of *Delta T Fractions Method* results (both methods require the temperature rise at setting to compute K_F and α_{cr} respectively), it can be assumed that the w/cm has a greater effect on the final temperature recorded (or calculated) than the temperature rise associated with setting. The calorimeter used for testing affects all results in a similar fashion.

Due to the fact the error or all three methods can considered reasonable, for the w/cm-dependent case, evaluation of each method should be heavily based on logistical considerations and the real-world application potential of the methods themselves. Of the three methods attempted, the *Direct-Fractions Method* is arguably the least applicable. Not only does it have the highest absolute error as compared to ASTM C 403 (2008) results, but it requires a full 7 days of thermal testing before initial and final set can be determined. Although this may be acceptable for some lab applications, it renders the method practically useless for field application. Also, due to its dependence on the final adiabatic temperature, results from this method are highly dependent on accurate determination of device losses. The one advantage that this method has over either of the other two is the fact that no knowledge of cement properties is needed for the calculation of set (which is not the case for the *DOH Fractions Method*).

As proposed, the only real disadvantage of the *DOH Fractions Method* is the fact that all calculations depend on the total heat of hydration of the cementitious materials (H_u). The calculation of H_u requires knowledge of the chemical properties of cementitious materials used as well as mixture proportions. These chemical properties are somewhat difficult to obtain and may not be available for concrete mixtures used for real-world application. Once H_u is determined, initial and final set can be calculated in real-time as the sample hydrates. Since *DOH Fractions Method* calculations are independent of temperatures recorded after setting has occurred, the *DOH Fractions Method is* not as dependent on knowledge of calorimeter losses as the *Direct Fractions Method*.

The *Delta T Fractions Method* appears to be the most applicable of the three methods considered. Not only does it have the least error when compared to ASTM C 403 (2008), but it is also the simplest and provides the most direct indicator of setting. A user would only have to monitor the device and wait for the pre-determined temperature rise that will result in initial and final set. This method is not highly dependent on device losses, and provides immediate set time results. The only disadvantage that was apparent is that the method is not highly mixture specific. If a larger range of mixture-dependent properties were examined it is possible that the pre-calibrated version of this method as discussed above would yield more consistent results than the *Delta T Fractions Method* as proposed.

Recommendations for the Fractions Method

Recommendations specifically for calculation of concrete setting by the *Fractions Method* and future work concerning the *Fractions Method* are as follows:

- w/cm-dependent K, α_{cr}, and ΔT values should be used in order to minimize error due to mixture proportions,
- means should be taken to avoid equilibrium effects due to a difference in calorimeter and specimen temperature at the onset of testing; all calorimeters should be allowed to acclimate to ambient temperatures for an appropriate amount of time before testing,
- the temperature sensor for calorimeter units should be utilized in a manner that it is in direct contact with the concrete, preferably in the center of the specimen, for the duration of testing,
- although measurements are not highly affected by the sampling rate of calorimeters, it is believed that, when possible a sampling of one point per minute should be used in order to minimize error due to interpolation, and

- the *Delta T Fractions Method* should be used over the other proposed *Fractions Method*s evaluated when determining setting of concrete due to both its accuracy and the logistical considerations discussed above.

6.3 Sensitivity of Set Times to Activation Energy Value

Although the maturity method is routinely applied in order to estimate in-place strength of concrete, an accurate approximation of the activation energy for the mixture being tested is not always known. ASTM C 1074 (2004) contains a method for the estimation of E, but states that an approximation of 40,000 to 45,000 J/mol may be used if maximum accuracy of the strength estimate is not required.

It would be convenient if a similar method could be applied to the use of the maturity method to predict setting characteristics. In order to investigate this possibility, a sensitivity analysis was performed in which E was varied by as much as 10,000 J/mol from the E value determined in Phase I of analysis for several mixtures. The w/cm-dependent *Delta T Fractions Method* and date from Calorimeter II was used for this analysis. The absolute error of *Delta T Fractions Method* setting times as compared to ASTM C 403 (2008) is plotted in Figure 6.37 for various activation energies.

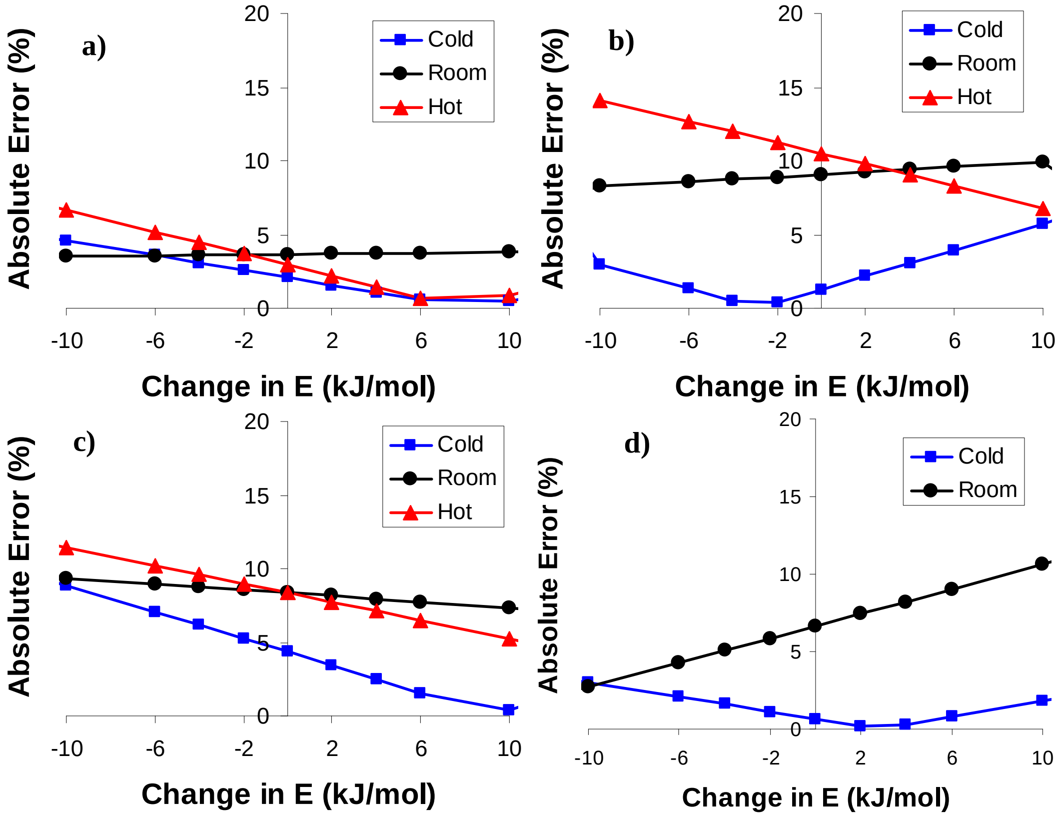

Figure 6.37: Effect of Change in E for a. Control, b. w/c 0.36, c. 30%C, and d. 50% Slag

As can be seen in Figure 6.37, a change in E of ±10,000 J/mol has little effect on the absolute error of the w/cm-dependent *Delta T Fractions Method*. All setting values still compare to ASTM C 403 (2008) within the ± 20% required by Criterion b in Section 6.2.3.2. When compared to the absolute error calculated for the *Delta T Fractions Method* using the best-fit activation energy, a change in E of up to 10,000 J/mol yielded, at most, a 5 percent change in absolute error calculated. Based on this analysis, it was determined that a range of 30,000 to 35,000 J/mol be used for maturity calculations for the determination of concrete setting if a mixture-specific value cannot be determined.

6.4 Conclusions and Recommendations

In this chapter, both Phase I and Phase II of analysis were discussed in detail. In Phase I, two methods to calculate the activation energy were presented, and it was determined that penetration resistance-based activation energy values should be used for the application of the equivalent age maturity method to concrete setting. For Phase II analysis, six separate methods for the calculation of initial and final set from semi-adiabatic temperature profiles were presented, and results were compared to setting results obtained by ASTM C 403 (2008).

Major findings concerning the general methodology of Phase II of the testing program are as follows:

- Sample size, ambient temperature, and device insulation properties have a significant effect on setting times obtained from thermal methods and can be avoided if test data are collected by semi-adiabatic calorimeters that meet the requirements defined by RILEM TC 119 TCE 1 (1997), and all data are adjusted to account for device losses.
- Differences in temperature between samples tested through thermal methods and penetration resistance-based methods may have a significant effect on values obtained. Comparison of results between methods requires the measurement of concrete/mortar temperature and the application of the equivalent age maturity method to account for the effect of temperature on all setting measurements.

- Thermal device sampling interval may have a significant effect on setting times obtained through thermal methods due to practical limits on data smoothing and significant variation between subsequent data points.
- Small fluctuations in the measurement of temperature caused by the testing apparatus may have a significant effect on setting times obtained through the *Fractions Methods* due to the relatively small change in temperature that occurs prior to concrete setting. This effect is much less critical for setting times obtained through the *Derivatives Methods*.
- Temperature data collected with a temperature sensor that was in direct contact with the concrete were determined to be more representative of the actual concrete temperatures than data for which this was not the case.

Major findings concerning the calculation of setting times as per the *Derivatives Method* are as follows:

- In general, *Derivatives Method* Results were found to be dependent on the w/cm of the mixture tested.
- Thermal device sampling interval has a significant effect on setting times obtained through the *Derivatives Method*, especially when a direct calculation of the slope of smoothed temperature data is used to obtain derivatives.
- The use of a direct calculation of the slope of the temperature profile using a large smoothing interval yielded results which compare reasonably well with penetration resistance results, yet fall slightly outside of the established criteria

for acceptability. Therefore, the *Direct-Slope Derivatives Method* should not be considered an adequate indicator of concrete setting.

- The use of a high-order polynomial fit to calculate the first derivative used for the prediction of setting yielded inconsistent results and higher than acceptable error when compared to penetration resistance results, therefore, the *Polynomial Derivatives Method* should not be considered an adequate indicator of concrete setting.
- The use of the hydration equation (Equation 2.8) to calculate the first derivative used for the prediction of setting yielded the most reliable results of the three variations of the *Derivatives Method* evaluated. However, final set values were not able to be calculated within the error range established as acceptable when compared to penetration resistance results. Therefore, the *DOH Derivatives Method* should not be considered an adequate indicator of concrete setting.
- The *DOH Derivatives Method* is considered the most applicable of the various *Derivatives Method*s evaluated due to the fact that it can be used to provide a real-time calculation of final set

Major findings concerning the calculation of setting times as per the *Fractions Method* are as follows:

- *Fractions Method* results were also found to be dependent on the w/cm of the mixture tested, but to a lesser extent than the *Derivatives Method*.

- The location of semi-adiabatic calorimeter temperature probe was found to have a significant effect on setting times obtained through the various versions of the *Fractions Method* evaluated.
- Defining setting in terms of the ratio of the temperature rise at setting over the maximum temperature rise yielded similar results to other *Fractions Method*s. However, this method is considered impractical due to the need to wait until the maximum temperature is reached before setting can be calculated. Based on the criteria defined in Section 6.2.3.2, The *Direct Fractions Method* should not be considered an adequate indicator of concrete setting.
- Defining setting in terms of a degree of hydration that occurs at setting also yielded results comparable to other *Fractions Method*s. Based on the criteria defined in Section 6.2.3.2, the w/cm-dependent *DOH Fractions Method* may be considered an adequate indicator of concrete setting, but is considered impractical due to the dependence on cement chemistry or similar methods to calculate total heat of hydration of cementitious materials of each mixture tested.
- Defining setting in terms of a specific temperature rise that occurs prior to setting yielded the most accurate results as compared to penetration resistance methods. Based on the criteria defined in Section 6.2.3.2, both the constant and the w/cm-dependent cases of the *Delta T Fractions Method* may be considered an adequate indicator of concrete setting. This method was considered the most applicable of the *Fractions Method*s due to its inherent simplicity and the ability to calculate setting in real time.

Recommendations based on work as described in this chapter for calculation of concrete setting by thermal methods as well as future research are as follows:

- Only semi-adiabatic calorimeters should be used for the determination of setting times through thermal methods, and all semi-adiabatic thermal data should be adjusted for device specific losses.
- The equivalent age maturity method should be applied to all setting measurements to account for the effect of temperature on all concrete setting times. An activation energy value range of 30,000 to 35,000 J/mol should be used for maturity calculations for the determination of concrete setting if a mixture-specific value is not available.
- Calorimeter sampling rate should be limited to no less than one point per minute for the calculation of concrete setting through semi-adiabatic calorimetry.
- Means should be taken to avoid equilibrium effects due to a difference in calorimeter and specimen temperature at the onset of testing; all calorimeters should be allowed to reach equilibrium with surrounding temperatures for at least two hours before the start of testing.
- The temperature sensor for calorimeter units should be in direct contact with the concrete, preferably in the center of the specimen, for the duration of testing.
- It is recommended to use the w/cm-dependent *Delta T Fractions Method* to determine setting of concrete through semi-adiabatic calorimetry.

Chapter 7

Conclusions and Recommendations

7.1 Summary of Work

The use of thermal testing to quantify the setting process of concrete has recently been investigated as a possible alternative to the penetration resistance-based method currently used. Several test methods, which utilize a variety of thermal testing devices and analysis methodologies, have been proposed. In 2007 a draft specification for the determination of setting through the analysis of thermal data from insulated concrete specimens was proposed to the American Society for Testing and Materials (ASTM) entitled the *Standard Test Method for Determining Setting Time of Concrete by the Temperature Method*. The purpose of this project is to evaluate this draft ASTM specification and other test methods as compared to the currently accepted penetration resistance test method (ASTM C 403), and, if possible, to prepare a test protocol for a thermal method that accurately and reliably estimates the setting times of concrete.

In order to evaluate the proposed thermal methods for determining concrete set times, two separate studies were undertaken. First, the precision of ASTM C 403 (2008) was investigated and the previously mentioned draft specification was evaluated in a round robin study undertaken by ASTM Committee C09.21.01. Separately, other test methods and analysis techniques were evaluated in a comprehensive laboratory testing program.

7.1.1 ASTM Round Robin Study

In the ASTM round robin study, three concrete mixtures were examined by 12 independent laboratories. For each mixture, all participants conducted both thermal testing as per the draft ASTM specification, and penetration resistance testing as per ASTM C 403 (2008). Several thermal devices were used, ranging from minimally insulated devices to devices that may be considered semi-adiabatic calorimeters. All testing was completed under laboratory ambient temperature conditions.

Based on these results, a precision statement for ASTM C 403 (2008) was developed for setting times ranging from 307 - 574 minutes for concrete samples taken from a single batch of concrete. Furthermore, the proposed ASTM standard and two additional analysis methods were evaluated by comparing setting times obtained through thermal methods to those obtained by ASTM C 403 (2008). In order to investigate the sensitivity of the proposed ASTM specification to various testing parameters, a sensitivity study was conducted to examine the effects of sample size, sample type, thermal device properties, and analysis type.

7.1.2 Laboratory Testing Program

For the laboratory testing program, seven independent mixtures with varying mixture proportions and cementitious materials were produced and tested at three temperatures. For each concrete batch produced, thermal testing was conducted with the use of semi-adiabatic calorimeters, and penetration resistance testing was conducted as per ASTM C 403 (2008). Temperatures were recorded for all specimens and all measurements of concrete age were converted to maturity with the use of the equivalent age maturity method. Semi-adiabatic calorimeter temperature data were corrected for device-specific losses, and all analysis was performed on this "nearly" adiabatic temperature profile.

In order to fully evaluate the possibility of the use of semi-adiabatic testing to quantify concrete setting, six individual analysis types were examined. As can be seen in Figure 7.1, these analysis methods can be broken down into either the *Fractions Method* or the *Derivatives Method*.

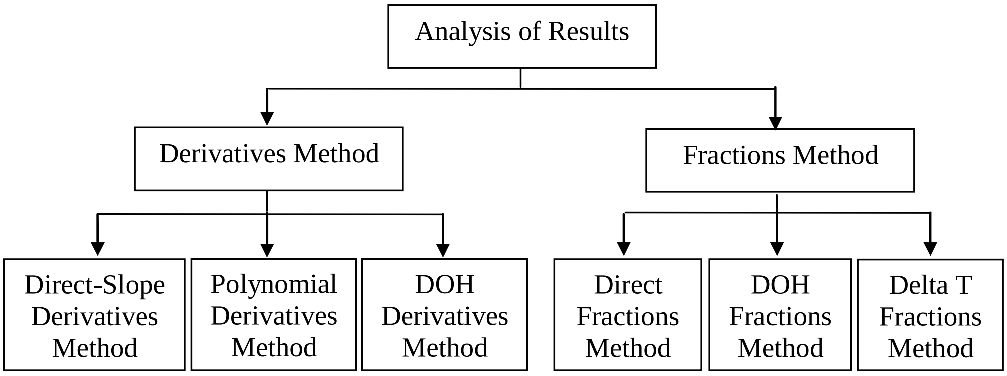

Figure 7.1: Analysis methods evaluated in the laboratory testing program for determining setting of concrete through thermal methods

For the *Derivatives Method*, initial and final set were defined as the times at which the maximum first and second derivative of concrete temperature were reached, and three methods were evaluated for the calculation of the derivatives. In the *Direct-Slope Derivatives Method*, the derivatives were calculated by directly determining the slope of the temperature profile using a large smoothing interval. For the *Polynomial Derivatives Method* and the *DOH Derivatives Method*, derivatives were obtained through the calculation of closed-form derivatives to a sixth-order polynomial and exponential hydration curve fit to the temperature data, respectively. In order to account for the effects of w/cm on setting, a w/cm independent and a w/cm dependent version of each method was evaluated.

To evaluate the *Fractions Method*, three individual definitions of concrete setting were used. For the *Direct Fractions Method*, setting was defined as the time at which a pre-defined ratio of the temperature rise over the maximum temperature rise measured occurs. Similarly, for the *DOH Fractions Method*, setting was defined as the time at which a specific degree of hydration is reached, and, for the *Delta T Fractions Method*, setting was defined as the time at which a pre-defined temperature rise occurs. Again, in order to account for the effects of w/cm on setting, a w/cm independent and a w/cm dependent version of each method was evaluated.

Each method was calibrated using data from ASTM C 403 (2008) testing, and then evaluated by comparing setting times obtained through thermal methods to those obtained as per ASTM C 403 (2008).

7.2 Conclusions

7.2.1 ASTM Round Robin Study Conclusions

Conclusions concerning the testing and analysis of results from the ASTM round robin study are as follows:

- Sample size, sample type, insulatory properties of the thermal testing device and data analysis method were found to have a very significant effect on thermal data and setting times obtained from thermal testing.
- As written, the thermal methods for determining time of set of concrete as proposed in the draft ASTM specification should not be considered an indicator of the setting time of a concrete mixture based on the criteria defined in Section 3.6.2.3.
- Of the three analytical methods evaluated, setting times obtained through methods that defined setting as the time at which a pre-defined a ratio of the temperature rise over the maximum temperature rise measured occurs compared most accurately with those obtained as per ASTM C 403 (2008).
- Of the thermal devices used in this study, results obtained by using a moderately-insulated single chamber device (Category II device) with multiple open-top specimen chambers yielded the most accurate results when compared to results obtained as per ASTM C 403 (2008) for testing under laboratory temperature conditions.
- Even when analysis was limited to the use of the Direct Fractions Method for Category II thermal devices, setting times obtained through thermal methods did not provide accurate estimates of setting times obtained from ASTM C 403 (2008).

7.2.2 Laboratory Testing Program Conclusions

Conclusions concerning the testing and analysis of results from the laboratory testing program are as follows:

- Sample size, ambient temperature, and device insulation properties have a significant effect on setting times obtained from thermal methods and effects due to these properties can be avoided if test data are collected by semi-adiabatic calorimeters that meet the requirements defined by RILEM TC 119 TCE 1 (1997), and all data are adjusted to account for device losses.
- Thermal device sampling interval may have a significant effect on setting times obtained through thermal methods. Practical limits on data smoothing and significant variation between subsequent data points were noted for the larger sampling interval used (15-minutes). These problems were not noted for the smaller sampling interval (1-minute).
- Small fluctuations in the measurement of temperature caused by the testing apparatus (e.g. effects due to a sample and device reaching equilibrium) may have a significant effect on setting times obtained through thermal methods due to the relatively small change in temperature that occurs prior to concrete setting.
- Temperature data collected with a temperature sensor that was in direct contact with the concrete was more representative of the actual concrete temperatures than data for which this was not the case.
- Differences in temperature between samples tested through thermal methods and penetration resistance-based methods may have a significant effect on

values obtained. Comparison of results between methods requires the measurement of concrete/mortar temperature and the application of the equivalent age maturity method to account for the effect of temperature on all setting measurements.

- Based on the criteria defined in Section 6.2.3.2, the *Direct-Slope Derivatives Method,* the *Polynomial Derivatives Method*, and the *DOH Derivatives Method* do not provide accurate estimates of setting and should not be used to estimate setting times of concrete.
- Based on the defined criteria, the *Direct Fractions Method* does not provide accurate estimates of setting and should not be used to estimate setting times of concrete.
- Based on the defined criteria, the w/cm dependent *DOH Fractions Method* may be considered an adequate indicator of concrete setting, but is considered impractical due to the dependence on cement chemistry or similar methods to calculate the total heat of hydration of cementitious materials of each mixture tested.
- Based on the defined criteria, both the w/cm independent and w/cm dependent *Delta T Fractions Method* may be considered adequate indicators of concrete setting. This method is considered the most applicable of the *Fractions Methods* due to its inherent simplicity and the ability to calculate setting in real time.

- An activation energy value range of 30,000 to 35,000 J/mol may be used for maturity calculations for the determination of concrete setting if a mixture-specific value is not available.

7.3 Recommendations

Based on the work described in this thesis the following recommendations can be offered:

- Only semi-adiabatic calorimeters should be used for the determination of setting times through thermal methods, and all semi-adiabatic thermal data should be adjusted for device specific losses.
- The equivalent age maturity method should be applied to all setting measurements to account for the effect of temperature on all concrete setting times. An activation energy value range of 30,000 to 35,000 J/mol should be used for maturity calculations for the determination of concrete setting if a mixture-specific value is not available.
- Calorimeter sampling rate should be limited to no less than one point per minute for the calculation of concrete setting through semi-adiabatic calorimetry.
- Means should be taken to avoid equilibrium effects due to a difference in calorimeter and specimen temperature at the onset of testing; all calorimeters should be allowed to reach equilibrium with surrounding temperatures for at least two hours before the start of testing.

- The temperature sensor for calorimeter units should be utilized in a manner that it is in direct contact with the concrete, preferably in the center of the specimen, for the duration of testing.
- It is recommended to use the w/cm dependent *Delta T Fractions Method* to determine setting of concrete through semi-adiabatic calorimetry.

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Appendix A

Draft ASTM Specification

Standard Guide for Analysis of Thermal Setting Time Data[1]

This standard is issued under the fixed designation X XXXX; 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 method is used for evaluating thermal data gathered from paste, mortar, or concrete measurements for setting time.

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:

C 219 Terminology Relating to Hydraulic Cement

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[1] This Practice/Guide is under the jurisdiction of ASTM Committee *C09 on Concrete and Concrete Aggregates* and is the direct responsibility of Subcommittee *C09.23.01 on Setting Time*.

Current edition approved XXX. XX, XXXX. Published XX XXXX.

3. Terminology

3.1 *Definitions*-see Terminology C 219

3.1.1 *peak temperature time, n*–time at which the peak temperature occurs during the main hydration peak.

3.1.2 *primary setting time, n*–time at the maximum curvature of the time-temperature curve.

3.1.3 *secondary setting time, n*–time at inflection point of the time-temperature curve.

3.1.4 *time-temperature curve, n*–graph of the time versus temperature data on linear scales.

4. Summary of Practice

4.1 The evaluation of data is based on a forth order polynomial fit of the time-temperature curve. The primary setting is calculated as the third derivative of the fit of the time-temperature curve. The secondary setting time is when the curvature is zero of the fit of the time-temperature curve.

5. Significance and Use

5.1 This test method provides a procedure for evaluating the setting time of concrete from thermal analysis data. For most time-temperature curves produced with semi-adiabatic calorimetry methods the shape has a point of the maximum curvature and an inflection point.

6. Procedure

6.1 Plot the time data on the horizontal axis and the temperature on the vertical axis (Note 1). Commercially available data graphing programs can be used to determine a best fit of a forth order polynomial (Note 2). A forth-order polynomial best fit often requires trimming of data after the maximum temperature has been reached to have a good fit. The trimming of the initial readings as the temperature measuring device and specimen temperature reach equilibrium may be required to achieve a good fit. The forth-order polynomial is represented by the following equation:

$$T = at^4 + bt^3 + ct^2 + dt + e \qquad (1)$$

where:

a, b, c, d, and e = coefficients and constant to be determined from best fit of graphing program

T = temperature

t = time

NOTE 1: Often ambient temperature data is collected at the same time as the specimen temperature data. Swings in ambient temperature can skew the specimen time-temperature curve. Subtracting the ambient temperature data from the specimen temperature data at the same time can reduce the skew. When this is done the specimen temperature data minus the ambient temperature is graphed versus the time. The forth order polynomial fit is determined from this graph.

NOTE 2: A Microsoft excel spreadsheet can be accessed through the ASTM website (www.astm.org) that determines the primary and secondary setting times from time temperature data input.

6.2 Determine the primary setting time from the coefficients of the forth order polynomial fit and the following equation:

$$P = -\frac{b}{4a} \tag{2}$$

6.3 Determine the secondary setting time from the coefficients of the forth order polynomial fit from the following equations. The equation for second derivative is:

$$\frac{d^2T}{dt^2} = 12at^2 + 6bt + 2c \tag{3}$$

The secondary setting time is the time (t) when the second derivative is equal to zero:

$$12at^2 + 6bt + 2c = 0 \tag{4}$$

This can be found by solving the quadratic equations:

$$S_1 = \frac{-6b + \sqrt{(6b)^2 - 4(12a)(2c)}}{2(12a)} \tag{5}$$

$$S_2 = \frac{-6b - \sqrt{(6b)^2 - 4(12a)(2c)}}{2(12a)} \tag{6}$$

where S_1 and S_2 are possible solutions to the quadratic equation. The secondary setting time is either S_1 and S_2, whichever is greater then the primary setting time and less then the time at the peak temperature.

7. Report

7.1 Graph of the time-temperature curve of the specimen with the forth order polynomial fit.

7.2 Graph of the time-temperature curve of the ambient temperature.

7.3 Primary setting time and secondary setting time.

8. Keywords

A1.1.1 calorimetry; primary setting time; secondary setting time; thermal setting time;

APPENDIX

(Nonmandatory Information)

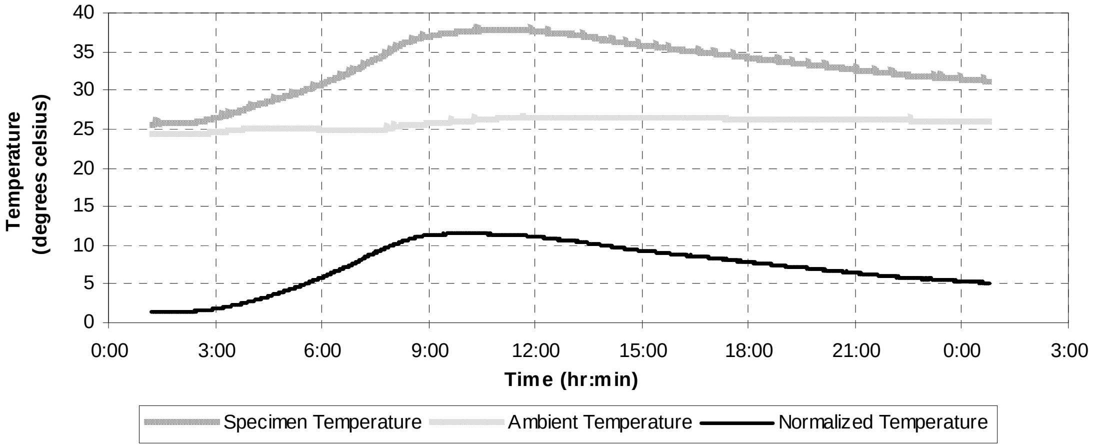
X1. EXAMPLE OF ANALYSIS OF THERMAL SETTING TIME DATA

X1.1 *Introduction*-The following example illustrates the procedure for determining the primary and secondary setting times from thermal setting time data.

X1.2 *Data Analysis*

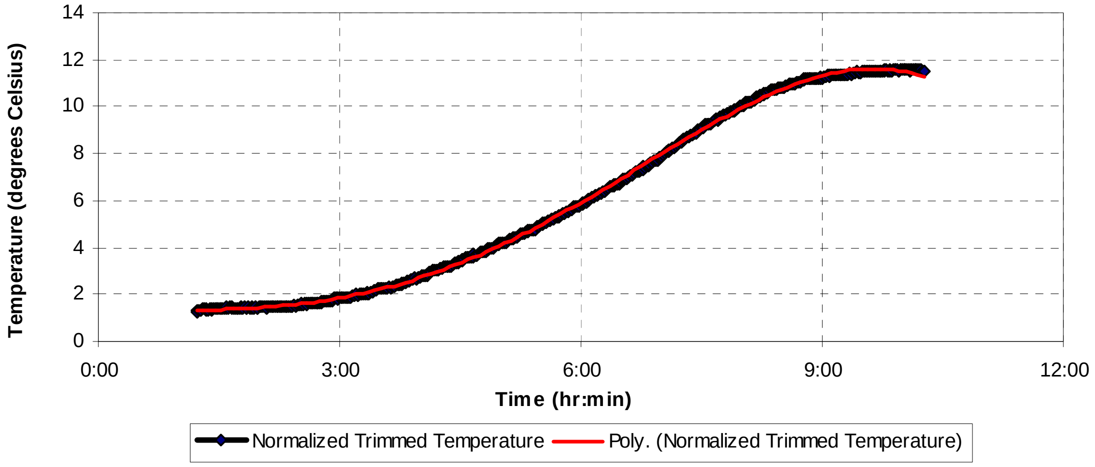
X1.2.1 The specimen time-temperature data and ambient time-temperature data is shown in Fig. X.1.

Figure X.1 Time-temperature data of specimen, ambient, and normalized specimen



X1.2.2 The ambient time-temperature data is subtracted from the specimen time-temperature data which is the normalized time-temperature data also shown in Fig. X.1. The initial time-temperature data where the measured temperature of the apparatus is reaching equilibrium with specimen temperature is removed from the data set. The data after the peak temperature is also removed to give a better forth order polynomial fit. The new data set is seen in Fig. X.2 with the forth order polynomial fit.

Figure X.2 Trimmed time-temperature of normalized specimen data with forth order polynomial fit



The forth order polynomial fit from Fig X.2 is displayed in Equ. X.1

$$T = -1840.2t^4 + 1210.2t^3 - 146.32t^2 + 8.9848t + 1.0950 \qquad \text{(X.1)}$$

Using Equ.1 in section:

a = -1840.2

b = 1210.2

c = -146.32

X1.2.3 The primary setting time in days is determined according to Equ. 2 and converted to hours by multiplying by 24 as seen in Equ. X.2.

$$P = \frac{-1210.2}{4(-1840.2)} \cdot 24 = 3.95 \qquad \text{(X.2)}$$

X1.2.4 The possible secondary setting times are determined in days according to Equ. 5 and Equ. 6 and converted to hours as seen in Equ. X.3 and Equ. X.4.

$$S_1 = \frac{-(6)1210.2 + \sqrt{(6 \cdot 1210.2)^2 - 4(12)(-1840.2)(2)(-146.32)}}{2(12)(-1840.2)} \cdot 24 = 1.13 \quad \text{(X.3)}$$

$$S_1 = \frac{-(6)1210.2 - \sqrt{(6 \cdot 1210.2)^2 - 4(12)(-1840.2)(2)(-146.32)}}{2(12)(-1840.2)} \cdot 24 = 6.76 \quad \text{(X.4)}$$

The secondary setting time is 6.76 hours since it is greater then the primary setting time and less then time at the peak temperature.

X1.3 *Example report*-The report of the pertinent data is shown in Fig. X.3.

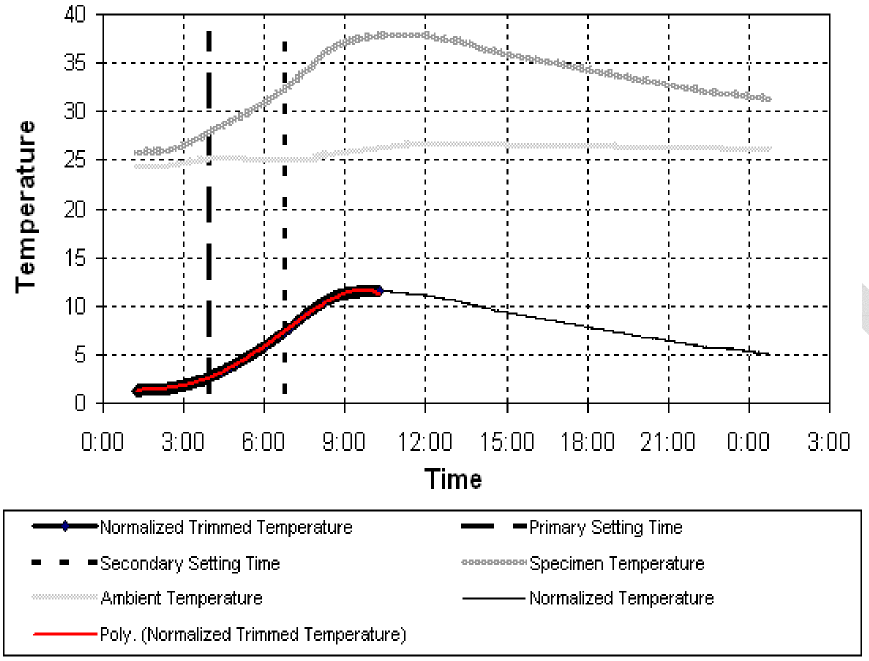
Figure X.3 Report of the example of analysis of thermal setting time data

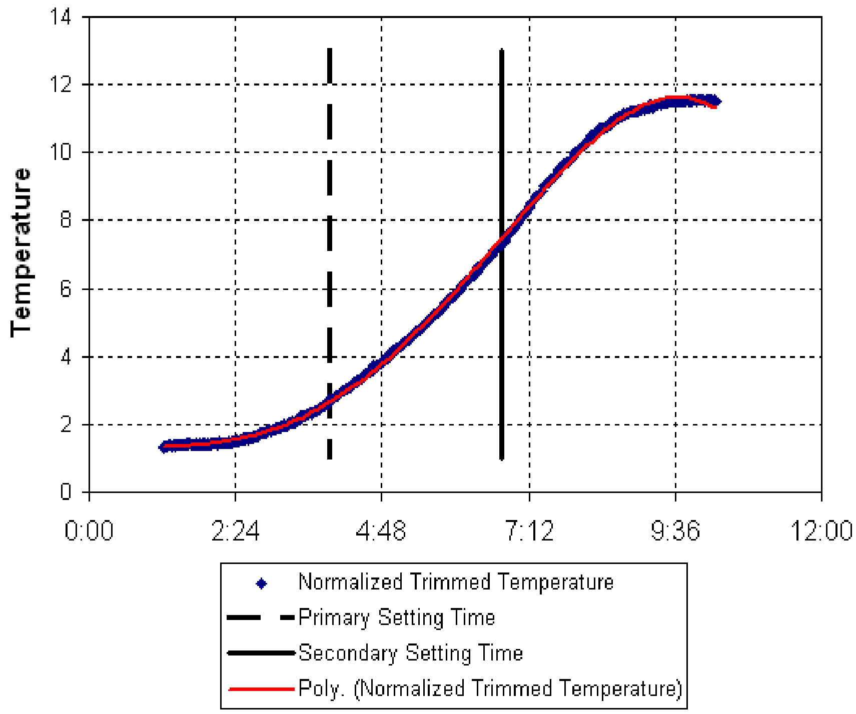
Description Appendix Report Example X.1

Primary Setting Time (hr:min) **3:56**

Secondary Setting Time (hr:min) **6:45**

Date of analysis: 9/30/2008





Appendix B

ASTM Round Robin Supplementary Information

In this section, pictures of the various thermal devices (Category I, II, and III) used in the ASTM round robin study will be presented.

Figure B.1: Category I Device (Group 3)

Figure B.2: Category I Device (Group 9)

Figure B.3 Example of the Category II – 4x8 Device used by

Groups 3, 4, 5, 6, 7, 9, and 10

Figure B.4: Example of the Category II – 3x6 Device used by

Groups 3, 5, 6, 7, 9, and 10

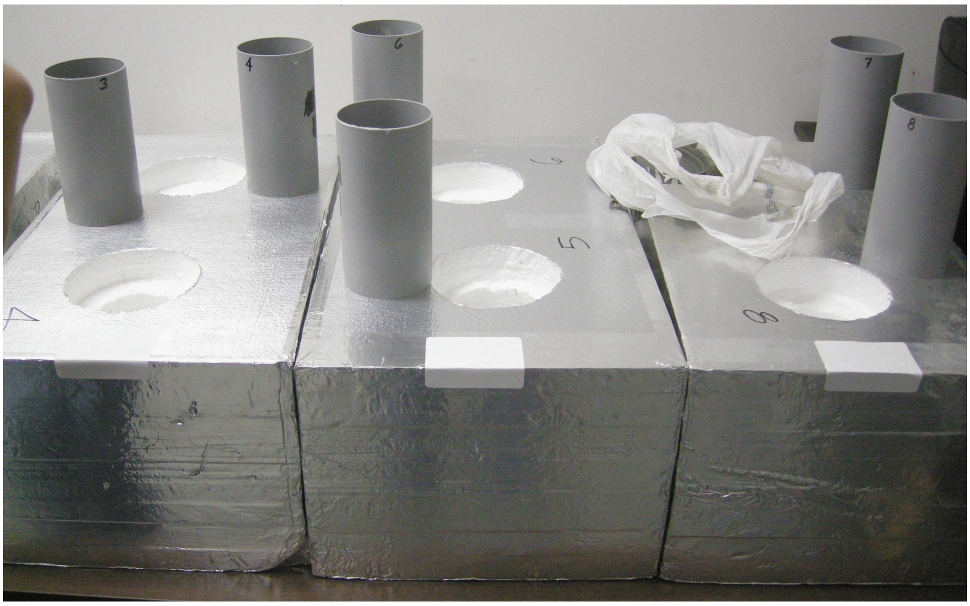


Figure B.5: Category II Device (Group 8)

Figure B.6: Category II Device (Group 8)

Figure B.7: Category III Device (Group 4)

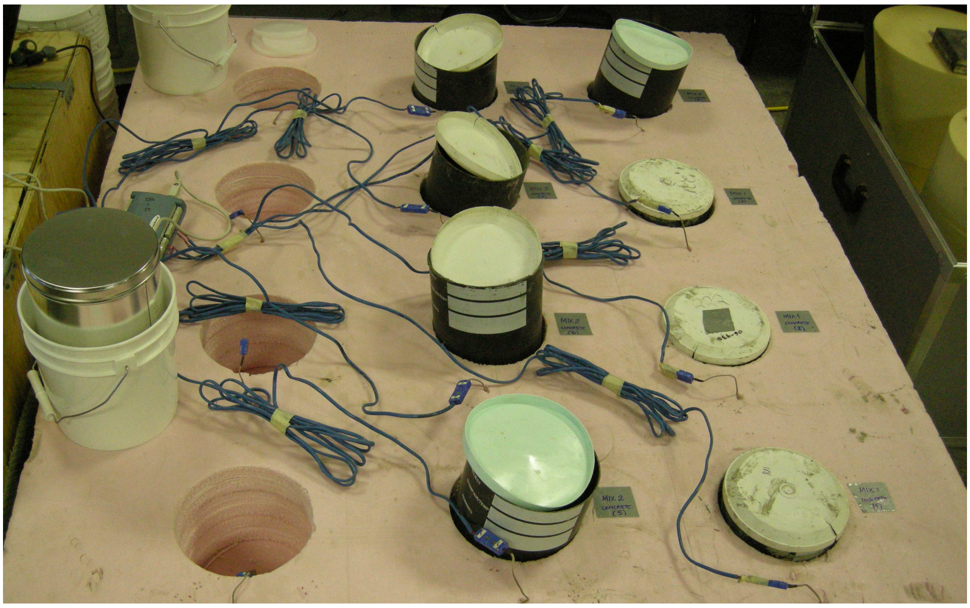


Figure B.8: Category III Device (Group 5)

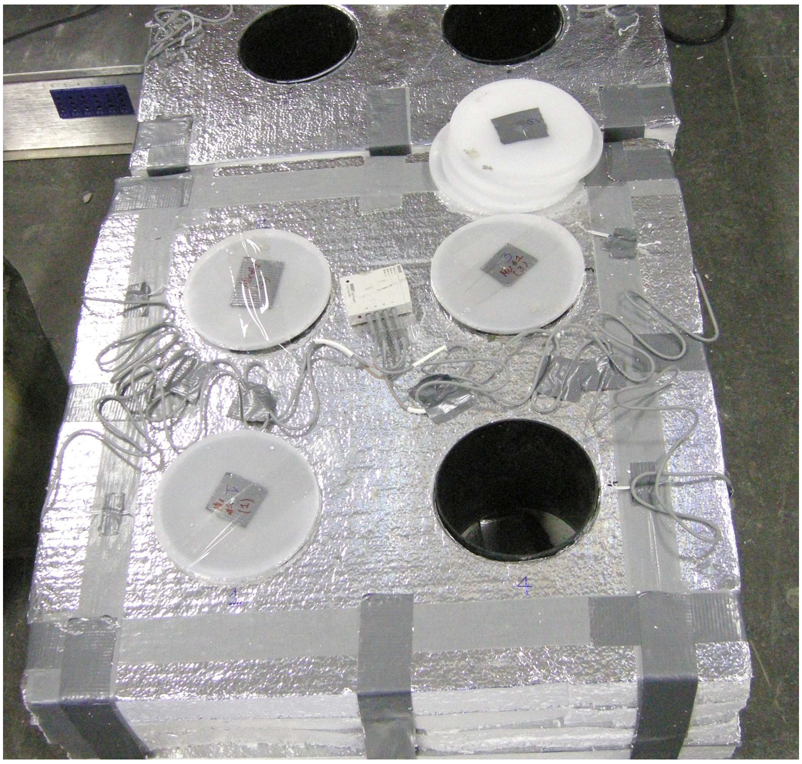


Figure B.9: Category III Device (Group 6)

Figure B.10: Category III Device (Group 8)

Figure B.11: Category III Device (Group9)

Figure B.12: Category III Device (Group 10)

Figure B.13: Category III Device (Group 12)

Appendix C

Compressive Strength Test Results

Appendix C contains compressive strength testing results for each batch of concrete tested. All compressive strength testing was completed as per ASTM C 39 (2004).

Table C.1: Compressive strength test results for Control

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	1	7.76	8.20	8.45
	2	25.36	23.55	25.49
	28	48.49	49.14	48.99
Room	1	21.44	20.25	19.94
	2	28.99	27.91	28.82
	28	49.84	50.33	50.24
Hot	1	24.60	26.04	26.07
	2	30.60	30.62	31.25
	28	48.50	49.95	48.24

Table C.2: Compressive strength test results for 30% C

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	2	11.48	11.47	10.83
	4	21.78	21.43	22.85
	28	38.37	38.88	39.26
Room (2)	1	14.19	14.03	15.60
	2	20.16	21.26	21.49
	28	51.88	52.42	50.07
Hot	1	19.08	19.06	18.78
	2	22.72	24.89	23.50
	28	50.42	49.51	47.95

Table C.3: Compressive strength test results for 30% F

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	2	11.36	11.58	12.51
	4	17.20	19.31	17.69
	28	32.59	31.82	31.96
Room	1	8.34	8.59	8.35
	2	14.13	14.75	14.82
	28	44.06	44.02	43.38
Hot	1	17.01	15.58	16.61
	2	21.02	20.94	20.23
	35	59.57	56.84	56.28

Table C.4: Compressive strength test results for 50% Slag

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	1	10.25	11.24	11.14
	2	18.54	17.97	18.68
	28	35.59	35.15	35.31
Room	1	11.93	10.78	11.27
	2	22.15	21.88	22.10
	28	55.09	53.50	53.80
Hot	1	18.48	19.92	19.68
	2	28.39	28.74	-
	28	51.36	53.68	54.56

Table C.5: Compressive strength test results for w/c 0.36

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	1	23.54	23.61	24.11
	2	39.87	42.25	39.47
	28	63.67	62.67	62.42
Room	1	27.72	29.56	26.77
	2	34.20	33.91	34.72
	28	59.13	56.41	58.77
Hot	1	34.43	35.88	32.01
	2	37.78	38.51	37.67
	28	47.57	53.30	49.25

Table C.6: Compressive strength test results for w/c 0.40

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	1	20.60	19.82	20.79
	2	29.81	27.53	29.10
	28	53.73	56.90	57.85
Room	1	23.32	25.40	24.60
	2	31.21	30.02	28.96
	28	52.45	50.44	49.46
Hot	1	28.68	26.46	29.42
	2	34.10	34.96	33.78
	28	49.83	54.53	51.86

Table C.7: Compressive strength test results for w/c 0.48

Batch Identification	Concrete Age (Days)	Compressive Strength (MPa)		
Cold	1	13.11	13.84	13.95
	2	21.31	20.66	20.50
	28	55.57	54.46	54.94
Room	1	19.02	22.01	20.47
	2	27.29	27.63	28.08
	28	47.71	45.80	45.63
Hot	1	25.72	23.15	23.76
	2	27.53	-	26.82
	28	42.08	44.89	45.09

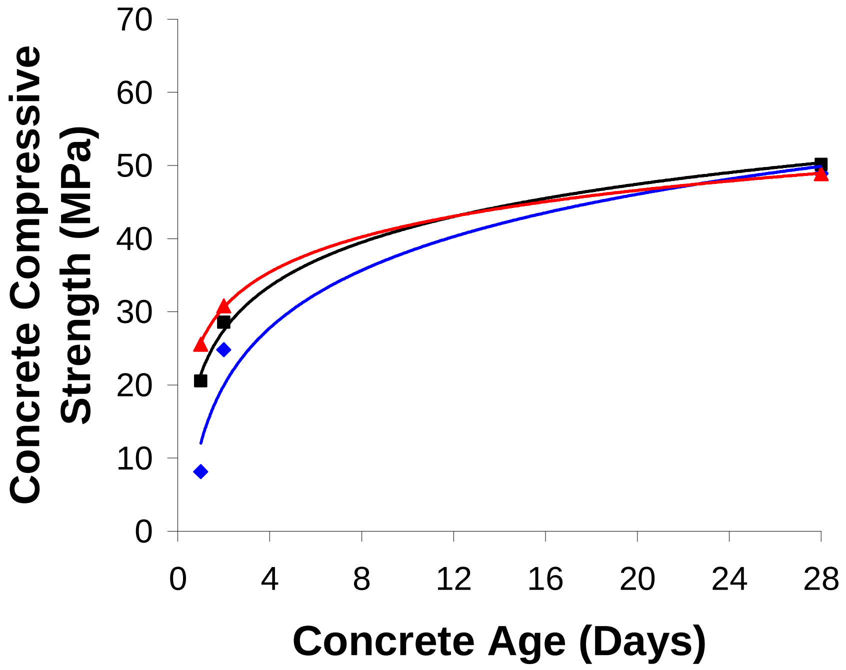


Figure C.1: Compressive strength test results for Control

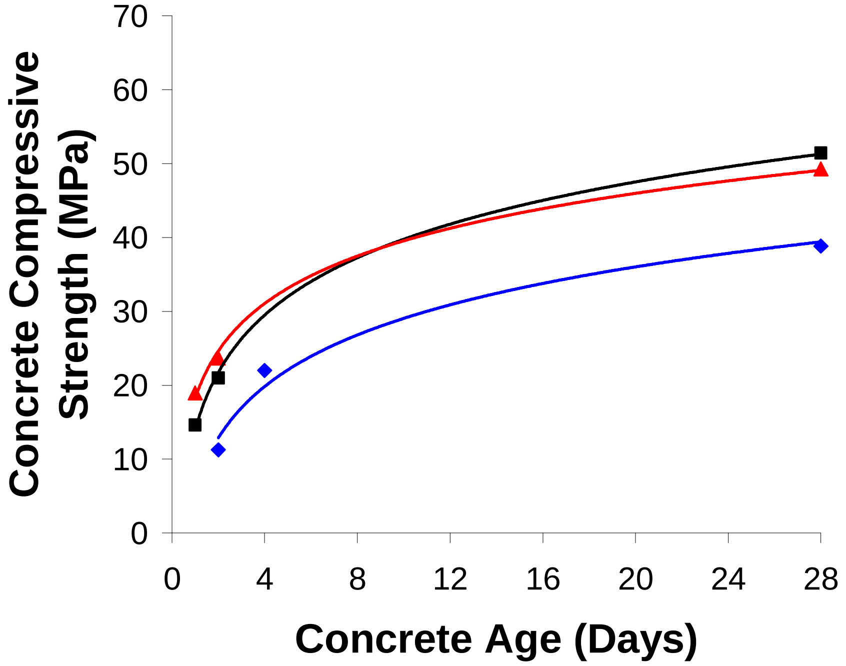


Figure C.2: Compressive strength test results for 30%C

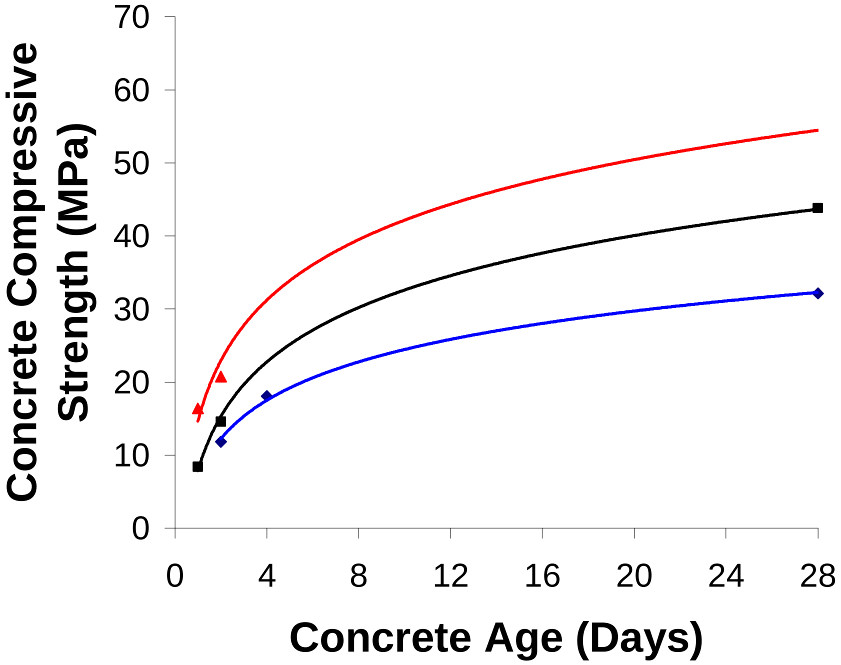


Figure C.3: Compressive strength test results for 30 % F

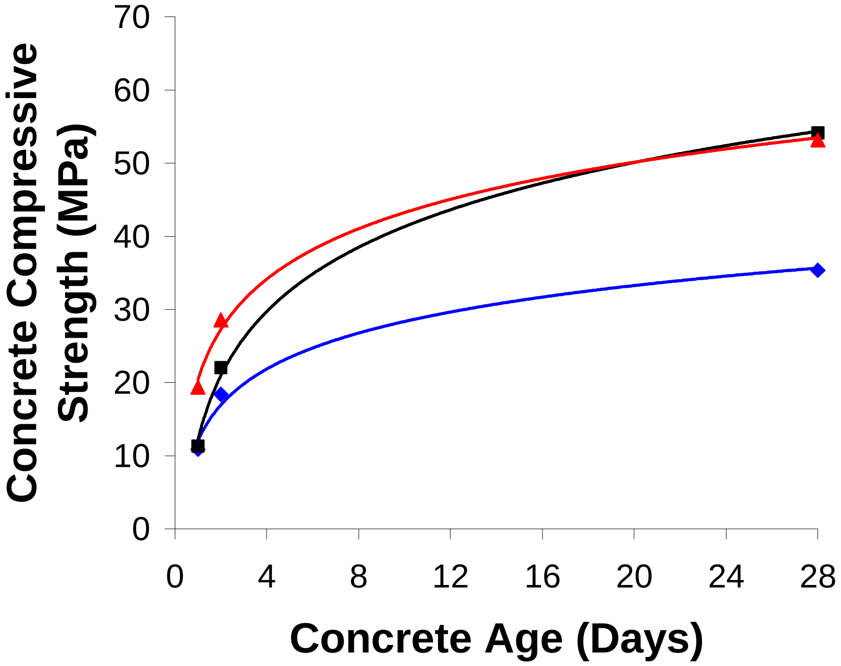


Figure C.4: Compressive strength test results for 50% Slag

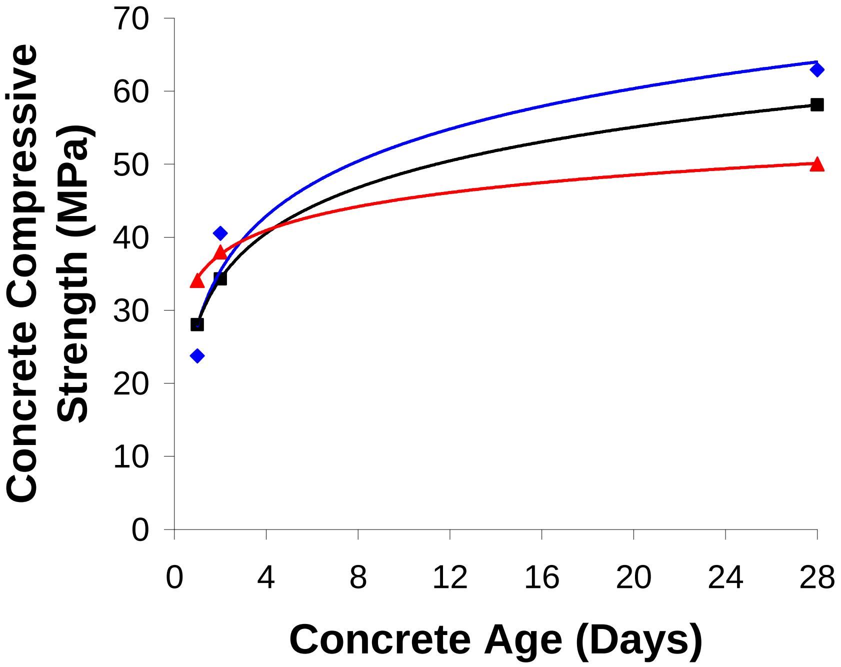


Figure C.5: Compressive strength test results for w/c 0.36

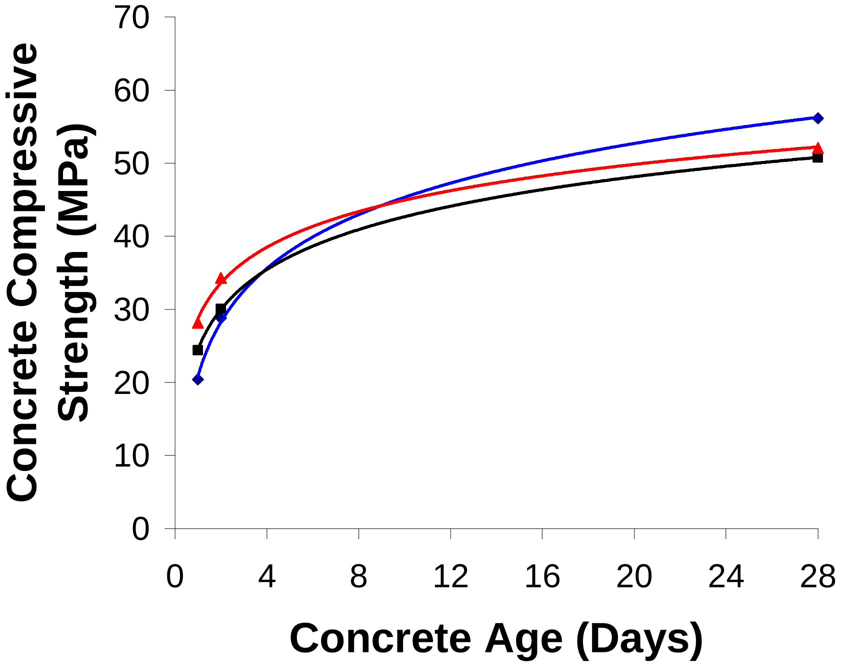


Figure C.6: Compressive strength test results for w/c 0.40

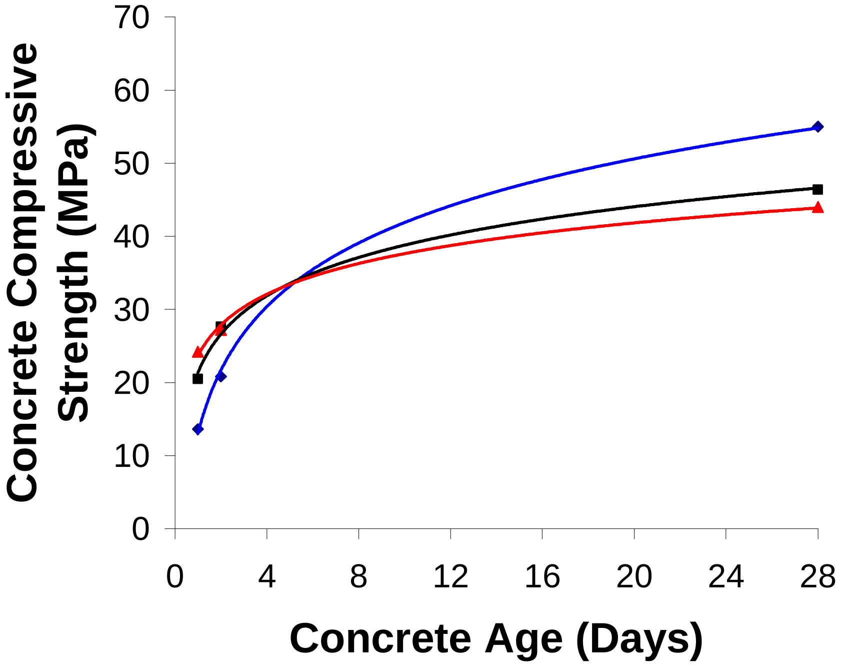


Figure C.7: Compressive strength test results for w/c 0.48

Appendix D

Penetration Resistance Test Results

Appendix D contains results from penetration resistance testing for each batch of concrete tested.

D.1 Penetration Resistance Results as per ASTM C 403 (2008)

Penetration resistance test results are presented in the following figures. All penetration resistance testing was completed as per ASTM C 403 (2008). All three batches of concrete (Hot, Room, and Cold) for each mixture are plotted on the same graph for comparison purposes. Data points shown represent actual penetration resistance readings while the regression analysis is represented by a solid line.

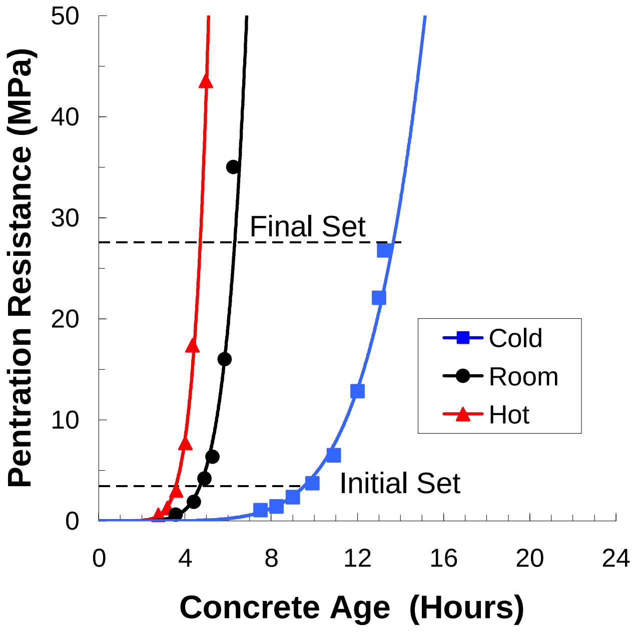


Figure D.1: Penetration resistance test results for Control

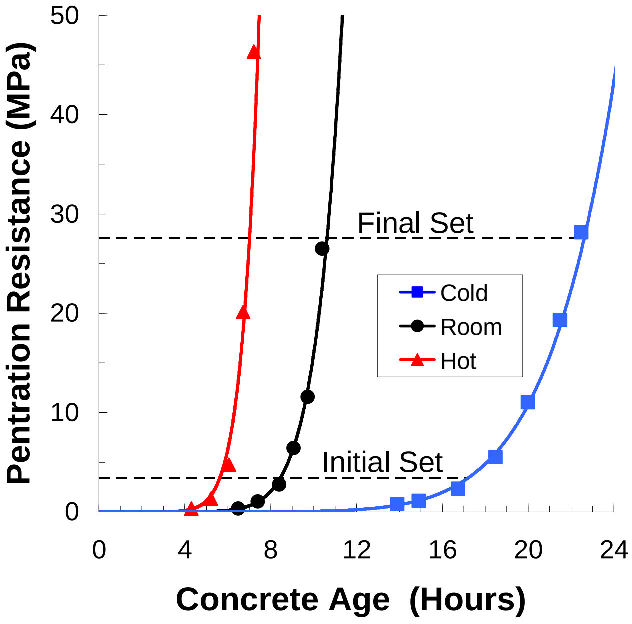


Figure D.2: Penetration resistance test results for 30% C

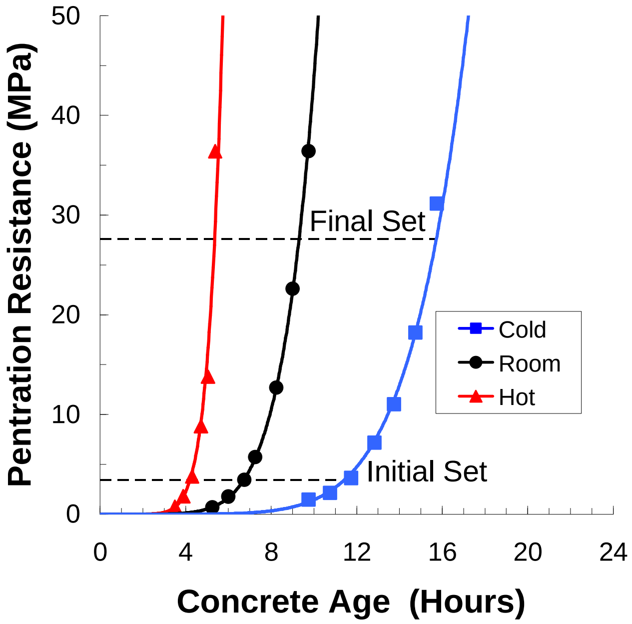


Figure D.3: Penetration resistance test results for 30% F

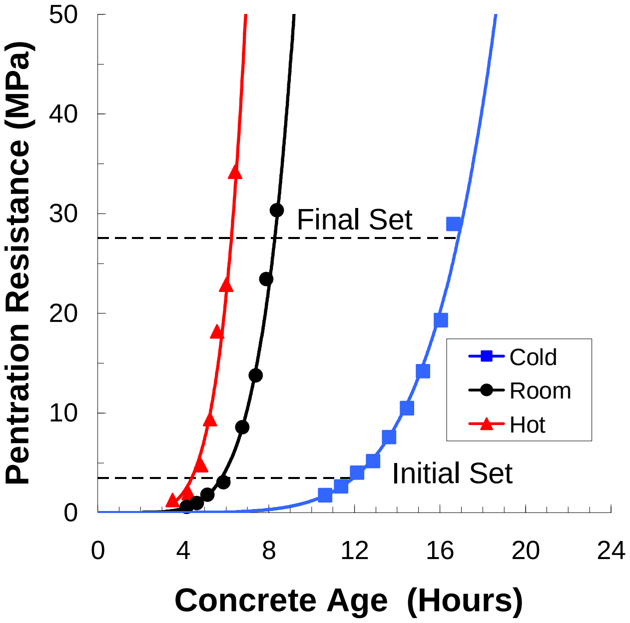


Figure D.4: Penetration resistance test results for 50% Slag

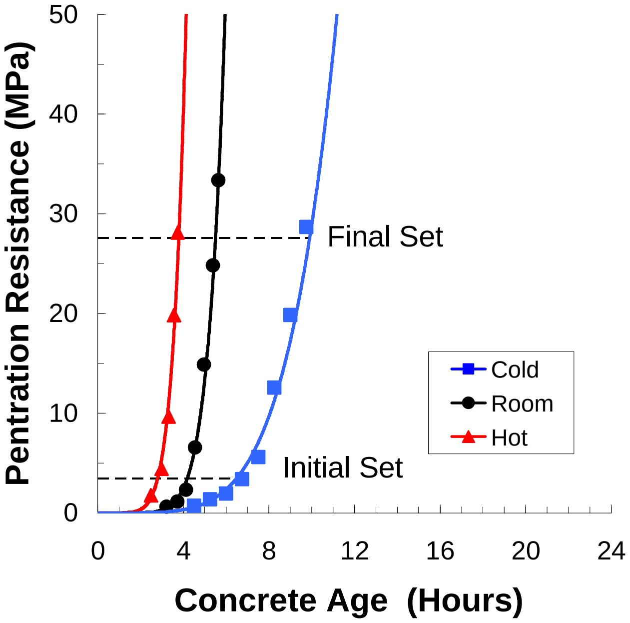


Figure D.5: Penetration resistance test results for w/c 0.36

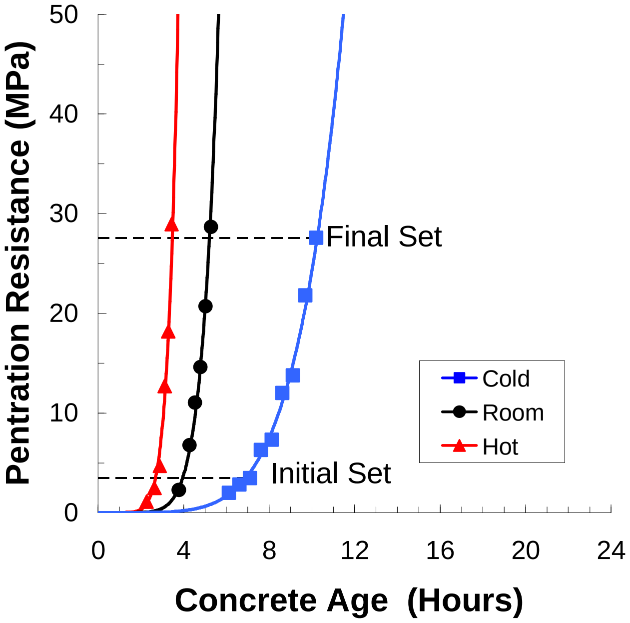


Figure D.6: Penetration resistance test results for w/c 0.40

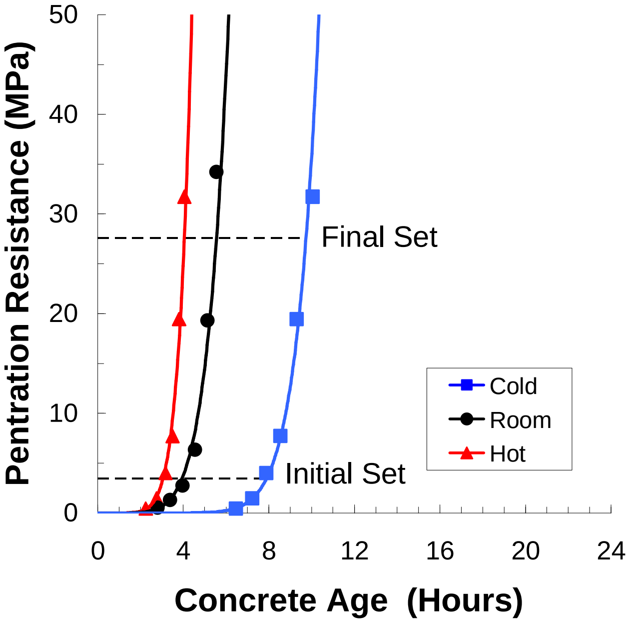


Figure D.7: Penetration resistance test results for w/c 0.48

D.2 Mortar Temperature Results

Mortar temperature for each ASTM C 403 (2008) sample was recorded during tested and is presented in the following figures. All three batches of concrete (Hot, Room, and Cold) for each mixture are plotted on the same graph. Data points shown are for distinguishing between data series only, and do not represent actual data points.

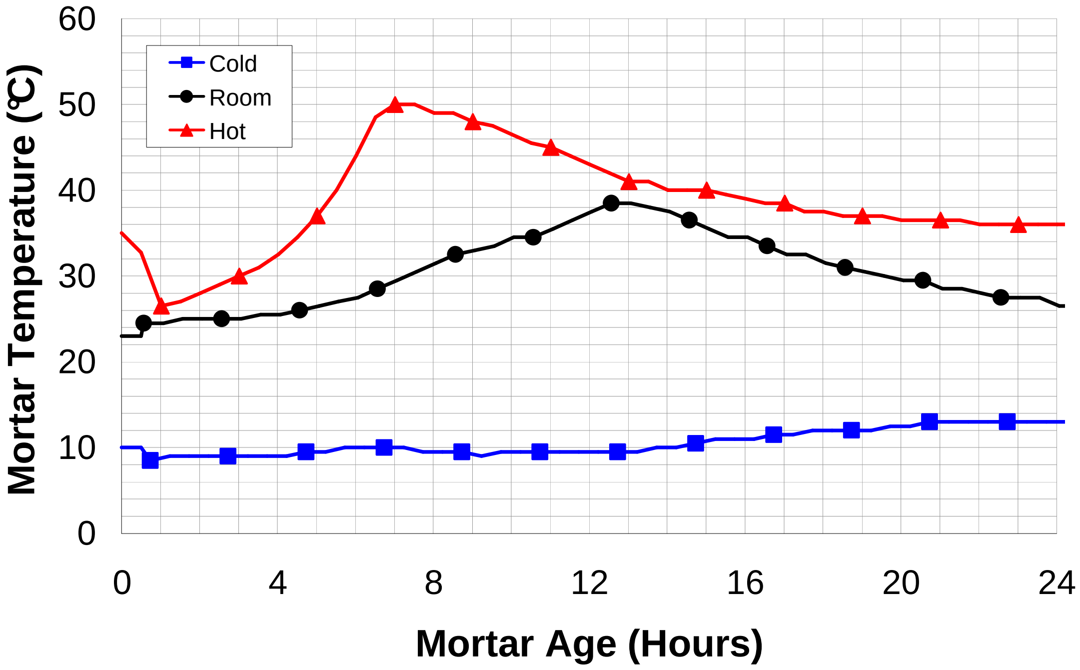


Figure D.8: Mortar temperature results for Control

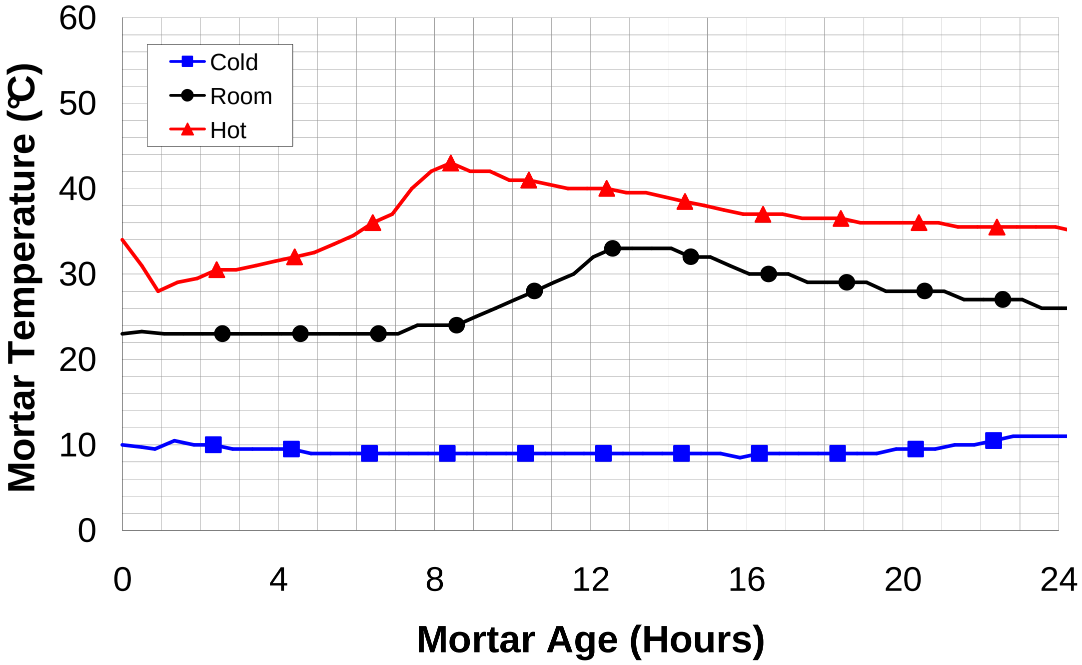


Figure D.9: Mortar temperature results for 30% C

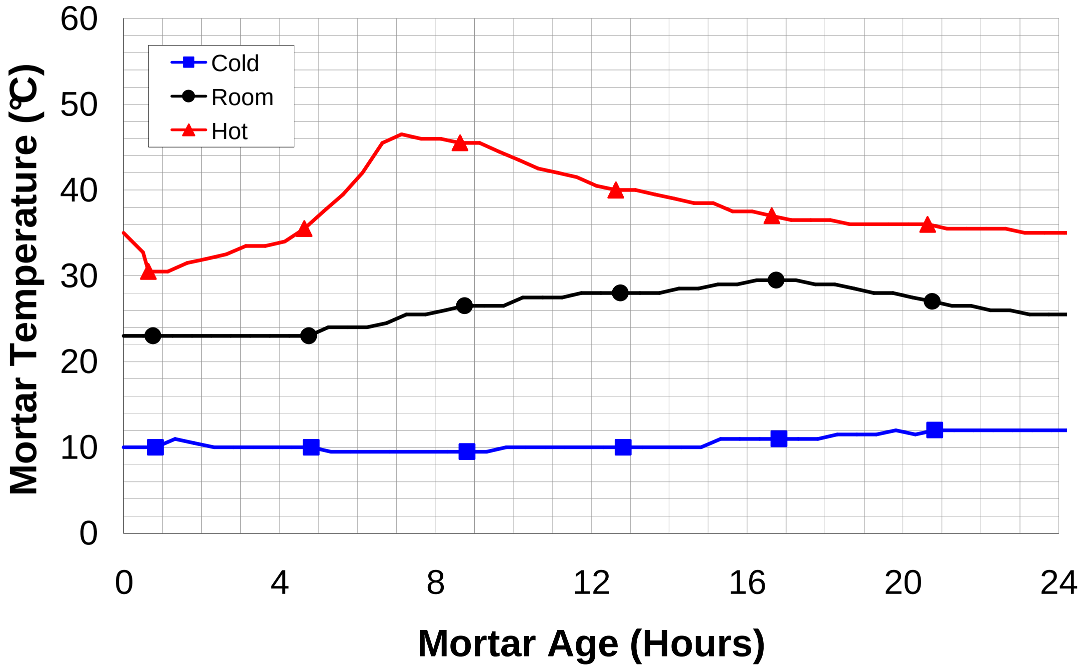


Figure D.10: Mortar temperature results for 30 % F

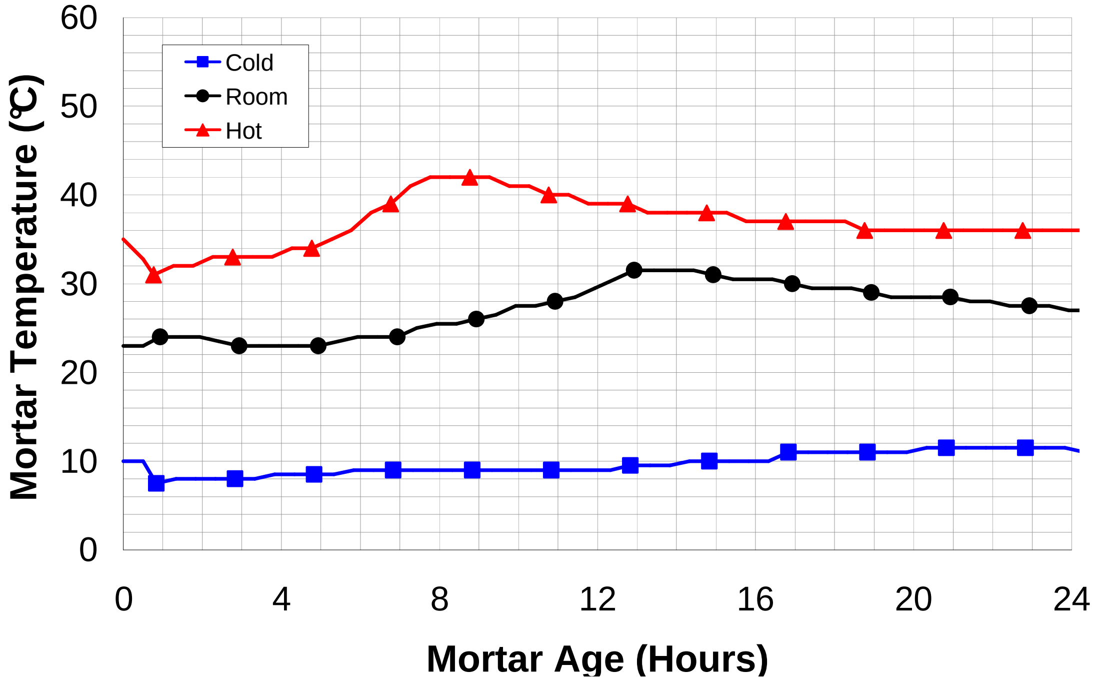


Figure D.11: Mortar temperature results 50 % Slag

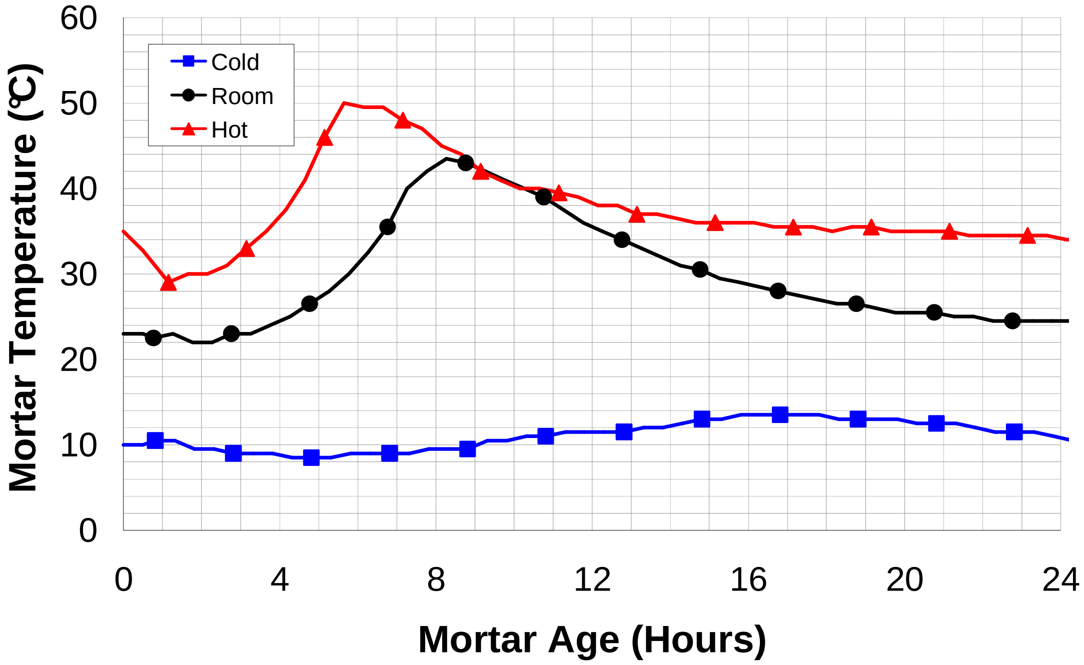


Figure D.12: Mortar temperature results for w/c 0.36

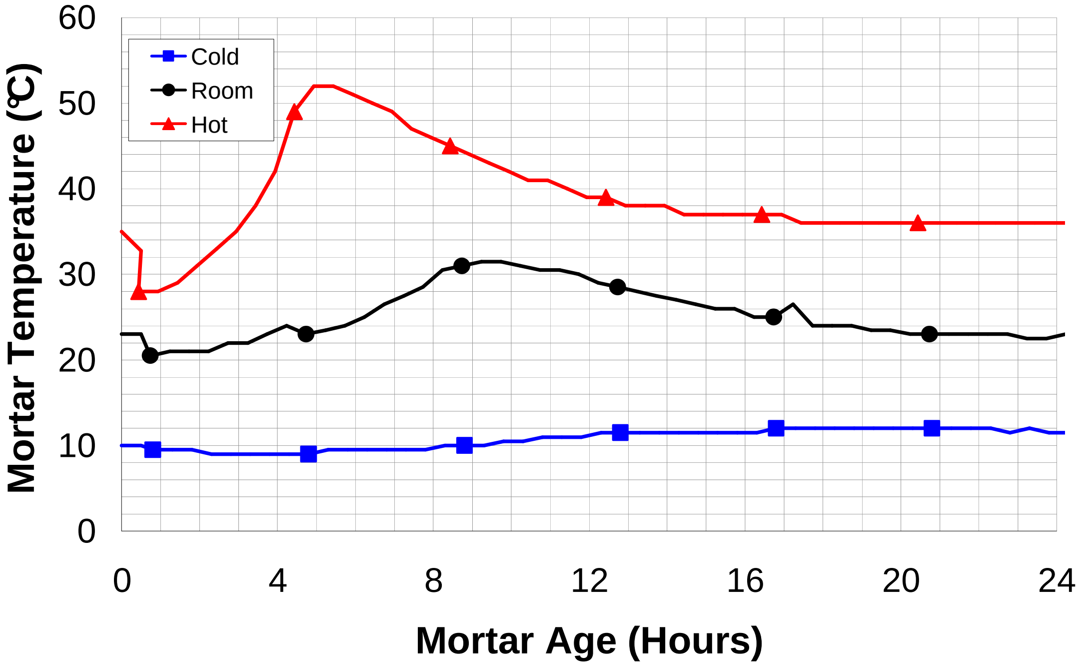


Figure D.13: Mortar temperature results for w/c 0.40

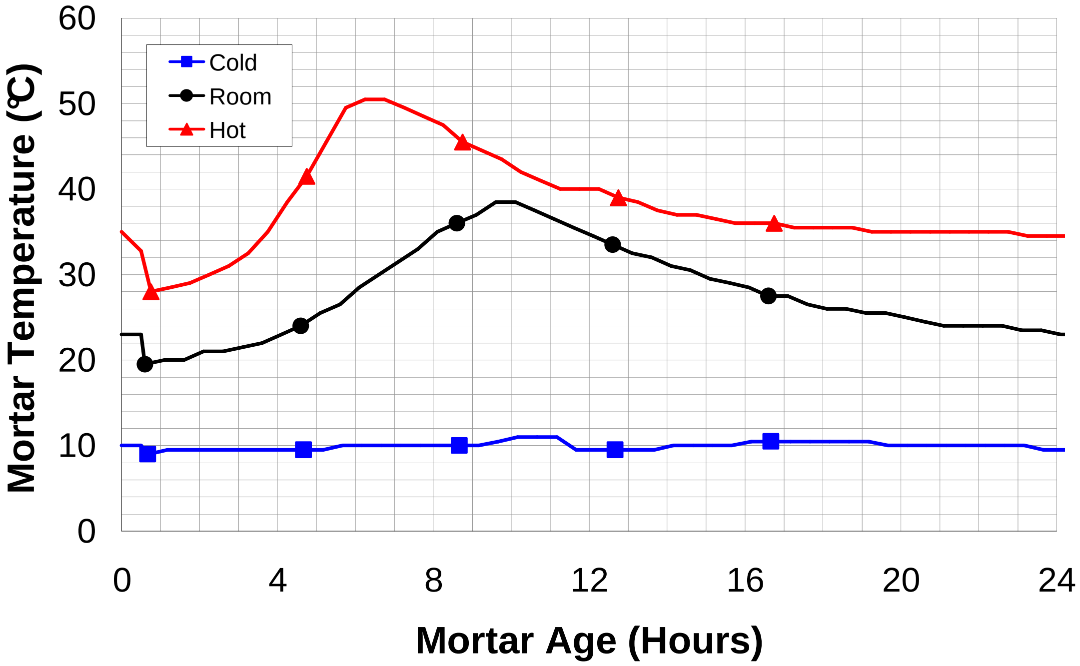


Figure D.14: Mortar temperature results for w/c 0.48

C.3 Penetration Resistance Results Adjusted for Maturity

Penetration resistance results adjusted for maturity using the “best fit” activation energy are presented in the following figures. All penetration resistance testing was completed as per ASTM C 403 (2008). The equivalent age maturity method was used to convert concrete age to concrete equivalent age. An activation energy value was used such that the error between initial and final set points for the three batches (Hot, Room, and Cold), was minimized. Data points shown represent actual penetration resistance readings while regression analysis is represented by a solid line.

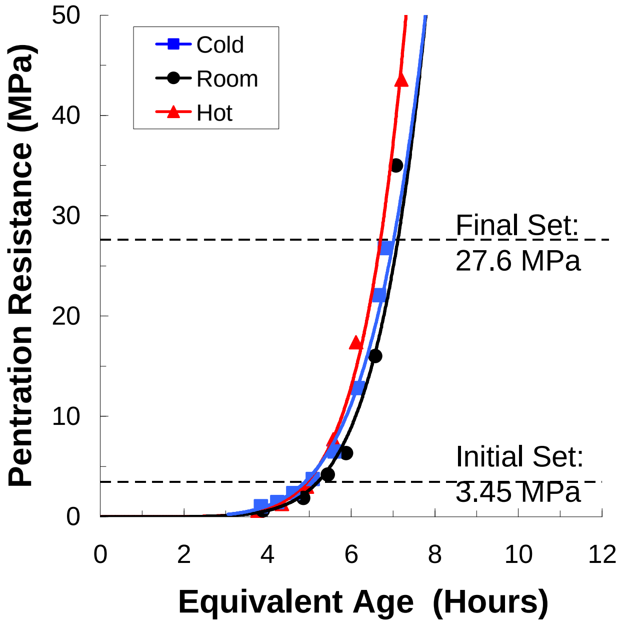


Figure D.15: Penetration resistance versus equivalent age for Control

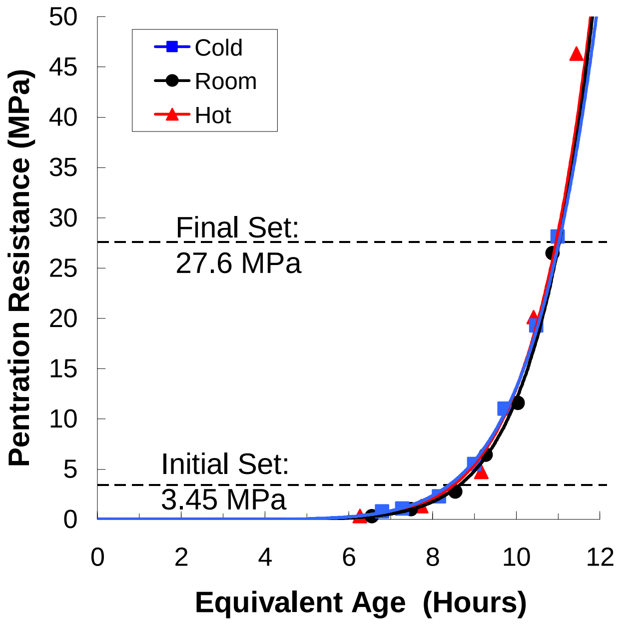


Figure D.16: Penetration resistance versus equivalent age for 30% C

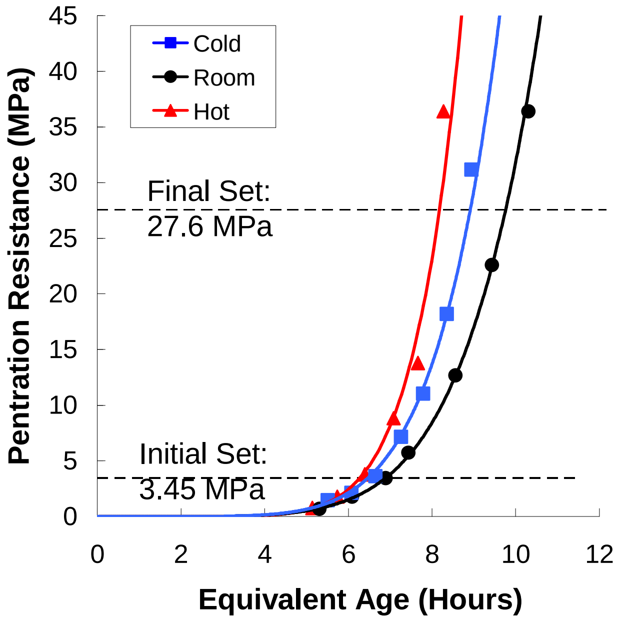


Figure D.17: Penetration resistance versus equivalent age for 30 % F

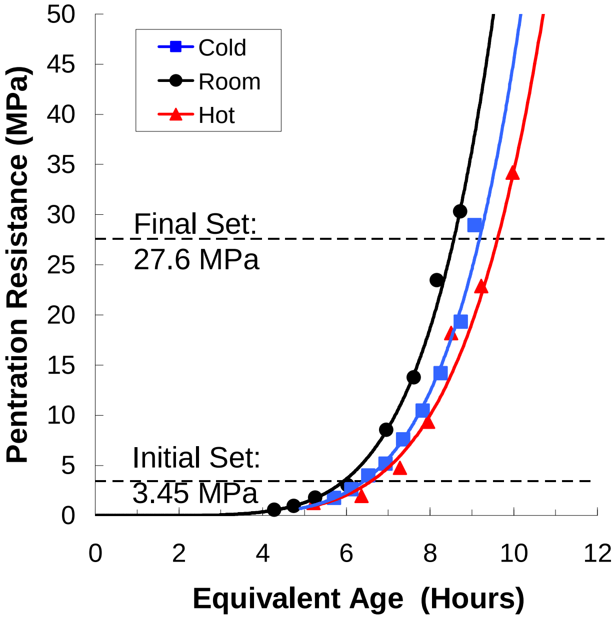


Figure D.18: Penetration resistance versus equivalent age for 50% Slag

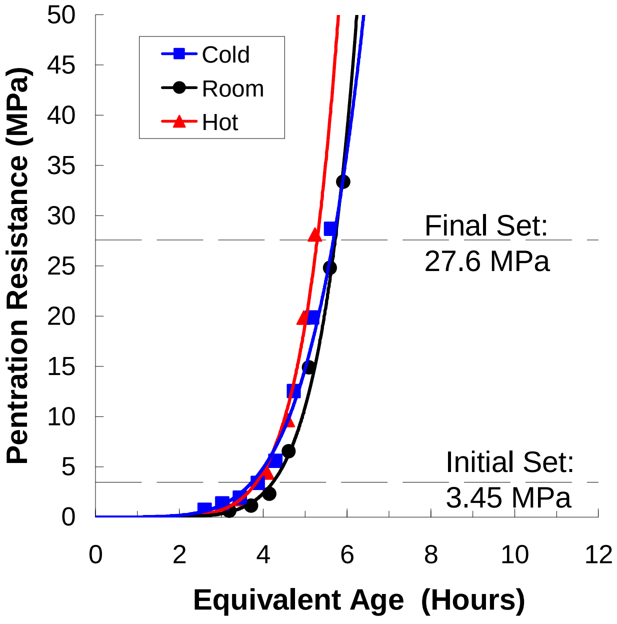


Figure D.19: Penetration resistance versus equivalent age for w/c 0.36

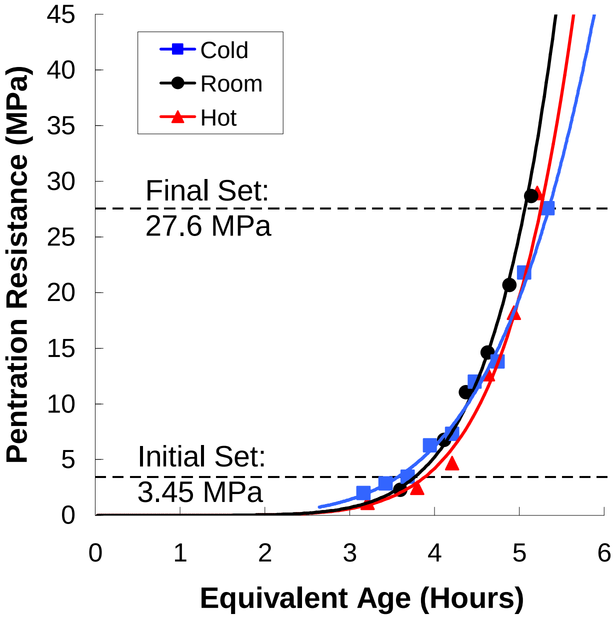


Figure D.20: Penetration resistance versus equivalent age for w/c 0.40

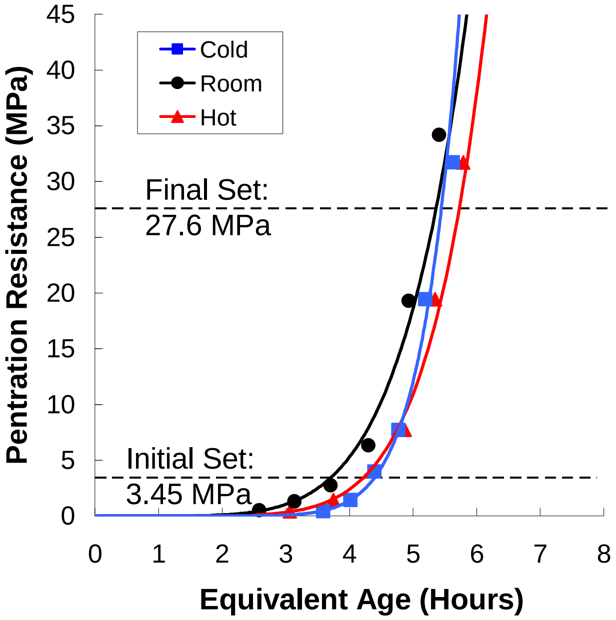


Figure D.21: Penetration resistance versus equivalent age for w/c 0.48

Appendix E

Thermal Test Results

Appendix E contains isothermal calorimetry and semi-adiabatic calorimetry test results for each batch of concrete tested.

E.1 Isothermal Calorimetry Test Results

Results from isothermal calorimetry tests for each concrete batch tested are presented in the following figures. All three batches of concrete (Hot, Room, and Cold) for each mixture are plotted on the same graph. Data points shown are for distinguishing between data series only, and do not represent actual data points. Data were normalized per gram of cement (g_{cem}) tested.

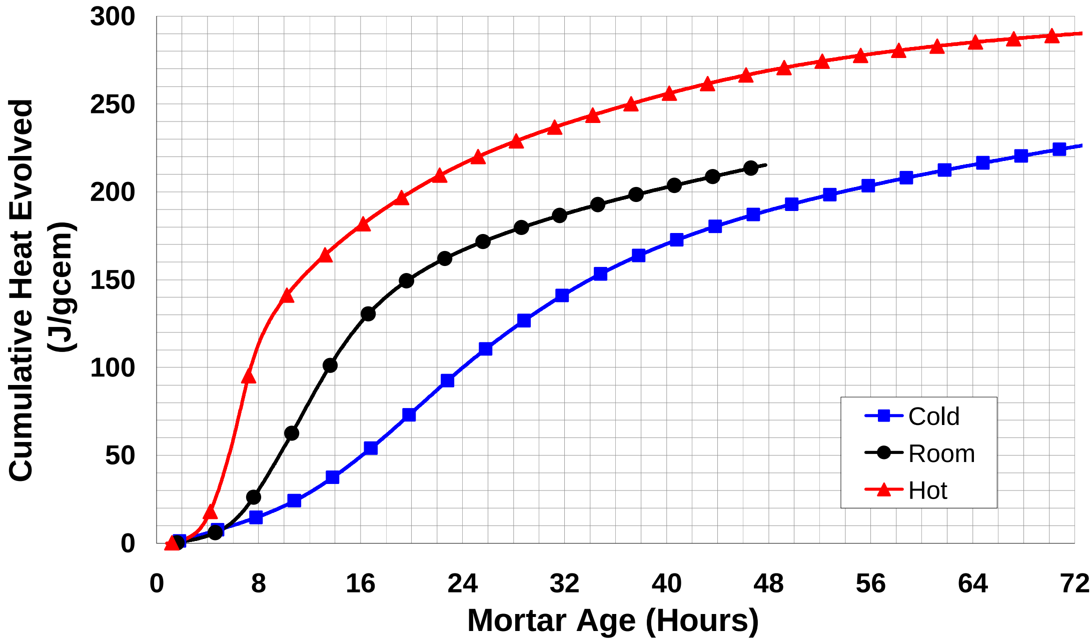


Figure E.1: Isothermal calorimetry test results (energy) for Control

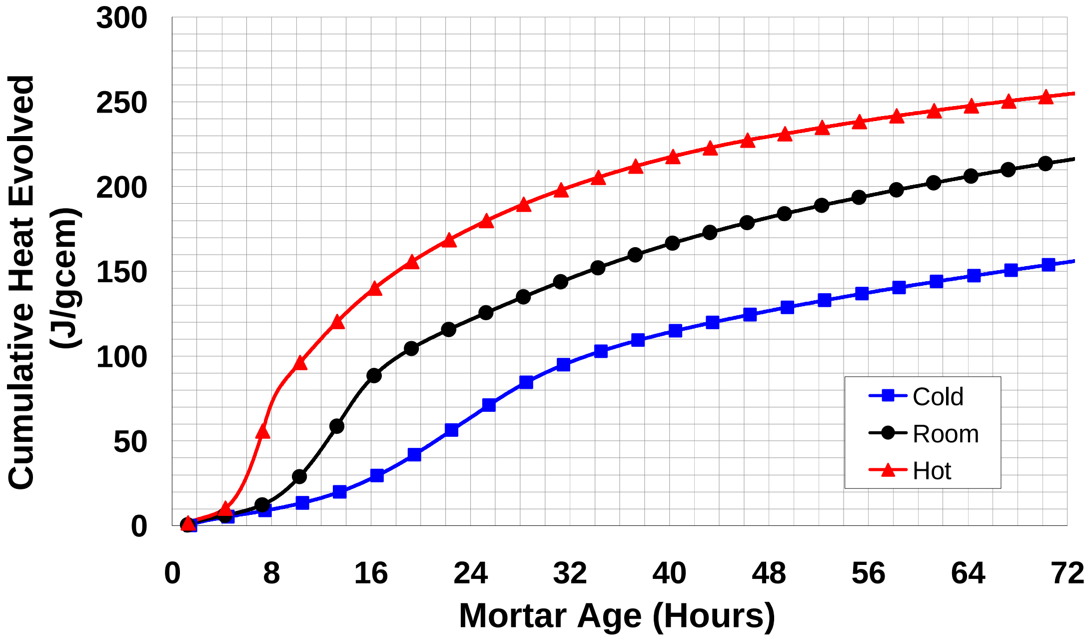


Figure E.2: Isothermal calorimetry test results (energy) for 30% C

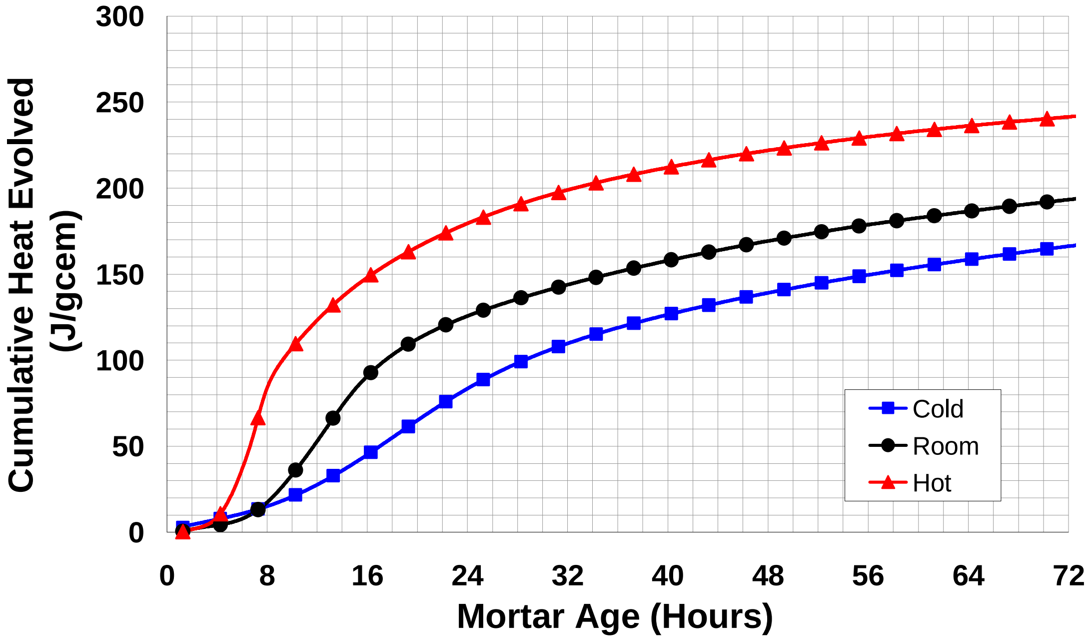


Figure E.3: Isothermal calorimetry test results (energy) for 30% F

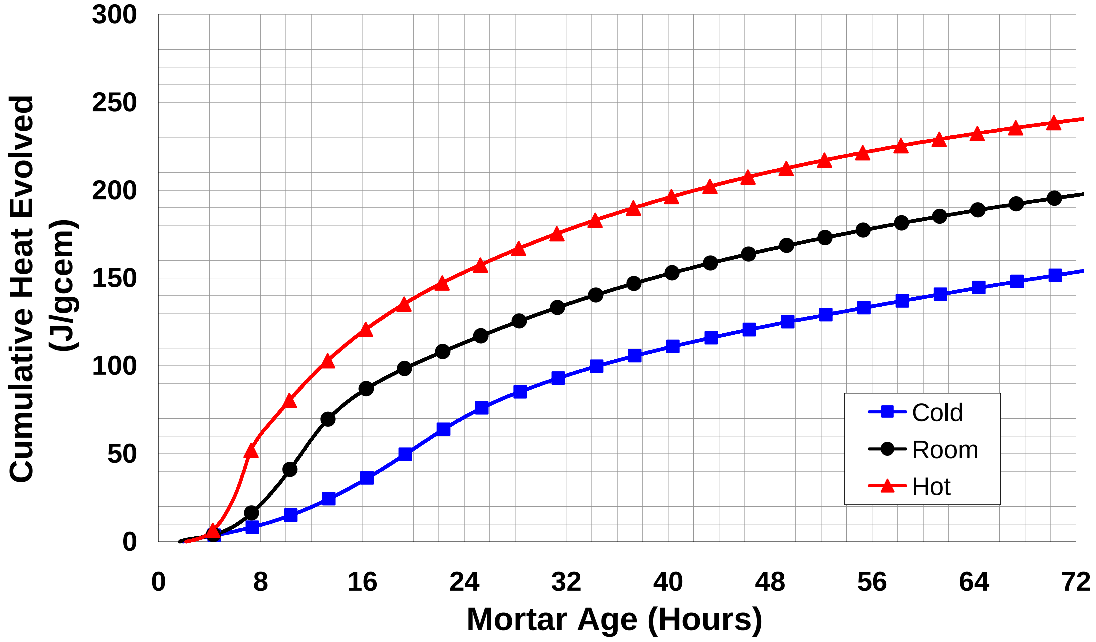


Figure E.4: Isothermal calorimetry test results (energy) for 50% Slag

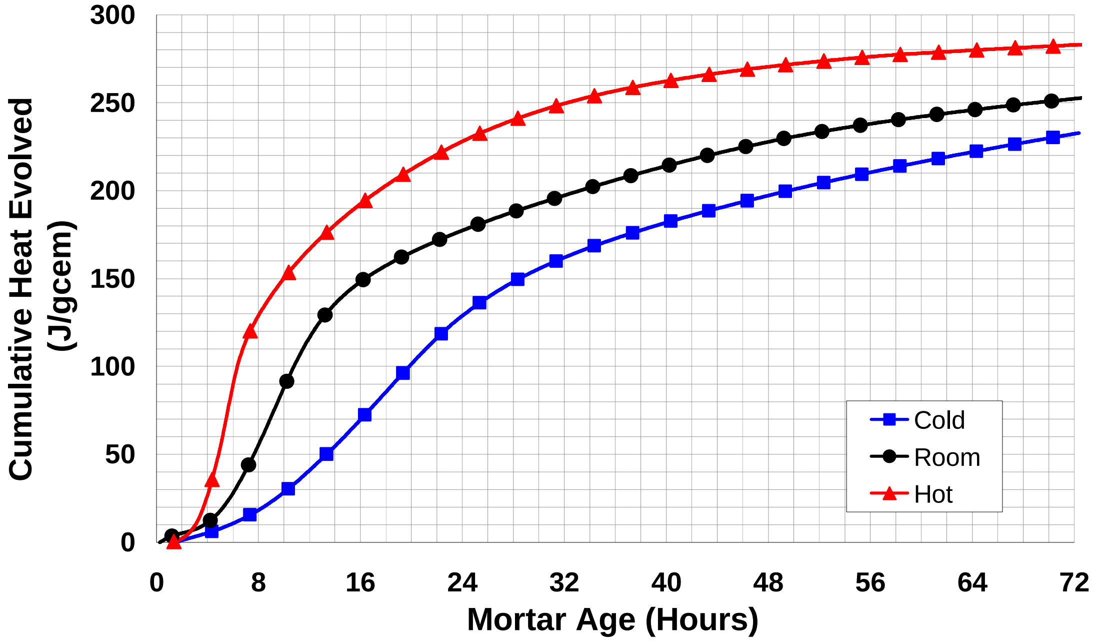


Figure E.5: Isothermal calorimetry test results (energy) for w/c 0.36

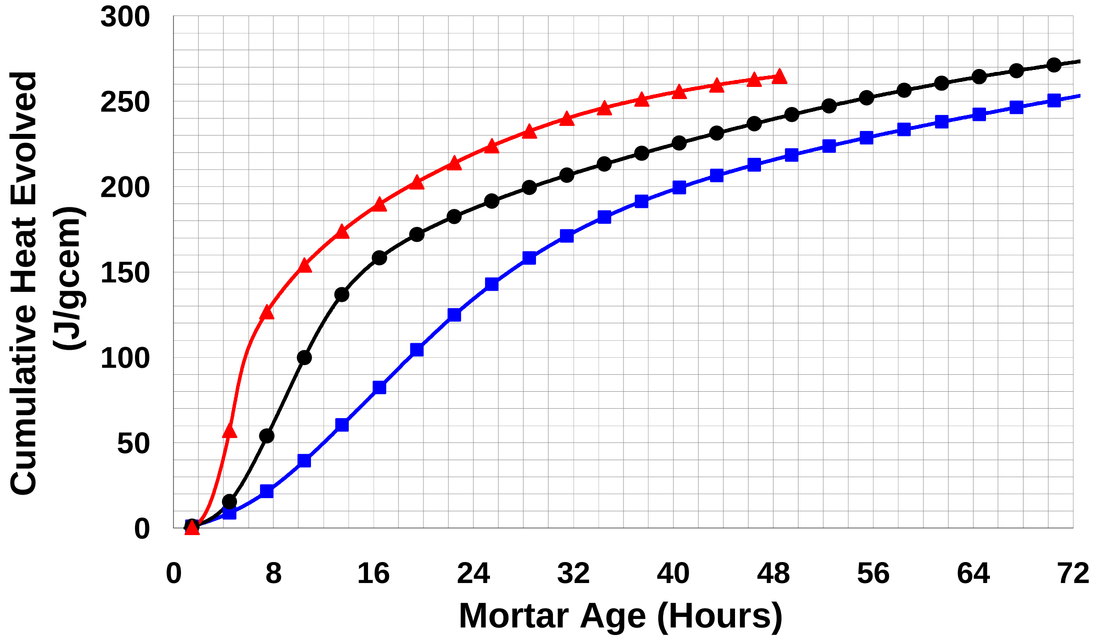


Figure E.6: Isothermal calorimetry test results (energy) for w/c 0.40

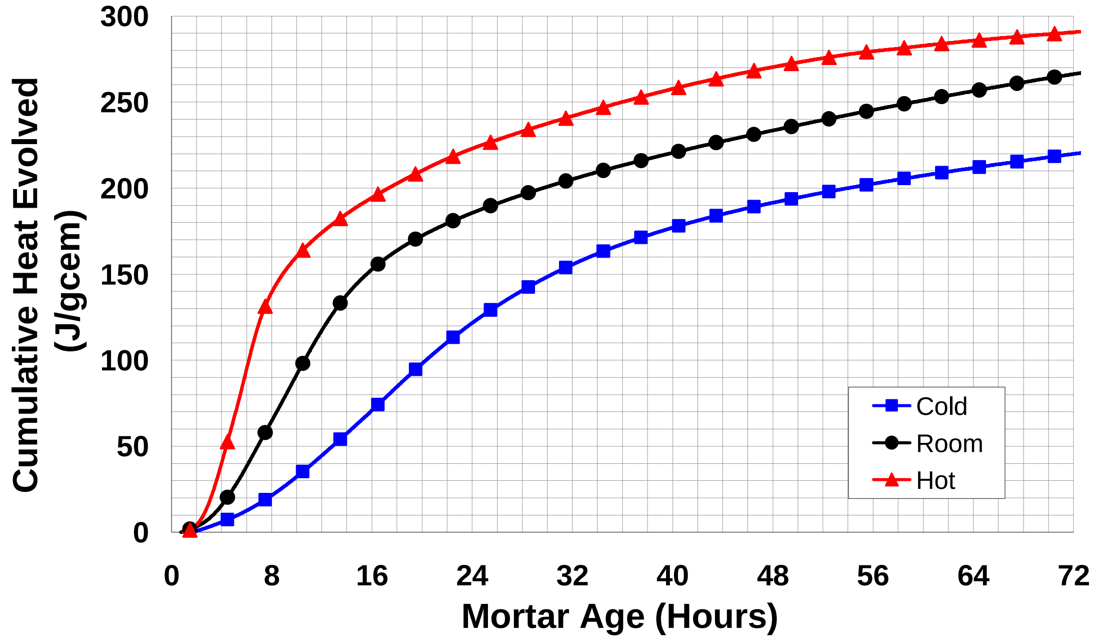


Figure E.7: Isothermal calorimetry test results (energy) for w/c 0.48

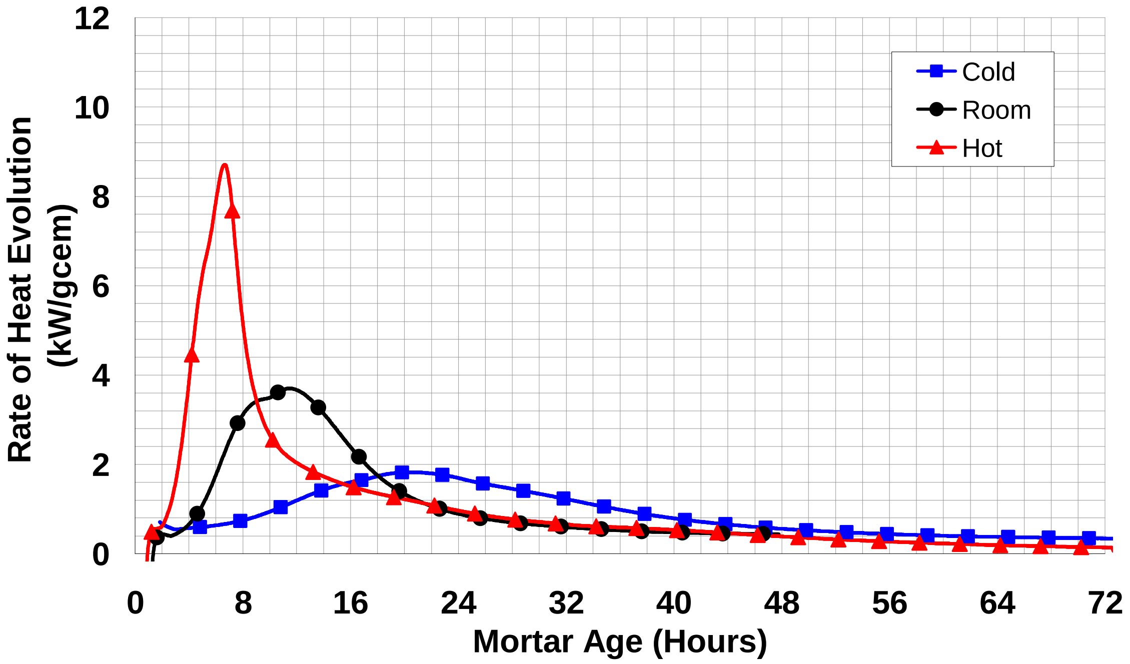


Figure E.8: Isothermal Calorimetry test results (Power) for Control

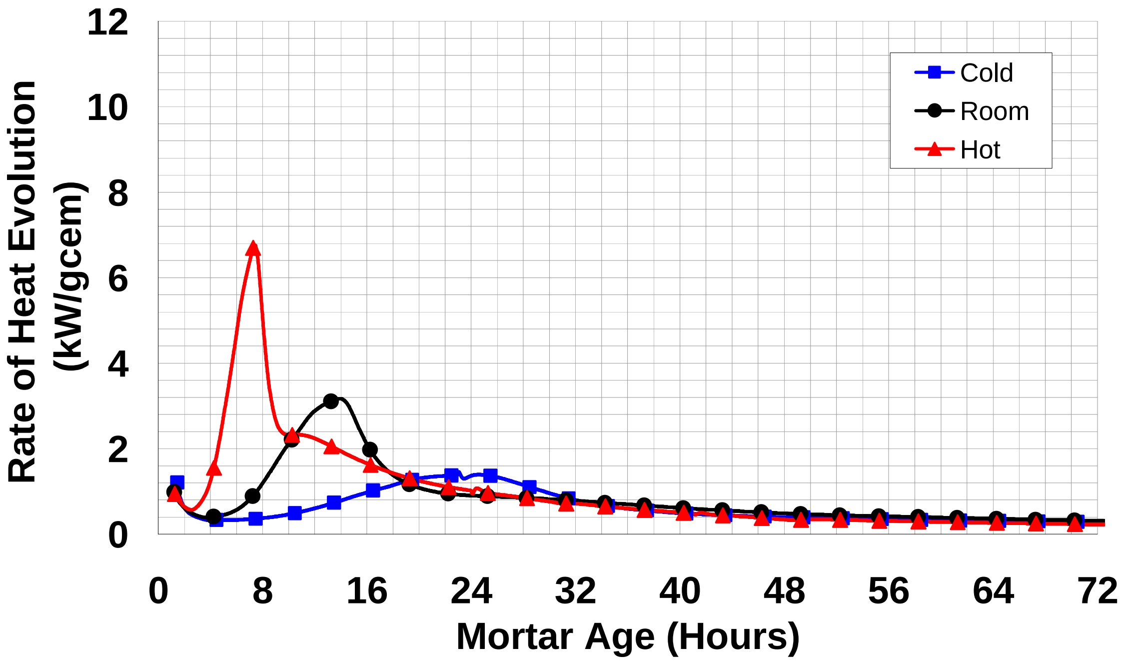


Figure E.9: Isothermal calorimetry test results (power) for 30% C

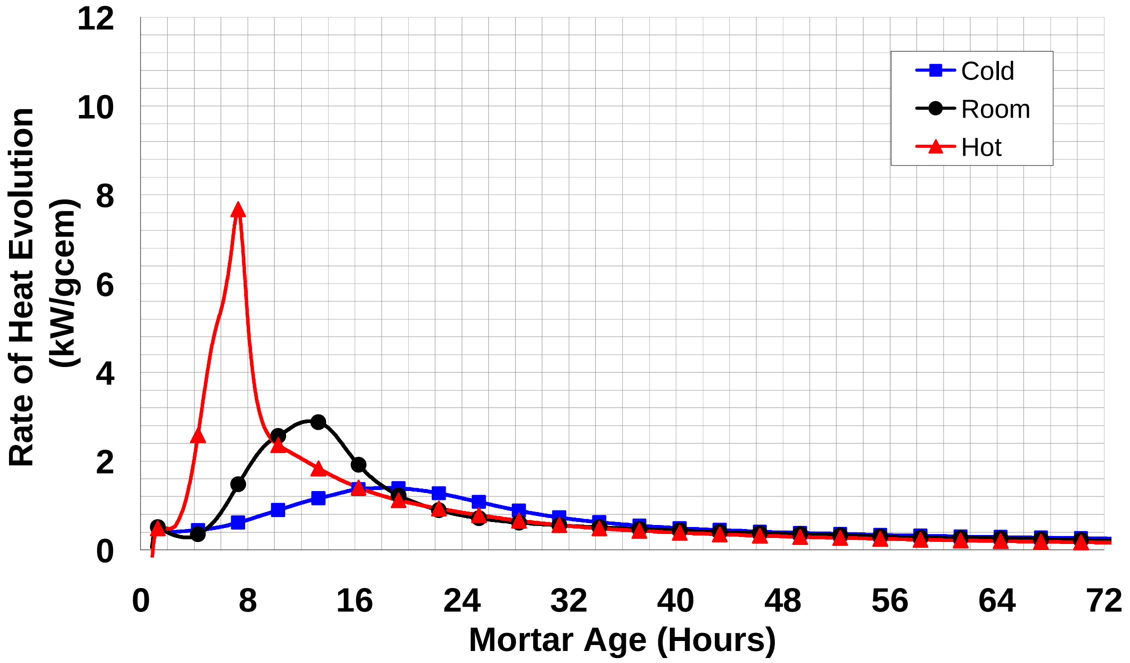


Figure E.10: Isothermal calorimetry test results (power) for 30% F

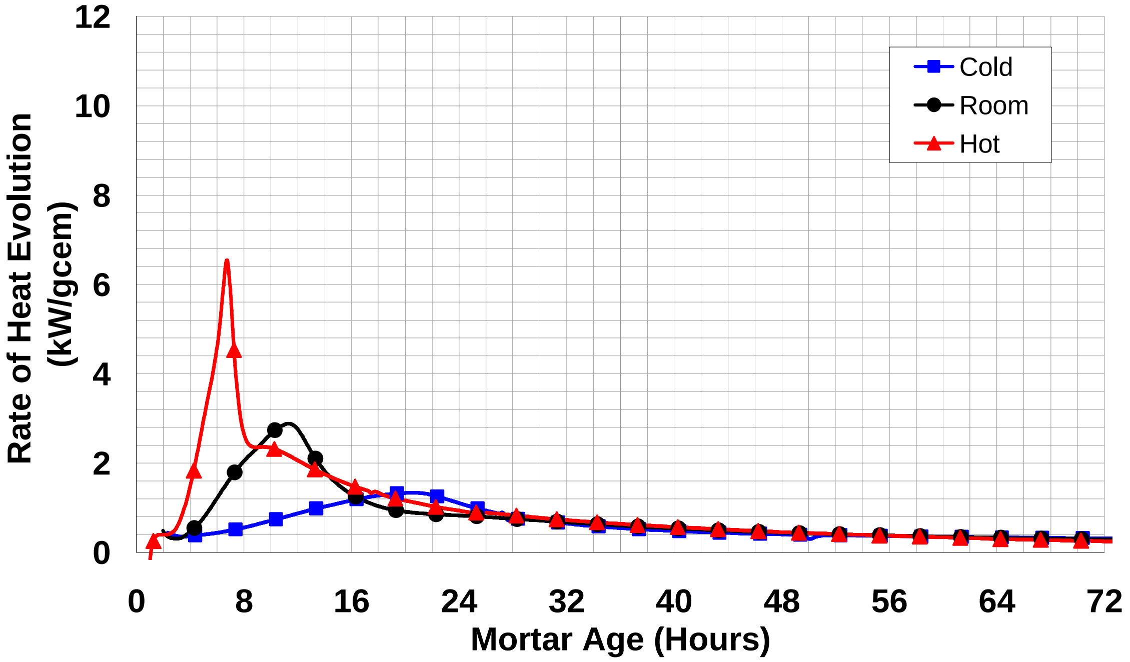


Figure E.11: Isothermal calorimetry test results (power) for 50% Slag

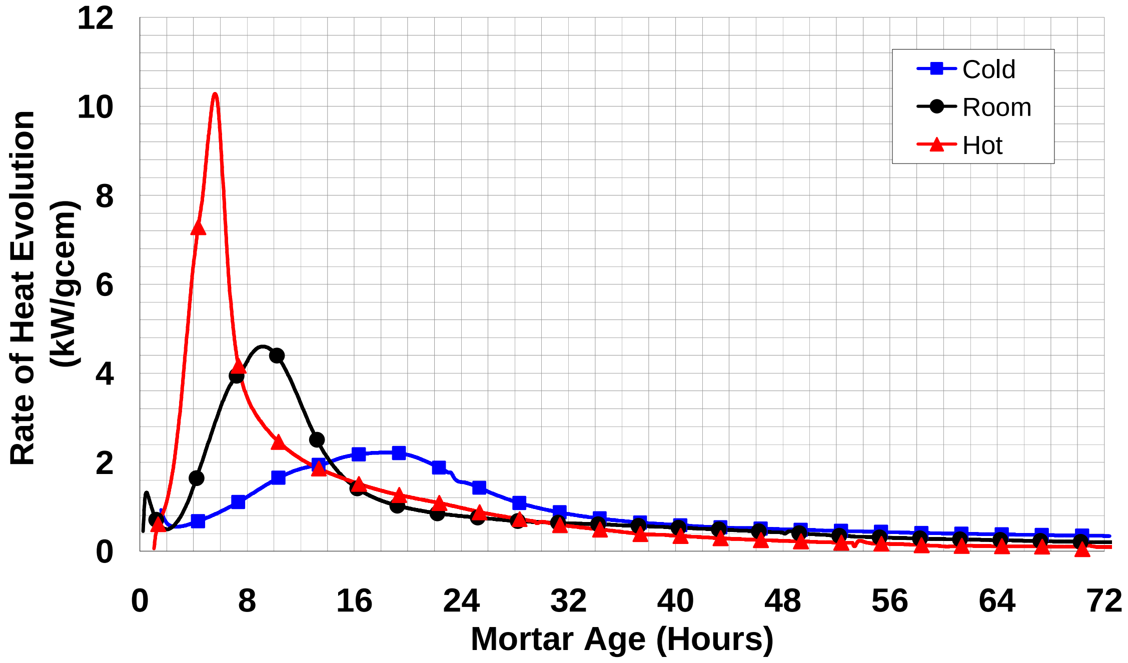


Figure E.12: Isothermal calorimetry test results (power) for w/c 0.36

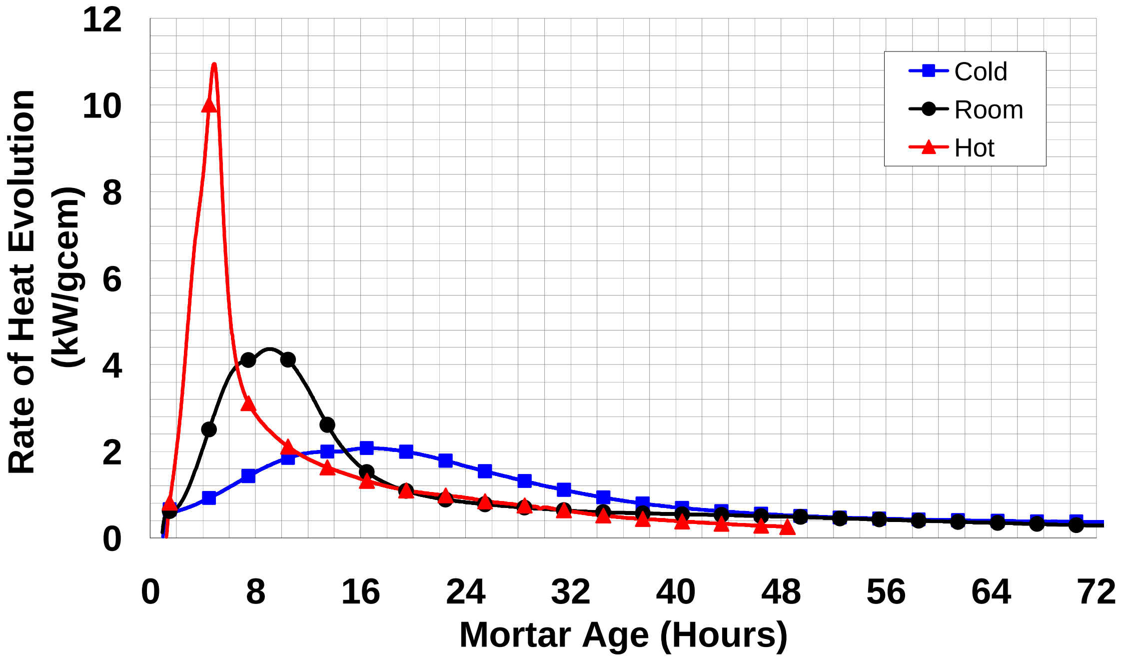


Figure E.13: Isothermal calorimetry test results (power) for w/c 0.40

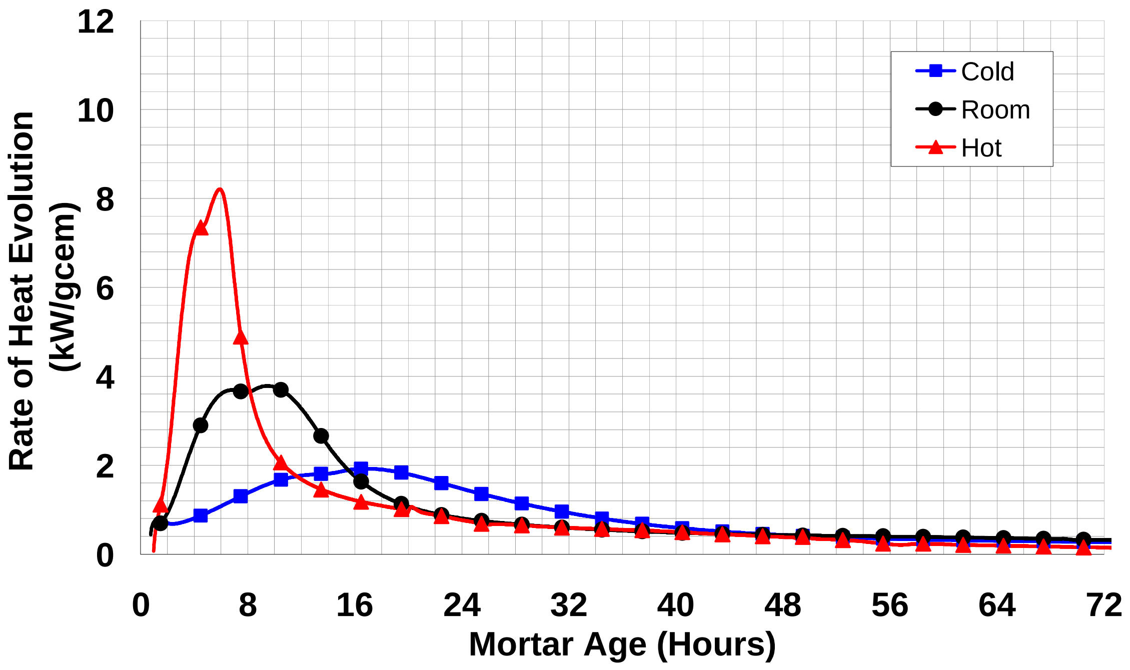


Figure E.14: Isothermal calorimetry test results (power) for w/c 0.48

E.2 Semi-adiabatic Calorimetry Test Results

Select data from semi-adiabatic tests for each concrete batch tested for Calorimeter I and Calorimeter II are presented in the following tables. One point for every hour of testing is shown. Data were collected at one-minute intervals for Calorimeter I and 15-minute intervals for Calorimeter II.

Table E.1: Select data from Calorimeter I for Control – Cold

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	8.8	49.0	23.8	97.0	15.0
2.0	9.2	50.0	23.6	98.0	14.9
3.0	9.5	51.0	23.4	99.0	14.8
4.0	9.9	52.0	23.1	100.0	14.7
5.0	10.1	53.0	22.9	101.0	14.6
6.0	10.5	54.0	22.7	102.0	14.5
7.0	10.8	55.0	22.5	103.0	14.4
8.0	11.1	56.0	22.2	104.0	14.3
9.0	11.4	57.0	22.0	105.0	14.2
10.0	11.8	58.0	21.7	106.0	14.1
11.0	12.2	59.0	21.5	107.0	14.0
12.0	12.7	60.0	21.3	108.0	13.9
13.0	13.3	61.0	21.0	109.0	13.8
14.0	14.0	62.0	20.8	110.0	13.8
15.0	14.8	63.0	20.6	111.0	13.7
16.0	15.7	64.0	20.4	112.0	13.6
17.0	16.7	65.0	20.2	113.0	13.5
18.0	17.8	66.0	20.0	114.0	13.4
19.0	19.0	67.0	19.7	115.0	13.4
20.0	20.3	68.0	19.5	116.0	13.3
21.0	21.6	69.0	19.3	117.0	13.2
22.0	22.8	70.0	19.1	118.0	13.1
23.0	24.0	71.0	18.9	119.0	13.1
24.0	24.9	72.0	18.7	120.0	13.0
25.0	25.6	73.0	18.5	121.0	12.9
26.0	26.1	74.0	18.3	122.0	12.9
27.0	26.4	75.0	18.2	123.0	12.8
28.0	26.6	76.0	18.0	124.0	12.8
29.0	26.7	77.0	17.8	125.0	12.7
30.0	26.7	78.0	17.6	126.0	12.6
31.0	26.6	79.0	17.5	127.0	12.6
32.0	26.6	80.0	17.3	128.0	12.5
33.0	26.5	81.0	17.1	129.0	12.5
34.0	26.4	82.0	17.0	130.0	12.4
35.0	26.2	83.0	16.8	131.0	12.4
36.0	26.1	84.0	16.7	132.0	12.3
37.0	26.0	85.0	16.5	133.0	12.3
38.0	25.8	86.0	16.4	134.0	12.2
39.0	25.6	87.0	16.2	135.0	12.2
40.0	25.5	88.0	16.1	136.0	12.1
41.0	25.3	89.0	16.0	137.0	12.1
42.0	25.1	90.0	15.8	138.0	12.1
43.0	24.9	91.0	15.7	139.0	12.0
44.0	24.8	92.0	15.6	140.0	12.0
45.0	24.6	93.0	15.4	141.0	12.0
46.0	24.3	94.0	15.3	142.0	11.9
47.0	24.1	95.0	15.2	143.0	11.9
48.0	24.0	96.0	15.1	144.0	11.8

Table E.2: Select data from Calorimeter I for Control – Room

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
-	-	49.0	35.5	97.0	27.1
2.0	25.0	50.0	35.2	98.0	27.0
3.0	25.3	51.0	34.9	99.0	26.9
4.0	25.8	52.0	34.7	100.0	26.8
5.0	26.5	53.0	34.4	101.0	26.8
6.0	27.7	54.0	34.1	102.0	26.7
7.0	29.1	55.0	33.9	103.0	26.6
8.0	30.7	56.0	33.7	104.0	26.5
9.0	32.5	57.0	33.4	105.0	26.5
10.0	34.1	58.0	33.2	106.0	26.4
11.0	35.9	59.0	32.9	107.0	26.3
12.0	38.1	60.0	32.7	108.0	26.3
13.0	40.3	61.0	32.5	109.0	26.2
14.0	41.5	62.0	32.3	110.0	26.1
15.0	42.2	63.0	32.0	111.0	26.1
16.0	42.6	64.0	31.8	112.0	26.0
17.0	42.9	65.0	31.6	113.0	25.9
18.0	43.1	66.0	31.4	114.0	25.9
19.0	43.2	67.0	31.2	115.0	25.8
20.0	43.2	68.0	31.0	116.0	25.8
21.0	43.1	69.0	30.8	117.0	25.7
22.0	43.0	70.0	30.6	118.0	25.6
23.0	42.8	71.0	30.4	119.0	25.6
24.0	42.6	72.0	30.2	120.0	25.5
25.0	42.4	73.0	30.0	121.0	25.5
26.0	42.1	74.0	29.9	122.0	25.4
27.0	41.9	75.0	29.7	123.0	25.4
28.0	41.6	76.0	29.6	124.0	25.4
29.0	41.4	77.0	29.4	125.0	25.3
30.0	41.1	78.0	29.3	126.0	25.3
31.0	40.8	79.0	29.2	127.0	25.2
32.0	40.5	80.0	29.0	128.0	25.2
33.0	40.2	81.0	28.9	129.0	25.1
34.0	39.9	82.0	28.8	130.0	25.1
35.0	39.6	83.0	28.6	131.0	25.1
36.0	39.3	84.0	28.5	132.0	25.0
37.0	39.0	85.0	28.4	133.0	25.0
38.0	38.7	86.0	28.3	134.0	25.0
39.0	38.4	87.0	28.1	135.0	25.0
40.0	38.1	88.0	28.0	136.0	24.9
41.0	37.8	89.0	27.9	137.0	24.9
42.0	37.5	90.0	27.8	138.0	24.9
43.0	37.2	91.0	27.7	139.0	24.8
44.0	36.9	92.0	27.6	140.0	24.8
45.0	36.6	93.0	27.5	141.0	24.8
46.0	36.3	94.0	27.4	142.0	24.8
47.0	36.0	95.0	27.3	143.0	24.7
48.0	35.7	96.0	27.2	144.0	24.7

Table E.3: Select data from Calorimeter I for Control – Hot

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	34.2	49.0	44.7	97.0	36.3
2.0	34.2	50.0	44.4	98.0	36.2
3.0	34.8	51.0	44.1	99.0	36.1
4.0	36.6	52.0	43.8	100.0	36.1
5.0	39.8	53.0	43.5	101.0	36.0
6.0	45.3	54.0	43.2	102.0	36.0
7.0	49.0	55.0	42.9	103.0	35.9
8.0	51.4	56.0	42.6	104.0	35.8
9.0	53.4	57.0	42.3	105.0	35.8
10.0	54.9	58.0	42.1	106.0	35.7
11.0	56.1	59.0	41.8	107.0	35.6
12.0	56.8	60.0	41.6	108.0	35.6
13.0	57.2	61.0	41.3	109.0	35.5
14.0	57.3	62.0	41.1	110.0	35.5
15.0	57.3	63.0	40.9	111.0	35.4
16.0	57.2	64.0	40.6	112.0	35.4
17.0	57.0	65.0	40.4	113.0	35.3
18.0	56.8	66.0	40.2	114.0	35.3
19.0	56.6	67.0	40.0	115.0	35.3
20.0	56.3	68.0	39.8	116.0	35.2
21.0	56.0	69.0	39.7	117.0	35.2
22.0	55.7	70.0	39.5	118.0	35.1
23.0	55.3	71.0	39.3	119.0	35.1
24.0	54.9	72.0	39.1	120.0	35.0
25.0	54.6	73.0	39.0	121.0	35.0
26.0	54.2	74.0	38.8	122.0	35.0
27.0	53.7	75.0	38.7	123.0	35.0
28.0	53.3	76.0	38.5	124.0	34.9
29.0	52.9	77.0	38.4	125.0	34.9
30.0	52.4	78.0	38.3	126.0	34.9
31.0	52.0	79.0	38.1	127.0	34.8
32.0	51.5	80.0	38.0	128.0	34.8
33.0	51.1	81.0	37.9	129.0	34.8
34.0	50.6	82.0	37.7	130.0	34.8
35.0	50.2	83.0	37.6	131.0	34.8
36.0	49.8	84.0	37.5	132.0	34.7
37.0	49.3	85.0	37.4	133.0	34.7
38.0	48.9	86.0	37.3	134.0	34.7
39.0	48.5	87.0	37.2	135.0	34.7
40.0	48.0	88.0	37.1	136.0	34.6
41.0	47.6	89.0	37.0	137.0	34.6
42.0	47.3	90.0	36.9	138.0	34.6
43.0	46.9	91.0	36.8	139.0	34.6
44.0	46.5	92.0	36.7	140.0	34.5
45.0	46.1	93.0	36.6	141.0	34.5
46.0	45.8	94.0	36.5	142.0	34.5
47.0	45.4	95.0	36.4	143.0	34.5
48.0	45.0	96.0	36.4	144.0	34.5

Table E.4: Select data from Calorimeter II for Control – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	26.3	97.0	18.2
2.0	10.3	50.0	26.1	98.0	18.1
3.0	9.9	51.0	26.0	99.0	17.9
4.0	10.2	52.0	25.7	100.0	17.9
5.0	10.5	53.0	25.7	101.0	17.7
6.0	10.7	54.0	25.5	102.0	17.6
7.0	11.0	55.0	25.3	103.0	17.4
8.0	11.4	56.0	25.1	104.0	17.3
9.0	11.7	57.0	25.0	105.0	17.3
10.0	11.9	58.0	24.8	106.0	17.0
11.0	12.4	59.0	24.6	107.0	16.9
12.0	13.1	60.0	24.4	108.0	16.8
13.0	13.7	61.0	24.3	109.0	16.8
14.0	14.2	62.0	24.1	110.0	16.6
15.0	15.1	63.0	23.9	111.0	16.5
16.0	16.1	64.0	23.7	112.0	16.4
17.0	17.1	65.0	23.5	113.0	16.1
18.0	18.2	66.0	23.2	114.0	16.2
19.0	19.5	67.0	23.0	115.0	16.0
20.0	20.8	68.0	22.8	116.0	15.8
21.0	22.1	69.0	22.8	117.0	15.7
22.0	23.3	70.0	22.6	118.0	15.7
23.0	24.3	71.0	22.3	119.0	15.7
24.0	25.5	72.0	22.1	120.0	15.6
25.0	26.0	73.0	22.1	121.0	15.5
26.0	26.6	74.0	21.9	122.0	15.3
27.0	26.9	75.0	21.7	123.0	15.3
28.0	27.2	76.0	21.5	124.0	15.0
29.0	27.3	77.0	21.4	125.0	15.0
30.0	27.5	78.0	21.2	126.0	14.9
31.0	27.5	79.0	21.0	127.0	14.8
32.0	27.5	80.0	20.8	128.0	14.8
33.0	27.6	81.0	20.8	129.0	14.8
34.0	27.5	82.0	20.6	130.0	14.6
35.0	27.5	83.0	20.2	131.0	14.6
36.0	27.4	84.0	20.1	132.0	14.4
37.0	27.3	85.0	20.1	133.0	14.4
38.0	27.4	86.0	19.9	134.0	14.4
39.0	27.3	87.0	19.6	135.0	14.1
40.0	27.3	88.0	19.5	136.0	14.2
41.0	27.1	89.0	19.4	137.0	14.2
42.0	27.0	90.0	19.3	138.0	14.1
43.0	27.0	91.0	19.0	139.0	14.0
44.0	27.0	92.0	19.0	140.0	14.0
45.0	26.8	93.0	18.8	141.0	13.8
46.0	26.6	94.0	18.8	142.0	13.8
47.0	26.6	95.0	18.6	143.0	13.8
48.0	26.3	96.0	18.5	144.0	13.6

Table E.5: Select data from Calorimeter II for Control – Room

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
-	-	49.0	38.0	97.0	30.1
2.0	24.5	50.0	37.9	98.0	30.2
3.0	25.1	51.0	37.8	99.0	30.0
4.0	25.7	52.0	37.6	100.0	29.9
5.0	26.3	53.0	37.2	101.0	30.0
6.0	27.3	54.0	37.1	102.0	29.8
7.0	28.6	55.0	36.8	103.0	29.6
8.0	30.1	56.0	36.5	104.0	29.6
9.0	31.7	57.0	36.5	105.0	29.6
10.0	33.3	58.0	36.1	106.0	29.5
11.0	34.9	59.0	36.0	107.0	29.5
12.0	36.8	60.0	35.8	108.0	29.5
13.0	39.1	61.0	35.5	109.0	29.3
14.0	40.5	62.0	35.2	110.0	29.1
15.0	41.3	63.0	35.3	111.0	29.0
16.0	41.9	64.0	35.1	112.0	29.0
17.0	42.4	65.0	34.7	113.0	29.1
18.0	42.6	66.0	34.6	114.0	29.0
19.0	42.7	67.0	34.4	115.0	28.9
20.0	43.0	68.0	34.2	116.0	28.8
21.0	43.0	69.0	34.0	117.0	28.8
22.0	43.0	70.0	33.9	118.0	28.6
23.0	42.9	71.0	33.8	119.0	28.5
24.0	42.9	72.0	33.6	120.0	28.5
25.0	42.9	73.0	33.4	121.0	28.5
26.0	42.7	74.0	33.2	122.0	28.4
27.0	42.5	75.0	33.0	123.0	28.4
28.0	42.5	76.0	32.9	124.0	28.2
29.0	42.4	77.0	32.8	125.0	28.1
30.0	42.2	78.0	32.6	126.0	28.1
31.0	42.0	79.0	32.4	127.0	28.0
32.0	41.8	80.0	32.3	128.0	27.9
33.0	41.4	81.0	32.1	129.0	27.9
34.0	41.3	82.0	32.0	130.0	27.7
35.0	41.1	83.0	31.9	131.0	27.7
36.0	40.9	84.0	31.9	132.0	27.8
37.0	40.6	85.0	31.7	133.0	27.5
38.0	40.6	86.0	31.6	134.0	27.6
39.0	40.3	87.0	31.3	135.0	27.5
40.0	40.1	88.0	31.3	136.0	27.5
41.0	39.9	89.0	31.1	137.0	27.5
42.0	39.7	90.0	31.1	138.0	27.4
43.0	39.3	91.0	30.8	139.0	27.3
44.0	39.0	92.0	30.8	140.0	27.3
45.0	39.0	93.0	30.6	141.0	27.4
46.0	38.8	94.0	30.6	142.0	27.4
47.0	38.7	95.0	30.4	143.0	27.4
48.0	38.4	96.0	30.2	144.0	27.2

Table E.6: Select data from Calorimeter II for Control – Hot

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
-	-	49.0	48.1	97.0	38.8
2.0	32.5	50.0	47.9	98.0	38.7
3.0	33.5	51.0	47.5	99.0	38.6
4.0	35.4	52.0	47.2	100.0	38.4
5.0	38.3	53.0	46.9	101.0	38.4
6.0	43.0	54.0	46.7	102.0	38.2
7.0	47.2	55.0	46.5	103.0	38.2
8.0	49.3	56.0	46.1	104.0	38.0
9.0	51.2	57.0	46.0	105.0	38.0
10.0	52.7	58.0	45.6	106.0	38.0
11.0	53.9	59.0	45.3	107.0	37.8
12.0	54.8	60.0	45.1	108.0	37.8
13.0	55.4	61.0	44.7	109.0	37.6
14.0	55.8	62.0	44.7	110.0	37.5
15.0	56.0	63.0	44.4	111.0	37.5
16.0	55.9	64.0	44.2	112.0	37.5
17.0	56.1	65.0	44.0	113.0	37.2
18.0	56.1	66.0	43.7	114.0	37.4
19.0	56.1	67.0	43.5	115.0	37.2
20.0	55.9	68.0	43.3	116.0	37.2
21.0	55.9	69.0	43.0	117.0	37.0
22.0	55.9	70.0	43.0	118.0	37.0
23.0	55.6	71.0	42.6	119.0	36.9
24.0	55.3	72.0	42.4	120.0	36.8
25.0	55.2	73.0	42.4	121.0	36.8
26.0	55.0	74.0	42.0	122.0	36.8
27.0	54.8	75.0	42.0	123.0	36.6
28.0	54.5	76.0	41.8	124.0	36.6
29.0	54.3	77.0	41.7	125.0	36.6
30.0	54.2	78.0	41.5	126.0	36.4
31.0	53.7	79.0	41.4	127.0	36.4
32.0	53.3	80.0	41.1	128.0	36.4
33.0	53.1	81.0	40.8	129.0	36.2
34.0	52.8	82.0	40.7	130.0	36.2
35.0	52.6	83.0	40.7	131.0	36.3
36.0	52.2	84.0	40.4	132.0	36.2
37.0	51.7	85.0	40.4	133.0	36.0
38.0	51.6	86.0	40.3	134.0	36.0
39.0	51.2	87.0	40.2	135.0	36.0
40.0	50.8	88.0	39.9	136.0	36.0
41.0	50.4	89.0	39.8	137.0	36.0
42.0	50.2	90.0	39.6	138.0	36.0
43.0	50.0	91.0	39.6	139.0	36.0
44.0	49.6	92.0	39.4	140.0	35.8
45.0	49.4	93.0	39.2	141.0	35.8
46.0	48.9	94.0	39.2	142.0	35.7
47.0	48.7	95.0	39.1	143.0	35.6
48.0	48.3	96.0	38.8	144.0	35.6

Table E.7: Select data from Calorimeter I for 30%C – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	11.7	49.0	20.1	97.0	14.3
2.0	11.9	50.0	19.9	98.0	14.2
3.0	12.2	51.0	19.8	99.0	14.1
4.0	12.3	52.0	19.7	100.0	14.1
5.0	12.4	53.0	19.5	101.0	14.0
6.0	12.5	54.0	19.4	102.0	13.9
7.0	12.6	55.0	19.3	103.0	13.9
8.0	12.7	56.0	19.1	104.0	13.8
9.0	12.8	57.0	19.0	105.0	13.7
10.0	12.9	58.0	18.9	106.0	13.7
11.0	13.0	59.0	18.7	107.0	13.6
12.0	13.1	60.0	18.6	108.0	13.5
13.0	13.3	61.0	18.5	109.0	13.5
14.0	13.4	62.0	18.3	110.0	13.4
15.0	13.7	63.0	18.2	111.0	13.4
16.0	13.9	64.0	18.1	112.0	13.3
17.0	14.2	65.0	17.9	113.0	13.3
18.0	14.6	66.0	17.8	114.0	13.3
19.0	15.1	67.0	17.7	115.0	13.2
20.0	15.7	68.0	17.5	116.0	13.2
21.0	16.3	69.0	17.4	117.0	13.2
22.0	17.1	70.0	17.2	118.0	13.1
23.0	17.8	71.0	17.1	119.0	13.1
24.0	18.6	72.0	17.0	120.0	13.1
25.0	19.4	73.0	16.8	121.0	13.1
26.0	20.2	74.0	16.7	122.0	13.0
27.0	20.8	75.0	16.6	123.0	13.0
28.0	21.3	76.0	16.5	124.0	13.0
29.0	21.6	77.0	16.3	125.0	12.9
30.0	21.7	78.0	16.2	126.0	12.9
31.0	21.8	79.0	16.1	127.0	12.9
32.0	21.8	80.0	16.0	128.0	12.8
33.0	21.7	81.0	15.9	129.0	12.8
34.0	21.7	82.0	15.7	130.0	12.8
35.0	21.6	83.0	15.7	131.0	12.8
36.0	21.6	84.0	15.5	132.0	12.7
37.0	21.5	85.0	15.4	133.0	12.7
38.0	21.4	86.0	15.3	134.0	12.7
39.0	21.3	87.0	15.2	135.0	12.6
40.0	21.2	88.0	15.1	136.0	12.6
41.0	21.1	89.0	15.0	137.0	12.6
42.0	20.9	90.0	14.9	138.0	12.6
43.0	20.8	91.0	14.8	139.0	12.5
44.0	20.7	92.0	14.7	140.0	12.5
45.0	20.6	93.0	14.6	141.0	12.5
46.0	20.4	94.0	14.6	142.0	12.5
47.0	20.3	95.0	14.5	143.0	12.5
48.0	20.2	96.0	14.4	144.0	12.4

Table E.8: Select data from Calorimeter I for 30%C – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	23.5	49.0	34.8	97.0	26.9
2.0	23.9	50.0	34.5	98.0	26.8
3.0	24.1	51.0	34.3	99.0	26.7
4.0	24.3	52.0	34.0	100.0	26.6
5.0	24.4	53.0	33.8	101.0	26.6
6.0	24.7	54.0	33.5	102.0	26.5
7.0	25.0	55.0	33.3	103.0	26.5
8.0	25.6	56.0	33.1	104.0	26.4
9.0	26.5	57.0	32.8	105.0	26.3
10.0	27.7	58.0	32.6	106.0	26.3
11.0	29.4	59.0	32.4	107.0	26.2
12.0	31.6	60.0	32.2	108.0	26.2
13.0	33.7	61.0	32.0	109.0	26.2
14.0	34.8	62.0	31.8	110.0	26.1
15.0	35.5	63.0	31.6	111.0	26.1
16.0	36.1	64.0	31.4	112.0	26.0
17.0	36.8	65.0	31.2	113.0	26.0
18.0	37.4	66.0	31.0	114.0	25.9
19.0	37.9	67.0	30.8	115.0	25.9
20.0	38.4	68.0	30.6	116.0	25.8
21.0	38.8	69.0	30.4	117.0	25.8
22.0	39.0	70.0	30.2	118.0	25.7
23.0	39.3	71.0	30.0	119.0	25.7
24.0	39.5	72.0	29.9	120.0	25.6
25.0	39.6	73.0	29.7	121.0	25.6
26.0	39.7	74.0	29.5	122.0	25.5
27.0	39.7	75.0	29.4	123.0	25.5
28.0	39.7	76.0	29.2	124.0	25.5
29.0	39.7	77.0	29.1	125.0	25.4
30.0	39.6	78.0	28.9	126.0	25.4
31.0	39.5	79.0	28.8	127.0	25.4
32.0	39.4	80.0	28.7	128.0	25.4
33.0	39.3	81.0	28.6	129.0	25.3
34.0	39.1	82.0	28.5	130.0	25.3
35.0	38.8	83.0	28.4	131.0	25.3
36.0	38.6	84.0	28.2	132.0	25.3
37.0	38.3	85.0	28.1	133.0	25.3
38.0	38.0	86.0	28.0	134.0	25.2
39.0	37.7	87.0	27.9	135.0	25.2
40.0	37.4	88.0	27.8	136.0	25.2
41.0	37.2	89.0	27.7	137.0	25.2
42.0	36.9	90.0	27.6	138.0	25.2
43.0	36.6	91.0	27.5	139.0	25.2
44.0	36.3	92.0	27.4	140.0	25.1
45.0	36.0	93.0	27.3	141.0	25.1
46.0	35.7	94.0	27.2	142.0	25.1
47.0	35.4	95.0	27.1	143.0	25.1
48.0	35.1	96.0	27.0	144.0	25.0

Table E.9: Select data from Calorimeter I for 30%C – Hot

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	33.8	49.0	44.8	97.0	37.9
2.0	34.2	50.0	44.6	98.0	37.8
3.0	34.4	51.0	44.3	99.0	37.8
4.0	34.7	52.0	44.1	100.0	37.7
5.0	35.4	53.0	43.9	101.0	37.6
6.0	36.9	54.0	43.6	102.0	37.6
7.0	39.8	55.0	43.4	103.0	37.5
8.0	43.1	56.0	43.2	104.0	37.5
9.0	44.8	57.0	43.0	105.0	37.4
10.0	46.4	58.0	42.7	106.0	37.3
11.0	47.8	59.0	42.5	107.0	37.3
12.0	49.0	60.0	42.3	108.0	37.2
13.0	50.0	61.0	42.1	109.0	37.2
14.0	50.8	62.0	41.9	110.0	37.2
15.0	51.4	63.0	41.8	111.0	37.1
16.0	51.9	64.0	41.6	112.0	37.0
17.0	52.2	65.0	41.4	113.0	37.0
18.0	52.4	66.0	41.2	114.0	37.0
19.0	52.5	67.0	41.1	115.0	36.9
20.0	52.5	68.0	40.9	116.0	36.9
21.0	52.4	69.0	40.8	117.0	36.9
22.0	52.3	70.0	40.6	118.0	36.8
23.0	52.1	71.0	40.5	119.0	36.8
24.0	52.0	72.0	40.3	120.0	36.7
25.0	51.7	73.0	40.2	121.0	36.7
26.0	51.5	74.0	40.1	122.0	36.7
27.0	51.2	75.0	39.9	123.0	36.6
28.0	50.9	76.0	39.8	124.0	36.6
29.0	50.6	77.0	39.7	125.0	36.6
30.0	50.3	78.0	39.6	126.0	36.6
31.0	50.0	79.0	39.5	127.0	36.5
32.0	49.7	80.0	39.4	128.0	36.5
33.0	49.4	81.0	39.3	129.0	36.5
34.0	49.1	82.0	39.1	130.0	36.4
35.0	48.8	83.0	39.1	131.0	36.4
36.0	48.5	84.0	39.0	132.0	36.4
37.0	48.3	85.0	38.8	133.0	36.4
38.0	47.9	86.0	38.8	134.0	36.3
39.0	47.6	87.0	38.7	135.0	36.3
40.0	47.4	88.0	38.6	136.0	36.3
41.0	47.1	89.0	38.5	137.0	36.3
42.0	46.8	90.0	38.4	138.0	36.3
43.0	46.5	91.0	38.3	139.0	36.2
44.0	46.2	92.0	38.3	140.0	36.2
45.0	45.9	93.0	38.2	141.0	36.2
46.0	45.6	94.0	38.1	142.0	36.2
47.0	45.3	95.0	38.0	143.0	36.1
48.0	45.1	96.0	37.9	144.0	36.1

Table E.10: Select data from Calorimeter II for 30%C – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	22.3	97.0	17.4
2.0	11.7	50.0	22.2	98.0	17.4
3.0	11.7	51.0	22.1	99.0	17.2
4.0	11.7	52.0	22.2	100.0	17.0
5.0	11.9	53.0	22.1	101.0	17.0
6.0	11.9	54.0	22.0	102.0	16.9
7.0	12.1	55.0	21.9	103.0	16.7
8.0	12.1	56.0	21.9	104.0	16.8
9.0	12.3	57.0	21.7	105.0	16.9
10.0	12.3	58.0	21.5	106.0	16.5
11.0	12.7	59.0	21.6	107.0	16.5
12.0	12.7	60.0	21.5	108.0	16.5
13.0	13.0	61.0	21.3	109.0	16.2
14.0	13.0	62.0	21.3	110.0	16.2
15.0	13.3	63.0	21.1	111.0	16.3
16.0	13.5	64.0	21.2	112.0	16.2
17.0	13.9	65.0	20.9	113.0	16.0
18.0	14.5	66.0	20.9	114.0	15.9
19.0	14.8	67.0	20.9	115.0	15.9
20.0	15.6	68.0	20.6	116.0	15.8
21.0	16.1	69.0	20.5	117.0	15.9
22.0	16.7	70.0	20.4	118.0	15.7
23.0	17.8	71.0	20.2	119.0	15.7
24.0	18.6	72.0	20.1	120.0	15.7
25.0	19.3	73.0	20.1	121.0	15.6
26.0	20.3	74.0	19.9	122.0	15.4
27.0	21.0	75.0	19.9	123.0	15.4
28.0	21.4	76.0	19.6	124.0	15.3
29.0	22.1	77.0	19.6	125.0	15.3
30.0	22.3	78.0	19.6	126.0	15.1
31.0	22.3	79.0	19.3	127.0	15.1
32.0	22.6	80.0	19.3	128.0	15.1
33.0	22.6	81.0	19.1	129.0	15.0
34.0	22.6	82.0	18.9	130.0	15.0
35.0	22.6	83.0	18.9	131.0	14.8
36.0	22.7	84.0	18.8	132.0	14.8
37.0	22.6	85.0	18.6	133.0	14.8
38.0	22.6	86.0	18.6	134.0	14.7
39.0	22.7	87.0	18.4	135.0	14.7
40.0	22.5	88.0	18.2	136.0	14.7
41.0	22.7	89.0	18.2	137.0	14.6
42.0	22.6	90.0	18.2	138.0	14.6
43.0	22.5	91.0	18.0	139.0	14.5
44.0	22.5	92.0	17.8	140.0	14.5
45.0	22.6	93.0	17.9	141.0	14.3
46.0	22.4	94.0	17.7	142.0	14.2
47.0	22.4	95.0	17.6	143.0	14.4
48.0	22.3	96.0	17.4	144.0	14.2

Table E.11: Select data from Calorimeter II for 30%C – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	36.9	97.0	28.5
2.0	24.0	50.0	36.6	98.0	28.2
3.0	24.2	51.0	36.4	99.0	28.2
4.0	24.3	52.0	36.2	100.0	28.0
5.0	24.5	53.0	36.0	101.0	28.0
6.0	24.7	54.0	35.8	102.0	28.0
7.0	25.0	55.0	35.5	103.0	27.8
8.0	25.7	56.0	35.2	104.0	27.7
9.0	26.6	57.0	35.2	105.0	27.5
10.0	27.7	58.0	34.9	106.0	27.5
11.0	29.4	59.0	34.6	107.0	27.5
12.0	31.6	60.0	34.4	108.0	27.3
13.0	33.4	61.0	34.2	109.0	27.3
14.0	34.3	62.0	34.0	110.0	27.3
15.0	35.1	63.0	34.0	111.0	27.1
16.0	35.7	64.0	33.5	112.0	26.8
17.0	36.2	65.0	33.5	113.0	26.8
18.0	36.9	66.0	33.3	114.0	26.8
19.0	37.3	67.0	32.9	115.0	26.8
20.0	37.9	68.0	32.7	116.0	26.6
21.0	38.2	69.0	32.5	117.0	26.6
22.0	38.6	70.0	32.3	118.0	26.6
23.0	39.0	71.0	32.3	119.0	26.4
24.0	39.2	72.0	32.1	120.0	26.4
25.0	39.4	73.0	31.9	121.0	26.3
26.0	39.4	74.0	31.7	122.0	26.3
27.0	39.7	75.0	31.5	123.0	26.1
28.0	39.9	76.0	31.2	124.0	26.1
29.0	39.9	77.0	31.2	125.0	26.1
30.0	39.9	78.0	30.9	126.0	25.9
31.0	39.9	79.0	30.9	127.0	25.9
32.0	39.9	80.0	30.7	128.0	25.8
33.0	39.9	81.0	30.5	129.0	25.8
34.0	39.7	82.0	30.5	130.0	25.7
35.0	39.6	83.0	30.3	131.0	25.7
36.0	39.6	84.0	30.1	132.0	25.5
37.0	39.3	85.0	29.9	133.0	25.5
38.0	39.2	86.0	29.9	134.0	25.5
39.0	39.0	87.0	29.7	135.0	25.5
40.0	38.8	88.0	29.7	136.0	25.3
41.0	38.6	89.0	29.3	137.0	25.3
42.0	38.4	90.0	29.3	138.0	25.3
43.0	38.3	91.0	29.3	139.0	25.1
44.0	37.9	92.0	29.1	140.0	25.1
45.0	37.7	93.0	28.9	141.0	25.1
46.0	37.5	94.0	28.9	142.0	25.1
47.0	37.3	95.0	28.7	143.0	25.1
48.0	37.1	96.0	28.5	144.0	24.9

Table E.12: Select data from Calorimeter II for 30%C – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	48.9	97.0	40.6
2.0	34.1	50.0	48.8	98.0	40.5
3.0	34.8	51.0	48.2	99.0	40.4
4.0	35.3	52.0	48.0	100.0	40.4
5.0	36.1	53.0	47.8	101.0	40.3
6.0	37.8	54.0	47.5	102.0	40.0
7.0	41.1	55.0	47.4	103.0	40.0
8.0	44.3	56.0	47.3	104.0	40.0
9.0	45.9	57.0	47.1	105.0	39.7
10.0	47.5	58.0	46.8	106.0	39.7
11.0	49.0	59.0	46.4	107.0	39.6
12.0	50.3	60.0	46.3	108.0	39.6
13.0	51.3	61.0	46.2	109.0	39.4
14.0	52.1	62.0	45.9	110.0	39.3
15.0	52.9	63.0	45.6	111.0	39.3
16.0	53.6	64.0	45.4	112.0	39.2
17.0	53.9	65.0	45.2	113.0	39.3
18.0	54.2	66.0	45.2	114.0	39.0
19.0	54.5	67.0	44.9	115.0	39.0
20.0	54.7	68.0	44.7	116.0	38.8
21.0	54.7	69.0	44.5	117.0	38.8
22.0	54.7	70.0	44.4	118.0	38.9
23.0	54.6	71.0	44.1	119.0	38.8
24.0	54.4	72.0	44.1	120.0	38.7
25.0	54.3	73.0	43.8	121.0	38.6
26.0	54.2	74.0	43.7	122.0	38.6
27.0	54.0	75.0	43.5	123.0	38.4
28.0	54.0	76.0	43.3	124.0	38.5
29.0	53.6	77.0	43.1	125.0	38.2
30.0	53.4	78.0	43.1	126.0	38.2
31.0	53.1	79.0	42.8	127.0	38.2
32.0	53.1	80.0	42.9	128.0	38.1
33.0	52.7	81.0	42.6	129.0	38.1
34.0	52.5	82.0	42.5	130.0	38.0
35.0	52.3	83.0	42.3	131.0	38.0
36.0	52.1	84.0	42.1	132.0	38.0
37.0	51.7	85.0	42.1	133.0	37.8
38.0	51.8	86.0	42.0	134.0	37.9
39.0	51.4	87.0	41.8	135.0	37.9
40.0	51.1	88.0	41.8	136.0	37.7
41.0	50.9	89.0	41.6	137.0	37.8
42.0	50.6	90.0	41.4	138.0	37.6
43.0	50.2	91.0	41.4	139.0	37.6
44.0	50.1	92.0	41.1	140.0	37.6
45.0	49.7	93.0	41.0	141.0	37.6
46.0	49.6	94.0	41.0	142.0	37.6
47.0	49.4	95.0	40.8	143.0	37.5
48.0	49.2	96.0	40.8	144.0	37.4

Table E.13: Select data from Calorimeter I for 30%F – Cold

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	11.5	49.0	19.3	97.0	13.1
2.0	11.6	50.0	19.2	98.0	13.0
3.0	11.8	51.0	19.0	99.0	13.0
4.0	12.0	52.0	18.7	100.0	12.9
5.0	12.1	53.0	18.6	101.0	12.8
6.0	12.2	54.0	18.4	102.0	12.8
7.0	12.4	55.0	18.2	103.0	12.7
8.0	12.6	56.0	18.0	104.0	12.7
9.0	12.8	57.0	17.8	105.0	12.6
10.0	13.0	58.0	17.7	106.0	12.5
11.0	13.4	59.0	17.5	107.0	12.5
12.0	13.8	60.0	17.3	108.0	12.4
13.0	14.2	61.0	17.2	109.0	12.4
14.0	14.8	62.0	17.0	110.0	12.3
15.0	15.4	63.0	16.8	111.0	12.3
16.0	16.1	64.0	16.7	112.0	12.2
17.0	16.8	65.0	16.6	113.0	12.2
18.0	17.6	66.0	16.4	114.0	12.2
19.0	18.4	67.0	16.2	115.0	12.1
20.0	19.2	68.0	16.1	116.0	12.1
21.0	19.9	69.0	16.0	117.0	12.0
22.0	20.6	70.0	15.8	118.0	12.0
23.0	21.1	71.0	15.7	119.0	12.0
24.0	21.6	72.0	15.6	120.0	11.9
25.0	21.9	73.0	15.4	121.0	11.9
26.0	22.1	74.0	15.3	122.0	11.8
27.0	22.3	75.0	15.2	123.0	11.8
28.0	22.3	76.0	15.1	124.0	11.8
29.0	22.3	77.0	14.9	125.0	11.7
30.0	22.3	78.0	14.8	126.0	11.7
31.0	22.2	79.0	14.7	127.0	11.7
32.0	22.1	80.0	14.6	128.0	11.6
33.0	22.0	81.0	14.5	129.0	11.6
34.0	21.8	82.0	14.4	130.0	11.6
35.0	21.7	83.0	14.3	131.0	11.6
36.0	21.6	84.0	14.2	132.0	11.5
37.0	21.4	85.0	14.1	133.0	11.5
38.0	21.3	86.0	14.0	134.0	11.5
39.0	21.1	87.0	13.9	135.0	11.5
40.0	20.9	88.0	13.8	136.0	11.4
41.0	20.8	89.0	13.7	137.0	11.4
42.0	20.6	90.0	13.6	138.0	11.4
43.0	20.4	91.0	13.5	139.0	11.4
44.0	20.2	92.0	13.4	140.0	11.4
45.0	20.0	93.0	13.4	141.0	11.4
46.0	19.9	94.0	13.3	142.0	11.4
47.0	19.7	95.0	13.2	143.0	11.4
48.0	19.5	96.0	13.2	144.0	11.4

Table E.14: Select data from Calorimeter I for 30%F – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	25.0	49.0	33.2	97.0	27.3
2.0	25.1	50.0	33.0	98.0	27.2
3.0	25.2	51.0	32.8	99.0	27.1
4.0	25.4	52.0	32.6	100.0	27.0
5.0	25.7	53.0	32.5	101.0	27.0
6.0	26.3	54.0	32.3	102.0	26.9
7.0	27.0	55.0	32.2	103.0	26.8
8.0	27.8	56.0	32.0	104.0	26.8
9.0	28.7	57.0	31.9	105.0	26.7
10.0	29.6	58.0	31.7	106.0	26.7
11.0	30.5	59.0	31.6	107.0	26.6
12.0	31.3	60.0	31.4	108.0	26.6
13.0	32.3	61.0	31.3	109.0	26.5
14.0	33.5	62.0	31.1	110.0	26.5
15.0	34.8	63.0	31.0	111.0	26.4
16.0	35.6	64.0	30.8	112.0	26.3
17.0	36.0	65.0	30.7	113.0	26.3
18.0	36.3	66.0	30.6	114.0	26.2
19.0	36.4	67.0	30.4	115.0	26.1
20.0	36.6	68.0	30.3	116.0	26.1
21.0	36.7	69.0	30.2	117.0	26.0
22.0	36.7	70.0	30.0	118.0	26.0
23.0	36.8	71.0	29.9	119.0	25.9
24.0	36.8	72.0	29.8	120.0	25.8
25.0	36.7	73.0	29.6	121.0	25.8
26.0	36.7	74.0	29.5	122.0	25.7
27.0	36.6	75.0	29.4	123.0	25.7
28.0	36.6	76.0	29.3	124.0	25.6
29.0	36.4	77.0	29.2	125.0	25.6
30.0	36.3	78.0	29.1	126.0	25.6
31.0	36.2	79.0	29.0	127.0	25.5
32.0	36.1	80.0	28.9	128.0	25.5
33.0	36.0	81.0	28.8	129.0	25.5
34.0	35.8	82.0	28.7	130.0	25.4
35.0	35.7	83.0	28.6	131.0	25.4
36.0	35.5	84.0	28.5	132.0	25.4
37.0	35.3	85.0	28.4	133.0	25.3
38.0	35.2	86.0	28.3	134.0	25.3
39.0	35.0	87.0	28.2	135.0	25.2
40.0	34.8	88.0	28.1	136.0	25.2
41.0	34.6	89.0	28.0	137.0	25.2
42.0	34.4	90.0	27.9	138.0	25.1
43.0	34.3	91.0	27.8	139.0	25.1
44.0	34.1	92.0	27.7	140.0	25.1
45.0	33.9	93.0	27.6	141.0	25.0
46.0	33.7	94.0	27.5	142.0	25.0
47.0	33.5	95.0	27.5	143.0	25.0
48.0	33.3	96.0	27.4	144.0	24.9

Table E.15: Select data from Calorimeter I for 30%F – Hot

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	33.2	49.0	43.0	97.0	37.5
2.0	33.5	50.0	42.8	98.0	37.4
3.0	33.8	51.0	42.6	99.0	37.3
4.0	34.8	52.0	42.4	100.0	37.3
5.0	36.7	53.0	42.2	101.0	37.2
6.0	39.7	54.0	42.1	102.0	37.2
7.0	43.8	55.0	41.9	103.0	37.1
8.0	45.8	56.0	41.7	104.0	37.0
9.0	47.4	57.0	41.5	105.0	37.0
10.0	48.6	58.0	41.3	106.0	36.9
11.0	49.5	59.0	41.2	107.0	36.9
12.0	50.2	60.0	41.0	108.0	36.8
13.0	50.7	61.0	40.9	109.0	36.8
14.0	51.0	62.0	40.7	110.0	36.7
15.0	51.1	63.0	40.5	111.0	36.7
16.0	51.1	64.0	40.4	112.0	36.7
17.0	51.0	65.0	40.3	113.0	36.6
18.0	50.9	66.0	40.1	114.0	36.6
19.0	50.7	67.0	40.0	115.0	36.6
20.0	50.5	68.0	39.9	116.0	36.5
21.0	50.2	69.0	39.8	117.0	36.5
22.0	50.0	70.0	39.7	118.0	36.5
23.0	49.7	71.0	39.6	119.0	36.4
24.0	49.4	72.0	39.5	120.0	36.4
25.0	49.1	73.0	39.3	121.0	36.4
26.0	48.8	74.0	39.2	122.0	36.3
27.0	48.6	75.0	39.1	123.0	36.3
28.0	48.3	76.0	39.0	124.0	36.3
29.0	48.0	77.0	39.0	125.0	36.3
30.0	47.7	78.0	38.8	126.0	36.2
31.0	47.5	79.0	38.8	127.0	36.2
32.0	47.2	80.0	38.7	128.0	36.2
33.0	46.9	81.0	38.6	129.0	36.2
34.0	46.6	82.0	38.5	130.0	36.1
35.0	46.4	83.0	38.4	131.0	36.1
36.0	46.1	84.0	38.4	132.0	36.1
37.0	45.8	85.0	38.3	133.0	36.1
38.0	45.6	86.0	38.2	134.0	36.1
39.0	45.3	87.0	38.1	135.0	36.0
40.0	45.1	88.0	38.1	136.0	36.0
41.0	44.8	89.0	38.0	137.0	36.0
42.0	44.6	90.0	37.9	138.0	36.0
43.0	44.3	91.0	37.8	139.0	36.0
44.0	44.1	92.0	37.8	140.0	35.9
45.0	43.9	93.0	37.7	141.0	35.9
46.0	43.6	94.0	37.7	142.0	35.9
47.0	43.4	95.0	37.6	143.0	35.9
48.0	43.2	96.0	37.6	144.0	35.9

Table E.16: Select data from Calorimeter II for 30%F – Cold

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
-	-	49.0	21.5	97.0	15.5
2.0	13.0	50.0	21.3	98.0	15.5
3.0	12.9	51.0	21.2	99.0	15.5
4.0	12.9	52.0	21.0	100.0	15.1
5.0	13.0	53.0	20.8	101.0	15.3
6.0	13.2	54.0	20.8	102.0	15.1
7.0	13.2	55.0	20.5	103.0	14.9
8.0	13.4	56.0	20.4	104.0	14.9
9.0	13.6	57.0	20.3	105.0	14.9
10.0	13.9	58.0	20.0	106.0	14.9
11.0	14.3	59.0	20.1	107.0	14.6
12.0	14.7	60.0	20.0	108.0	14.5
13.0	15.2	61.0	19.8	109.0	14.5
14.0	15.9	62.0	19.7	110.0	14.3
15.0	16.3	63.0	19.5	111.0	14.5
16.0	17.0	64.0	19.2	112.0	14.3
17.0	17.8	65.0	19.2	113.0	14.3
18.0	18.8	66.0	19.2	114.0	14.1
19.0	19.5	67.0	19.0	115.0	14.0
20.0	20.4	68.0	18.8	116.0	14.0
21.0	21.1	69.0	18.8	117.0	13.9
22.0	21.8	70.0	18.5	118.0	13.9
23.0	22.3	71.0	18.4	119.0	13.8
24.0	22.6	72.0	18.4	120.0	3.1
25.0	23.0	73.0	18.1	121.0	3.1
26.0	23.3	74.0	18.1	122.0	3.2
27.0	23.3	75.0	17.9	123.0	3.0
28.0	23.3	76.0	17.7	124.0	3.0
29.0	23.4	77.0	17.6	125.0	2.9
30.0	23.4	78.0	17.6	126.0	2.8
31.0	23.4	79.0	17.4	127.0	2.8
32.0	23.4	80.0	17.4	128.0	2.6
33.0	23.2	81.0	17.3	129.0	2.6
34.0	23.1	82.0	17.1	130.0	2.5
35.0	22.9	83.0	17.1	131.0	2.7
36.0	23.0	84.0	17.0	132.0	2.4
37.0	22.9	85.0	16.7	133.0	2.7
38.0	22.8	86.0	16.7	134.0	2.4
39.0	22.6	87.0	16.5	135.0	2.4
40.0	22.6	88.0	16.3	136.0	2.4
41.0	22.6	89.0	16.3	137.0	2.3
42.0	22.3	90.0	16.2	138.0	2.4
43.0	22.3	91.0	16.1	139.0	2.2
44.0	22.1	92.0	16.0	140.0	2.2
45.0	21.9	93.0	15.9	141.0	2.2
46.0	21.8	94.0	15.8	142.0	2.2
47.0	21.6	95.0	15.7	143.0	2.1
48.0	21.6	96.0	15.5	144.0	2.0

Table E.17: Select data from Calorimeter II for 30%F – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	35.5	97.0	29.8
2.0	24.6	50.0	35.3	98.0	29.8
3.0	24.9	51.0	35.2	99.0	29.5
4.0	25.1	52.0	35.1	100.0	29.6
5.0	25.6	53.0	34.9	101.0	29.6
6.0	26.2	54.0	34.7	102.0	29.4
7.0	26.8	55.0	34.7	103.0	29.3
8.0	27.6	56.0	34.5	104.0	29.3
9.0	28.4	57.0	34.4	105.0	29.4
10.0	29.3	58.0	34.3	106.0	29.2
11.0	30.4	59.0	34.0	107.0	29.0
12.0	31.2	60.0	34.0	108.0	29.0
13.0	32.1	61.0	33.9	109.0	29.0
14.0	33.3	62.0	33.7	110.0	28.9
15.0	34.8	63.0	33.7	111.0	28.7
16.0	35.7	64.0	33.4	112.0	28.8
17.0	36.1	65.0	33.3	113.0	28.6
18.0	36.5	66.0	33.3	114.0	28.5
19.0	36.7	67.0	33.2	115.0	28.5
20.0	36.8	68.0	33.0	116.0	28.4
21.0	37.0	69.0	33.0	117.0	28.4
22.0	37.2	70.0	32.7	118.0	28.4
23.0	37.4	71.0	32.6	119.0	28.3
24.0	37.4	72.0	32.4	120.0	28.3
25.0	37.4	73.0	32.2	121.0	28.0
26.0	37.6	74.0	32.1	122.0	28.2
27.0	37.5	75.0	32.1	123.0	28.0
28.0	37.5	76.0	31.9	124.0	28.1
29.0	37.6	77.0	31.8	125.0	27.9
30.0	37.7	78.0	31.7	126.0	27.8
31.0	37.6	79.0	31.7	127.0	27.9
32.0	37.3	80.0	31.4	128.0	27.7
33.0	37.4	81.0	31.5	129.0	27.8
34.0	37.2	82.0	31.3	130.0	27.8
35.0	37.2	83.0	31.2	131.0	27.5
36.0	37.1	84.0	31.2	132.0	27.4
37.0	37.0	85.0	31.0	133.0	27.6
38.0	36.8	86.0	30.9	134.0	27.4
39.0	36.7	87.0	30.8	135.0	27.4
40.0	36.6	88.0	30.7	136.0	27.3
41.0	36.6	89.0	30.5	137.0	27.3
42.0	36.5	90.0	30.6	138.0	27.3
43.0	36.3	91.0	30.4	139.0	27.3
44.0	36.1	92.0	30.4	140.0	27.1
45.0	36.0	93.0	30.1	141.0	27.1
46.0	36.0	94.0	30.1	142.0	27.1
47.0	35.8	95.0	30.1	143.0	27.1
48.0	35.5	96.0	29.9	144.0	27.0

Table E.18: Select data from Calorimeter I for 50% Slag – Cold

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	9.1	49.0	18.1	97.0	14.0
2.0	9.3	50.0	18.1	98.0	13.9
3.0	9.6	51.0	18.0	99.0	13.9
4.0	9.8	52.0	17.9	100.0	13.8
5.0	10.0	53.0	17.8	101.0	13.8
6.0	10.2	54.0	17.7	102.0	13.7
7.0	10.4	55.0	17.6	103.0	13.6
8.0	10.6	56.0	17.5	104.0	13.6
9.0	10.8	57.0	17.4	105.0	13.5
10.0	11.1	58.0	17.3	106.0	13.5
11.0	11.3	59.0	17.2	107.0	13.4
12.0	11.6	60.0	17.1	108.0	13.4
13.0	12.0	61.0	17.0	109.0	13.3
14.0	12.3	62.0	16.9	110.0	13.3
15.0	12.8	63.0	16.8	111.0	13.2
16.0	13.3	64.0	16.7	112.0	13.2
17.0	13.8	65.0	16.6	113.0	13.1
18.0	14.4	66.0	16.5	114.0	13.1
19.0	15.0	67.0	16.4	115.0	13.0
20.0	15.6	68.0	16.3	116.0	13.0
21.0	16.2	69.0	16.2	117.0	12.9
22.0	16.8	70.0	16.2	118.0	12.9
23.0	17.4	71.0	16.1	119.0	12.8
24.0	17.9	72.0	16.0	120.0	12.8
25.0	18.3	73.0	15.9	121.0	12.8
26.0	18.5	74.0	15.8	122.0	12.7
27.0	18.7	75.0	15.7	123.0	12.7
28.0	18.9	76.0	15.6	124.0	12.7
29.0	19.0	77.0	15.5	125.0	12.7
30.0	19.0	78.0	15.4	126.0	12.6
31.0	19.1	79.0	15.4	127.0	12.6
32.0	19.1	80.0	15.3	128.0	12.6
33.0	19.1	81.0	15.2	129.0	12.5
34.0	19.1	82.0	15.1	130.0	12.5
35.0	19.0	83.0	15.0	131.0	12.5
36.0	19.0	84.0	14.9	132.0	12.5
37.0	19.0	85.0	14.9	133.0	12.4
38.0	19.0	86.0	14.8	134.0	12.4
39.0	18.9	87.0	14.7	135.0	12.4
40.0	18.8	88.0	14.6	136.0	12.4
41.0	18.7	89.0	14.6	137.0	12.3
42.0	18.7	90.0	14.5	138.0	12.3
43.0	18.6	91.0	14.4	139.0	12.3
44.0	18.5	92.0	14.3	140.0	12.3
45.0	18.4	93.0	14.3	141.0	12.2
46.0	18.4	94.0	14.2	142.0	12.2
47.0	18.3	95.0	14.1	143.0	12.2
48.0	18.2	96.0	14.1	144.0	12.2

Table E.19: Select data from Calorimeter I for 50% Slag – Room

Concrete Age (Hours)	Concrete Temperature (°C)	Concrete Age (Hours)	Concrete Temperature (°C)	Concrete Age (Hours)	Concrete Temperature (°C)
-	-	49.0	35.3	97.0	27.9
2.0	25.5	50.0	35.1	98.0	27.8
3.0	25.6	51.0	34.9	99.0	27.7
4.0	25.7	52.0	34.7	100.0	27.6
5.0	26.0	53.0	34.5	101.0	27.5
6.0	26.5	54.0	34.4	102.0	27.4
7.0	27.2	55.0	34.2	103.0	27.3
8.0	28.0	56.0	34.0	104.0	27.3
9.0	28.9	57.0	33.8	105.0	27.2
10.0	29.9	58.0	33.6	106.0	27.1
11.0	31.2	59.0	33.4	107.0	27.0
12.0	32.8	60.0	33.3	108.0	26.9
13.0	34.0	61.0	33.1	109.0	26.8
14.0	34.6	62.0	32.9	110.0	26.8
15.0	35.1	63.0	32.7	111.0	26.7
16.0	35.6	64.0	32.5	112.0	26.6
17.0	36.0	65.0	32.3	113.0	26.5
18.0	36.3	66.0	32.2	114.0	26.5
19.0	36.6	67.0	32.0	115.0	26.4
20.0	36.9	68.0	31.8	116.0	26.3
21.0	37.1	69.0	31.6	117.0	26.3
22.0	37.3	70.0	31.5	118.0	26.2
23.0	37.5	71.0	31.3	119.0	26.1
24.0	37.6	72.0	31.2	120.0	26.1
25.0	37.7	73.0	31.0	121.0	26.0
26.0	37.8	74.0	30.8	122.0	26.0
27.0	37.8	75.0	30.7	123.0	25.9
28.0	37.9	76.0	30.5	124.0	25.8
29.0	37.9	77.0	30.4	125.0	25.8
30.0	37.9	78.0	30.2	126.0	25.7
31.0	37.8	79.0	30.1	127.0	25.7
32.0	37.8	80.0	30.0	128.0	25.6
33.0	37.7	81.0	29.8	129.0	25.6
34.0	37.6	82.0	29.7	130.0	25.6
35.0	37.5	83.0	29.5	131.0	25.5
36.0	37.4	84.0	29.4	132.0	25.4
37.0	37.3	85.0	29.3	133.0	25.4
38.0	37.1	86.0	29.2	134.0	25.3
39.0	37.0	87.0	29.0	135.0	25.3
40.0	36.8	88.0	28.9	136.0	25.3
41.0	36.7	89.0	28.8	137.0	25.2
42.0	36.5	90.0	28.7	138.0	25.2
43.0	36.3	91.0	28.5	139.0	25.1
44.0	36.1	92.0	28.4	140.0	25.1
45.0	36.0	93.0	28.3	141.0	25.0
46.0	35.8	94.0	28.2	142.0	25.0
47.0	35.6	95.0	28.1	143.0	25.0
48.0	35.4	96.0	28.0	144.0	24.9

Table E.20: Select data from Calorimeter I for 50% Slag – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	33.7	49.0	45.7	97.0	38.3
2.0	33.8	50.0	45.4	98.0	38.3
3.0	34.0	51.0	45.2	99.0	38.2
4.0	34.4	52.0	44.9	100.0	38.1
5.0	35.4	53.0	44.7	101.0	38.1
6.0	37.2	54.0	44.5	102.0	38.0
7.0	40.4	55.0	44.2	103.0	37.9
8.0	42.6	56.0	44.0	104.0	37.8
9.0	44.1	57.0	43.8	105.0	37.8
10.0	45.5	58.0	43.6	106.0	37.7
11.0	46.7	59.0	43.4	107.0	37.6
12.0	47.8	60.0	43.2	108.0	37.6
13.0	48.7	61.0	43.0	109.0	37.5
14.0	49.5	62.0	42.8	110.0	37.5
15.0	50.2	63.0	42.6	111.0	37.4
16.0	50.7	64.0	42.4	112.0	37.3
17.0	51.2	65.0	42.2	113.0	37.3
18.0	51.5	66.0	42.1	114.0	37.3
19.0	51.7	67.0	41.9	115.0	37.2
20.0	51.9	68.0	41.7	116.0	37.2
21.0	52.0	69.0	41.6	117.0	37.1
22.0	52.0	70.0	41.4	118.0	37.1
23.0	52.0	71.0	41.3	119.0	37.0
24.0	52.0	72.0	41.1	120.0	37.0
25.0	51.8	73.0	41.0	121.0	36.9
26.0	51.7	74.0	40.8	122.0	36.9
27.0	51.6	75.0	40.7	123.0	36.8
28.0	51.4	76.0	40.6	124.0	36.8
29.0	51.2	77.0	40.4	125.0	36.7
30.0	51.0	78.0	40.3	126.0	36.7
31.0	50.8	79.0	40.2	127.0	36.7
32.0	50.6	80.0	40.1	128.0	36.6
33.0	50.3	81.0	39.9	129.0	36.6
34.0	50.1	82.0	39.8	130.0	36.6
35.0	49.8	83.0	39.7	131.0	36.6
36.0	49.5	84.0	39.6	132.0	36.5
37.0	49.2	85.0	39.5	133.0	36.5
38.0	48.9	86.0	39.4	134.0	36.5
39.0	48.6	87.0	39.3	135.0	36.4
40.0	48.3	88.0	39.2	136.0	36.4
41.0	48.0	89.0	39.1	137.0	36.4
42.0	47.7	90.0	39.0	138.0	36.3
43.0	47.4	91.0	38.9	139.0	36.3
44.0	47.1	92.0	38.8	140.0	36.3
45.0	46.8	93.0	38.7	141.0	36.3
46.0	46.5	94.0	38.6	142.0	36.3
47.0	46.2	95.0	38.5	143.0	36.2
48.0	45.9	96.0	38.4	144.0	36.2

Table E.21: Select data from Calorimeter II for 50% Slag – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	20.2	97.0	16.9
2.0	9.5	50.0	20.2	98.0	16.7
3.0	9.6	51.0	20.1	99.0	16.7
4.0	9.8	52.0	20.0	100.0	16.3
5.0	9.8	53.0	20.0	101.0	16.5
6.0	10.0	54.0	20.0	102.0	16.4
7.0	10.2	55.0	20.0	103.0	16.4
8.0	10.5	56.0	20.0	104.0	16.4
9.0	10.6	57.0	20.0	105.0	16.1
10.0	11.0	58.0	19.6	106.0	15.9
11.0	11.2	59.0	19.8	107.0	16.0
12.0	11.5	60.0	19.7	108.0	15.9
13.0	11.8	61.0	19.7	109.0	15.9
14.0	12.2	62.0	19.5	110.0	15.7
15.0	12.8	63.0	19.4	111.0	15.7
16.0	13.1	64.0	19.2	112.0	15.5
17.0	13.7	65.0	19.3	113.0	15.6
18.0	14.3	66.0	19.3	114.0	15.5
19.0	15.1	67.0	19.3	115.0	15.5
20.0	15.7	68.0	19.1	116.0	15.5
21.0	16.5	69.0	19.1	117.0	15.3
22.0	17.0	70.0	18.7	118.0	15.1
23.0	17.8	71.0	19.0	119.0	15.1
24.0	18.3	72.0	18.8	120.0	5.1
25.0	18.8	73.0	18.8	121.0	5.0
26.0	19.0	74.0	18.6	122.0	5.1
27.0	19.4	75.0	18.6	123.0	5.0
28.0	19.5	76.0	18.2	124.0	4.6
29.0	19.6	77.0	18.4	125.0	4.8
30.0	19.9	78.0	18.3	126.0	4.9
31.0	20.0	79.0	18.3	127.0	4.8
32.0	20.0	80.0	18.3	128.0	4.8
33.0	20.1	81.0	18.1	129.0	4.8
34.0	20.1	82.0	17.8	130.0	4.3
35.0	20.2	83.0	17.9	131.0	4.5
36.0	20.1	84.0	17.9	132.0	4.5
37.0	20.5	85.0	17.9	133.0	4.5
38.0	20.2	86.0	17.7	134.0	4.5
39.0	20.4	87.0	17.7	135.0	4.5
40.0	20.2	88.0	17.4	136.0	4.1
41.0	20.4	89.0	17.5	137.0	4.2
42.0	20.3	90.0	17.4	138.0	4.2
43.0	20.4	91.0	17.2	139.0	4.1
44.0	20.4	92.0	17.2	140.0	4.2
45.0	20.2	93.0	17.2	141.0	4.2
46.0	20.0	94.0	16.8	142.0	3.8
47.0	20.3	95.0	16.9	143.0	4.1
48.0	20.2	96.0	16.9	144.0	4.0

Table E.22: Select data from Calorimeter II for 50% Slag – Room

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
-	-	49.0	38.7	97.0	31.4
2.0	25.8	50.0	38.6	98.0	31.3
3.0	26.0	51.0	38.5	99.0	31.1
4.0	26.2	52.0	38.3	100.0	31.1
5.0	26.5	53.0	38.2	101.0	30.9
6.0	27.1	54.0	37.9	102.0	30.7
7.0	28.0	55.0	37.7	103.0	30.7
8.0	28.9	56.0	37.7	104.0	30.7
9.0	29.8	57.0	37.5	105.0	30.6
10.0	30.9	58.0	37.3	106.0	30.4
11.0	32.3	59.0	37.1	107.0	30.4
12.0	34.2	60.0	37.0	108.0	30.4
13.0	34.9	61.0	36.7	109.0	30.3
14.0	35.8	62.0	36.7	110.0	30.1
15.0	36.1	63.0	36.5	111.0	30.1
16.0	36.6	64.0	36.3	112.0	30.1
17.0	37.3	65.0	36.0	113.0	29.8
18.0	37.6	66.0	36.0	114.0	29.8
19.0	38.1	67.0	35.7	115.0	29.6
20.0	38.3	68.0	35.6	116.0	29.5
21.0	38.5	69.0	35.4	117.0	29.5
22.0	38.9	70.0	35.3	118.0	29.5
23.0	39.1	71.0	35.1	119.0	29.3
24.0	39.3	72.0	34.8	120.0	29.3
25.0	39.6	73.0	34.8	121.0	29.1
26.0	39.6	74.0	34.6	122.0	29.2
27.0	39.9	75.0	34.4	123.0	29.1
28.0	40.0	76.0	34.2	124.0	28.9
29.0	40.2	77.0	34.2	125.0	28.6
30.0	40.2	78.0	34.0	126.0	28.5
31.0	40.3	79.0	33.8	127.0	28.4
32.0	40.3	80.0	33.6	128.0	28.5
33.0	40.2	81.0	33.6	129.0	28.3
34.0	40.1	82.0	33.3	130.0	28.2
35.0	40.3	83.0	33.1	131.0	28.2
36.0	40.2	84.0	33.1	132.0	28.1
37.0	40.2	85.0	32.9	133.0	28.1
38.0	39.9	86.0	32.7	134.0	28.0
39.0	39.9	87.0	32.7	135.0	28.0
40.0	39.9	88.0	32.5	136.0	28.0
41.0	39.9	89.0	32.3	137.0	28.0
42.0	39.7	90.0	32.1	138.0	27.8
43.0	39.5	91.0	32.1	139.0	27.8
44.0	39.5	92.0	32.0	140.0	28.0
45.0	39.3	93.0	32.0	141.0	27.9
46.0	39.2	94.0	31.6	142.0	27.7
47.0	39.0	95.0	31.6	143.0	27.6
48.0	38.8	96.0	31.4	144.0	27.8

Table E.23: Select data from Calorimeter I for w/c 0.36 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	25.4	97.0	13.6
2.0	11.0	50.0	25.0	98.0	13.5
3.0	11.3	51.0	24.7	99.0	13.4
4.0	11.6	52.0	24.3	100.0	13.3
5.0	11.9	53.0	24.0	101.0	13.2
6.0	12.3	54.0	23.6	102.0	13.1
7.0	12.7	55.0	23.3	103.0	13.0
8.0	13.3	56.0	22.9	104.0	12.9
9.0	14.1	57.0	22.6	105.0	12.8
10.0	15.1	58.0	22.3	106.0	12.7
11.0	16.3	59.0	21.9	107.0	12.6
12.0	17.7	60.0	21.6	108.0	12.5
13.0	19.4	61.0	21.3	109.0	12.4
14.0	21.4	62.0	20.9	110.0	12.3
15.0	23.6	63.0	20.6	111.0	12.2
16.0	25.9	64.0	20.3	112.0	12.2
17.0	27.8	65.0	20.0	113.0	12.1
18.0	28.9	66.0	19.7	114.0	12.1
19.0	29.6	67.0	19.5	115.0	12.0
20.0	30.0	68.0	19.2	116.0	11.9
21.0	30.2	69.0	18.9	117.0	11.8
22.0	30.4	70.0	18.7	118.0	11.8
23.0	30.6	71.0	18.4	119.0	11.7
24.0	30.7	72.0	18.1	120.0	11.7
25.0	30.7	73.0	17.9	121.0	11.6
26.0	30.8	74.0	17.6	122.0	11.6
27.0	30.8	75.0	17.4	123.0	11.5
28.0	30.7	76.0	17.2	124.0	11.5
29.0	30.7	77.0	17.0	125.0	11.4
30.0	30.6	78.0	16.7	126.0	11.4
31.0	30.4	79.0	16.5	127.0	11.3
32.0	30.3	80.0	16.3	128.0	11.3
33.0	30.1	81.0	16.1	129.0	11.2
34.0	29.9	82.0	15.9	130.0	11.2
35.0	29.6	83.0	15.7	131.0	11.2
36.0	29.4	84.0	15.6	132.0	11.1
37.0	29.1	85.0	15.4	133.0	11.1
38.0	28.8	86.0	15.2	134.0	11.0
39.0	28.5	87.0	15.0	135.0	11.0
40.0	28.3	88.0	14.9	136.0	11.0
41.0	27.9	89.0	14.7	137.0	11.0
42.0	27.7	90.0	14.6	138.0	10.9
43.0	27.3	91.0	14.4	139.0	10.9
44.0	27.0	92.0	14.3	140.0	10.9
45.0	26.7	93.0	14.1	141.0	10.9
46.0	26.4	94.0	14.0	142.0	10.8
47.0	26.0	95.0	13.9	143.0	10.8
48.0	25.7	96.0	13.7	144.0	10.8

Table E.24: Select data from Calorimeter I for w/c 0.36 – Room

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	22.5	49.0	33.3	97.0	25.0
2.0	22.6	50.0	32.9	98.0	24.9
3.0	22.8	51.0	32.5	99.0	24.9
4.0	23.3	52.0	32.2	100.0	24.9
5.0	24.3	53.0	31.8	101.0	24.8
6.0	26.0	54.0	31.5	102.0	24.8
7.0	28.5	55.0	31.2	103.0	24.8
8.0	31.8	56.0	31.0	104.0	24.7
9.0	36.3	57.0	30.7	105.0	24.7
10.0	39.8	58.0	30.4	106.0	24.7
11.0	41.7	59.0	30.2	107.0	24.6
12.0	43.0	60.0	29.9	108.0	24.6
13.0	44.0	61.0	29.6	109.0	24.5
14.0	44.8	62.0	29.4	110.0	24.5
15.0	45.2	63.0	29.2	111.0	24.5
16.0	45.5	64.0	28.9	112.0	24.4
17.0	45.7	65.0	28.7	113.0	24.4
18.0	45.7	66.0	28.5	114.0	24.3
19.0	45.6	67.0	28.3	115.0	24.3
20.0	45.5	68.0	28.1	116.0	24.2
21.0	45.3	69.0	27.9	117.0	24.2
22.0	45.1	70.0	27.7	118.0	24.1
23.0	44.8	71.0	27.5	119.0	24.1
24.0	44.5	72.0	27.3	120.0	24.1
25.0	44.1	73.0	27.2	121.0	24.0
26.0	43.7	74.0	27.0	122.0	24.0
27.0	43.3	75.0	26.9	123.0	23.9
28.0	42.9	76.0	26.8	124.0	23.9
29.0	42.4	77.0	26.7	125.0	23.9
30.0	42.0	78.0	26.5	126.0	23.9
31.0	41.5	79.0	26.5	127.0	23.9
32.0	41.0	80.0	26.4	128.0	23.8
33.0	40.5	81.0	26.3	129.0	23.8
34.0	40.1	82.0	26.2	130.0	23.8
35.0	39.6	83.0	26.1	131.0	23.8
36.0	39.1	84.0	26.0	132.0	23.7
37.0	38.6	85.0	25.9	133.0	23.7
38.0	38.1	86.0	25.8	134.0	23.7
39.0	37.6	87.0	25.7	135.0	23.7
40.0	37.2	88.0	25.6	136.0	23.6
41.0	36.7	89.0	25.5	137.0	23.6
42.0	36.2	90.0	25.5	138.0	23.6
43.0	35.8	91.0	25.4	139.0	23.5
44.0	35.3	92.0	25.3	140.0	23.5
45.0	34.9	93.0	25.2	141.0	23.5
46.0	34.5	94.0	25.2	142.0	23.4
47.0	34.0	95.0	25.1	143.0	23.4
48.0	33.7	96.0	25.0	144.0	23.4

Table E.25: Select data from Calorimeter I for w/c 0.36 – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	35.6	49.0	44.6	97.0	37.0
2.0	36.2	50.0	44.3	98.0	37.0
3.0	37.8	51.0	44.0	99.0	36.9
4.0	41.6	52.0	43.6	100.0	36.9
5.0	49.3	53.0	43.4	101.0	36.8
6.0	53.6	54.0	43.1	102.0	36.7
7.0	56.9	55.0	42.8	103.0	36.7
8.0	59.2	56.0	42.6	104.0	36.7
9.0	60.6	57.0	42.3	105.0	36.6
10.0	61.3	58.0	42.1	106.0	36.6
11.0	61.7	59.0	41.9	107.0	36.5
12.0	61.9	60.0	41.6	108.0	36.5
13.0	61.9	61.0	41.4	109.0	36.4
14.0	61.8	62.0	41.2	110.0	36.4
15.0	61.6	63.0	41.0	111.0	36.4
16.0	61.3	64.0	40.8	112.0	36.3
17.0	60.9	65.0	40.6	113.0	36.3
18.0	60.4	66.0	40.4	114.0	36.3
19.0	59.9	67.0	40.3	115.0	36.2
20.0	59.3	68.0	40.1	116.0	36.2
21.0	58.7	69.0	39.9	117.0	36.1
22.0	58.1	70.0	39.7	118.0	36.1
23.0	57.5	71.0	39.6	119.0	36.1
24.0	56.8	72.0	39.5	120.0	36.1
25.0	56.2	73.0	39.3	121.0	36.0
26.0	55.6	74.0	39.2	122.0	36.0
27.0	54.9	75.0	39.1	123.0	36.0
28.0	54.3	76.0	38.9	124.0	36.0
29.0	53.7	77.0	38.8	125.0	36.0
30.0	53.1	78.0	38.7	126.0	35.9
31.0	52.6	79.0	38.6	127.0	35.9
32.0	52.0	80.0	38.4	128.0	35.9
33.0	51.5	81.0	38.3	129.0	35.9
34.0	51.0	82.0	38.2	130.0	35.9
35.0	50.4	83.0	38.1	131.0	35.8
36.0	49.9	84.0	38.0	132.0	35.8
37.0	49.4	85.0	37.9	133.0	35.8
38.0	49.0	86.0	37.8	134.0	35.8
39.0	48.5	87.0	37.8	135.0	35.8
40.0	48.0	88.0	37.7	136.0	35.7
41.0	47.6	89.0	37.6	137.0	35.7
42.0	47.2	90.0	37.5	138.0	35.7
43.0	46.8	91.0	37.4	139.0	35.7
44.0	46.4	92.0	37.3	140.0	35.7
45.0	46.0	93.0	37.3	141.0	35.7
46.0	45.7	94.0	37.2	142.0	35.7
47.0	45.3	95.0	37.1	143.0	35.7
48.0	44.9	96.0	37.1	144.0	35.6

Table E.26: Select data from Calorimeter II for w/c 0.36 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	29.5	97.0	16.8
2.0	12.0	50.0	29.2	98.0	16.8
3.0	11.6	51.0	28.8	99.0	16.4
4.0	11.8	52.0	28.7	100.0	16.4
5.0	12.2	53.0	28.4	101.0	16.2
6.0	12.7	54.0	28.0	102.0	16.1
7.0	13.0	55.0	27.5	103.0	16.0
8.0	13.6	56.0	27.3	104.0	15.8
9.0	14.3	57.0	26.7	105.0	15.6
10.0	15.7	58.0	26.7	106.0	15.5
11.0	16.9	59.0	26.3	107.0	15.3
12.0	18.5	60.0	26.0	108.0	15.3
13.0	20.5	61.0	25.8	109.0	15.1
14.0	22.6	62.0	25.3	110.0	14.9
15.0	25.0	63.0	24.8	111.0	14.9
16.0	27.6	64.0	24.8	112.0	14.6
17.0	29.5	65.0	24.4	113.0	14.6
18.0	30.7	66.0	24.1	114.0	14.6
19.0	31.2	67.0	23.7	115.0	14.6
20.0	31.7	68.0	23.5	116.0	14.3
21.0	31.9	69.0	23.0	117.0	14.4
22.0	32.3	70.0	22.8	118.0	14.2
23.0	32.7	71.0	22.5	119.0	14.0
24.0	32.9	72.0	22.3	120.0	14.0
25.0	33.0	73.0	22.1	121.0	13.8
26.0	33.2	74.0	21.7	122.0	13.8
27.0	33.2	75.0	21.3	123.0	13.6
28.0	33.5	76.0	21.3	124.0	13.6
29.0	33.6	77.0	21.0	125.0	13.5
30.0	33.6	78.0	20.8	126.0	13.4
31.0	33.5	79.0	20.5	127.0	13.4
32.0	33.4	80.0	20.3	128.0	13.4
33.0	33.1	81.0	19.9	129.0	13.1
34.0	33.2	82.0	19.8	130.0	13.0
35.0	33.1	83.0	19.5	131.0	13.0
36.0	32.9	84.0	19.3	132.0	13.0
37.0	32.7	85.0	19.1	133.0	13.0
38.0	32.7	86.0	18.9	134.0	12.8
39.0	32.2	87.0	18.6	135.0	12.7
40.0	32.1	88.0	18.6	136.0	12.7
41.0	31.9	89.0	18.3	137.0	12.6
42.0	31.7	90.0	17.9	138.0	12.6
43.0	31.3	91.0	17.9	139.0	12.4
44.0	31.3	92.0	17.7	140.0	12.4
45.0	30.7	93.0	17.5	141.0	12.4
46.0	30.4	94.0	17.4	142.0	12.3
47.0	30.2	95.0	17.1	143.0	12.2
48.0	29.8	96.0	16.9	144.0	12.2

Table E.27: Select data from Calorimeter II for w/c 0.36 – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	37.4	97.0	27.0
2.0	22.4	50.0	37.0	98.0	26.9
3.0	22.8	51.0	36.6	99.0	27.0
4.0	23.4	52.0	36.2	100.0	26.8
5.0	24.7	53.0	35.9	101.0	26.7
6.0	26.4	54.0	35.5	102.0	26.5
7.0	28.9	55.0	35.3	103.0	26.5
8.0	32.3	56.0	35.0	104.0	26.5
9.0	36.7	57.0	34.7	105.0	26.4
10.0	40.1	58.0	34.3	106.0	26.2
11.0	41.9	59.0	34.2	107.0	26.3
12.0	43.3	60.0	33.6	108.0	26.3
13.0	44.2	61.0	33.4	109.0	26.1
14.0	45.1	62.0	33.2	110.0	26.1
15.0	45.5	63.0	33.0	111.0	25.9
16.0	46.1	64.0	32.7	112.0	25.9
17.0	46.4	65.0	32.5	113.0	25.9
18.0	46.5	66.0	32.1	114.0	25.7
19.0	46.7	67.0	32.1	115.0	25.7
20.0	46.7	68.0	31.9	116.0	25.7
21.0	46.7	69.0	31.6	117.0	25.7
22.0	46.6	70.0	31.3	118.0	25.5
23.0	46.5	71.0	31.2	119.0	25.5
24.0	46.4	72.0	31.0	120.0	25.5
25.0	46.2	73.0	30.8	121.0	25.5
26.0	45.9	74.0	30.6	122.0	25.5
27.0	45.8	75.0	30.3	123.0	25.3
28.0	45.4	76.0	30.2	124.0	25.3
29.0	44.9	77.0	29.8	125.0	25.3
30.0	44.8	78.0	29.7	126.0	25.1
31.0	44.2	79.0	29.6	127.0	25.1
32.0	44.0	80.0	29.3	128.0	25.2
33.0	43.4	81.0	29.3	129.0	24.9
34.0	43.0	82.0	29.0	130.0	24.9
35.0	42.8	83.0	29.0	131.0	24.9
36.0	42.2	84.0	28.8	132.0	24.9
37.0	41.9	85.0	28.7	133.0	24.7
38.0	41.6	86.0	28.7	134.0	24.7
39.0	41.2	87.0	28.3	135.0	24.7
40.0	40.8	88.0	28.1	136.0	24.7
41.0	40.3	89.0	27.9	137.0	24.7
42.0	40.0	90.0	27.9	138.0	24.7
43.0	39.7	91.0	27.7	139.0	24.7
44.0	39.3	92.0	27.7	140.0	24.7
45.0	38.8	93.0	27.5	141.0	24.5
46.0	38.5	94.0	27.3	142.0	24.5
47.0	38.2	95.0	27.4	143.0	24.5
48.0	37.8	96.0	27.2	144.0	24.4

Table E.28: Select data from Calorimeter II for w/c 0.36 – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	48.8	97.0	39.5
2.0	34.6	50.0	48.6	98.0	39.4
3.0	36.4	51.0	48.3	99.0	39.4
4.0	39.6	52.0	48.0	100.0	39.2
5.0	46.4	53.0	47.6	101.0	39.2
6.0	51.9	54.0	47.4	102.0	39.2
7.0	55.1	55.0	47.0	103.0	39.1
8.0	57.7	56.0	46.9	104.0	38.8
9.0	59.4	57.0	46.6	105.0	38.8
10.0	60.5	58.0	46.2	106.0	38.8
11.0	61.0	59.0	46.0	107.0	38.7
12.0	61.5	60.0	45.6	108.0	38.6
13.0	61.7	61.0	45.5	109.0	38.5
14.0	61.7	62.0	45.2	110.0	38.4
15.0	61.9	63.0	45.0	111.0	38.4
16.0	61.9	64.0	44.8	112.0	38.2
17.0	61.7	65.0	44.6	113.0	38.2
18.0	61.6	66.0	44.4	114.0	38.1
19.0	61.1	67.0	44.1	115.0	38.0
20.0	61.0	68.0	43.9	116.0	38.0
21.0	60.5	69.0	43.7	117.0	37.9
22.0	60.1	70.0	43.5	118.0	37.9
23.0	59.5	71.0	43.3	119.0	37.9
24.0	59.0	72.0	43.1	120.0	37.7
25.0	58.5	73.0	42.9	121.0	37.6
26.0	58.1	74.0	42.8	122.0	37.8
27.0	57.7	75.0	42.6	123.0	37.5
28.0	57.2	76.0	42.4	124.0	37.5
29.0	56.8	77.0	42.2	125.0	37.5
30.0	56.4	78.0	42.0	126.0	37.5
31.0	55.8	79.0	41.9	127.0	37.3
32.0	55.4	80.0	41.8	128.0	37.2
33.0	55.1	81.0	41.7	129.0	37.2
34.0	54.5	82.0	41.4	130.0	37.3
35.0	54.1	83.0	41.3	131.0	37.3
36.0	53.7	84.0	41.1	132.0	37.1
37.0	53.3	85.0	41.1	133.0	37.1
38.0	52.8	86.0	41.0	134.0	37.1
39.0	52.4	87.0	40.8	135.0	37.1
40.0	52.2	88.0	40.6	136.0	37.1
41.0	51.7	89.0	40.5	137.0	36.9
42.0	51.4	90.0	40.5	138.0	36.9
43.0	50.9	91.0	40.3	139.0	36.8
44.0	50.5	92.0	40.2	140.0	36.8
45.0	50.2	93.0	40.0	141.0	36.7
46.0	50.0	94.0	40.0	142.0	36.8
47.0	49.6	95.0	39.8	143.0	36.8
48.0	49.1	96.0	39.6	144.0	36.8

Table E.29: Select data from Calorimeter I for w/c 0.40 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	10.3	49.0	23.3	97.0	14.4
2.0	10.8	50.0	23.0	98.0	14.3
3.0	11.1	51.0	22.8	99.0	14.2
4.0	11.4	52.0	22.5	100.0	14.1
5.0	11.7	53.0	22.3	101.0	14.0
6.0	12.0	54.0	22.0	102.0	13.9
7.0	12.4	55.0	21.8	103.0	13.8
8.0	12.9	56.0	21.6	104.0	13.8
9.0	13.5	57.0	21.3	105.0	13.7
10.0	14.2	58.0	21.1	106.0	13.6
11.0	15.0	59.0	20.8	107.0	13.5
12.0	16.0	60.0	20.6	108.0	13.4
13.0	17.0	61.0	20.4	109.0	13.4
14.0	18.1	62.0	20.2	110.0	13.3
15.0	19.3	63.0	20.0	111.0	13.2
16.0	20.7	64.0	19.7	112.0	13.2
17.0	22.1	65.0	19.5	113.0	13.1
18.0	23.6	66.0	19.3	114.0	13.0
19.0	25.0	67.0	19.1	115.0	13.0
20.0	26.3	68.0	18.9	116.0	12.9
21.0	27.2	69.0	18.7	117.0	12.8
22.0	27.8	70.0	18.5	118.0	12.8
23.0	28.1	71.0	18.2	119.0	12.7
24.0	28.3	72.0	18.1	-	-
25.0	28.3	73.0	17.9	-	-
26.0	28.3	74.0	17.7	-	-
27.0	28.2	75.0	17.5	-	-
28.0	28.0	76.0	17.3	-	-
29.0	27.9	77.0	17.1	-	-
30.0	27.7	78.0	17.0	-	-
31.0	27.5	79.0	16.8	-	-
32.0	27.3	80.0	16.6	-	-
33.0	27.1	81.0	16.5	-	-
34.0	26.9	82.0	16.3	-	-
35.0	26.6	83.0	16.2	-	-
36.0	26.4	84.0	16.0	-	-
37.0	26.2	85.0	15.9	-	-
38.0	25.9	86.0	15.7	-	-
39.0	25.7	87.0	15.6	-	-
40.0	25.5	88.0	15.5	-	-
41.0	25.2	89.0	15.3	-	-
42.0	25.0	90.0	15.2	-	-
43.0	24.7	91.0	15.1	-	-
44.0	24.5	92.0	15.0	-	-
45.0	24.2	93.0	14.8	-	-
46.0	24.0	94.0	14.7	-	-
47.0	23.7	95.0	14.6	-	-
48.0	23.5	96.0	14.5	-	-

Table E.30: Select data from Calorimeter I for w/c 0.40 – Room

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	22.3	49.0	34.4	97.0	25.6
2.0	22.6	50.0	34.1	98.0	25.5
3.0	23.0	51.0	33.8	99.0	25.4
4.0	24.0	52.0	33.5	100.0	25.3
5.0	25.5	53.0	33.2	101.0	25.2
6.0	27.6	54.0	32.9	102.0	25.2
7.0	30.2	55.0	32.6	103.0	25.1
8.0	33.6	56.0	32.3	104.0	25.1
9.0	37.5	57.0	32.0	105.0	25.0
10.0	40.2	58.0	31.8	106.0	24.9
11.0	41.8	59.0	31.5	107.0	24.9
12.0	42.9	60.0	31.3	108.0	24.8
13.0	43.8	61.0	31.0	109.0	24.8
14.0	44.4	62.0	30.7	110.0	24.7
15.0	44.8	63.0	30.5	111.0	24.7
16.0	45.1	64.0	30.3	112.0	24.6
17.0	45.2	65.0	30.0	113.0	24.6
18.0	45.2	66.0	29.8	114.0	24.5
19.0	45.2	67.0	29.6	115.0	24.5
20.0	45.0	68.0	29.4	116.0	24.4
21.0	44.8	69.0	29.2	117.0	24.3
22.0	44.7	70.0	29.0	118.0	24.3
23.0	44.4	71.0	28.9	119.0	24.2
24.0	44.2	72.0	28.7	120.0	24.2
25.0	43.9	73.0	28.5	121.0	24.1
26.0	43.6	74.0	28.3	122.0	24.1
27.0	43.3	75.0	28.2	123.0	24.1
28.0	43.0	76.0	28.0	124.0	24.0
29.0	42.6	77.0	27.9	125.0	24.0
30.0	42.1	78.0	27.7	126.0	24.0
31.0	41.7	79.0	27.6	127.0	24.0
32.0	41.3	80.0	27.4	128.0	23.9
33.0	40.8	81.0	27.3	129.0	23.9
34.0	40.4	82.0	27.2	130.0	23.9
35.0	40.0	83.0	27.0	131.0	23.9
36.0	39.5	84.0	26.9	132.0	23.8
37.0	39.1	85.0	26.8	133.0	23.8
38.0	38.7	86.0	26.7	134.0	23.8
39.0	38.3	87.0	26.6	135.0	23.8
40.0	37.9	88.0	26.5	136.0	23.8
41.0	37.5	89.0	26.4	137.0	23.7
42.0	37.0	90.0	26.3	138.0	23.7
43.0	36.7	91.0	26.1	139.0	23.7
44.0	36.3	92.0	26.0	140.0	23.7
45.0	35.9	93.0	25.9	141.0	23.7
46.0	35.5	94.0	25.8	142.0	23.6
47.0	35.1	95.0	25.7	143.0	23.6
48.0	34.8	96.0	25.6	144.0	23.6

Table E.31: Select data from Calorimeter I for w/c 0.40 – Hot

Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)	Concrete Age (Hours)	Concrete Temperature (℃)
1.0	34.2	49.0	43.5	97.0	36.3
2.0	34.7	50.0	43.2	98.0	36.2
3.0	36.8	51.0	42.9	99.0	36.2
4.0	41.4	52.0	42.7	100.0	36.1
5.0	48.7	53.0	42.4	101.0	36.1
6.0	52.5	54.0	42.1	102.0	36.0
7.0	55.3	55.0	41.9	103.0	36.0
8.0	57.4	56.0	41.6	104.0	36.0
9.0	58.6	57.0	41.4	105.0	35.9
10.0	59.2	58.0	41.1	106.0	35.9
11.0	59.5	59.0	40.9	107.0	35.8
12.0	59.7	60.0	40.7	108.0	35.8
13.0	59.7	61.0	40.4	109.0	35.7
14.0	59.7	62.0	40.3	110.0	35.7
15.0	59.5	63.0	40.0	111.0	35.7
16.0	59.3	64.0	39.9	112.0	35.6
17.0	59.0	65.0	39.7	113.0	35.6
18.0	58.6	66.0	39.5	114.0	35.5
19.0	58.2	67.0	39.3	115.0	35.5
20.0	57.7	68.0	39.1	116.0	35.5
21.0	57.1	69.0	39.0	117.0	35.4
22.0	56.6	70.0	38.8	118.0	35.4
23.0	56.0	71.0	38.7	119.0	35.4
24.0	55.4	72.0	38.6	120.0	35.4
25.0	54.9	73.0	38.4	121.0	35.3
26.0	54.3	74.0	38.3	122.0	35.3
27.0	53.7	75.0	38.2	123.0	35.3
28.0	53.1	76.0	38.0	124.0	35.3
29.0	52.5	77.0	37.9	125.0	35.3
30.0	52.0	78.0	37.8	126.0	35.3
31.0	51.4	79.0	37.7	127.0	35.3
32.0	50.9	80.0	37.6	128.0	35.2
33.0	50.3	81.0	37.5	129.0	35.2
34.0	49.8	82.0	37.4	130.0	35.2
35.0	49.3	83.0	37.3	131.0	35.2
36.0	48.8	84.0	37.2	132.0	35.2
37.0	48.3	85.0	37.1	133.0	35.2
38.0	47.8	86.0	37.0	134.0	35.1
39.0	47.4	87.0	36.9	135.0	35.1
40.0	46.9	88.0	36.9	136.0	35.1
41.0	46.5	89.0	36.8	137.0	35.1
42.0	46.1	90.0	36.7	138.0	35.1
43.0	45.7	91.0	36.7	139.0	35.1
44.0	45.2	92.0	36.6	140.0	35.1
45.0	44.9	93.0	36.5	141.0	35.0
46.0	44.5	94.0	36.5	142.0	35.0
47.0	44.2	95.0	36.4	143.0	35.0
48.0	43.8	96.0	36.3	144.0	35.0

Table E.32: Select data from Calorimeter II for w/c 0.40 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	27.5	97.0	17.4
2.0	11.5	50.0	27.5	98.0	17.4
3.0	11.4	51.0	27.1	99.0	17.4
4.0	11.6	52.0	26.9	100.0	17.3
5.0	11.8	53.0	26.5	101.0	16.9
6.0	12.1	54.0	26.6	102.0	16.9
7.0	12.6	55.0	26.3	103.0	16.7
8.0	13.0	56.0	26.1	104.0	16.7
9.0	13.7	57.0	25.7	105.0	16.6
10.0	14.5	58.0	25.4	106.0	16.4
11.0	15.2	59.0	25.1	107.0	16.1
12.0	16.5	60.0	25.1	108.0	16.0
13.0	17.6	61.0	24.8	109.0	16.1
14.0	18.9	62.0	24.6	110.0	15.9
15.0	20.2	63.0	24.2	111.0	15.7
16.0	21.8	64.0	24.1	112.0	15.7
17.0	23.4	65.0	23.7	113.0	15.5
18.0	25.3	66.0	23.7	114.0	15.4
19.0	26.8	67.0	23.3	115.0	15.4
20.0	28.0	68.0	23.1	116.0	15.2
21.0	29.0	69.0	22.8	117.0	15.2
22.0	29.6	70.0	22.6	118.0	15.2
23.0	29.8	71.0	22.2	119.0	14.9
24.0	30.3	72.0	22.3	120.0	15.0
25.0	30.3	73.0	22.0	-	-
26.0	30.4	74.0	21.8	-	-
27.0	30.4	75.0	21.6	-	-
28.0	30.4	76.0	21.4	-	-
29.0	30.2	77.0	20.9	-	-
30.0	30.2	78.0	20.8	-	-
31.0	30.3	79.0	20.6	-	-
32.0	30.3	80.0	20.6	-	-
33.0	30.1	81.0	20.4	-	-
34.0	29.9	82.0	20.1	-	-
35.0	29.8	83.0	19.9	-	-
36.0	29.8	84.0	19.7	-	-
37.0	29.5	85.0	19.6	-	-
38.0	29.5	86.0	19.4	-	-
39.0	29.3	87.0	19.2	-	-
40.0	29.1	88.0	19.0	-	-
41.0	28.8	89.0	18.7	-	-
42.0	28.8	90.0	18.5	-	-
43.0	28.7	91.0	18.5	-	-
44.0	28.5	92.0	18.3	-	-
45.0	28.3	93.0	18.2	-	-
46.0	28.1	94.0	18.2	-	-
47.0	28.0	95.0	17.6	-	-
48.0	27.9	96.0	17.7	-	-

Table E.33: Select data from Calorimeter II for w/c 0.40 – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	37.7	97.0	28.0
2.0	22.6	50.0	37.5	98.0	27.8
3.0	23.2	51.0	37.0	99.0	27.9
4.0	24.3	52.0	36.8	100.0	27.6
5.0	25.9	53.0	36.4	101.0	27.4
6.0	28.2	54.0	36.3	102.0	27.5
7.0	31.0	55.0	35.9	103.0	27.5
8.0	34.5	56.0	35.5	104.0	27.3
9.0	38.4	57.0	35.3	105.0	27.1
10.0	40.8	58.0	35.1	106.0	27.1
11.0	42.1	59.0	34.7	107.0	26.9
12.0	43.1	60.0	34.5	108.0	26.9
13.0	44.0	61.0	34.3	109.0	26.9
14.0	44.7	62.0	33.9	110.0	26.7
15.0	45.3	63.0	33.7	111.0	26.7
16.0	45.5	64.0	33.5	112.0	26.7
17.0	45.8	65.0	33.3	113.0	26.5
18.0	45.8	66.0	33.1	114.0	26.4
19.0	46.0	67.0	32.7	115.0	26.4
20.0	46.0	68.0	32.5	116.0	26.2
21.0	46.0	69.0	32.3	117.0	26.2
22.0	45.8	70.0	32.1	118.0	26.2
23.0	45.8	71.0	32.1	119.0	26.1
24.0	45.5	72.0	31.7	120.0	25.9
25.0	45.5	73.0	31.4	121.0	25.9
26.0	45.4	74.0	31.3	122.0	25.9
27.0	45.1	75.0	31.1	123.0	25.7
28.0	44.9	76.0	31.1	124.0	25.7
29.0	44.6	77.0	30.9	125.0	25.8
30.0	44.5	78.0	30.7	126.0	25.5
31.0	44.0	79.0	30.4	127.0	25.5
32.0	43.7	80.0	30.4	128.0	25.5
33.0	43.3	81.0	30.1	129.0	25.4
34.0	43.1	82.0	30.0	130.0	25.4
35.0	42.6	83.0	29.8	131.0	25.4
36.0	42.3	84.0	29.8	132.0	25.2
37.0	41.9	85.0	29.6	133.0	25.1
38.0	41.5	86.0	29.4	134.0	25.1
39.0	41.2	87.0	29.4	135.0	25.1
40.0	41.0	88.0	29.4	136.0	25.0
41.0	40.5	89.0	29.1	137.0	25.0
42.0	40.2	90.0	28.9	138.0	25.0
43.0	39.7	91.0	28.8	139.0	25.0
44.0	39.5	92.0	28.6	140.0	24.8
45.0	39.2	93.0	28.6	141.0	24.8
46.0	38.7	94.0	28.4	142.0	24.8
47.0	38.5	95.0	28.2	143.0	24.8
48.0	38.2	96.0	28.0	144.0	24.6

Table E.34: Select data from Calorimeter II for w/c 0.40 – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	46.6	97.0	38.9
2.0	31.2	50.0	46.3	98.0	38.7
3.0	33.6	51.0	46.0	99.0	38.7
4.0	37.2	52.0	45.6	100.0	38.7
5.0	43.5	53.0	45.5	101.0	38.5
6.0	48.5	54.0	45.2	102.0	38.4
7.0	51.1	55.0	45.1	103.0	38.2
8.0	53.1	56.0	44.8	104.0	38.2
9.0	54.6	57.0	44.6	105.0	38.3
10.0	55.7	58.0	44.3	106.0	38.0
11.0	56.2	59.0	44.2	107.0	38.1
12.0	56.6	60.0	43.9	108.0	38.1
13.0	56.9	61.0	43.7	109.0	37.9
14.0	57.0	62.0	43.6	110.0	37.8
15.0	57.3	63.0	43.4	111.0	37.8
16.0	57.3	64.0	43.2	112.0	37.7
17.0	57.4	65.0	42.9	113.0	37.6
18.0	57.2	66.0	42.7	114.0	37.6
19.0	57.0	67.0	42.6	115.0	37.4
20.0	56.7	68.0	42.4	116.0	37.5
21.0	56.4	69.0	42.3	117.0	37.5
22.0	56.2	70.0	42.1	118.0	37.2
23.0	55.8	71.0	42.0	119.0	37.4
24.0	55.6	72.0	41.6	120.0	37.1
25.0	55.2	73.0	41.6	121.0	37.1
26.0	54.7	74.0	41.5	122.0	37.1
27.0	54.3	75.0	41.3	123.0	37.1
28.0	54.0	76.0	41.2	124.0	37.0
29.0	53.5	77.0	41.1	125.0	36.9
30.0	53.1	78.0	40.9	126.0	36.9
31.0	52.7	79.0	40.9	127.0	36.9
32.0	52.4	80.0	40.6	128.0	36.9
33.0	52.0	81.0	40.4	129.0	36.8
34.0	51.4	82.0	40.3	130.0	36.9
35.0	51.1	83.0	40.2	131.0	36.8
36.0	50.8	84.0	40.0	132.0	36.8
37.0	50.4	85.0	40.0	133.0	36.7
38.0	50.1	86.0	39.9	134.0	36.7
39.0	49.8	87.0	39.7	135.0	36.7
40.0	49.3	88.0	39.5	136.0	36.6
41.0	49.0	89.0	39.7	137.0	36.7
42.0	48.6	90.0	39.4	138.0	36.5
43.0	48.4	91.0	39.2	139.0	36.5
44.0	48.0	92.0	39.3	140.0	36.5
45.0	47.6	93.0	39.2	141.0	36.4
46.0	47.5	94.0	39.1	142.0	36.4
47.0	47.2	95.0	39.0	143.0	36.3
48.0	46.8	96.0	39.0	144.0	36.3

Table E.35: Select data from Calorimeter I for w/c 0.48 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	11.6	49.0	22.0	97.0	14.1
2.0	11.7	50.0	21.7	98.0	14.0
3.0	12.0	51.0	21.5	99.0	13.9
4.0	12.3	52.0	21.2	100.0	13.8
5.0	12.6	53.0	21.0	101.0	13.7
6.0	13.0	54.0	20.7	102.0	13.7
7.0	13.5	55.0	20.5	103.0	13.6
8.0	14.1	56.0	20.3	104.0	13.5
9.0	14.8	57.0	20.0	105.0	13.4
10.0	15.6	58.0	19.8	106.0	13.4
11.0	16.5	59.0	19.6	107.0	13.3
12.0	17.5	60.0	19.4	108.0	13.2
13.0	18.6	61.0	19.2	109.0	13.2
14.0	19.7	62.0	19.0	110.0	13.1
15.0	21.0	63.0	18.7	111.0	13.0
16.0	22.2	64.0	18.5	112.0	13.0
17.0	23.4	65.0	18.3	113.0	12.9
18.0	24.5	66.0	18.2	114.0	12.9
19.0	25.5	67.0	18.0	115.0	12.8
20.0	26.4	68.0	17.8	116.0	12.8
21.0	27.1	69.0	17.6	117.0	12.7
22.0	27.6	70.0	17.4	118.0	12.7
23.0	27.8	71.0	17.3	119.0	12.6
24.0	27.9	72.0	17.1	120.0	12.6
25.0	27.9	73.0	16.9	121.0	12.5
26.0	27.8	74.0	16.8	122.0	12.5
27.0	27.7	75.0	16.6	123.0	12.4
28.0	27.5	76.0	16.5	124.0	12.4
29.0	27.3	77.0	16.3	125.0	12.3
30.0	27.0	78.0	16.2	126.0	12.3
31.0	26.8	79.0	16.1	127.0	12.3
32.0	26.5	80.0	15.9	128.0	12.2
33.0	26.3	81.0	15.8	129.0	12.2
34.0	26.0	82.0	15.7	130.0	12.1
35.0	25.7	83.0	15.5	131.0	12.1
36.0	25.5	84.0	15.4	132.0	12.1
37.0	25.2	85.0	15.3	133.0	12.1
38.0	24.9	86.0	15.2	134.0	12.0
39.0	24.6	87.0	15.1	135.0	12.0
40.0	24.4	88.0	14.9	136.0	12.0
41.0	24.1	89.0	14.8	137.0	11.9
42.0	23.8	90.0	14.7	138.0	11.9
43.0	23.5	91.0	14.6	139.0	11.9
44.0	23.3	92.0	14.5	140.0	11.9
45.0	23.0	93.0	14.4	141.0	11.8
46.0	22.8	94.0	14.3	142.0	11.8
47.0	22.5	95.0	14.2	143.0	11.8
48.0	22.2	96.0	14.1	144.0	11.8

Table E.36: Select data from Calorimeter I for w/c 0.48 – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	21.8	49.0	34.5	97.0	26.1
2.0	22.2	50.0	34.2	98.0	26.0
3.0	22.9	51.0	33.9	99.0	25.9
4.0	24.0	52.0	33.7	100.0	25.8
5.0	25.6	53.0	33.4	101.0	25.7
6.0	27.6	54.0	33.1	102.0	25.6
7.0	30.0	55.0	32.9	103.0	25.5
8.0	32.8	56.0	32.6	104.0	25.4
9.0	35.8	57.0	32.4	105.0	25.3
10.0	38.4	58.0	32.2	106.0	25.2
11.0	40.1	59.0	31.9	107.0	25.2
12.0	41.2	60.0	31.7	108.0	25.1
13.0	41.9	61.0	31.4	109.0	25.0
14.0	42.3	62.0	31.2	110.0	24.9
15.0	42.7	63.0	31.0	111.0	24.8
16.0	42.9	64.0	30.7	112.0	24.7
17.0	43.0	65.0	30.5	113.0	24.6
18.0	43.1	66.0	30.3	114.0	24.5
19.0	43.0	67.0	30.1	115.0	24.5
20.0	42.9	68.0	29.9	116.0	24.4
21.0	42.7	69.0	29.7	117.0	24.3
22.0	42.5	70.0	29.5	118.0	24.2
23.0	42.3	71.0	29.3	119.0	24.1
24.0	42.0	72.0	29.2	120.0	24.0
25.0	41.7	73.0	29.0	121.0	23.9
26.0	41.4	74.0	28.8	122.0	23.9
27.0	41.2	75.0	28.7	123.0	23.8
28.0	40.9	76.0	28.5	124.0	23.7
29.0	40.6	77.0	28.4	125.0	23.7
30.0	40.3	78.0	28.3	126.0	23.6
31.0	40.0	79.0	28.1	127.0	23.6
32.0	39.7	80.0	28.0	128.0	23.5
33.0	39.4	81.0	27.9	129.0	23.5
34.0	39.1	82.0	27.8	130.0	23.4
35.0	38.8	83.0	27.6	131.0	23.4
36.0	38.4	84.0	27.5	132.0	23.3
37.0	38.1	85.0	27.4	133.0	23.3
38.0	37.8	86.0	27.3	134.0	23.2
39.0	37.5	87.0	27.1	135.0	23.2
40.0	37.2	88.0	27.0	136.0	23.1
41.0	36.9	89.0	26.9	137.0	23.1
42.0	36.6	90.0	26.8	138.0	23.0
43.0	36.3	91.0	26.7	139.0	23.0
44.0	35.9	92.0	26.6	140.0	22.9
45.0	35.6	93.0	26.5	141.0	22.8
46.0	35.3	94.0	26.4	142.0	22.8
47.0	35.0	95.0	26.3	143.0	22.8
48.0	34.8	96.0	26.2	144.0	22.7

Table E.37: Select data from Calorimeter I for w/c 0.48 – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	35.0	49.0	44.6	97.0	36.9
2.0	35.8	50.0	44.3	98.0	36.8
3.0	38.0	51.0	44.0	99.0	36.7
4.0	42.0	52.0	43.7	100.0	36.7
5.0	47.9	53.0	43.4	101.0	36.6
6.0	51.6	54.0	43.2	102.0	36.6
7.0	54.0	55.0	42.9	103.0	36.5
8.0	55.6	56.0	42.7	104.0	36.4
9.0	56.7	57.0	42.4	105.0	36.4
10.0	57.3	58.0	42.2	106.0	36.3
11.0	57.6	59.0	41.9	107.0	36.3
12.0	57.7	60.0	41.7	108.0	36.2
13.0	57.6	61.0	41.5	109.0	36.2
14.0	57.4	62.0	41.3	110.0	36.1
15.0	57.2	63.0	41.1	111.0	36.1
16.0	56.9	64.0	40.9	112.0	36.0
17.0	56.7	65.0	40.7	113.0	36.0
18.0	56.3	66.0	40.5	114.0	36.0
19.0	56.1	67.0	40.3	115.0	35.9
20.0	55.7	68.0	40.1	116.0	35.9
21.0	55.4	69.0	39.9	117.0	35.9
22.0	55.0	70.0	39.8	118.0	35.8
23.0	54.6	71.0	39.6	119.0	35.8
24.0	54.2	72.0	39.5	120.0	35.7
25.0	53.8	73.0	39.3	121.0	35.7
26.0	53.4	74.0	39.2	122.0	35.7
27.0	52.9	75.0	39.0	123.0	35.7
28.0	52.5	76.0	38.9	124.0	35.6
29.0	52.1	77.0	38.8	125.0	35.6
30.0	51.7	78.0	38.6	126.0	35.6
31.0	51.2	79.0	38.5	127.0	35.6
32.0	50.8	80.0	38.4	128.0	35.5
33.0	50.4	81.0	38.3	129.0	35.5
34.0	50.0	82.0	38.1	130.0	35.5
35.0	49.6	83.0	38.0	131.0	35.4
36.0	49.2	84.0	37.9	132.0	35.4
37.0	48.8	85.0	37.8	133.0	35.4
38.0	48.4	86.0	37.7	134.0	35.4
39.0	48.0	87.0	37.6	135.0	35.4
40.0	47.6	88.0	37.5	136.0	35.3
41.0	47.3	89.0	37.5	137.0	35.3
42.0	46.9	90.0	37.4	138.0	35.3
43.0	46.5	91.0	37.3	139.0	35.3
44.0	46.2	92.0	37.2	140.0	35.3
45.0	45.8	93.0	37.1	141.0	35.3
46.0	45.5	94.0	37.0	142.0	35.3
47.0	45.2	95.0	37.0	143.0	35.2
48.0	44.9	96.0	36.9	144.0	35.2

Table E.38: Select data from Calorimeter II for w/c 0.48 – Cold

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	25.4	97.0	17.6
2.0	12.1	50.0	25.4	98.0	17.6
3.0	11.6	51.0	25.2	99.0	17.4
4.0	11.9	52.0	24.5	100.0	17.2
5.0	12.2	53.0	24.7	101.0	17.1
6.0	12.7	54.0	24.6	102.0	17.0
7.0	13.2	55.0	24.3	103.0	17.0
8.0	13.9	56.0	24.2	104.0	17.0
9.0	14.5	57.0	23.9	105.0	16.8
10.0	15.4	58.0	23.5	106.0	16.5
11.0	16.2	59.0	23.6	107.0	16.8
12.0	17.4	60.0	23.3	108.0	16.4
13.0	18.3	61.0	23.3	109.0	16.4
14.0	19.6	62.0	23.1	110.0	16.2
15.0	20.8	63.0	22.7	111.0	16.2
16.0	21.9	64.0	22.4	112.0	15.9
17.0	23.6	65.0	22.3	113.0	15.9
18.0	24.6	66.0	22.3	114.0	15.8
19.0	25.5	67.0	22.1	115.0	15.8
20.0	26.7	68.0	21.8	116.0	15.6
21.0	27.6	69.0	21.8	117.0	15.5
22.0	28.1	70.0	21.3	118.0	15.4
23.0	28.5	71.0	21.4	119.0	15.5
24.0	28.7	72.0	21.1	0.0	0.0
25.0	28.9	73.0	21.0	0.0	0.0
26.0	29.1	74.0	21.0	0.0	0.0
27.0	29.1	75.0	20.8	0.0	0.0
28.0	28.6	76.0	20.4	0.0	0.0
29.0	28.8	77.0	20.3	0.0	0.0
30.0	28.7	78.0	20.3	0.0	0.0
31.0	28.7	79.0	20.1	0.0	0.0
32.0	28.5	80.0	20.1	0.0	0.0
33.0	28.4	81.0	19.9	0.0	0.0
34.0	28.0	82.0	19.5	0.0	0.0
35.0	28.1	83.0	19.6	0.0	0.0
36.0	27.8	84.0	19.4	0.0	0.0
37.0	27.6	85.0	19.3	0.0	0.0
38.0	27.6	86.0	19.2	0.0	0.0
39.0	27.4	87.0	19.0	0.0	0.0
40.0	27.0	88.0	18.6	0.0	0.0
41.0	27.0	89.0	18.7	0.0	0.0
42.0	26.9	90.0	18.7	0.0	0.0
43.0	26.7	91.0	18.5	0.0	0.0
44.0	26.5	92.0	18.3	0.0	0.0
45.0	26.2	93.0	18.2	0.0	0.0
46.0	25.9	94.0	17.9	0.0	0.0
47.0	25.7	95.0	17.9	0.0	0.0
48.0	25.7	96.0	17.8	0.0	0.0

Table E.39: Select data from Calorimeter II for w/c 0.48 – Room

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
1.0	21.9	49.0	36.7	97.0	27.8
2.0	22.4	50.0	36.5	98.0	27.8
3.0	23.0	51.0	36.1	99.0	27.6
4.0	24.2	52.0	36.0	100.0	27.6
5.0	25.7	53.0	35.6	101.0	27.5
6.0	27.8	54.0	35.4	102.0	27.2
7.0	30.3	55.0	35.2	103.0	27.2
8.0	33.0	56.0	34.9	104.0	27.0
9.0	35.8	57.0	34.7	105.0	27.0
10.0	38.4	58.0	34.4	106.0	26.7
11.0	40.0	59.0	34.2	107.0	26.7
12.0	41.0	60.0	34.0	108.0	26.7
13.0	41.6	61.0	33.8	109.0	26.4
14.0	42.2	62.0	33.6	110.0	26.4
15.0	42.4	63.0	33.4	111.0	26.4
16.0	42.7	64.0	33.0	112.0	26.3
17.0	42.9	65.0	33.0	113.0	26.3
18.0	43.2	66.0	32.7	114.0	26.0
19.0	43.2	67.0	32.5	115.0	26.0
20.0	43.1	68.0	32.3	116.0	25.9
21.0	43.1	69.0	32.0	117.0	25.9
22.0	43.1	70.0	31.8	118.0	25.6
23.0	42.9	71.0	31.7	119.0	25.6
24.0	42.7	72.0	31.5	120.0	25.6
25.0	42.5	73.0	31.3	121.0	25.5
26.0	42.4	74.0	31.1	122.0	25.3
27.0	42.0	75.0	31.0	123.0	25.3
28.0	42.0	76.0	30.7	124.0	25.3
29.0	41.9	77.0	30.6	125.0	25.1
30.0	41.6	78.0	30.4	126.0	25.1
31.0	41.2	79.0	30.4	127.0	25.1
32.0	41.2	80.0	30.1	128.0	24.9
33.0	40.9	81.0	30.1	129.0	24.8
34.0	40.6	82.0	29.9	130.0	24.8
35.0	40.4	83.0	29.7	131.0	24.8
36.0	40.0	84.0	29.4	132.0	24.6
37.0	39.9	85.0	29.4	133.0	24.6
38.0	39.7	86.0	29.4	134.0	24.6
39.0	39.5	87.0	29.0	135.0	24.5
40.0	39.0	88.0	29.0	136.0	24.3
41.0	38.8	89.0	28.8	137.0	24.3
42.0	38.6	90.0	28.6	138.0	24.2
43.0	38.4	91.0	28.6	139.0	24.2
44.0	38.2	92.0	28.4	140.0	24.2
45.0	37.9	93.0	28.3	141.0	24.0
46.0	37.5	94.0	28.1	142.0	24.0
47.0	37.3	95.0	28.1	143.0	24.0
48.0	37.0	96.0	27.8	144.0	23.9

Table E.40: Select data from Calorimeter II for w/c 0.48 – Hot

Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature	Concrete Age	Concrete Temperature
(Hours)	(℃)	(Hours)	(℃)	(Hours)	(℃)
-	-	49.0	49.4	97.0	39.8
2.0	37.3	50.0	49.1	98.0	39.6
3.0	39.8	51.0	48.8	99.0	39.7
4.0	44.9	52.0	48.6	100.0	39.4
5.0	51.7	53.0	48.2	101.0	39.2
6.0	54.8	54.0	47.9	102.0	39.2
7.0	56.9	55.0	47.6	103.0	39.2
8.0	58.3	56.0	47.4	104.0	39.0
9.0	59.3	57.0	47.1	105.0	39.0
10.0	59.8	58.0	46.9	106.0	38.8
11.0	60.1	59.0	46.5	107.0	38.8
12.0	60.1	60.0	46.2	108.0	38.8
13.0	60.3	61.0	46.0	109.0	38.5
14.0	60.3	62.0	45.7	110.0	38.5
15.0	60.0	63.0	45.5	111.0	38.3
16.0	60.0	64.0	45.3	112.0	38.3
17.0	59.7	65.0	45.1	113.0	38.2
18.0	59.6	66.0	44.7	114.0	38.2
19.0	59.4	67.0	44.6	115.0	38.0
20.0	59.4	68.0	44.4	116.0	38.0
21.0	59.0	69.0	44.3	117.0	37.8
22.0	58.8	70.0	44.0	118.0	37.8
23.0	58.5	71.0	43.6	119.0	37.7
24.0	58.2	72.0	43.6	120.0	37.7
25.0	57.9	73.0	43.4	121.0	37.5
26.0	57.5	74.0	43.2	122.0	37.6
27.0	57.0	75.0	43.0	123.0	37.6
28.0	56.8	76.0	42.8	124.0	37.4
29.0	56.4	77.0	42.7	125.0	37.4
30.0	56.0	78.0	42.5	126.0	37.2
31.0	55.8	79.0	42.3	127.0	37.3
32.0	55.3	80.0	42.2	128.0	37.2
33.0	54.8	81.0	42.0	129.0	37.2
34.0	54.5	82.0	41.7	130.0	37.2
35.0	54.1	83.0	41.7	131.0	37.0
36.0	53.9	84.0	41.4	132.0	37.0
37.0	53.5	85.0	41.4	133.0	37.0
38.0	53.0	86.0	41.2	134.0	36.8
39.0	52.8	87.0	41.1	135.0	36.8
40.0	52.3	88.0	40.9	136.0	36.8
41.0	52.0	89.0	40.7	137.0	36.6
42.0	51.7	90.0	40.5	138.0	36.7
43.0	51.3	91.0	40.4	139.0	36.6
44.0	51.1	92.0	40.4	140.0	36.6
45.0	50.7	93.0	40.2	141.0	36.4
46.0	50.3	94.0	40.0	142.0	36.5
47.0	49.9	95.0	39.8	143.0	36.5
48.0	49.7	96.0	39.9	144.0	36.3