FINAL REPORT

AUTOGENOUS ACCELERATED CURING OF CONCRETE CYLINDERS

Part III

Temperature Relationships

by

Larry M. Cook Highway Engineer Trainee

(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

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PREFACE

The Research Council's studies of early determination of compressive strength of concrete stored in water baths at elevated temperatures were initiated in 1967 as a part of the State funded research program. The results of this research were presented by K. H. McGhee in his report entitled "Water Bath Accelerated Curing of Concrete".

Under the work plan by L. M. Cook entitled "An Investigation of the Moisture-Temperature Relationships — Autogenous Accelerated Curing for Early Determination of Concrete Strength Potential", the study was extended to autogenous curing. The extended study was approved for financing under Federal Highway Planning and Research Funds on May 14, 1969. The objectives of this project were:

- 1. To extend knowledge of the thermal and moisture behavior of concrete subjected to high curing temperatures during autogenous curing.
- 2. To examine the influence that variables such as cement type, cement factor, water-cement ratio, and admixtures have on moisture and temperature.
- 3. To correlate the accelerated strengths of autogenously cured cylinders with those of 28 and 91 day old moist cured cylinders.

Concurrently with the Council's research project, ASTM Committee C-9 was developing standard methods of testing, Several questions raised during the ASTM efforts were closely related to the Council's work. As a result of a discussion with Federal Highway Administration personnel in October 1969, a limited study of the curing container characteristics and storage conditions was undertaken to supplement the major project effort.

The total project ultimately involved preparation of approximately 300 batches of concrete in the laboratory with all of the necessary testing. Calibration of moisture measuring instrumentation and continuous recording of temperature and moisture for the test specimens resulted in voluminous data.

For maximum intelligibility and usefulness, the report on this project has been subdivided into five parts as follows:

Part I		Strength Results
Part II	anches.	Development of a Moisture Measuring Method
Part III	(METHOD)	Temperature Relationships
Part IV	04-35KD	Moisture Relationships
Part V	as Days	ASTM Cooperative Testing Program with Additional Emphasis on the Influence of Container and Storage Characteristics (Supplemented by Data on Water Bath Curing From an Earlier Council Project)

In Part V, it was deemed desirable to include data from the earlier study by McGhee so as to give a comprehensive picture of the Council's portion of the ASTM Cooperative Testing Program. While some of the work reported in Part V was not a part of the autogenous curing study, most of it was done as a part of the project so that its inclusion in the project report seems logical.

Each part of the report contains sufficient background information to enable it to stand alone as coverage of the aspect of the project reflected in its title. The titles, in general, reflect the project objectives. Taken together, these five reports represent the final report on the study of Autogenous Accelerated Curing of Concrete Cylinders.

SUMMARY

Forty-eight different concrete mixes were designed to investigate the influence of cement types (II, III, and V), cement contents (450, 550, and 650 lb. per cu. yd.), water cement ratios (0.4, 0.5, and 0.6), admixtures (accelerator, retarder, and air entraining agent), and initial mixture temperatures ($50^{\circ}F$, $70^{\circ}F$, and $90^{\circ}F$) on the temperature development of autogenous accelerated cured concrete cylinders.

A total of 940 concrete cylinders were made incorporating autogenously cured cylinders, 28 day, 91 day, and 1 year moist cured cylinders, and autogenously cured cylinders instrumented for temperature and moisture measurements.

The project involved routine testing for slump, unit weight, air content, time of set, and compressive strengths. Additional measurements of temperature development and moisture movement were made.

This report describes the influence of concrete mixture variables and initial mixture temperatures on the temperatures developed during autogenous accelerated curing and the relationships among the temperature parameters resulting from the measurements.

The report lists eleven observations and conclusions.

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FINAL REPORT

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INTRODUCTION

Curing a concrete cylinder autogenously (by a self-generated increase in curing temperature) inside a container sufficiently insulated to prevent heat and moisture loss will provide an accelerated strength comparable to that obtained by applying external heat. (1) Application of autogenous curing to rapid testing of concrete has been extensively studied experimentally in Canada. (1) The Virginia Highway Research Council's studies of this method have been described in Part I of this report. (2)

Since in autogenous curing the heat is derived internally, the accelerating process is self-regulating, which does away with the necessity for determining a proper time of set before commencing acceleration and decreases the adverse effect of heat on the reactions of calcium sulfate and calcium aluminate.

Because the temperatures are generated internally and thus cannot be related to a fixed external temperature, a detailed understanding of the heat development during autogenous curing is important.

OBJECTIVES

The objectives of this investigation were as follows:

- 1. To supplement the current understanding of the thermal behavior of concrete subjected to high curing temperatures developed during autogenous curing typical of that being utilized for rapid strength determination. (3, 4, 5)
- 2. To examine the influence that variables such as cement type, cement factor, water-cement ratio, and admixtures have on the temperature development.
- 3. To examine the influence of initial mixture temperatures over a range from 50°F to 90°F on the temperature and total heat developed during curing and the compressive strengths attained.

BACKGROUND ON THERMAL PROPERTIES OF FRESH HYDRAULIC CEMENT CONCRETE

Thermal Properties

The thermal properties of concrete are important because of the transfer of heat between concrete and the surrounding environment. This transfer of heat results in large temperature changes which can be either beneficial or detrimental to the physical and chemical properties of concrete.

In general, an increase in curing temperature increases the rate and extent of cement hydration. In the presence of adequate moisture, a rise in curing temperature accelerates the chemical reactions and results in increased early strength; however, some investigators (6,7) have shown that while higher temperatures during curing increase early strength they have a detrimental effect on the strength after about 7 days. This detrimental effect on strength at later ages can be explained in terms of the aluminate and silicate reactions. In general, there are two ways in which early curing temperatures will affect the properties of the hydrated cement. (6)

The existence of a sulfate content for cement at which strength and other important properties are optimized is well established. $^{(8)}$ As the concrete curing temperature rises, the setting process is accelerated and the early strength is increased. This increased rate of reaction requires, for the proper retardation of the C_3A , that the solubility of gypsum be increased beyond that necessary for the proper setting of concrete at normal moist curing temperatures $^{(9)}$. Nevertheless, the solubility of gypsum decreases rather than increases $^{(9,10)}$ with increasing temperature. Brown $^{(9)}$ showed that the solubility of gypsum reaches a maximum at approximately 100^{0} F and begins to decrease rapidly at approximately 150^{0} F. Thus for curing at higher than normal temperatures the cement behaves as one below its optimum with resultant reduced strength.

Temperature also affects the extent of hydration of both the alumina and silica compounds. Verbeck⁽⁶⁾ has studied the effect of temperature on the degree of hydration of C₂S and C₃S, and his results are shown in Figure 1. This figure shows the degrees of hydration at ages from 1 day to 5 years when these compounds are hydrated at different initial temperatures. At the age of 1 day, the higher the hydration temperature, the higher the degree of hydration; but at later ages, the opposite is true. The rapid hydration produced by the high temperature tends to form a thick dense layer, or shell, around the cement grains, thus retarding the subsequent hydration; this shell also accounts for part of the reduction in strength at later ages. There is evidence⁽⁶⁾ that the rapid hydration reactions also cause a nonuniform distribution of the hydrated cement within the gel structure, which retards full strength development.

Thus the technical literature indicates that there will be optimum curing temperatures above which the accelerated or early strength development will decrease. Recent results from studies of accelerated curing substantiate this fact. (1,2,11,12)

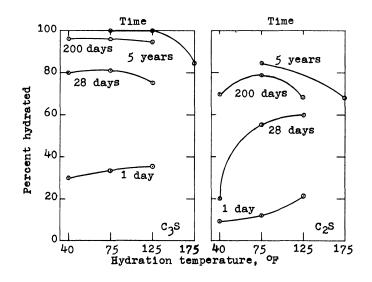


Figure 1. Effect of temperature on degree of hydration of C₂S and C₃S. (From reference 6.)

Adiabatic Temperature Rise

Heat is produced by the chemical hydration of the cement and, because of the mass of concrete, the resulting heat energy is unable to leave the paste. The result is an adiabatic temperature rise in mass concrete.

The property of adiabatic temperature rise is a function of the amount of heat generated, which is dependent upon the initial mixture temperature, the type of cement (i.e., heat of hydration), the cement content, the water-cement ratio, and the admixtures used. (13, 14)

In research conducted at the University of California, ⁽¹⁵⁾ it was shown that for adiabatically cured concretes the values for thermal conductivity, specific heat, and calculated diffusivity varied with curing temperature and time in a manner substantially different from that for identical concretes cured at 70°F and 100 percent relative humidity.

Knight and Johnson, ⁽¹⁵⁾ at the University of California, found that for adiabatically cured concrete sealed in metal casting forms, the relationship between specific heat and mean temperature for normal moist cured concrete did not hold true. The results of their work are shown in Figure 2.

If the adiabatic temperature is known, the heat of hydration of the cement paste may be computed for any time or corresponding temperature by using the following expression:

$$H = \frac{T_{ad}S}{1.8C}$$

where:

H = Heat of hydration of cement paste, calories/gram.

T_{ad} = Adiabatic temperature, ^oF

S = Specific heat, Btu/lb/OF

C = Proportion of cement in concrete by weight

The calculated relationship plotted in Figure 2 between specific heat and adiabatic curing temperature was derived from the following expression:

$$S = \frac{0.173H}{\Delta T_{ad}}$$

where:

 $S = Specific heat, Btu/lb/^{O}F$

H = Heat of hydration of cement, calories/gram.

 $\Delta T_{ad} = Adiatatic temperature rise, {}^{O}F$

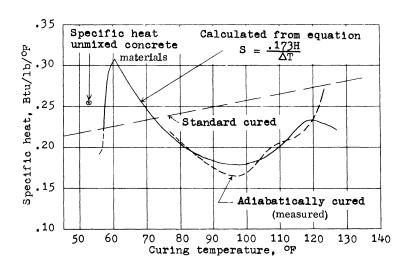


Figure 2. Effect of curing temperature on specific heat of concrete for adiabatically cured concrete. (From reference 15.)

In general, if heat is applied either externally or internally at the center of a concrete cylinder, a temperature gradient will develop between the outer surface and the center of the cylinder. The steepness of this temperature gradient depends primarily on the temperature differential between the center and outer surface of the cylinder.

Typical thermal gradients resulting from steam curing a concrete cylinder, as reported by Hanson, $^{(16)}$ are shown in Figure 3. The cylinders were cast in 5/16-inch thick steel molds and were allowed to cure for 1 hour at 76° F prior to steaming. The cylinders were then subjected to different rates of temperature rise as shown in Figure 3. After a steaming period of 50 minutes at temperatures necessary to cause a rate of temperature rise of 80° F per hour, the temperature differential was about 33° F, whereas it took 2 hours of steaming at a rate of rise of 20° per hour to establish a temperature differential of 8° F.

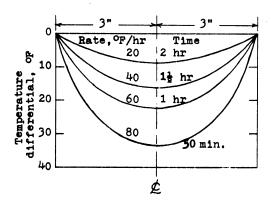


Figure 3. Temperature gradients in fresh concrete cylinder under steam curing conditions. (From reference 16.)

For rates of temperature rise below approximately 10°F per hour, the temperature differential was not significant, and for adiabatically cured cylinders the temperature differential was very near zero. A pilot study was made during this investigation to determine the temperature gradients for low rates of heat loss, and the results are discussed in Appendix A.

In autogenous accelerated curing, the accelerated process is achieved through an adiabatic temperature rise generated by the concrete's own heat of hydration. There is little open air space around the cylinder; therefore, there is no medium available to absorb the heat generated by the concrete. Consequently, the transfer of heat, which results in detrimentally high temperature gradients, does not exist.

The normal parameters involved in describing heat flow, i.e., thermal conductivity and specific heat, are not adequate for describing the thermal behavior for adiabatically cured concrete. The rate of temperature rise and the total heat generated for adiabatic curing can best be explained through the use of temperature-time curves and the integration of the area under the temperature-time curves, respectively.

Temperature has important influences not only on the rate of chemical reactions during hydration but also on the movements of moisture and the resulting humidities.

Water retention is necessary because hydration of cement takes place only in the water filled capillaries, and the cement gel can form only when sufficient water is available both for the chemical reactions and for the filling of the gel pores being formed.

The purpose of curing is to maintain the concrete in a saturated, or nearly saturated, condition until the originally water-filled space in the fresh cement paste has been replaced to the desired extent by the products of cement hydration.

Complete discussions of the methods and results of moisture measurements made in this experiment are included in Parts II and IV of this report. (17, 18)

EXPERIMENTAL TEST MATERIALS AND PROCEDURES

Variables

Table I lists the concrete mixture variables investigated in this study to determine their influence on temperature development for the autogenous accelerated curing method. The air content was held constant at 5.5 ± 0.5 percent for all mixtures.

TABLE I

CONCRETE VARIABLES INVESTIGATED

Initial mix temperatures 50°F, 70°F, 90°F

Heats of hydration Low, medium, high

Cement factors 450 lb/cu yd, 550 lb/cu yd, 650 lb/cu yd

Water cement ratios 0.4, 0.5, 0.6

Admixtures accelerator, retarder

Cements

In order to provide a range of heat liberation during curing, three types of portland cement were used in this experiment. One of the three cements met each of the specifications for types II, III, or V portland cement as set forth in ASTM C 150-68. (19)

Heat evolution tests were conducted by the Portland Cement Association on all three cements at temperatures of 50°F, 70°F, and 90°F using a conduction calorimeter, (20) and the results of these tests are shown in Figures 4, 5, and 6. A more detailed discussion of the cements used is presented in Part I of this report. (2)

A discussion of the aggregates, admixtures, and water used is presented in Appendix B.

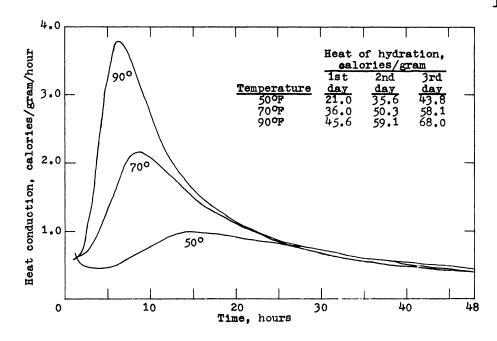


Figure 4. Rate of heat evolution plotted against time for type V cement and a water-cement ratio of 0.4.

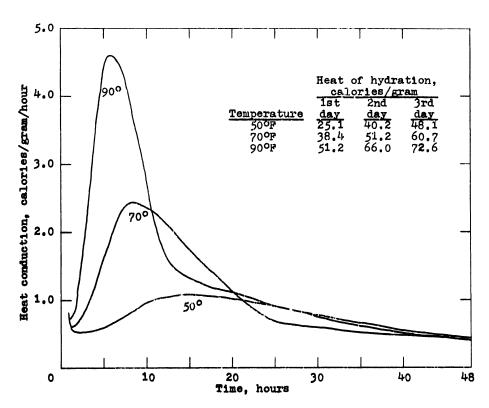


Figure 5. Rate of heat evolution plotted against time for type II cement and a water-cement ratio of 0.4.

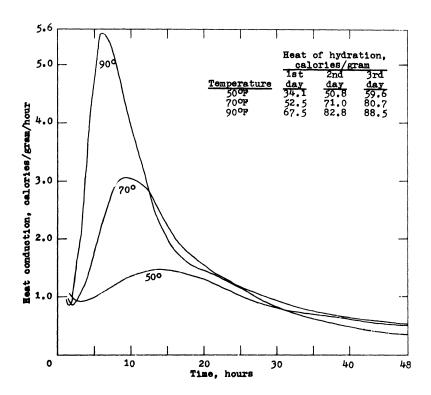


Figure 6. Rate of heat evolution plotted against time for type III cement and a water-cement ratio of 0.4.

Concrete Mixture Design

The recommended practice for selecting proportions for concrete (ACI 613-54)⁽²¹⁾ was used for the design of all mixtures. The concrete mixture schedules describing the three phases of the experiment are presented in Appendix C.

Mixing and Testing Procedure

Mixing and testing were in accordance with ASTM C 192. Mixing was accomplished in a Lancaster pan mixer as follows:

- 1. Fine aggregate and cement were placed in the mixer and mixed for 30 seconds.
- 2. Water was added to mixer and mixed for 1 minute.
- 3. Coarse aggregate was added to mixer and mixed for 2 minutes. Thus the total initial mixing time was $3\frac{1}{2}$ minutes.

When a retarding or accelerating admixture was used, part of the mixing water contained the retarder or accelerator and the remaining water contained the air-entraining admixture.

Immediately after initial mixing, the temperature of the concrete was recorded, then tests for slump, air content, and density were conducted in accordance with ASTM C 143, C 231, and C 138, respectively⁽¹⁹⁾ If the results of these tests did not meet the requirements previously established for the project the batch was discarded. Provided the requirements were met, the concrete from the slump test was returned to the mixer and the concrete was remixed for 1 minute. Concrete used in the air test was always discarded.

All cylinders were made according to ASTM C 192 procedures. The two cylinders from each batch for autogenous curing were cast simultaneously so that fluctuations in temperature would be the same for both cylinders. The cylinders designated for moist curing were stored in a moist room meeting the requirements of ASTM C 511 and were cured according to ASTM C 192 procedures.

The cylinders for autogenous curing were formed in metal single-use molds, sealed with metal lids, and then placed inside three plastic bags. The plastic bags were used to ensure no moisture loss, to reduce further the volume of air between the cylinder and inner lining of the autogenous container, and to act as a handle for removing the cylinder from the autogenous container.

The cylinder, in the plastic bags, was lowered into the autogenous container, and the plastic bags were sealed with a wire tie. As soon as the plastic bags were sealed, the autogenous container was closed and sealed with its lever-lock ring. All autogenous curing containers were sealed in less than 30 minutes after time ZERO* and were then stored in a room in which the ambient temperature was the same as the initial mix temperature of the concrete. For Phase III, as described in Appendix C, the room temperature was kept at 50° F, 70° F, or 90° F, to correspond to the respective initial mixture temperature.

The autogenous cylinders were cured for 47 hours, then removed from the containers and allowed to cool at room temperature for 1 hour before capping. The cylinders were capped at 48 hours and tested in compression at 49 hours, according to ASTM C 617 and C 39 procedures, respectively. At the prescribed time of test, the standard moist cured cylinders were removed from the moist room, capped, and tested according to ASTM C 617 and C 39.

^{*}Time "ZERO" is the time at which the mixing water is added to the cement.

Development of Temperature and Maturity Data

Temperatures in the autogenously cured specimens were recorded automatically for two cylinders from the same batch of concrete for each of the 48 mixtures. Temperatures recorded during the experiment ranged from 53°F to 159°F.

The maximum temperature differential between any two temperature curves representing the same concrete batch ranged from $0.25^{\circ}F$ to $2.00^{\circ}F$, with a differential of $3.00^{\circ}F$ occurring twice. Since the average differential was $1.33^{\circ}F$; or $\pm 0.67^{\circ}F$ of the average temperature, only one of the curves resulting from each mixture was analyzed.

A typical temperature-time curve is shown in Figure 7. The following parameters, designated in Figure 7, were derived from each temperature curve:

A = Maximum autogenous temperature, ^oF

B = Autogenous temperature increase $(+\Delta T)$, ${}^{o}F$

C = Autogenous temperature decrease ($-\Delta T$), ^{O}F

D = Time to maximum temperature, hours

E = Initial rate of temperature rise, ^oF per hour

F = Average time of initial rate of temperature rise, hours

G = Final rate of temperature rise, ^oF per hour

H = Average time of final rate of temperature rise, hours

I = Change from initial to final rate of temperature rise, hours

J = Rate of temperature rise between initial and final set, ^oF per hour

K = Total heat generated, i.e., measured area, OF x hr.

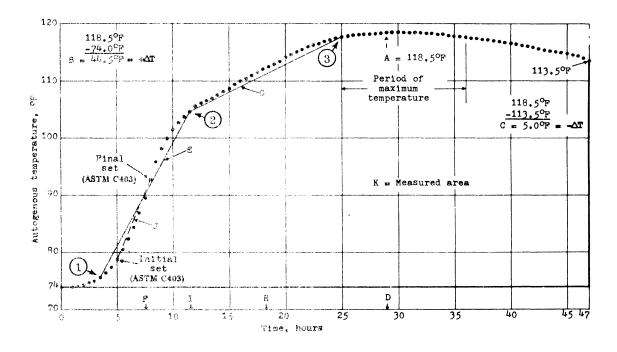


Figure 7. Typical temperature-time curve for autogenously cured concrete cylinder.

The beginning point (1) for determining the initial rate of temperature rise was established after observing the behavior of all temperature curves. It took approximately 3 hours for the temperature to rise 1°F; only 1/2 hour was required for the next 1°F temperature rise, which was an increase of 600 percent over that for the initial 3 hours. Therefore, point (1) was defined as the first plotted datum point occurring after the first 1°F temperature rise. Point (2) was determined by constructing tangents to the curve (tangents are not shown) and bisecting the interior angle formed by the tangents. Point (3) is designated as the beginning of the "period of maximum temperature" and was established after examining the behavior of the temperature curves in the vicinity of maximum temperature. The period of maximum temperature was defined as follows:

- 1. All data points having the value of maximum temperature.
- 2. All data points included in the 5-hour time intervals immediately preceding and following the maximum temperature data points, unless a 1°F temperature drop occurs within either of these 5-hour time intervals. If the 1°F temperature drop occurs at any time within either 5-hour interval, it is at that time that the period of maximum temperature begins and/or ends.

The initial and final rates of temperature rise are the average rates of temperature rise between points 1 and 2, and between points 2 and 3, respectively. A measure of the total heat generated during autogenous curing was determined by integrating the area bounded by the temperature curve on the left and top, by the 74° F temperature line on the bottom, and by the 47-hour line on the right. This measured area converted to $^{\circ}$ F x hours is a measure of the total heat generated.

The maturity, expressed in ^oF x hr, was determined for various combinations of time and temperature. The strength of concrete depends on both age and temperature and is therefore a function of

$$\sum (\text{Time x temperature}) = \sum (\text{Time x } \Delta T)$$

which is called maturity. The datum temperature from which ΔT is determined has been found experimentally $^{(22,23)}$ to be 11°F. At temperatures between the freezing point of water and 11°F, concrete will show a small increase in strength with time.

If the curing temperature varies during the curing period, then maturity is the integral of time and temperature expressed as a summation. The maturity relationship should be used with caution since it has been found (24) that changes in curing temperatures during the curing cycle drastically affect the results.

Maturities were determined for the following limiting combinations of temperature and time:

- 1. (Maximum autogenous temperature) x (Time to maximum temperature).
- 2. (Autogenous temperature increase) x (Time to maximum temperature).

- 3. (Total heat generated, i.e., measured area (in 2) above initial-mixture-temperature line) x 50° F x hr.
- 4. (Total heat generated above initial-mixture-temperature line) + (Initial mixture temperature 11°F) x 47 hours. This quantity is designated as the total maturity.

Analyzing Temperature and Maturity Data

The analysis of the temperature and maturity data was quite complex. A total of 5 mixture variables and 25 parameters were examined. Table II is a listing of these variables and parameters. For the three phases (Appendix C), more than 600 relationships for the 30 variables and parameters were investigated, and only those relationships showing a high degree of correlation or having a noticeable influence on the temperature and strength of autogenous cured concrete are discussed in the remainder of this report. Because many of the variables are interrelated, the discussion of results includes an evaluation of strength development as influenced by autogenous curing parameters. A full discussion of strength relationships was presented in Part I of this report. (2)

TABLE II

LIST OF VARIABLES AND PARAMETERS INVESTIGATED

Variables

Cement type	II, III, V
Cement content	450, 550, 650 lb/cu yd
Water cement ratio	0.4, 0.5, 0.6
Admixtures*	Accelerator and retarder
Initial mixture temperatures (IMT)	50 ^o F, 70 ^o F, 90 ^o F

^{*}Air content was constant at 5.5 ± 0.5 percent.

Parameters

Maximum autogenous temperature (T), ${}^{O}F$ Autogenous temperature increase ($+\Delta T$), ${}^{O}F$ Autogenous temperature decrease ($-\Delta T$), ${}^{O}F$ Initial rate of temperature rise, ${}^{O}F$ per hour Final rate of temperature rise, ${}^{O}F$ per hour Average time of initial rate of temperature rise, hours Average time of final rate of temperature rise, hours Change from initial to final rate of temperature rise, hours Time to maximum autogenous temperature, hours

TABLE II (Continued)

Time of initial set, hours
Time of final set, hours
Rate of temperature rise between initial and final set, ^oF per hour
Total heat generated (measured area), ^oF x hours
Autogenous cylinder strength, psi
28-day strength, psi
91-day strength, psi
28-day efficiency (Autogenous strength 28-day strength 100), percent
91-day efficiency (Autogenous strength 91-day strength 100), percent
Unit weight of concrete, pounds per cubic foot

Four maturity combinations as discussed on page 11 Logarithm of the four maturity combinations

RESULTS

Figure 8 shows three temperature-time curves representing low, medium, and high maximum autogenous temperatures which are typical of the data obtained. No movement of moisture between the top and bottom of the cylinders was detected, as described in Part IV of this report. (18) The absence of moisture movement indicates that the temperatures developed within the cylinders were fairly uniform.

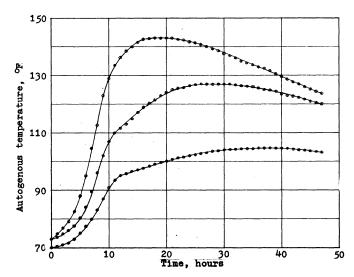


Figure 8. Typical temperature-time curves for autogenously cured concrete.

This fact was further substantiated by the results of the pilot study on thermal gradients as described in Appendix A. For Phases I and II (Appendix C), the range of temperature increase ($+\Delta T$) above initial mixture temperature was 35°F to 70°F, and the maximum autogenous temperature obtained ranged from 105°F to 143°F. Table III shows the temperature results from Phase III.

TABLE III
TEMPERATURE RESULTS FROM PHASE III

Initial mix temperature or	Maximum autogenous temperature, or	Temperature increase, or
50	Low 83 High 124	29 68
70	Low 113 High 140	41 69
90	Low 126 High 159	37 66

Influence of Mixture Variables on Temperature Parameters

The curves shown in presenting the influence of mixture variables on temperature parameters represent the average values for each measured parameter. In Figures 9 through 16 each datum point represents the average value for four cylinders where cement content is a variable and the average value for six cylinders where water-cement ratio or admixture is the variable. The influence of initial mixture temperatures (IMT) on the temperature parameters is discussed in the next section.

Figure 9 shows the influence of cement type, cement content, water-cement ratio, and admixtures on the maximum autogenous temperature. The admixtures and water-cement ratio had negligible effects on maximum temperature as compared with the effect of cement content and cement type.

The influence of the mixture variables on the initial rate of temperature rise is presented in Figure 10. As the water-cement ratio increased, the initial rate of temperature rise decreased for Type III cement. For Type II cement the initial rate of temperature rise decreased from W/C 0.4 to 0.5 but remained the same for W/C 0.5 to 0.6. The introduction of an accelerator to the mixture resulted in an increase in the initial rate from 41 to 49 percent (type II and III cements, respectively) over the rate for air entrainment only, whereas the use of a retarder reduced the rate by only 3 percent. As the cement content increased, the initial rate of temperature rise also increased.

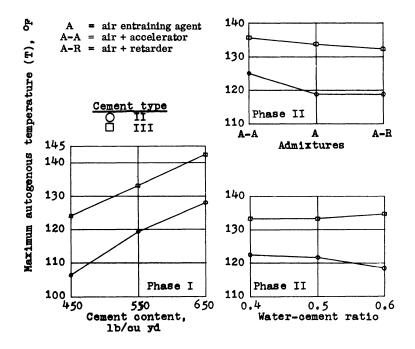


Figure 9. Influence of mixture variables on maximum autogenous temperature.

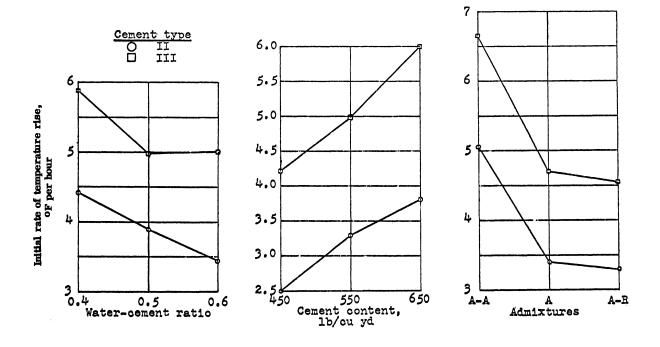


Figure 10. Influence of mixture variables on initial rate of temperature rise.

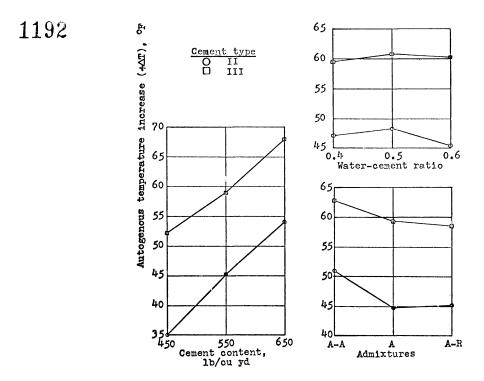


Figure 11. Influence of mixture variables on autogenous temperature increase (+ ΔT).

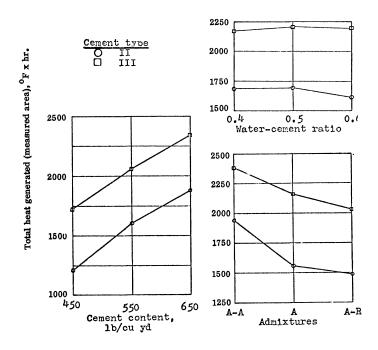


Figure 12. Influence of mixture variables on total heat generated.

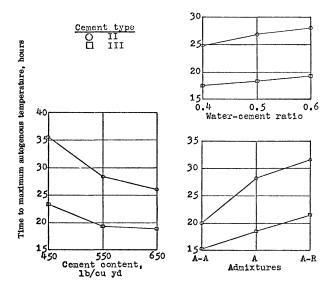


Figure 13. Influence of mixture variables on time to maximum autogenous temperature.

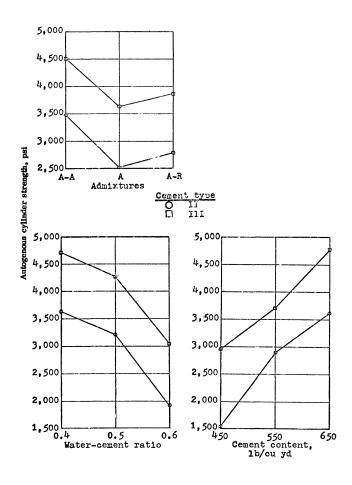


Figure 14. Influence of mixture variables on autogenous cylinder strength.

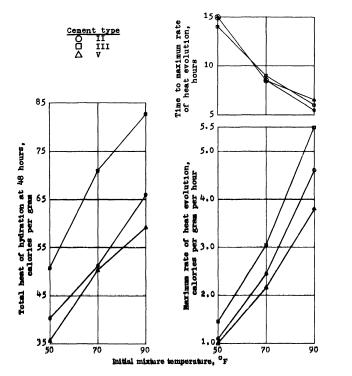


Figure 15. Results of heat of hydration tests conducted on paste made from each of the three cement types at initial mixture temperatures of 50°F, 70°F, and 90°F by Portland Cement Association; Data taken from Figures 4, 5, and 6.

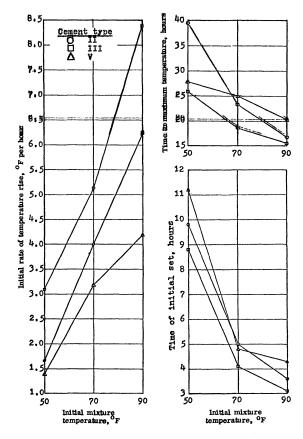


Figure 16. Influence of initial concrete mixture temperature on initial rate of temperature rise, time of initial set, and time to maximum temperature.

The influence of the mixture variables on the autogenous temperature increase is shown in Figure 11. The water-cement ratio had little effect on the temperature increase ($+\Delta T$) as compared with the effect of cement content and cement type. The admixtures affected the temperature increase ($+\Delta T$) as expected. The retarder affects this parameter in the same manner as air alone, i.e., the retarder delays the chemical reactions of the cements but does not substantially alter them.

Figure 12 shows the influence of the mixture variables on the total heat generated expressed in terms of the measured area under the temperature curve in ^oF/hr. Changes in the water cement ratio had little effect on the total heat generated, whereas admixtures and cement content caused large changes.

Figure 13 shows the influence of the mixture variables on the time required to reach the maximum autogenous temperature. The time to maximum autogenous temperature increased as the water-cement ratio increased and as the mixture was retarded by the presence of admixtures. Increases in cement content resulted in a decrease in the time to maximum temperature. These results would be expected since a decrease in time corresponds to an increase in the rate of cement hydration.

The influences of the mixture variables on the autogenous cylinder strengths are shown in Figure 14. These have been discussed in detail in Part I of this report. (2) As the cement content increased and the water-cement ratio decreased, the strength increased for cement types II and III. The effects of admixtures on strength is the same for both types of cements, the only difference being that the strengths for type III cement are approximately 1,000 psi higher than those for type II cement.

Influence of Initial Mixture Temperatures on Temperature Parameters

Figure 15 shows the relationship between IMT, cement type, and heat of hydration values as determined by the Portland Cement Association for the three cements used in this investigation. As the IMT was increased, the maximum rate of heat evolution (calories per gram per hour) increased for all three cements. This relationship in Figure 15 is very similar to that of Figure 16, which indicates the close relationship between rate of heat evolution of the cement and the initial rate of temperature rise of the concrete. This close relationship supports the validity of the method used to determine the initial rate of temperature rise.

Figure 15 also shows that as the IMT increases, the maximum rate of heat evolution is reached at an earlier age. The time differential at which the maximum rate is reached is less than 1 hour for all three cements at any particular IMT. The relationship between the times for the various IMT's is closely related to the time to maximum autogenous temperature and to the time of initial set, as shown in Figure 16.

Figure 15 further shows that as the IMT increases the total heat of hydration (calories per gram) at 48 hours increases and that the total heat evolved for the type III cement is considerably higher than it is for either the type II or type V cements. The relationship between these curves is very similar to the corresponding curves shown in the remaining figures of this section.

In order to better illustrate the influence of the IMT's on the temperature parameters and ultimately on the measured strengths, selected temperature parameters have been plotted against IMT and are shown in Figures 17 through 21.

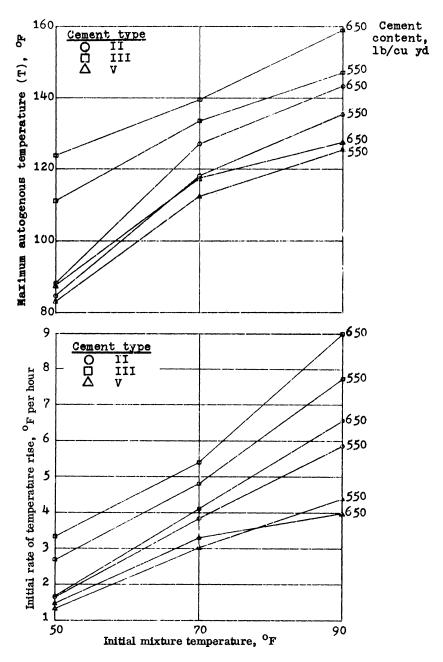


Figure 17. Maximum autogenous temperature and initial rate of temperature rise plotted against mixture temperature.

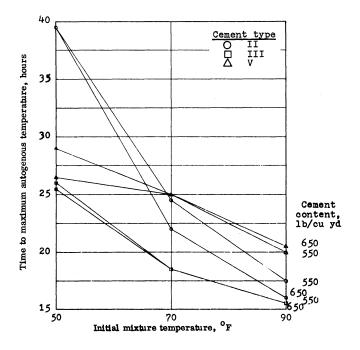


Figure 18. Relationship between time to maximum temperature and initial mixture temperature.

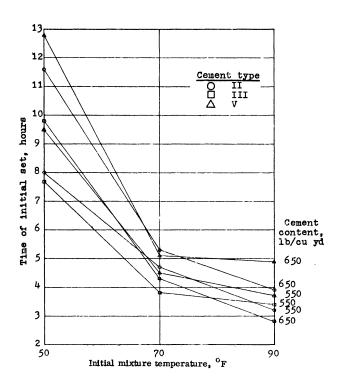


Figure 19. Relationship between time of initial set and initial mixture temperature.

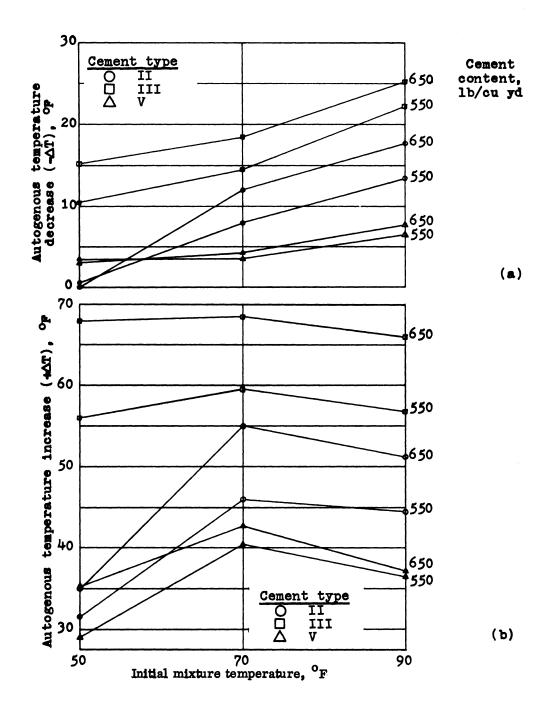


Figure 20. Autogenous temperature decrease and autogenous temperature increase plotted against initial mixture temperature.

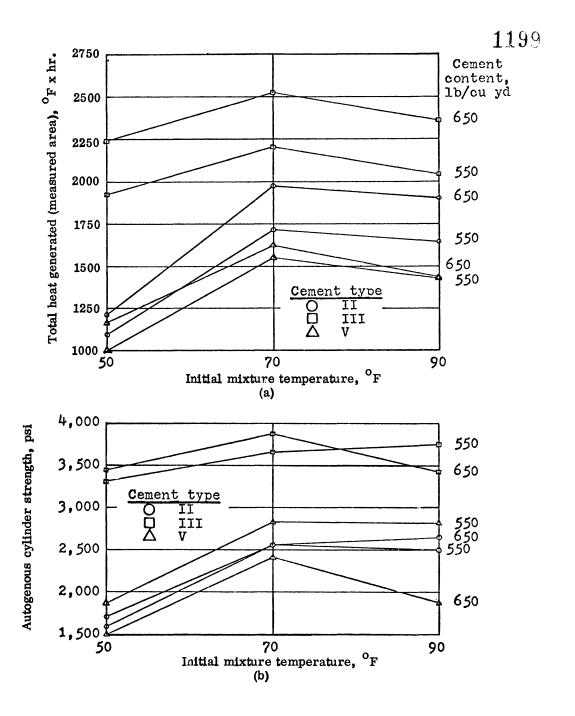


Figure 21. Total heat generated and autogenous cylinder strength plotted against initial mixture temperature.

Figure 17 demonstrates that as the IMT is increased, the maximum autogenous temperature and the initial rate of temperature rise increase. This behavior is expected since the application of heat to curing concrete acts as a catalyst to increase the rate of hydration and since for any IMT between 11°F and 100°F the temperature of the concrete will rise as long as sufficient water is available for cement hydration.

As the IMT is increased, hydration, and therefore the rate of temperature rise, increases; consequently, as seen in Figure 18, the time to reach the maximum autogenous temperature decreases. In this figure, as in the other figures of this group, the general influence of the IMT on the temperature parameters was more pronounced for the type II cement.

The time to reach maximum autogenous temperature (Figure 18) correlates very well with the time of initial set shown in Figure 19. The greatest influence of the IMT on time of set occurs between $50^{\circ}F$ and $70^{\circ}F$. For initial set, the time is reduced approximately 2.5 times from an IMT of $50^{\circ}F$ to $70^{\circ}F$, whereas the reduction is only 1.25 times from an IMT of $70^{\circ}F$ to $90^{\circ}F$.

The effect of IMT was such that for an IMT of 50° F the time of final set (not shown), for the three cements tested, ranged from 10.8 hours to 17.2 hours and for IMT's of 70° F and 90° F the times ranged from 4.4 hours to 7.2 hours.

Figure 20 (a) shows that as the IMT increases, the autogenous temperature decrease (- ΔT) increases, and Figure 20 (b) that the autogenous temperature increase (+ ΔT) increases from an IMT of $50^{\rm O}F$ to an IMT of $70^{\rm O}F$, but that the temperature increase (+ ΔT) decreases slightly from an IMT of $70^{\rm O}F$ to $90^{\rm O}F$. The reasons for this decrease in temperature increase (+ ΔT) are probably that changes in hydration temperature affect the optimum gypsum content and the rapid hydration induced by the high temperature causes the encapsulation of the C₂S and C₃S cement grains with a dense zone of hydration product, thus retarding the subsequent hydration as discussed earlier. The effect of initial mixture temperature on temperature increase (+ ΔT) was greater for cement types V and II than for type III.

The total heat generated also increases when the IMT is increased from $50^{\rm O}{\rm F}$ to $70^{\rm O}{\rm F}$ and then decreases from $70^{\rm O}{\rm F}$ to $90^{\rm O}{\rm F}$, as shown in Figure 21(a). In fact, the relative positions of the six curves to each other in Figure 21(a) are almost identical to those of the curves in Figure 20(b). The relationship between autogenous temperature increase (+ $\Delta{\rm T}$) and total heat generated was highly significant as shown in Figure 29 (page 29).

Since the total heat generated by the concrete is a measure of the degree of completeness of cement hydration, which in turn controls the development of concrete strength, the resulting relationship between the autogenous strength and the IMT is shown in Figure 21 (b). At some IMT between 70°F and 90°F, the strength development is either retarded or reaches a point at which no further gain in strength is realized as the IMT increases. This retardation of strength phenomenon is discussed in detail in Part I of this report. (2) The strengths of the types II and V cements were approximately the same, with the type III cement having a strength approximately 1,000 psi greater than the types II or V cements after 47 hours of autogenous curing.

Temperature Relationships

The relationships among the temperature parameters, without regard to the mixture variations, are of some interest and are given in Figures 22 through 31. Each plotted value in Figures 22 through 31 is the average value of two data points.

Figure 22 shows the relationship between maximum autogenous temperature and initial rate of temperature rise. The rate of temperature rise between 1°F per hour and 3°F per hour, which occurred with an IMT of 50°F, was associated with a greater change in the maximum autogenous temperature than when the rate of temperature rise was between 3°F per hour and 9°F per hour which occurred with IMT values of 70°F and 90°F. This effect results from the extended length of time for the maximum temperature to be reached for an IMT of 50°F, as shown in Figure 23. This figure presents the relationship between maximum autogenous temperature and time to maximum temperature. The overlap of the rate of temperature rise and the time to maximum temperature, Figures 22 and 23 respectively, for the three initial mixture temperatures results from the variations in mixture variables as discussed earlier. Generally, as the maximum autogenous temperature increases, the time to maximum temperature decreases. This would always be the case so long as the total heats evolved by the cements are approximately the same. The ranges in times to maximum temperature were as follows:

Initial mixture temperature, oF	Range of time to maximum temperature, hours		
50	25.5 - 39.5		
70	14.0 - 36.0		
90	15.5 - 20.5		

A change in the IMT was directly reflected in the relationship between maximum autogenous temperature and autogenous temperature increase ($\pm\Delta T$), as shown in Figure 24. For an IMT of $70^{\circ}F$ the temperature increase ranged from $35^{\circ}F$ to $70^{\circ}F$ with a maximum autogenous temperature differential of $\pm3^{\circ}F$. The maximum autogenous temperature difference between the IMT curves of $50^{\circ}F$ and $90^{\circ}F$ was $37^{\circ}F$, which reflects the 40 degrees difference in the IMT. For the three IMT's the relationships are given by a family of parallel straight lines and the correlation of data for each is highly significant.

The relationship between maximum autogenous temperature and total heat generated is identical to that shown in Figure 24. Because each of the three parameters (maximum autogenous temperature, rate of temperature rise, and total heat generated) are closely related, any one of the three can be utilized to characterize the time-temperature curve. Since maximum autogenous temperature is the easiest to measure, it is perhaps the most significant.

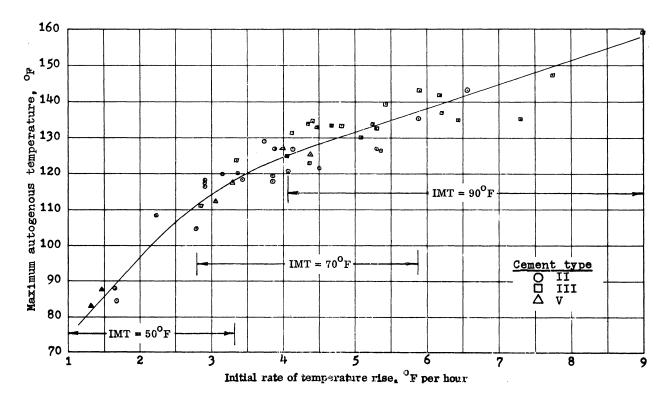


Figure 22. Relationship between maximum autogenous temperature and initial rate of temperature rise for all initial mixture temperatures.

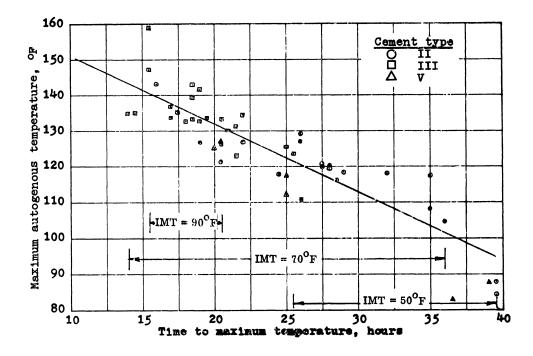


Figure 23. Relationship between maximum autogenous temperature and time to maximum temperature for all initial mixture temperatures.

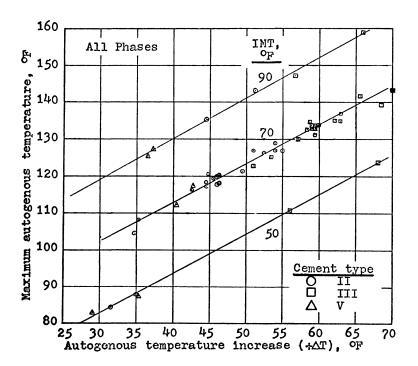


Figure 24. Relationship between maximum autogenous temperature and autogenous temperature increase ($+\Delta T$) for all initial mixture temperatures.

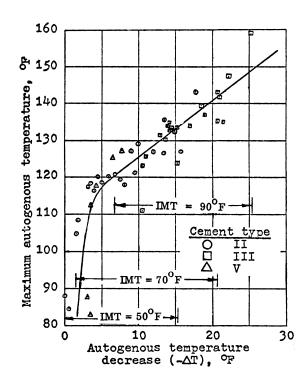


Figure 25. Relationship between maximum autogenous temperature and autogenous temperature decrease ($-\Delta T$).

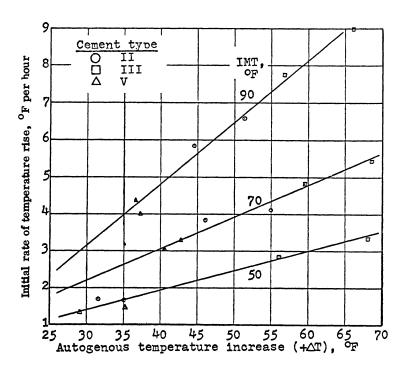


Figure 26. Relationship between initial rate of temperature rise and autogenous temperature increase for all initial mixture temperatures.

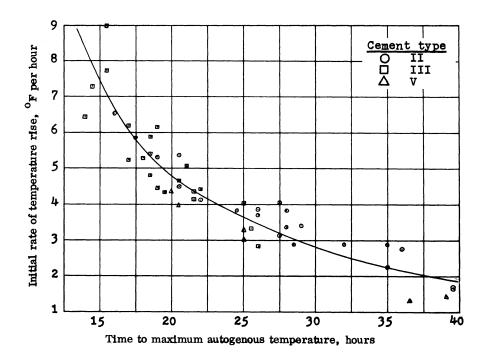


Figure 27. Relationship between initial rate of temperature rise and time to maximum temperature for all initial mixture temperatures.

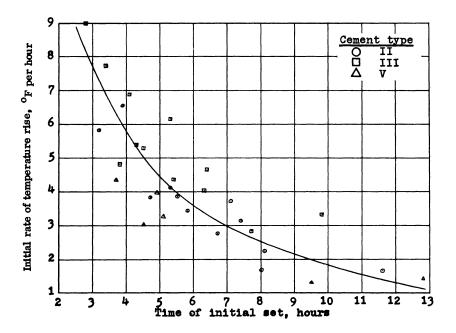


Figure 28. Relationship between initial rate of temperature rise and time of initial set for all initial mixture temperatures.

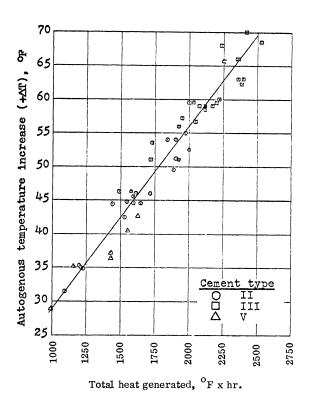


Figure 29. Relationship between autogenous temperature increase and total heat generated for all mixture temperatures.



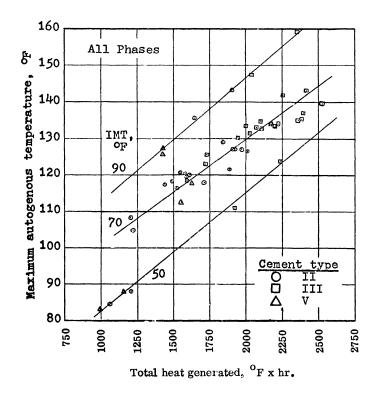


Figure 30. Relationship between maximum autogenous temperature and total heat generated for all initial mixture temperatures.

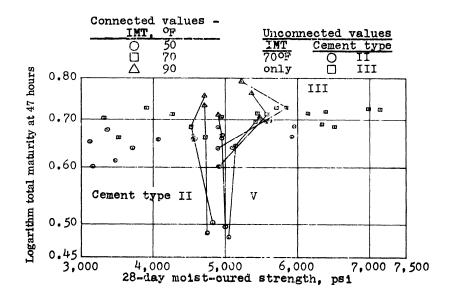


Figure 31. Relationship between logarithm of total maturity and 28-day strength for all initial mixture temperatures.

The relationship between maximum autogenous temperature and the subsequent autogenous temperature decrease (- ΔT) is shown in Figure 25. The temperature decrease (-ΔT) is negligible up to a maximum autogenous temperature of 115°F. Beyond this temperature the temperature decrease $(-\Delta T)$ increases rapidly. It is postulated that this phenomenon occurs because of changes in the specific heat of adiabatically cured concrete, as explained earlier. In Figure 2 it was shown that for temperatures between 80°F and 110°F the specific heat increases rapidly. At the low temperatures (80°F to 115°F), the rate of heat evolved from the concrete is low enough that the concrete-curing-container system is near temperature equilibrium by the time the maximum autogenous temperature is reached, which results in a low temperature decrease (- Δ T) in the concrete as the system temperature stabilizes. At temperatures above 115°F, the high specific heats increase the rate of heat evolved from the concrete. causing a lag in system temperature equilibrium and a larger temperature decrease $(-\Delta T)$ as temperature equilibrium is reached. It is also recognized that the autogenous containers are not perfect insulators, as evidenced by their heat retention curves shown in Part I of this report⁽²⁾, and that this fact is responsible for a minor portion of the heat loss. However, it is believed that the rate of heat loss would decrease considerably if the containers remained sealed for a longer period of time.

Figure 26 shows the relationship between the initial rate of temperature rise and the autogenous temperature increase ($+\Delta T$). As the rate of temperature rise increases, so does the autogenous temperature increase ($+\Delta T$). This relationship is affected by the IMT, as shown in Figure 26. The influence of the IMT on the rate of temperature rise becomes more pronounced with increasing IMT's.

Figure 27 demonstrates the relationship between the initial rate of temperature rise and the time to maximum temperature. This relationship was independent of the IMT as opposed to that shown in Figure 26. As the rate of temperature rise increases the time to maximum temperature decreases in the same manner as the heat of hydration curves developed for the cements and shown earlier in Figures 4, 5, and 6.

The relationship between the initial rate of temperature rise and the time of initial set is given in Figure 28. The curves in this figure and Figure 27 are generally the same, except for the shift in the time between the two curves. The data in Table IV, which were taken from these two curves, show that the time of initial set can be used to predict the time to reach the maximum autogenous temperature. The time of maximum autogenous temperature is four to five times the time of initial set.

Figure 29 presents the relationship between the autogenous temperature increase $(+\Delta T)$ and the total heat generated. The correlation between the temperature increase $(+\Delta T)$ and the total heat generated is highly significant. The main difference between this curve and that of Figure 30 is the influence of the IMT. The relationship in Figure 29 is independent of the IMT.

The maturity relationships discussed earlier were analyzed to determine their relationship to the temperature parameters. As a result of this study, it was concluded that the maturity relationships investigated for autogenously cured concrete were very poor.

TABLE IV

RELATIONSHIP BETWEEN TIME OF INITIAL SET AND TIME TO MAXIMUM TEMPERATURE FOR VARIOUS INITIAL RATES OF TEMPERATURE RISE

(Data taken from curves of Figures 27 and 28.)

Initial rate of temperature rise, ^O F per hr.	Time of initial set, hr.	Time to maximum temperature, hr.	Time of set Time of max. temp. (100), percent	Multiplier
2 3 4 5 6 7 8	9.4 7.0 5.5 4.5 3.9 3.3 2.9	37.75 28.75 23.25 19.25 17.00 15.50 14.25	24.9 24.3 23.7 23.4 22.9 21.3 20.4	4.0 4.1 4.2 4.3 4.4 4.7

The logarithm of the total maturity, as described earlier, did result in a straight line relationship when plotted against strength for an IMT of $70^{\circ}F$ for the data from Phases I and II. When Phase III data for IMT's of $50^{\circ}F$, $70^{\circ}F$, and $90^{\circ}F$ were plotted, the resulting straight line relationship was perpendicular to the straight line relationship for an IMT of $70^{\circ}F$, as shown in Figure 31.

OBSERVATIONS AND CONCLUSIONS

- 1. Test results indicate that the temperatures developed within the autogenously cured cylinders were uniform and temperature gradients were minimal.
- 2. For an initial mixture temperature of $70^{\rm O}F$ the range of temperature increase (+ ΔT) above the initial mixture temperature was $35^{\rm O}F$ to $70^{\rm O}F$ depending upon mixture variables.
- 3. The general order of influence of the mixture variables on the temperature parameters and strength is shown in Table V.

TABLE V

GENERAL ORDER OF INFLUENCE OF MIXTURE VARIABLES ON TEMPERATURE PARAMETERS AND STRENGTH

Refe Figure	r to Page	Temperature parameters and strength	Decreasing order of influence of mixture variables
			1 0 0 1 11
9	15	Maximum autogenous temperature (T)	1, 2, 3, and $4*$
10	15	Initial rate of temperature rise	3, 2, 1, and 4
11	16	Autogenous temperature increase (+ΔT)	1, 2, 3, and 4
12	16	Total heat generated	1, 2, 3, and 4
13	17	Time to maximum autogenous temperature	3, 2, 1, and 4
14	17	Autogenous cylinder strength	1, 4, 2, and 3
	2 = 3 =	cement content cement type admixture water-cement ratio	

- 4. There was a close relationship between the rate of heat evolution of the cement, as determined by the Portland Cement Association, and the initial rate of temperature rise of the concrete.
- 5. The general influence of the initial mixture temperature on the temperature parameters was more pronounced for the type II cement than for cement types III and V.
- 6. The influence of the initial mixture temperature was such that as the IMT changed from 50°F to 70°F to 90°F the maximum autogenous temperature and initial rate of temperature rise increased and the time to reach maximum autogenous temperature decreased.
- 7. The autogenous temperature increase ($+\Delta T$), the total heat generated, and the autogenous cylinder strength all increased when the IMT was raised from $50^{\circ}F$ to $70^{\circ}F$ but they then decreased when the IMT was raised from $70^{\circ}F$ to $90^{\circ}F$. The reasons for these decreases are probably that changes in hydration temperature affect the optimum gypsum content and the rapid hydration induced by the high temperature causes the encapsulation of the C_2S and C_3S cement grains with a dense zone of hydration product, thus retarding the subsequent hydration.

- 8. If the maximum autogenous strength was developed by the time the maximum autogenous temperature was reached it seems feasible, at least for the containers used in this experiment, that the total length of time for autogenous curing could be reduced by at least 8 hours. Since the longest time required to reach maximum temperature was 39.5 hours (Figure 23) the remaining 7.5 hours of a 47-hour curing period might be used as the period of time, depending on the working conditions, during which the cylinder may be removed from the autogenous container for strength testing without having any adverse effects on strength development.
- 9. Because each of the three parameters (maximum autogenous temperature, rate of temperature rise, and total heat generated) are closely related, any one of the three can be utilized to characterize the time-temperature curve. Since maximum autogenous temperature is the easiest to measure, it is perhaps the most significant.
- 10. The time of initial set can be used to predict the time to reach maximum autogenous temperature or vice versa. The time to reach maximum autogenous temperature is four to five times the time of initial set depending on the initial rate of temperature rise.
- 11. Maturity was analyzed to determine its relationship to the temperature parameters. As a result of this study, it was concluded that the maturity-temperature parameter relationships investigated for autogenously cured concrete were very poor.

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Temperature Monitoring

A pilot study was undertaken to determine the number of thermocouples needed in each cylinder to describe accurately the autogenous adiabatic temperature rise. Seven cylinders were made in metal single-use molds for investigation of various facets of temperature and moisture in concrete cylinders.

Two of these cylinders were instrumented with six thermocouples in each. The thermocouples were placed as shown in Figure 32. The initial concrete temperature was 67°F, and the tests were conducted at a room temperature of 72°F. The only difference between the two cylinders was that one cylinder was cured in still air while the other was cured in the draft created by a low speed fan at a distance of approximately 7 feet from the fan, which represented a more severe curing condition. Temperature data were recorded for 2 days, and the results showed an average differential of 0.9°F between the interior and exterior thermocouples in the draft cured cylinder. The range in differential temperatures was from 0°F to 2.5°F. The maximum concrete temperature was 80°F, and the maximum difference between the bottom and top thermocouples was 1.5°F. The temperature in the top of the cylinder was less than in the bottom, and the bottom temperature was less than that in the middle of the cylinder.

In the cylinder cured in still air, the maximum temperature differential between the interior and exterior of the cylinder was $0.2^{\rm O}F$, with a range from $0^{\rm O}F$ to $1^{\rm O}F$. Again, the maximum concrete temperature was $80^{\rm O}F$, and the maximum difference between the top and bottom of the cylinder was $1.5^{\rm O}F$. The temperatures in the top and bottom of the cylinder were equal but below the temperature in the middle of the cylinder.

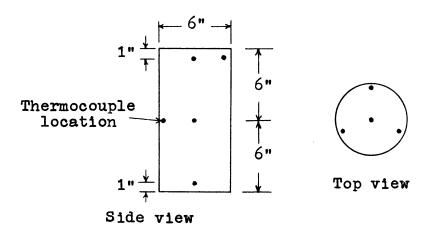


Figure 32. Locations of thermocouples in concrete cylinders used in temperature pilot study.

As a result of this pilot study, one copper-constantan thermocouple located in the center of the cylinder at a depth of 6 inches was used to record temperatures for the main investigation. The test results from the pilot study also showed that the temperature readings taken every hour would be sufficient; but since the temperature readings were recorded automatically, the interval was set for every half-hour.

All 96 thermocouples used in the actual experiment were tested under various controlled temperature conditions to check fabrication and variability. The maximum variability as recorded by a Honeywell multipoint temperature recorder was $\pm 1/4^{\circ}$ F.

APPENDIX B

Experimental Test Materials

Admixtures

The air-entraining agent was commercially marketed neutralized vinsol resin conforming to ASTM C 260. (19)

The retarding admixture was a water-reducing, retarding, nonair-entraining, metallic salt of hydroxylated carboxylic acid acid conforming to ASTM C 494, Type $D_{\bullet}^{(19)}$

Calcium chloride (CaCl $_2$) flakes, conforming to ASTM C 494, Type E and ASTM D 98 $_{\circ}$ Type 1, were used as the accelerator.

Water

The mixing water was from the Charlottesville, Virginia, water supply and was used at various temperatures as necessitated by the several initial mixture temperatures.

Aggregates

A crushed granite gneiss having a maximum size of 1 inch was used as coarse aggregate. The same type of aggregate, graded artificially, was used throughout the experiment. The coarse aggregate gradation was in conformance with ASTM standard C 136-67. (19)

The fine aggregate was a washed, natural silica sand conforming to ASTM standard C 33-67. (19) The same type and gradation of fine aggregate was used for the entire project.

A more detailed discussion of the experimental test materials is presented in Part I of this report. (2)

APPENDIX C

Mixture and Specimen Preparation Schedules

The experiment was divided into three phases. (25) Phase I was patterned after the ASTM Cooperative Testing Program (26) so that the results of this investigation could be related to the ASTM results. Phases II and III were designed such that the main variables were water-cement ratio and initial mix temperature, respectively.

The constants in Phase I were

Initial mixture temperature = 73° F

Slump = 2 to 3 inches

Air content = 5 to 6 percent

The mixture schedules for Phase I are given in Table VI.

TABLE VI
MIXTURE SCHEDULES FOR PHASE I

Type II cement			Type III cement		
Mix number	Cement, lb/cu yd	Admixtures*	Mix number	Cement, lb/cu yd	Admixtures*
I-a	450	A	I-g	450	A
I-b	450	A-R	I-h	450	A-R
I-c	550	A	I≕ <u>1</u>	550	A
I-d	550	A-R	I j	550	A-R
I-e	650	A	I⊶k	650	A
I-f	650	A⊸R	Il	650	A~R

^{*}A = Air-entraining agent; R = Retarder.

The constants in Phase II were:

Initial mixture temperature = 73° F

Cement factor = 550 lb/cu yd

Air content = 5 to 6 percent

The mixture schedules for Phase II are given in Table VII.

TABLE VII

MIXTURE SCHEDULES FOR PHASE II

Ty	Type II cement			e III cement	
Mix number	W/C ratio	Admixtures*	Mix number	W/C ratio	Admixtures*
II-a	0.4	A	II-j	0.4	A
II-b	0.4	A-R	II-k	0.4	A-R
II-c	0.4	A-A	II-1	0.4	A-A
II-d	0.5	A	II-m	0.5	A
II-e	0.5	A-R	II-n	0.5	A-R
II-f	0.5	A-A	II-o	0.5	A-A
II-g	0.6	A	II-p	0.6	A
II-h	0.6	A-R	II-q	0.6	A-R
II-i	0.6	A-A	II-r	0.6	A-A

^{*} A = Air only;

A-R = Air and retarder;

A-A = Air and accelerator.

The constants in Phase III were:

Water-cement ratio = 0.5

Admixture = Air only

Air content = 5 to 6 percent.

The mixture schedules for Phase III are given in Table VIII.

TABLE VIII
MIXTURE SCHEDULES FOR PHASE III

Initial			Cement	nt type		
mixture temperature	Mixture number	Cement, lb/cu yd	Mixture number	Cement, lb/cu yd	Mixture number	Cement, 1b/cu yd
50 ⁰ F	III-a III-b III-c	450* 550 650	III-d III-d	450* 550 650	III∞g III∞h III∽i	450* 550 650
73 ⁰ F	III.∞j III.∞k III.∞l	450* 550 650	III-m III-n III-o	450* 550 650	III=p III=r	450* 550 650
90 ⁰ F	III⊶s III-t III-u	450* 550 650	Ш-х Ш-х	450* 550 650	Ш-у Ш-г Ш-г1	450* 550 650

^{*}The mixtures having a cement factor of 450 lb/cu yd were so stiff (Slump = 0) that it was impossible to make properly formed cylinders. In order to use a cement factor of 450 lb/cu yd different aggregate gradations would have to be used and the mixtures redesigned. Since aggregate gradation was a constant, mixtures III-a, d, g, j, m, p, s, v, and y were not made.

The schedule for specimen preparation is shown in Table IX. There were three batches of concrete for each mixture. A more detailed discussion of the mixture and specimen preparation schedule is presented in Part I of this report. (2)

A total of 218 batches of concrete were made in the laboratory, of which 74 batches did not meet the air content or slump requirements established for the project.

Time of set tests were conducted for all mixes in Phases I and III. In both phases the test specimens were cured at ambient temperatures equal to the initial mixture temperature.

 $\begin{tabular}{ll} TABLE\ IX \\ SPECIMEN\ PREPARATION\ SCHEDULE \\ \end{tabular}$

Type of curing	Number of cylinders per batch			Total cylinders	
	Batch 1	Batch 2	Batch 3	per mixture	
Autogenous	2	2		4	
28-day standard*	2	2		4	
91-day standard	2	2		4	
1-year standard	2	2		4	
2-day standard			2	2	
Autogenous temperature**			2	2	
Time of set test***			-	-	
Totals	8	8	4	20	

^{*&}quot;Standard" means normal moist curing conditions.

^{**&}quot;Autogenous temperature" denotes cylinders instrumented for temperature measurements.

^{***}Cement-sand mortar for time of set test, in accordance with ASTM C 403, (19) was taken from the third batch of each mixture.