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Abnormal Stiffening of Concrete Containing Chemical Water-Reducing and Retarding Admixtures

Final Report

F. E. LEGG, JR.

November 1972

Michigan Department of State Highways
State Highways Building
Contract No. 71-1323
Lansing, Michigan

Department of Civil Engineering

T H E   U N I V E R S I T Y   O F   M I C H I G A N

COLLEGE OF ENGINEERING
Department of Civil Engineering

Final Report

ABNORMAL STIFFENING OF CONCRETE CONTAINING CHEMICAL WATER-REDUCING AND RETARDING ADMIXTURES

F. E. Legg, Jr.

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MICHIGAN DEPARTMENT OF STATE HIGHWAYS
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INTRODUCTION

Portland cement must necessarily be manufactured to have hardening properties in concrete suitable for average job conditions of mixing time, temperature, and consistency of the plastic concrete. Hot summer temperatures, low temperatures of the fall or winter, job delays, etc., sometimes make it desirable to modify the inherent concrete setting properties to better accommodate to job circumstances. Use of accelerating or retarding admixtures added during construction to compensate for specific field conditions is becoming widespread practice.

Retarding and water-reducing-retarding admixtures have been known for many years and were first designated as "dispersing agents." The use of retarding admixtures, as such, was first studied intensively by the U.S Bureau of Reclamation about 20 years ago and a comprehensive report was issued in 1955 (1). Significantly, even this first report called attention to the marked difference in behavior of the retarding admixtures with cements from different lots or different mills. Profound differences in setting time were noted for which there was no obvious explanation. To this day, the reasons for this anomalous behavior and means for predicting it are obscure. This is exemplified by two authoritative committee reports issued during 1971 calling attention to the necessity to test each combination of admixture with the job cement to determine their compatibility (2,3).

At the time of the first extensive examination of retarders by the U.S. Bureau of Reclamation, major interest centered around the desire to prevent "cold joints" between successive lifts or pours so as to insure watertight integrity of hydraulic structures. Use of retarder in the earlier pour enabled revibration so as to bond the partially hardened concrete with the newly placed concrete. Periodic Proctor penetration tests of mortar wet screened from the retarded concrete enabled assessment of the length of time during which consolidation by internal vibration was feasible. Screened mortar whose Proctor penetration attained 500 psi was considered to have reached the vibration limit. This has since been verified by others. A Proctor reading of 4000 psi indicated a concrete which had become so hard as to have reached "final" set and would have a strength if tested in the standard cylinder compression test of about 100 psi.

In the intervening years, retarders have been used to aid placement of concrete in bridge decks supported by steel continuous over two or more spans, not only to permit minor adjustment in deck elevation as dead load increased during construction but to alleviate cracking due to deflection of the partially hardened concrete in adjacent spans. Britton of the New York State Highway Department reported a large project using admixture retarded concrete for this purpose (4).

There was considerable uncertainty in the use of retarders to aid bridge deck placement, and research was undertaken resulting in NCHRP Report No. 106, "Revibration of Retarded Concrete for Continuous Bridge Decks" (5). One of the major findings of this research was that vigorous surface vibration could be successful in closing cracks in the partially hardened concrete if conducted before the concrete reached a penetration resistance (Proctor test on the screened mortar) of about 60 psi. Depending upon dosage, concrete temperatures, etc., this degree of set may be attained as soon as 1.5 hr after mixing during hot weather for unretarded concrete to as much as 6-8 hr, or more, for concrete with retarder if such delay in setting is desired. It should be emphasized that this degree of set is reached considerably earlier than the initial set, or "vibration limit" (500 psi pen.) employed in normal acceptance testing of chemical admixtures by ASTM Specification C 494, Chemical Admixtures for Concrete.

When using retarders in concrete, occasions have arisen where the expected retardation has not been achieved, and even acceleration of set has occurred, or other anomalous behavior has been encountered. Few of these cases have been well documented, usually because job circumstances simply did not permit getting all of the needed data. One case, however, was thoroughly reported (6). Reference 7 included as a part of Reference 8 reports extensive effort to link admixture behavior with the chemical composition of the cement. References 9, 10, and 11 and many others report similar studies. Such unusual, and unexpected, behavior in the construction of bridge decks in Michigan has led to the initiation of the present study.

INVESTIGATION APPROACHES—GENERAL CONSIDERATIONS

Several investigational approaches have been considered as potentially rewarding in resolving the uncertainties in use of retarding, or water-reducing and retarding, admixtures in Highway Department practice:

1. Study setting behavior of mortar wet-sieved from laboratory batches of concrete. Essentially, this would be an extension of the routine testing program required by ASTM C 494, Specifications for Chemical Admixtures for Concrete, with which all such admixtures are now required to comply, but it would study many combinations of cement brands, admixture dosages, mixing temperature, etc.

Although this approach has some advantage from the standpoint of more closely duplicating job use of the admixture, it is cumbersome and study of any great number of combinations of cements, aggregates, dosages, and admixture brands, etc., is almost prohibitive from the standpoint of time and effort. Also, use of fine and coarse aggregate in making trial batches of concrete introduces two more variables whose influence must be known in order to provide results that can be interpreted.

2. Determine setting behavior of trial batches of paste or mortar. This enables much more rapid progress in the number of cement, admixture, and dosage combinations which can be studied since smaller, more manageable batches of only a few hundred grams are needed. Use of standard Ottawa testing sand in preparation of the mortars makes them almost as easy to study as cement-water-admixture paste.

Indeed, many investigators have gone the route of studying the physical behavior of admixtures in mortars and pastes.

3. A research approach having great appeal for those concerned with the basic mechanism of cement setting is to observe the physical behavior and at intervals arrest further hydration by rapidly grinding the partially hydrated paste and washing it immediately in acetone to remove the free water. The residue is then analyzed by wet chemical, X-ray diffraction, DTA or other means to correlate the observed chemical changes with physical behavior. Young, for example, used this technique (9,10). Taplin (12) used a quick vacuum drying method which yielded interesting results. Verbeck and Foster reported nonevaporable water and vapor absorption as a measure of cement hydration (13). These are the "classical" methods, reports of which are in great abundance in the literature. Much of what is known about cement hydration has been discovered using these techniques.

4. Beginning with the work of Bates (14) and followed by Carlson (15), Forbrich (16), and Lerch (17), much excellent work was done on the heat liberated by hydrating cements. Carlson, in particular, noted a relationship between total heat liberation and setting and concluded, "Actually, the final set can probably be determined to a greater degree of reproducibility by heat measurements than by the usual method for determining final set" (Ref. 15, p. 366). To a considerable degree, this led to the work undertaken in the present investigation wherein continuous temperature history for the first 24 hr, or more, are recorded of cement-water pastes containing various dosages of water-reducing and retarding admixtures. The pastes are stored in vacuum flasks to conserve the heat liberated during the hardening period and temperatures determined by thermocouples embedded in the paste. Automatic recording of the temperature history makes this an attractive feature because manual attention is not needed. It is of course hoped that the characteristic heat release "signature" of the particular cement-admixture dosage combination can be associated with setting behavior.

In the present study, setting times of the pastes were determined simultaneously with the heat generation studies using two techniques, namely, Vicat penetration (ASTM C 191) and a new cone penetration test proposed by Kuntze and Hawkins (18). Details of the adopted techniques are given in the subsequent section and in Appendix D.

Extensive consideration was given in planning this research to the behavior of cement-water pastes relative to the stiffening of concrete itself. Despite the fact that all specifications for portland cement carry requirements using paste setting times with the Vicat or Gillmore apparatus, or both, the propriety of these tests is sometimes questioned. Berger reported extensively on this controversy (19). In partial defense of using paste Vicat setting times as indication of concrete performance in this research, both Tamas and Bruere recently reported considerable work with retarders using the Vicat apparatus (20,21).

The variables reported as influencing the setting behavior of concrete containing water-reducing retarders include the following:

1. Manufacturer and chemical composition of the admixture itself. Since the Michigan Department of State Highways uses air-entraining cement, Type 1A, almost exclusively in bridge deck construction, appreciable contribution of air by the chemical admixture itself is not desired. Therefore, only non-air-entraining admixtures of the hydroxylated carboxylic acid-type are normally used. Four such liquid admixtures were employed in the present program representing products from three manufacturers.

2. Admixture dosage. Generally, manufacturers now provide products which require 3 to 5 liq oz per 100 lb of cement for usual amounts of retardation. In this study, increments of 3, 5, and 7 liq oz per 100 lb of cement were

employed. The higher dosage of 7 oz was deliberately chosen to reveal setting behavior when using an excessive dosage.

3. Cement chemical composition and brand. A great variety of influences of the cement chemistry have at various times been alleged to control admixture response. Three lots of cement from two manufacturers were studies in this program and their chemical composition is reported in Appendix B, but effort has not been made to relate cement chemistry to physical behavior.

4. Concrete temperature. In the case of plain concrete without admixture, higher temperatures accelerate hardening. In hot weather, use of retarding admixture is often made to compensate for such rapid hardening. However, field difficulties are sometimes reported to the effect that just the reverse has occurred—instead of retardation with the admixture an acceleration of set has been experienced. In the present study, three temperatures were employed which cover the usual summer range, 60°, 75°, and 90°F.

5. Water-cement ratio. Most data indicates that increasing the water-cement ratio prolongs the setting time. The bulk of the work in this study was conducted on cement-water-admixture pastes having a fixed w/c = 0.35. This is about the maximum amount of water in a paste that can be tolerated without excessive bleeding and contrasts with a w/c of about 0.40 to 0.50 for average 6-sack bridge deck concrete. Concrete used in the present investigation had a w/c = 0.5, equivalent to 5.6 gal/sack.

EXPERIMENTAL PROCEDURES

From the outset of this investigation, it was decided to seek a technique for characterizing admixture performance which would demand least technician's time. It was recognized that more sophisticated equipment than conventionally used in cement or concrete testing might be needed to accomplish this goal. Conserving of technician's time is important when the elapsed time is considered during which observations must be made on retarded concrete to enable description of its setting behavior: this may be 10-15 hr, or more, and often is quite unpredicatble. Scheduling of personnel becomes a vexing problem.

Because behavior of the cement-water-admixture system seemed basic to this research, the major part of this work was done with cement pastes instead of mortars or concrete. Details in addition to the following are given in Appendix A.

1. PREPARATION OF PASTES

Mixing of the pastes was modeled after the procedure prescribed in ASTM C 305, Standard Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency except for two important modifications:

1.1 Temperature Control

In order to achieve the proper initial temperature of the paste (60°, 75°, or 90°F, respectively), it was necessary to temper the mixing water or otherwise change the temperature of the mix, for 75°F a portion of the mixing water was replaced with ice water and for 90°F, the entire amount of mixing water plus liquid admixture, if any, was heated to approximately 100°F. For 60°F, it was necessary to briefly immerse the mixing bowl with its contents in an ice water bath during the 10-min wait period. Generally, this required cooling to about 54°F to compensate for the heating during the final 1-min mixing.

1.2 Wait Period

During preliminary trials, it was found that certain admixture-cement combinations stiffened markedly almost at once, consequently a period of 10 min with the mixer stopped was introduced into the mixing sequence to give some semblance to a usual ready-mix operation where delay is experienced prior to placement. This would allow the very earliest chemical reactions to occur before beginning the measurements.

2. SETTING TIME

After preparation of the paste, a portion was immediately spooned into a container, lined with kitchen plastic, as used in ASTM C 359, Standard Method of Test for False Set of Portland Cement (Mortar Method) and the paste immediately stored in a constant temperature water bath maintained at 60°, 75°, or 90°F (± 0.3°F), as selected. The container was submerged to about 1/2 in. from the top.

Two types of setting time were determined on the paste specimen:

2.1 Vicat

ASTM Method C 191, Time of Setting of Hydraulic Cement by Vicat Needle was used. A plot of the setting time of each paste was made and the time picked off the curve for 25 mm and 5 mm penetration. The "final set" time was found to be thoroughly impractical for some of the retarded pastes since the curve approaches zero penetration almost asymptotically, and the time when "the needle does not sink visibly into the paste" is not clearly discernible.

2.2 Cone Set

For this test, a 20° blunted stainless steel cone penetrometer weighted to 250 g was used. Times for 30 mm and 5 mm penetration were picked off the penetration-time curves and were used in the tabulation. This apparatus is more fully described in Reference 18.

Figure 1 is a photograph of the paste penetrometers, paste receptacle and constant temperature water bath.

3. TEMPERATURE RISE TESTS

Simultaneously with filling the setting time container, a portion of the freshly mixed paste was spooned into a Styrofoam cup (75 mm diam, 85 mm high) into which a glass shielded thermocouple was centrally inserted and the assembly placed into a wide-mouth vacuum bottle. At the time of screwing on the bottle cover, an iron-constantan thermocouple was threaded down through a hole in the cover so that the thermocouple junction was positioned near the center bottom of the paste sample. The vacuum bottle was a commercially available "Dine-A-Liner" from which the removable plastic liner had been discarded. The Styrofoam cup essentially filled the vacuum bottle. The thermocouple shield was 5 mm O.D. glass tubing, 4 in. long, closed at the bottom end and was not recovered at the completion of the hardening period. Temperature of the paste (accurate to ± 1°F) was automatically recorded at intervals of slightly over 4 min for the duration of the test period.

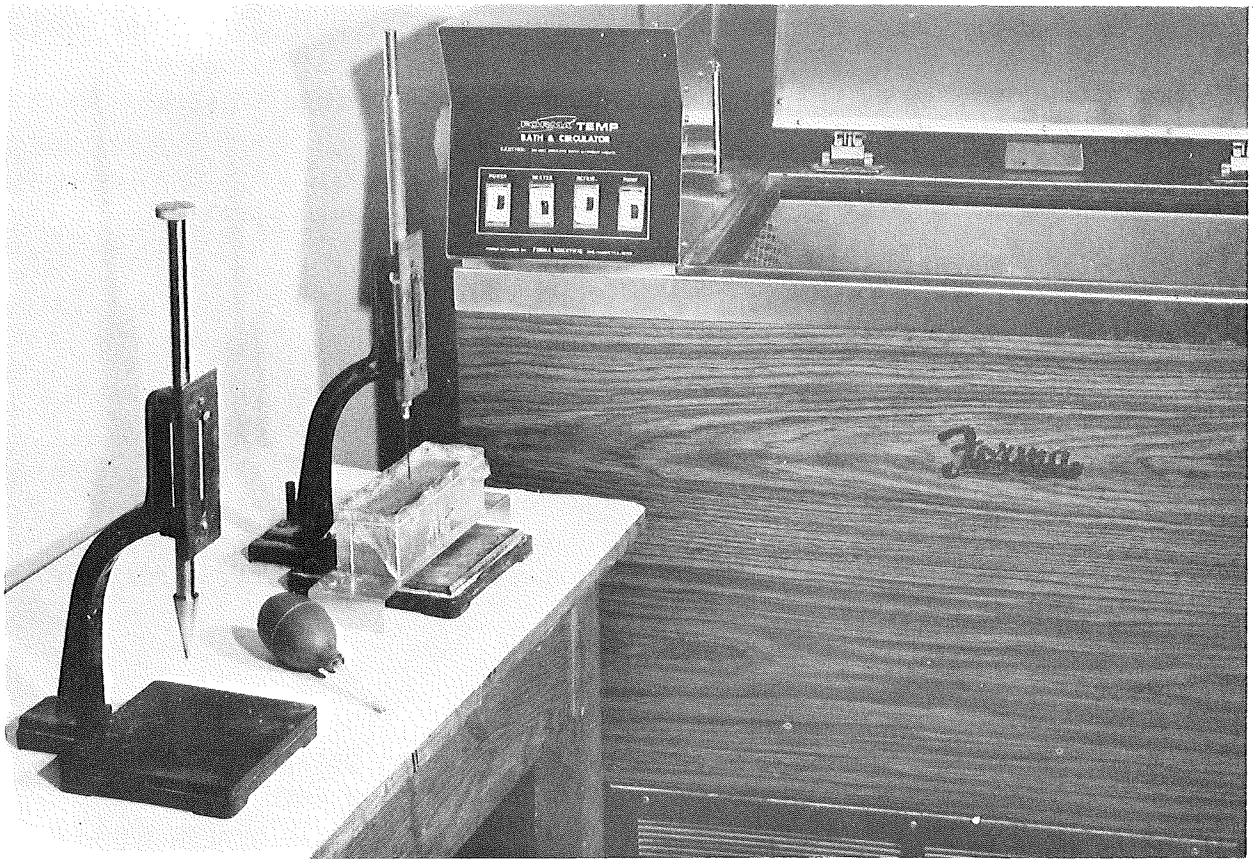

Figure 1. Vicat and cone cement paste penetrometers, paste setting time container, and constant temperature water bath.

Figure 2 gives a photograph of the automatic temperature recorder and four vacuum bottles, one of which is opened to display the cup, cover, and glass shield insert.

4. CONCRETE TESTS

A limited number of tests were made of 6-sack concrete using techniques similar to those provided in testing admixtures ASTM C 494, Specifications for Chemical Admixtures for Concrete, except that three temperatures were used for mixing and storage of the specimens during setting, i.e., 60°, 75°, and 90°F. Initial temperatures of the mix were attained by tempering the mix water with ice or with hot water as needed. Mixing times were again modified to more nearly resemble transit mix operations; an initial 3-min mix was employed followed by a 5-min rest period with the mixer stopped, and a final 3-min mixing.

4.1 Slump Tests

Slump of the concrete during the hardening period was made at 1/2-hr intervals until the concrete was so stiff as to have a 1/2-in. slump. During the hardening period, the portion of the batch used for slump testing was stored, respectively, in a thermostatically controlled oven set to maintain 90°F, or in the laboratory air for 75°F, or in a cooling chamber at 60°F. The concrete was covered with several layers of moist burlap during the wait period between slump determinations.

4.2 Penetration Resistance of Sieved Mortar

Penetration resistance of the wet-sieved mortar was determined at intervals using the apparatus shown in Figure 3. This technique is prescribed in ASTM C 403, Time of Setting of Concrete Mixtures by Penetration Resistance. Progressively smaller diameter Proctor needles shown in the figure were used to follow the hardening of the mortar until final set of 4000 psi was attained. The 6-in. diameter metal container was covered with a tight-fitting cover and stored in a water bath at 60°, 75°, or 90°F as scheduled.

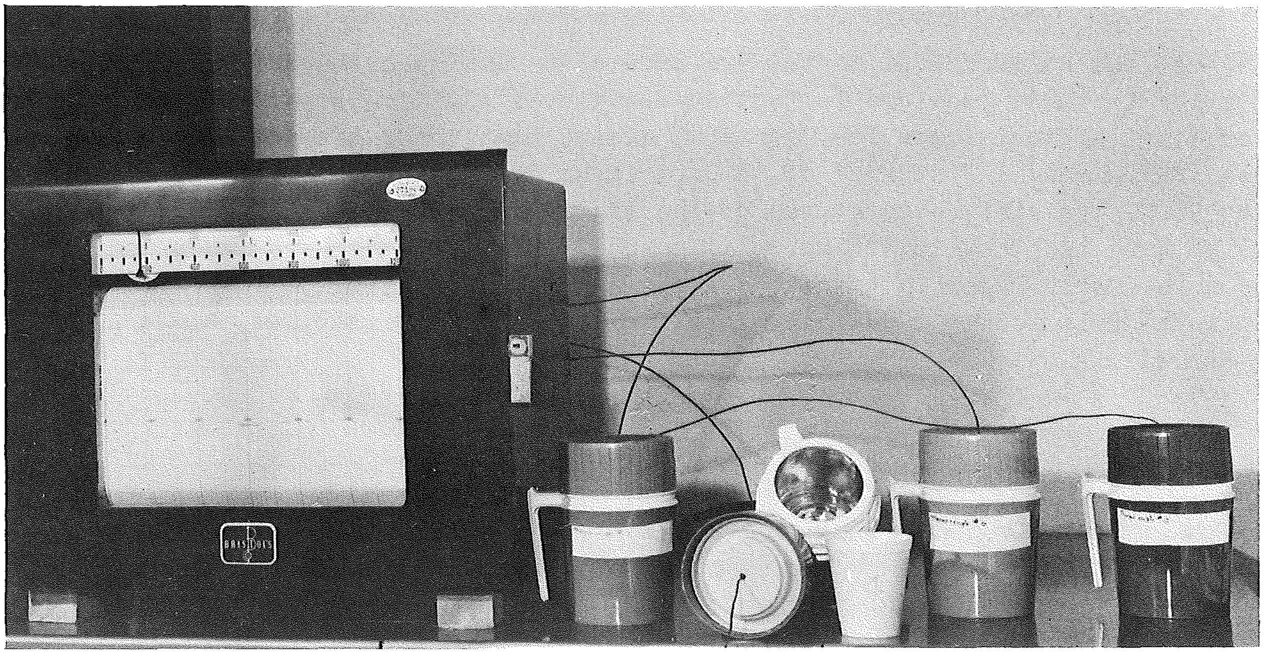

Figure 2. Automatic 10-point temperature recorder with four vacuum flasks, one opened to display paste cup, flask cover, and glass thermocouple protector.

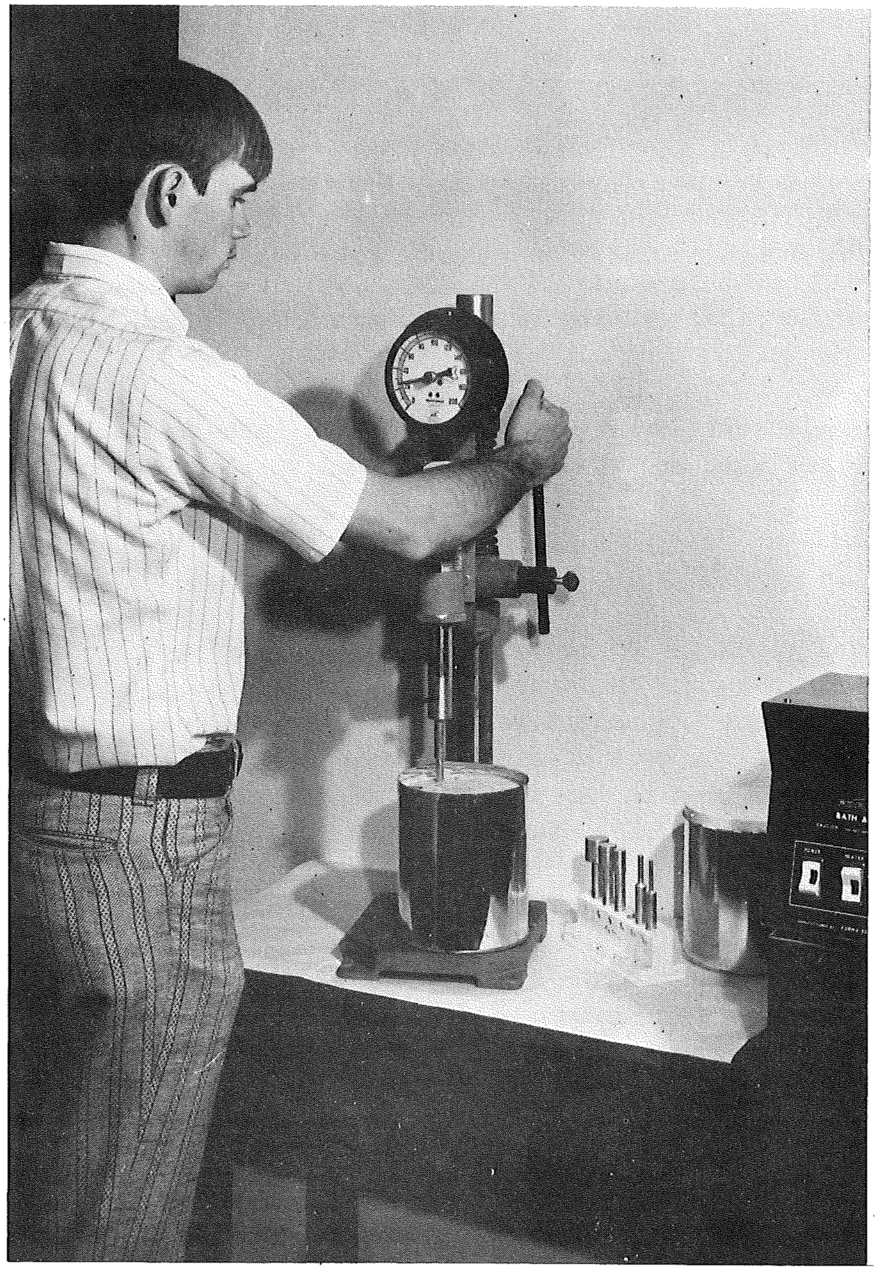

Figure 3. Apparatus used for time of setting of concrete by penetration resistance of sieved mortar.

TEST RESULTS

1. MATERIALS

Routine acceptance tests of the three lots of cement used in this investigation are shown in Appendix B. Cements Nos. 1 and 2 are from the same manufacturer and are much more finely ground than cement No. 3. Cement No. 1 was quite severely false setting by conventional C 359 tests and cement No. 2 mildly so.

Aggregate tests are shown in Appendix C. The aggregates were from laboratory stock and were produced from glacial deposits near Ann Arbor.

2. SCHEDULE OF CEMENT-ADMIXTURE-TEMPERATURE COMBINATIONS

Table I gives the schedule of batches studied in this investigation. It is noted that only one admixture (No. 1) was mixed in pastes combined with the severely false setting cement No. 1, whereas all dosage, temperature, and admixture combinations were studied in pastes with cements Nos. 2 and 3. In the case of concrete, due to the available time, only cement No. 3 was studied with admixtures Nos. 2, 3, and 4.

3. PASTES

Immediately after mixing each batch of paste, it was split and the portion for setting time was placed in the stainless steel false set container specified in ASTM C 359 and immediately placed in the constant temperature water bath (controlled to ± 0.2°C) and the portion for thermal studies inserted in the vacuum flask with thermocouple. Thermocouple readings started approximately 8 min after completion of mixing. Vicat and cone setting time readings were made during the progress of setting; in extreme cases these were as early as 10 min after mixing and as long as 24 hr after mixing.

A typical day's operation was to mix batches with a given admixture at a selected nominal temperature and at four dosages, namely, 0, 3, 5, and 7 fl oz per 100 lb of cement. These batches were then observed for setting properties and the paste temperature recorder operated for 24 hr to simultaneously observe heat generation of the four batches.

At the completion of the 24-hr period of temperature recording, values were scaled off the recorder chart and plotted on a compressed time scale similar to the upper portion of Figure 4. The Vicat and cone penetration values simultaneously determined were similarly plotted on the bottom portion of the

TABLE I

SCHEDULE OF BATCHES MADE IN THIS STUDY

Cement No.	Admixture No.	Dosage, oz/100 lb:	Temperature 60°F				Temperature 75°F				Temperature 90°F			
			0	3	5	7	0	3	5	7	0	3	5	7
		Pastes												
1	1		x	x	x	x	x	x	x	x	x	x	x	x
2	1		x	x	x	x	x	x	x	x	x	x	x	x
2	2		x	x	x	x	x	x	x	x	x	x	x	x
2	3		x	x	x	x	x	x	x	x	x	x	x	x
2	4		x	x	x	x	x	x	x	x	x	x	x	x
3	1		x	x	x	x	x	x	x	x	x	x	x	x
3	2		x	x	x	x	x	x	x	x	x	x	x	x
3	3		x	x	x	x	x	x	x	x	x	x	x	x
3	4		x	x	x	x	x	x	x	x	x	x	x	x
		Concrete												
3	2		x	x	x	x	x	x	x	x	x	x	x	x
3	3			x	x	x		x	x	x		x	x	x
3	4			x	x	x		x	x	x		x	x	x

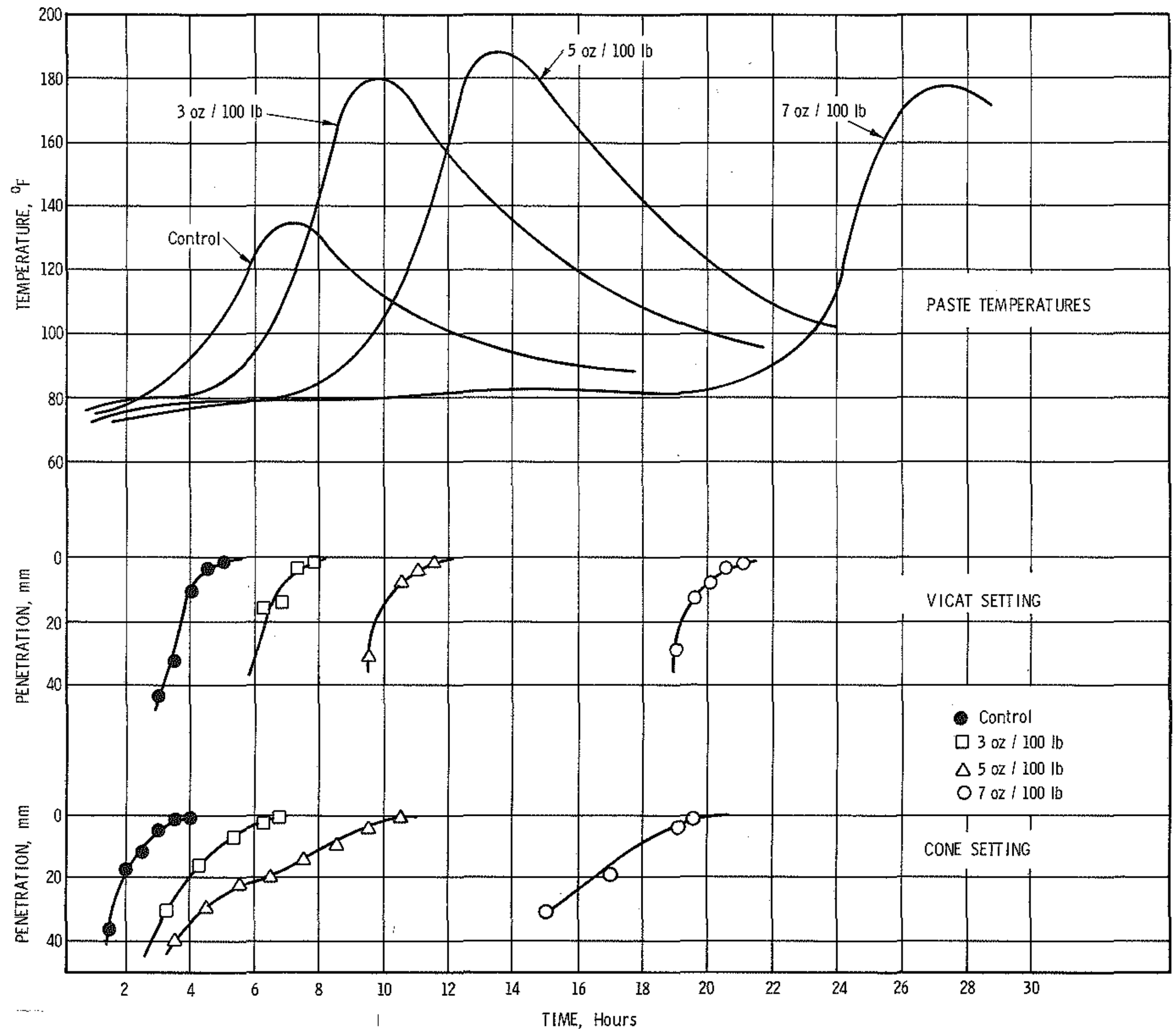

Figure 4. Adiabatic temperature rise and corresponding Vicat and cone setting of cement paste without admixture and at three dosages—normal behavior (cement No. 3, admixture No. 3, 75°F).

same sheet as shown in the figure. The original plotting sheet time scale was readable to the nearest 6 min.

In order to handle the considerable amount of data generated, values characterizing these curves were read off and summarized in tabular form, Appendix D, "Detailed Tests of Pastes" from which subsequent analyses have been made.

Data from the concrete tests was also plotted as exemplified by Figure 6. Contrary to usual practice, the penetration resistance curves have been plotted on a log scale in order to more accurately exhibit the 60 psi value, the reason for which will be discussed later. Values characterizing these curves were also read off and are summarized in Table II, "Tests of Concrete."

Brief discussion is made of these curves prior to making a detailed analysis of the data. Figure 4 has characteristics learned to be typical of "normal" paste behavior:

1. Successively greater admixture dosages prolong the time to reach peak temperatures of the pastes stored in the vacuum flasks.

2. Peak temperatures increase, or do not appreciably diminish as the dosage is increased.

3. Increased dosage causes both Vicat and cone setting time to be delayed roughly proportional to the time to reach peak temperature.

4. The time over which readable cone penetrations are obtained is much greater than for Vicat needle penetrations. This elongation of the time scale is considered advantageous since concrete itself is normally plastic over an appreciable period and the transition from plastic to a "solid" is not abrupt.

Figure 5 is presented from another day's operation to exemplify paste of quite different behavior:

1. Increased admixture dosages progressively diminished peak temperatures reached.

2. A very weak, and early, peak temperature was attained for the 7-oz dosage of this particular cement and admixture combination.

3. The 7-oz dosage caused reversal of the time sequence of Vicat set wherein it occurs only slightly later than the control without admixture but earlier than the 3- and 5-oz dosages.

4. The cone setting time is even more unusual in that the 7-oz dosage caused set to occur even earlier than the control and, further, the curve is

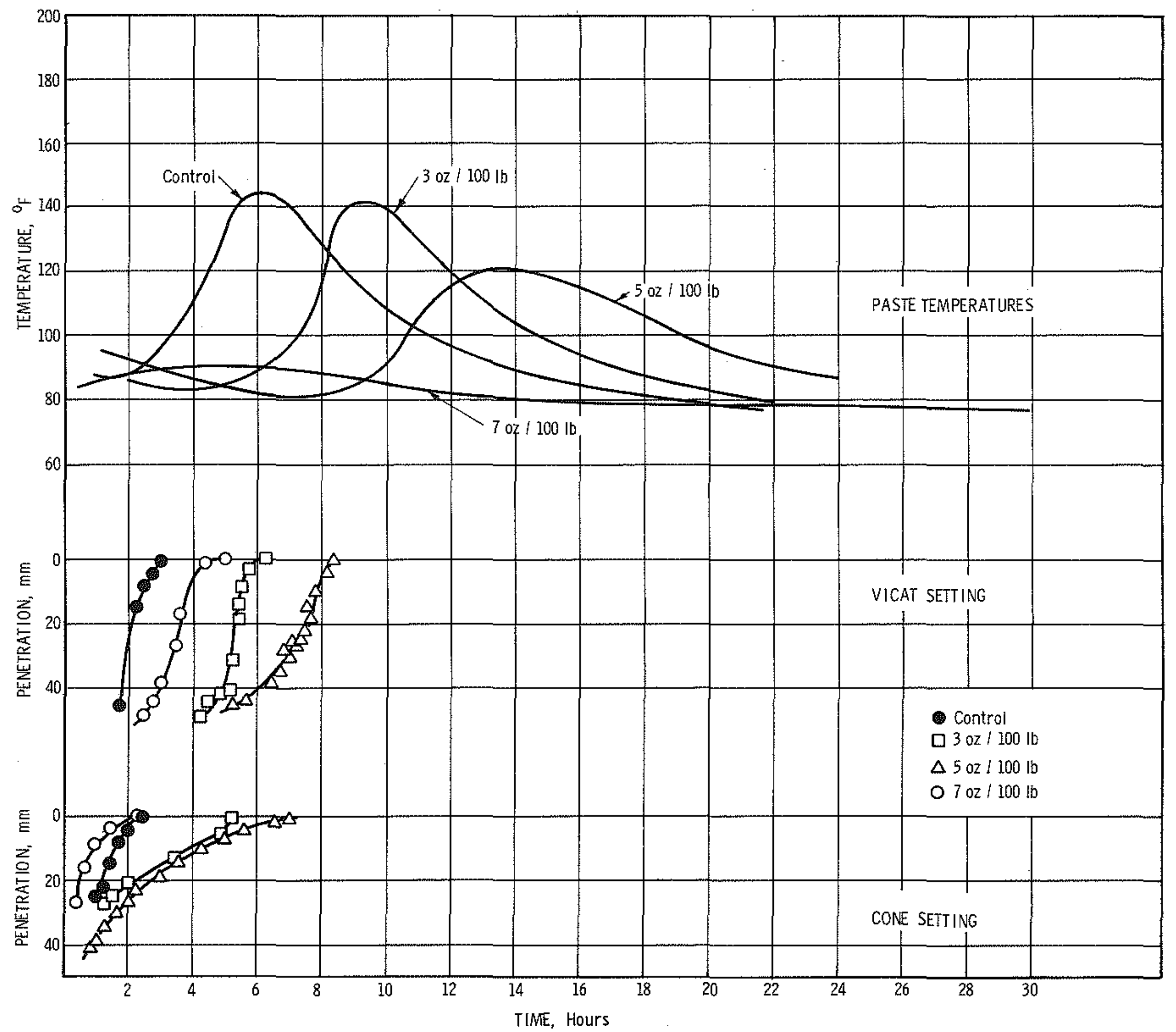

Figure 5. Same type of data as Figure 4—abnormal behavior (cement No. 1, admixture No. 1, 90°F).

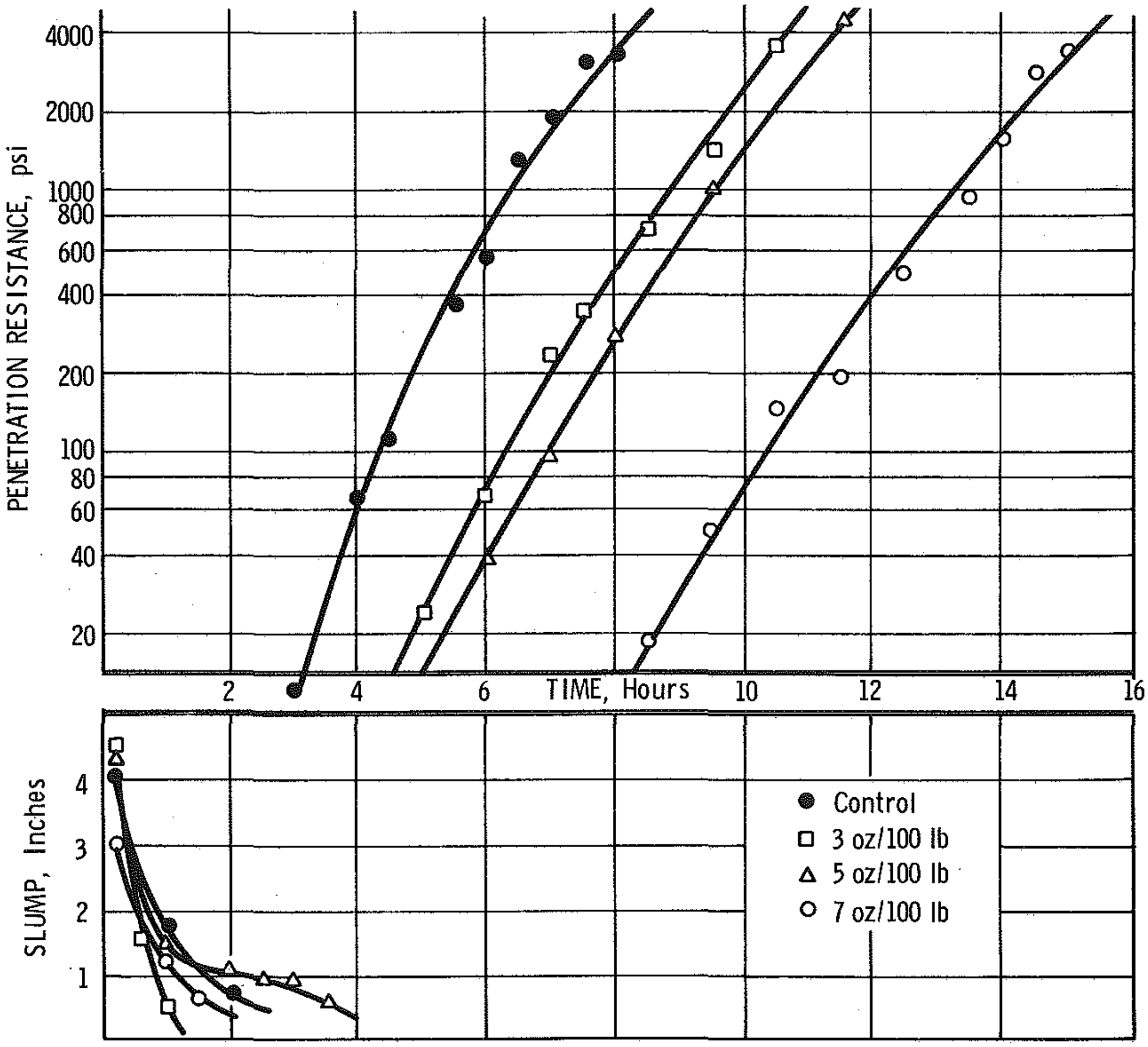

Figure 6. Penetration resistance and slump of concrete vs. time after mixing (cement No. 3, admixture No. 4, 60°F).

very steep indicating a relatively short time during which the paste is plastic, i.e., the transition from a "liquid" to a solid is brief.

Figures 7-10 display characteristics, derived from the tabulated data, of the control pastes without admixture. The four bars corresponding to each mix temperature for cements No. 2 and 3 indicate a reasonably satisfactory degree of concordance between repeat runs. A control mix was made each day, so that evaluation of four admixtures at a given temperature with a given cement yielded four control batches.

As would be expected, higher mixing temperatures hasten hydration and shorten time of setting and time to reach maximum temperature in the vacuum flasks. It will be noted that the time to attain initial cone set (arbitrarily selected as 30 mm penetration in this work) is, like the slump of concrete as will be presented later, relatively less sensitive to mixing temperature. Cements Nos. 2 and 3 respond somewhat differently to all four measures of hydration exhibited by these figures thus indicating their inherent differences in setting properties. The data on cement No. 1 is less complete on which to draw conclusions.

Data collected from the cement pastes indicated that in the vacuum flask tests the time to reach maximum temperature was the most promising measure for predicting setting performance; consequently, Figures 11-14 have been prepared giving in detail the retardation, or delay in time to reach maximum temperature with respect to the control, for all the cement-admixture-temperature combinations.

4. ADMIXTURE NO. 1

Figure 11 displays the behavior of admixture No. 1 with all three cements:

1. At 60°F or 75°F, increased dosage of admixture with cement No. 1 increased delay in reaching a temperature peak. At a temperature of 90°F and a 7-oz dosage, no peak in the temperature curve was observed, at least during the first 31 hr. Reference to the detailed data in Appendix D indicates this batch set prematurely.

2. With cement No. 2, an orderly progression of delay was noted at all three mix temperatures and at all three dosages.

3. With cement No. 3, anomalous behavior was observed at all three temperatures for the 7-oz dosage in that no peak of heat generation was recorded. This was again accompanied by anomalous setting behavior.

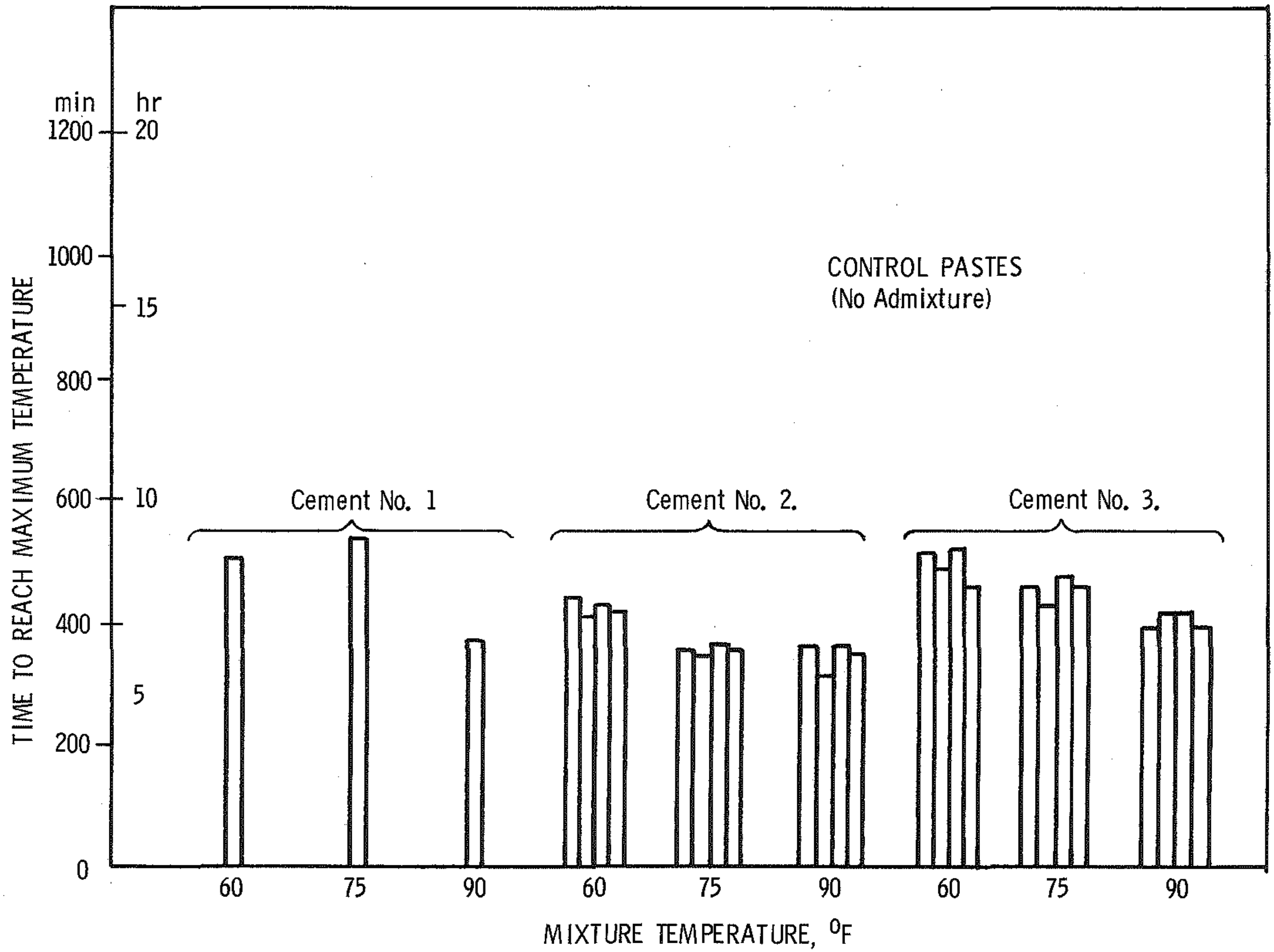

Figure 7. Time to reach maximum temperature for control pastes (no admixture). Four repeat tests at each mixing temperature for cements Nos. 2 and 3.

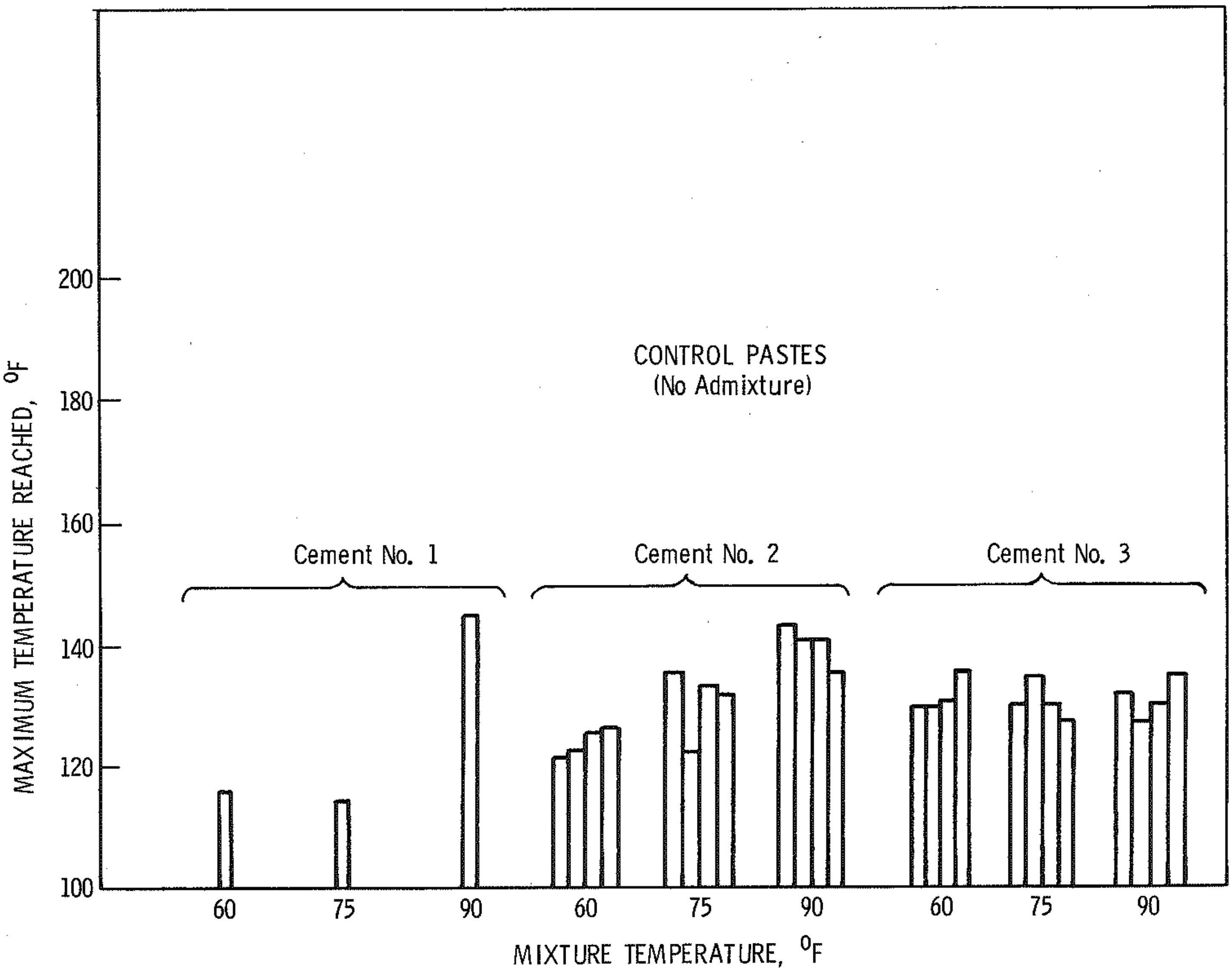

Figure 8. Maximum temperature attained in vacuum flasks—control pastes without admixture. Four repeat tests at each mixing temperature for cements Nos. 2 and 3.

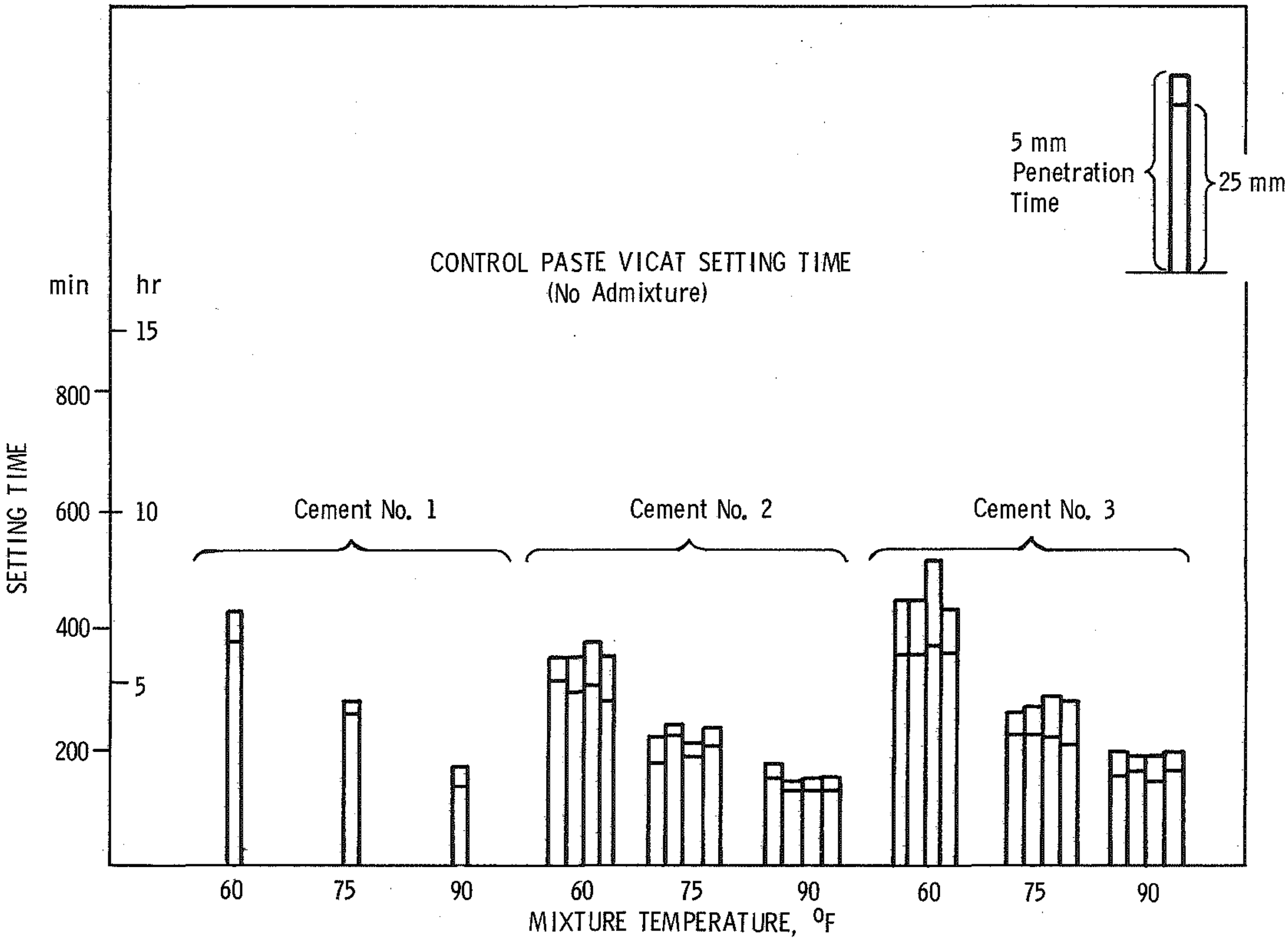

Figure 9. Control paste vicat setting time (no admixture). Four repeat tests for each mixing temperature for cements Nos. 2 and 3.

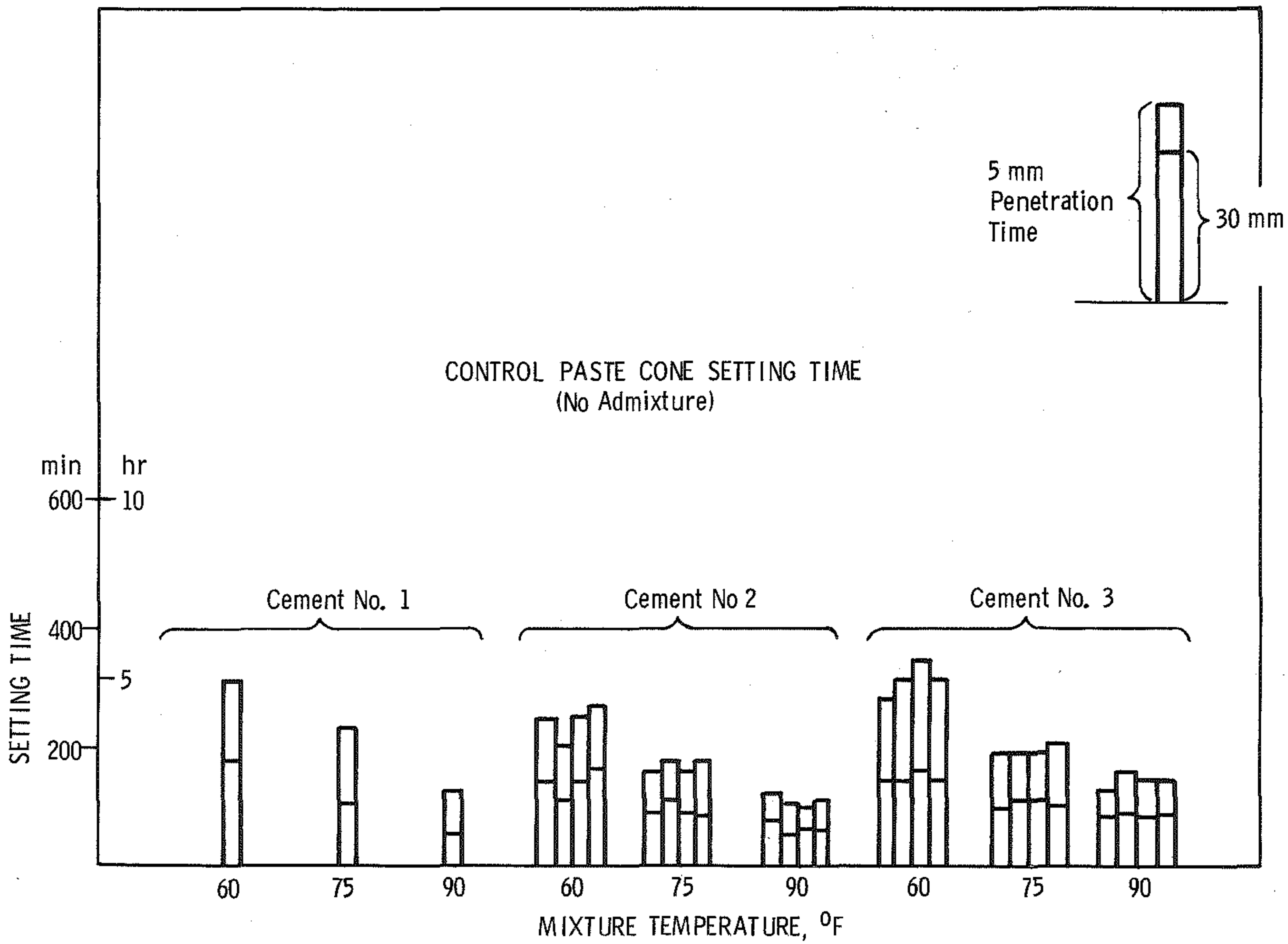

Figure 10. Control paste setting time using 20° blunted cone loaded to 250 g (no admixture). Four repeat tests for each mixing temperature for cements Nos. 2 and 3.

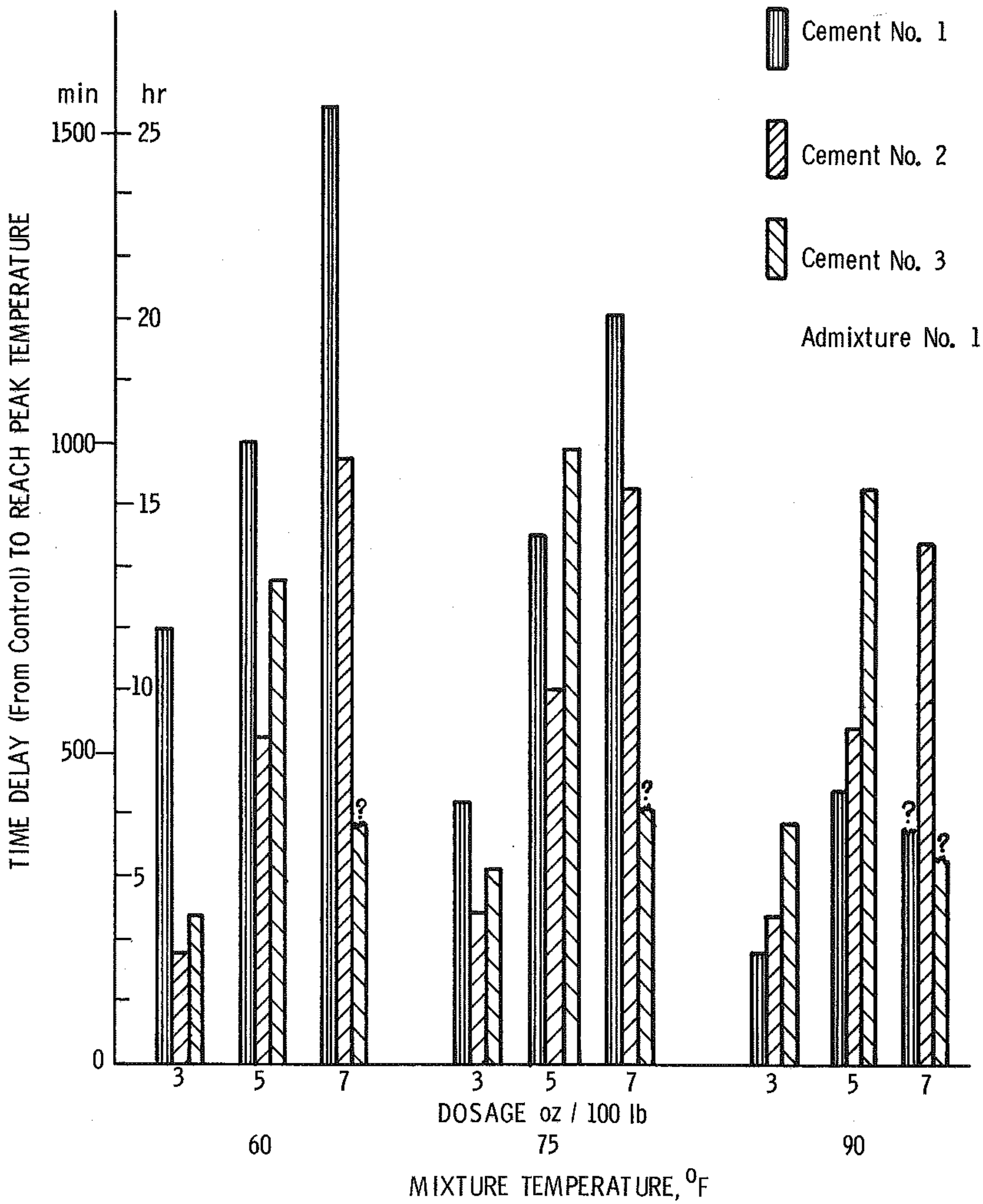

Figure 11. Delay from control in reaching peak temperatures of pastes made with admixture No. 1.

5. ADMIXTURE NO. 2

Figure 12 shows the delay of peak temperature attainment for admixture No. 2. Only cements Nos. 2 and 3 were incorporated with this admixture.

1. Two striking differences in behavior are noted with this admixture in combination with cement No. 3. At a 7-oz dosage at both 60°F and 75°F, the peak heat time was reached earlier than with the control without admixture, leading to a prediction of admixture causing set acceleration. No peak was observed at 90°F at 5-oz or 7-oz dosage. Reference again to Appendix D reveals anomalous setting in all four cases.

6. ADMIXTURE NO. 3

Figure 13 shows similar data for admixture No. 3 with the same two cements. The 7-oz dosage with cement No. 3 at 60° and at 90°F again shows no heat peak. Setting in both cases was anomalous.

7. ADMIXTURE NO. 4

The response of admixture No. 4 in Figure 14 is fairly regular and unusual setting behavior would not be predicted.

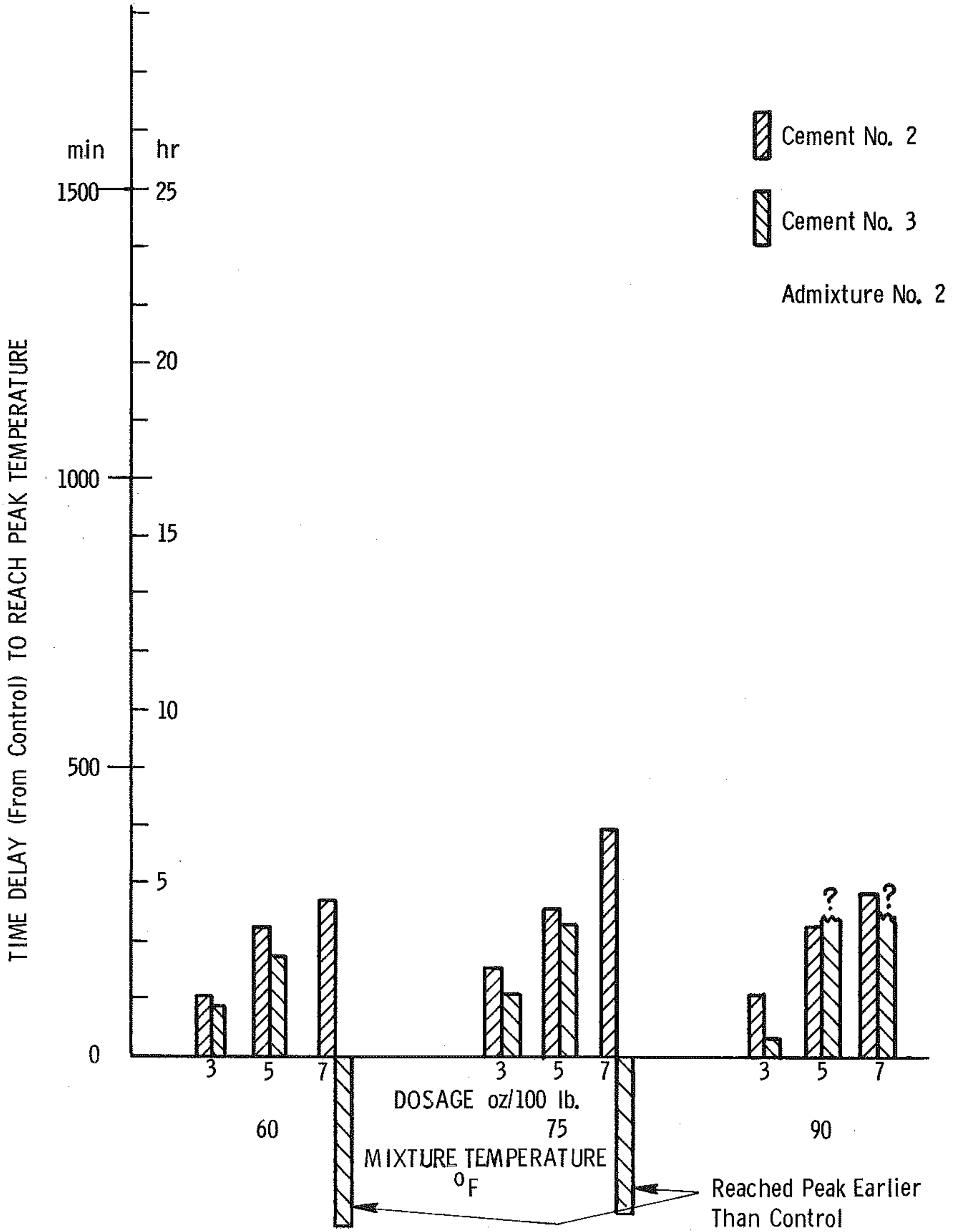

Figure 12. Delay from control in reaching peak temperature of pastes made with admixture No. 2.

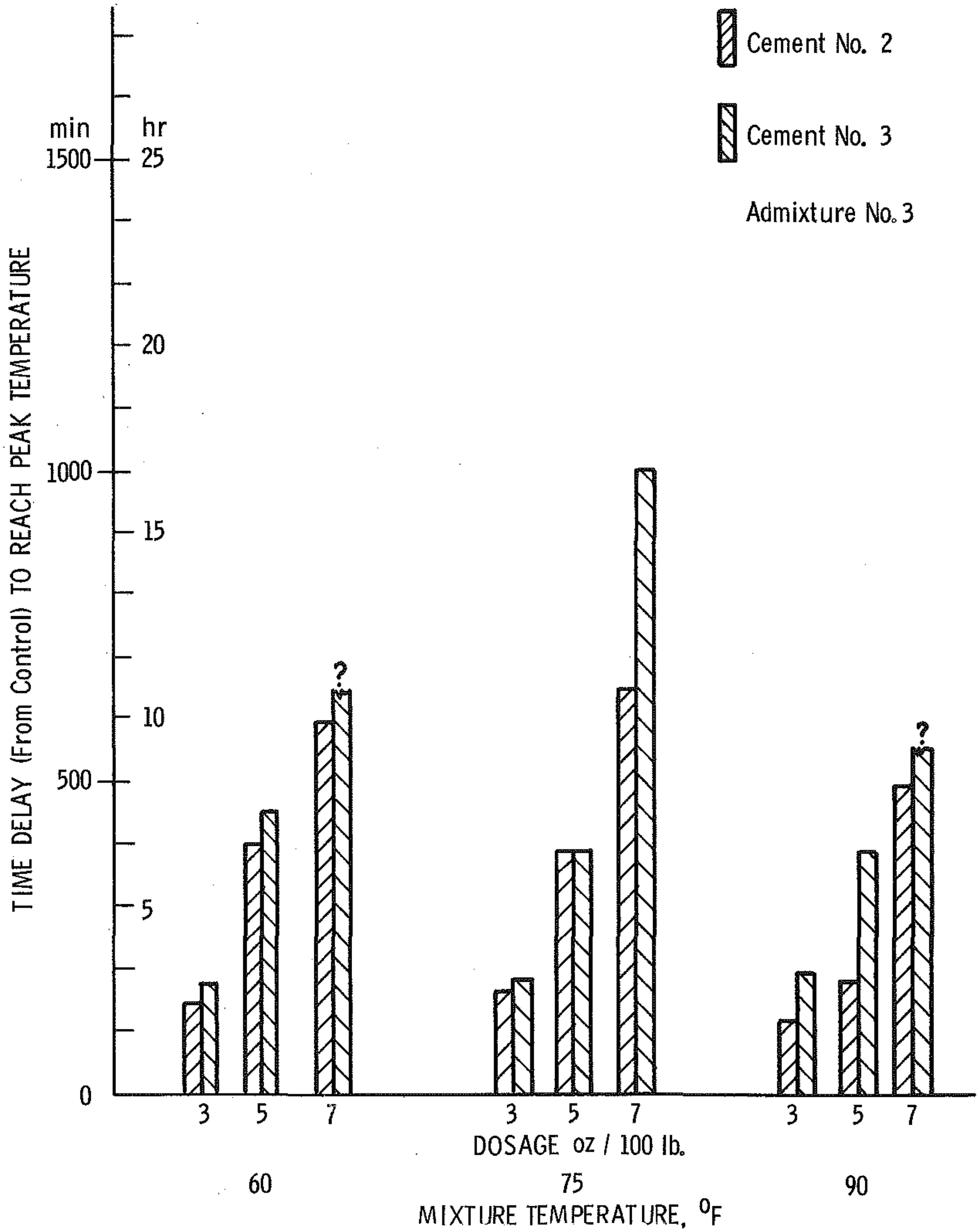

Figure 13. Delay from control in reaching peak temperature of pastes made with admixture No. 3.

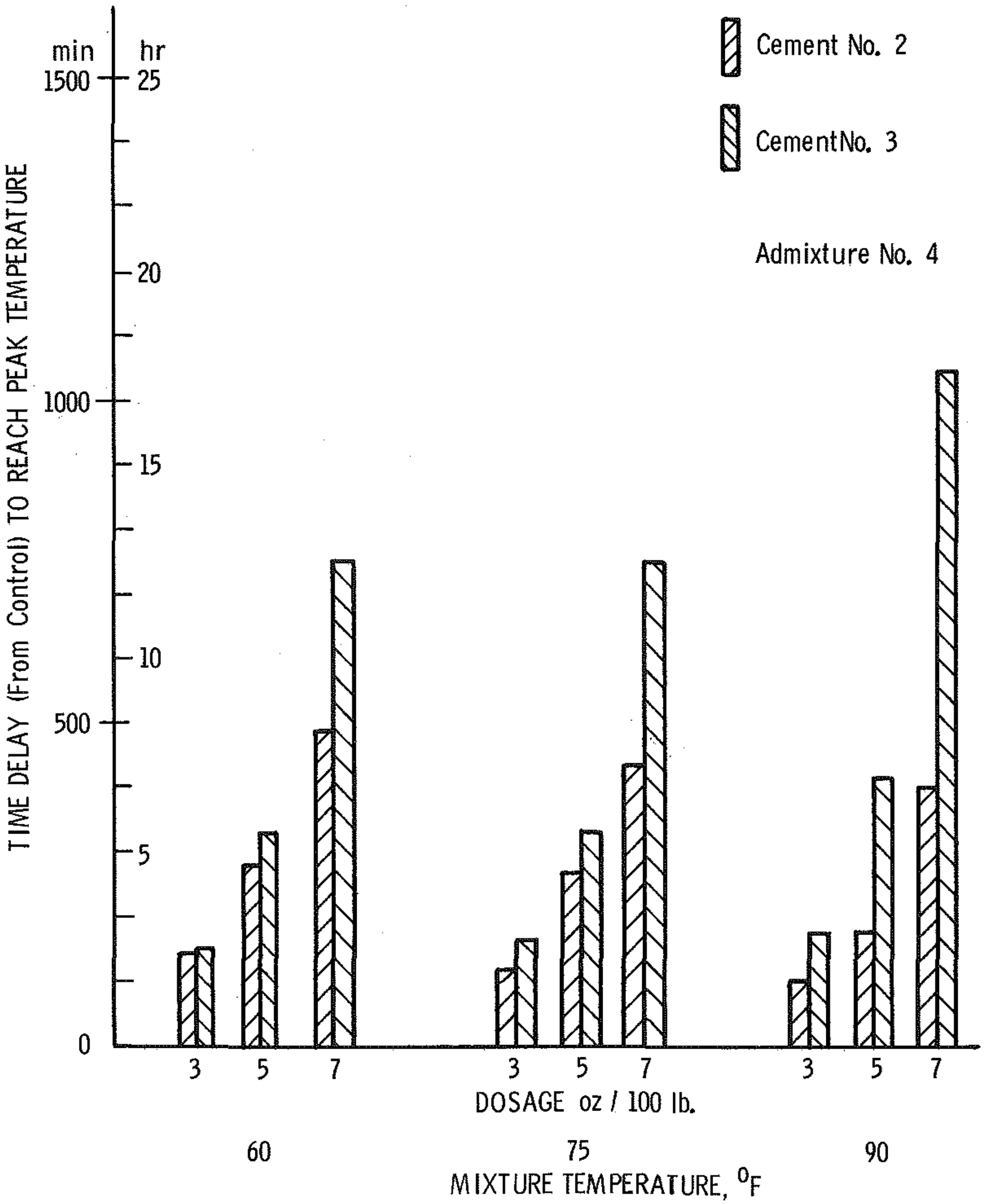

Figure 14. Delay from control in reaching peak temperature of pastes made with admixture No. 4.

CONCRETE TESTS

Due to limitations of the research contract, the time was brief during which concrete tests could be conducted. However, such tests were made with cement No. 3 with three admixtures: Nos. 2, 3, and 4, and at the same temperatures and dosages as used for the corresponding paste tests, namely, at 60°, 75°, and 90°F and at dosages of 0, 3, 5, and 7 oz per 100 lb of cement.

The concrete data is shown in detail in Table II. Subsequent analyses of the data emphasize two observations; namely, (1) time after mixing to reach 1/2 in. slump and, (2) time to reach 60 psi penetration resistance of the mortar wet-sieved from the concrete.

1. TIME TO REACH 1/2 INCH SLUMP

The time to reach 1/2 in. slump has practical job implications, particularly for slabs, in that it represents the length of time during which the concrete gives the impression of being workable to the average handler. Concrete stiffer than 1/2 in. slump cannot be readily shoveled, for example.

The data in Table II includes tests on 27 batches of concrete containing retarder and three control batches without retarder. The striking observation is made that in only six cases was the retarder successful in slowing the slump loss of comparable concrete with respect to the next lower increment of retarder. In several cases, additional retarder actually hastened stiffening as measured by the slump test. This accelerated rate of slump loss due to retarder has been reported previously (Ref. 22) and emphasizes misconceptions in the use of these chemical admixtures. Unless equipment is available by which the concrete can be given high-frequency revibration with substantial power input, the utility of the retarding admixtures is highly questionable.

These considerations inevitably bring up the alternative solution of obtaining retardation by cooling the concrete. Consider the present case of unretarded concrete at 60°F having a time to reach 60 psi penetration resistance of 240 min. With concrete mixed at 90°F, no amount of retarder No. 2 would be successful in prolonging the set this much. With retarder No. 4, the excessive dosage of 7 oz would have to be resorted to and with retarder No. 3, either the 5- or 7-oz dosage should be employed. Furthermore, there is independent evidence to indicate that concrete initially cast at 60°F will be superior to that cast at 90°F and the wisdom of using retarder to achieve this much set delay can be questioned.

TABLE II

TESTS OF CONCRETE

Cement No.	Admixture No.	Nominal Mixing Temp, °F	Admixture Dosage, oz/100 lb	Slump Test: Initial, in.	Slump Test: Time to Reach 1/2 in., min.	Penetration Tests of Sieved Mortar, Time to Reach, min: 60 psi	500 psi	4000 psi
3	Control (No Admix.)	60	0	4	150	240	340	490
		75	0	3-1/8	75	135	215	285
		90	0	3-3/4	70	112	155	230
	4	60	3	4-1/2	60	345	495	640
			5	4-1/2	225	390	525	690
			7	3-1/2	105	585	735	915
		75	3	4-1/2	90	205	285	360
			5	5-1/4	120	295	400	495
			7	3-3/8	120	435	545	645
		90	3	3	90	160	215	270
			5	3-1/2	110	210	270	330
			7	4-1/2	100	360	430	495
	3	60	3	2-1/2	135	295	405	585
			5	3-3/8	120	450	585	750
			7	2-1/2	195	615	795	975
		75	3	3-3/4	90	210	300	390
			5	4	90	335	435	530
			7	4	90	525	630	735
		90	3	3	80	165	210	285
			5	3-3/4	90	275	310	380
			7	3-1/2	90	395	465	525
	2	60	3	3-1/2	195	250	345	490
			5	2-3/4	105	345	450	585
			7	2-3/4	75	425	570	735
		75	3	3	180	195	255	330
			5	5-1/4	200	280	350	425
			7	4	90	335	395	455
		90	3	3	90	140	190	235
			5	3-1/4	105	200	250	290
			7	3-1/4	90	150	165	Indef.

2. TIME TO ATTAIN 60 PSI PENETRATION RESISTANCE

The column in Table II labeled time to reach 60 psi penetration resistance represents the time found by the NCHRP researchers (Ref. 5) within which vigorous revibration was successful in closing cracks in freshly cast bridge decks. With only one exception, it is noted that the values in this column progress in an orderly manner, i.e., lower temperatures and larger doses prolong the time to reach 60 psi penetration. The exception was the 7-oz dosage of admixture No. 2 at 90°F in which case attainment of 60 psi penetration resistance was hastened.

PREDICTION OF SETTING OF CONCRETE FROM PASTE TESTS

Analyses so far presented exhibit the complex interactions between cement brand, admixture brand, dosage, and mixing temperature. These analyses are not exhaustive, and further study may be made later.

Major interest in this research revolves around developing simple means to predict setting performance of job concrete, particularly to foretell unusual delay or acceleration of set. Until some future time when more is known of the chemistry of the reactions between the organic admixtures and cement constituents enabling prediction of performance, it now appears mandatory to use cement paste-admixture combinations containing the job materials in order to predict.

Reference was previously made to the long time use of Vicat setting time of cement paste as a measure of expected setting of concrete. In the present case, cone setting time of paste was also determined. It was found that the time for the paste to reach maximum temperature in the vacuum flask was related to both Vicat and cone setting time.

In the present investigation, 108 batches of paste were made. In 86 batches, simultaneous observation of time to reach peak temperature, Vicat set, and cone setting time were available. The 86 observations consolidate data from all batches regardless of cement brand, admixture brand, dosage, or mix temperature. Regression analysis of these yielded the following:

$$
\begin{aligned}
\text{Vicat setting Time (5 mm pen.)} &= 1.23\ t_p - 270 \text{ (in min)} \\
\text{Standard Error of Estimate} &= 150 \text{ min} \\
\text{Correlation Coefficient, } r &= 0.91, \text{ and}
\end{aligned}
$$

$$
\begin{aligned}
\text{Cone Setting Time (5 mm pen.)} &= 1.10\ t_p - 280 \text{ (in min)} \\
\text{Standard Error of Estimate} &= 139 \text{ min} \\
\text{Correlation Coefficient, } r &= 0.90
\end{aligned}
$$

Where: t_p = Time in minutes to reach maximum temperature

Thus, both Vicat and cone setting time (5 mm penetration) are highly correlated with time of reaching peak temperature in the vacuum flask tests, and the inference seems justified that the rapidly procured paste value will successfully predict setting performance of concrete.

In partial confirmation of the above, Figure 15 has been prepared to demonstrate the observed correlation between setting performance of concrete from

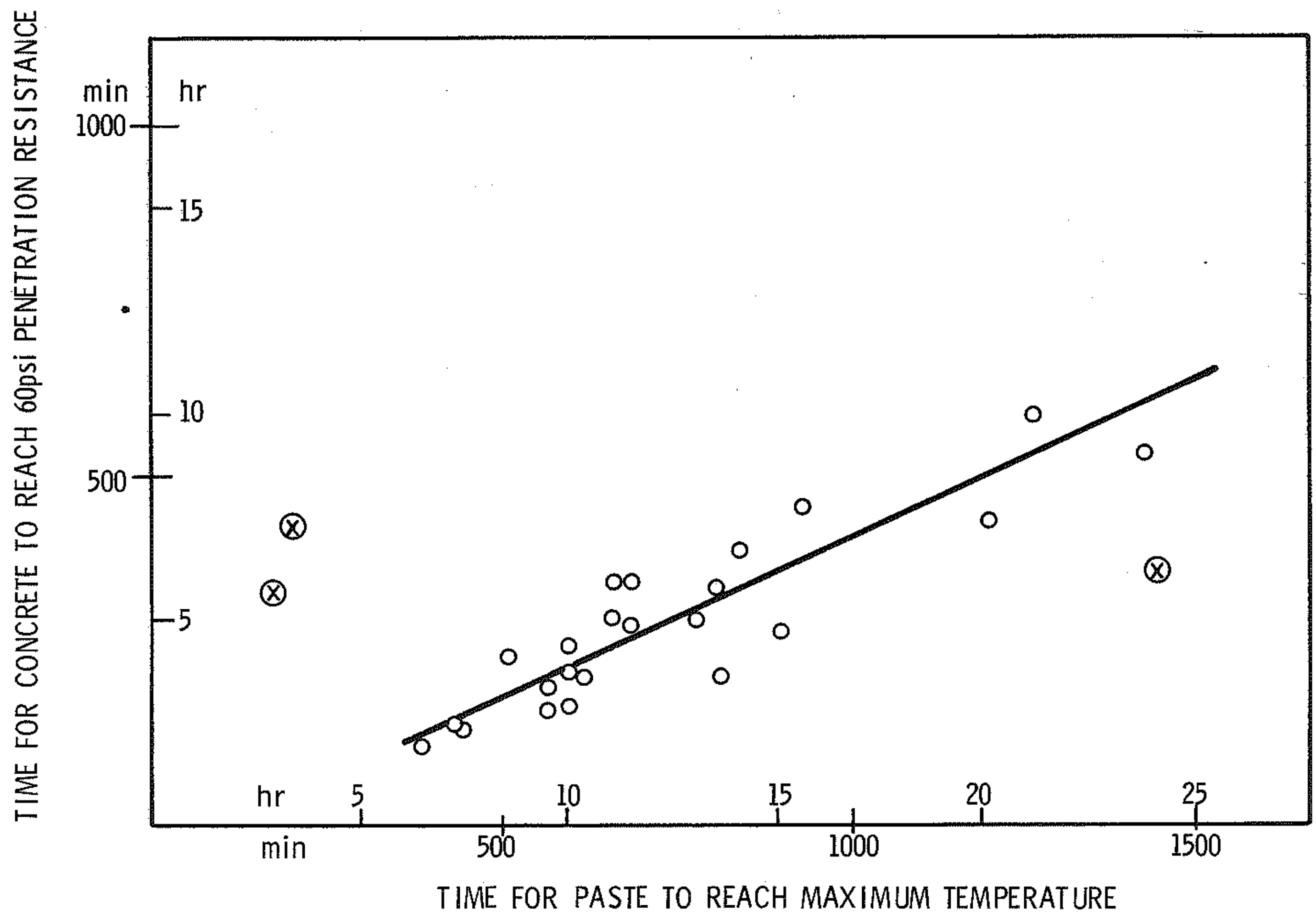

Figure 15. Time for concrete to reach 60 psi penetration resistance vs. time for paste to reach maximum temperature in vacuum flasks.

Table II and the time to reach maximum temperature in the paste tests. The limited amount of data on concrete necessarily restricts validity of the relationship, but if the three points marked "X" are discarded, regression analysis yields the following:

Time for concrete to reach 60 psi penetration resistance	=	$0.43\ t_p - 31$ (in min)
Standard error of estimate	=	56 min
Correlation Coefficient, r	=	0.90

Where: t_p = Time in minutes to reach maximum paste temperature

Again, a high degree of correlation is indicated. The two points marked "X" in the left-hand portion of the graph represent batches which had a severe drop in the expected maximum paste temperature, and the "X" in the right portion likewise has a less severe drop in maximum paste temperature. Four other paste batches for which concrete data are available did not develop significant temperature peaks in the vacuum flasks and will be the subject of subsequent discussion.

Examination of the paste data indicated, except for three batches, that one or more of the following conditions prevailed when abnormal setting of the paste was encountered:

1. No appreciable peak of maximum temperature developed during 24 hr or more after mixing for the pastes stored in the vacuum flask.

2. Increased dosage of retarder admixture failed to prolong the time for the paste to reach maximum temperature.

3. Increased admixture dosage was accompanied by appreciable drop in maximum temperature attained by the paste (roughly 20°F drop, or greater, for an additional 2 oz of admixture).

Table III has been prepared to assess applicability of these criteria. The nonconforming batches in the top portion of the table are from the 72 batches for which no data is available from the corresponding concrete, whereas those in the bottom portion are from the 36 paste batches for which corresponding concrete was made.

It is indicated in Table III that almost in all cases, only the 7-oz dosage is predicted to cause unusual setting. This is generally a higher dosage than the manufacturers recommend. In only three out of 108 batches was unusual paste setting encountered which was not predicted by the proposed heat release criteria. However, in four cases, unusual heat release and paste

TABLE III

APPLICATION OF ADMIXTURE PERFORMANCE CRITERIA TO PASTES AND CONCRETE

Cement No.	Admixture No.	Nominal Mixing Temp, °F	Admixture Dosage, oz/100 lb	Unusual Heat Release in Vacuum Flask	Abnormal Set		
					Paste		Concrete
					Vecat	Cone	
1	1	60	7	3		a	No data
1	1	75	7	3		a,b	No data
1	1	90	7	1	b	a,b	No data
2	1	90	7	3		b	No data
3	1	60	7	1		b	No data
3	1	75	7	1		a,b	No data
3	1	90	7	1	a,b	a,b	No data
2	2	90	7	3		a,b	No data
2	3	60	7	---		a	No data
2	3	75	7	---		a	No data
2	3	90	7	---		a	No data
3	2	60	7	2,3		a	d
3	2	75	7	2,3	a,b	a,b	d
3	2	90	5	1	a	a	---
3	2	90	7	1	a	a	a,c
3	3	60	7	1	c	c	---
3	3	90	7	1	c	a	---
3	4	90	7	3		a	---

Symbols:

Paste in Vacuum Flask

1 - No heat peak observed in paste

2 - Increased admixture dosage failed to prolong time for paste to reach heat peak

3 - Increased admixture dosage was accompanied by appreciable drop in maximum temperature attained by paste (greater drop than 20°F)

Setting Characteristics

a - Increased dose accompanied by more rapid initial set

b - Increased dosage failed to prolong, or diminished time for, final set

c - Increased dosage prolonged set excessively

d - Very rapid slump loss of concrete

setting behavior was not accompanied by unusual setting of the corresponding concrete. Thus, with respect to concrete, the criteria may be overly conservative. In no case, however, is it contemplated that "rejection" of the admixture be involved but simply a caution that excessive dosage may cause unusual setting.

CONCLUDING OBSERVATIONS

Work of others and the research undertaken under the present contract indicate that the performance of water-reducing retarders in concrete is a matter of considerable complexity. Variations in performance due to admixture dosage, mixing temperature, chemistry and perhaps physical properties of cement, chemistry of the admixture and other less well identified variables, suggest that no simple prediction will be forthcoming. Indeed, the usual recommendations now prevalent are that, prior to use, job cement and admixture be procured and trial batches of concrete be made to ascertain performance. This is a time-consuming, cumbersome operation and hardly warranted except for a very large construction project where much depends upon satisfactory performance of the admixture.

In the present work, an abbreviated test for predicting job setting performance of admixtures has been investigated. This procedure involves automatic recording of cement paste-admixture temperatures when the pastes are stored in vacuum thermos flasks to conserve the heat. The time-temperature "signature" generated by the paste is quite successful in detecting unusual setting of the cement-admixture combination. Although job cement and admixture are needed to perform the evaluation, less than 2 lb of cement is needed for each batch, and only about 1 lb per batch would be required if experience proves that the setting time tests can be foregone. If setting time tests be excluded, the present equipment owned by the department (except for six additional thermos flasks) could handle at least ten batches per day with an estimated expenditure of about six man-hours. It seems unlikely that even this work load would often be experienced due to the limited number of admixture-dosage-cement-temperature combinations needed to be evaluated by the MDSH.

It is envisioned that the proposed criteria will identify the dosages and mixing temperatures which for the particular cement and admixture combination unusual job setting of concrete can be expected.

Unusual setting of the concrete so far noted has been manifested by very rapid loss of slump, rapid attainment of 60 psi penetration resistance of wet-screened mortar, or excessively long time of setting (10-12 hr or more). Generally, such unusual setting occurred at high dosages of retarder (7-oz per 100 lb of cement). Good correlation was generally found between paste temperature measurements and paste setting time, the latter which in turn is considered well correlated with concrete performance. However, four batches of concrete displayed relatively normal setting characteristics whereas their vacuum flask paste counterparts predicted the reverse. This suggests the necessity of further work to determine if the different water-cement ratios of the two causes the difference. Another interpretation is that the paste test is very sensitive to small differences causing changes in setting characteristics, but the occasional oversensitivity should not be reason for too much concern in view of the fact that acceptance or rejection of the admixture is not involved.

A perplexing aspect of the study of retarding admixtures in concrete is that of ascertaining whether field forces place greater emphasis on prolongation of set as measured by penetration tests of sieved mortar or on preventing rapid loss of slump. The two measures of set are not at all well related to each other. Rapid loss of slump may, or may not, be accompanied by rapid attainment of high penetration resistance of sieved mortar as well demonstrated in Table II. Resolution of this matter will demand combined effort of field construction forces and laboratory personnel, and until such time that it is successfully resolved there is bound to be uncertainty as to the exact nature of the prediction most needed.

RECOMMENDATIONS

It is recommended that immediately before casting a bridge deck where water-reducing retarder will be employed, a generous amount of job cement be sent to the laboratory (25 lb or more), and a series of pastes be made up incorporating amounts of the brand of retarder to be used on the job and at temperatures appropriate for the season, probably 60° and 75°F for spring or fall construction and 75° and 90°F for summer. Dosages employed should span the manufacturer's recommendation, probably 0, 2, 4, and 6 oz per 100 lb of cement. Job experience may indicate the desirability of periodic laboratory samples during the course of the work, particularly if the time for casting the bridge deck is prolonged.

The pastes should be made up using the technique described in Appendix A.

Field personnel should be informed of the probability of job setting difficulties when one or more of the following conditions is observed for the cement-admixture pastes stored in the vacuum flasks:

1. No heat peak observed in the paste for 24 hr. This can often be immediately predicted when severe set occurs during the initial 10-min wait period during mixing the paste.

2. Increased dosage of admixture fails to prolong time for the paste to reach a heat peak.

3. Increased admixture dosage is accompanied by appreciable drop in maximum temperature attained by the paste (greater drop than 20°F for additional 2 oz increment).

Generally, this data should be available for advising field personnel by noon of the second day after receiving the cement.

The above contemplates foretelling job difficulties with setting time only. It is recognized that other job problems may arise when using admixtures such as unusual air-entrainment or lack of water reduction and will have to be separately handled.

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REFERENCES

1. Tuthill, L. H., and Gordon, W. A., "Properties and Uses of Initially Retarded Concrete," ACI Journal, Proc., Vol. 52, 1955, pp. 273-286.

2. "Admixtures in Concrete," Highway Research Board Special Report 119, 1971, pp. 14-20.

3. "Guide for Use of Admixtures in Concrete," Reported by ACI Committee 212, ACI Journal, Proc. Vol. 68, 1971, pp. 646-676.

4. Britton, H. B., "Continuous Integral Deck Construction; A Rational Approach to Placing Structural Deck on Three-Span Continous Bridge Deck Units," Highway Research Board Bulletin 362, 1962, pp. 8-16.

5. Hilsdorf, H. K., and Lott, J. L., "Revibration of Concrete for Continuous Bridge Decks," National Cooperative Highway Research Program Report 106, 1970, pp. 1-67.

6. Tuthill, L. H., Adams, R. F., Bailey, S. N., and Smith, R. W., "A Case of Abnormally Slow Hardening Concrete for Tunnel Lining," ACI Journal, Proc. Vol. 57, 1961, pp. 1091-1109.

7. Polivka, Milos, and Klein, Alexander, "Effect of Water-Reducing Admixtures and Set-Retarding Admixtures as Influenced by Portland Cement Composition," ASTM STP, No. 266, "Symposium on Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete," 1959, pp. 124-139.

8. "Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete," ASTM Special Technical Publication, STP No. 266, 1959, pp. 1-246.

9. Young, J. F., "Influence of Tricalcium Aluminate on the Hydration of Calcium-Silicates," Journal of American Ceramic Society, Vol. 52, 1969, pp. 44-46.

10. Young, J. F., "Effect of Organic Compounds on the Interconversion of Calcium Aluminate Hydrates: Hydration of Tricalcium Aluminate," Journal of the American Ceramic Society, Vol. 53, 1970, pp. 65-69.

11. Steinour, Harold H., "The Setting of Portland Cement—A Review of Theory, Performance and Control," Portland Cement Association Research Bulletin 98, November 1958, pp. 1-124.

12. Taplin, J. H., "A Method for Following the Hydration Reaction in Portland Cement Paste," Australian Journal of Applied Science, Vol. 10, 1959, pp. 329-345.

13. Verbeck, George, J., and Foster, Cecil W., "Long-Time Study of Cement Performance in Concrete, Chapter 6—The Heats of Hydration of the Cements," Proc., ASTM, Vol. 50, 1950, pp. 1235-1262.

14. Bates, P. H., "Variations in Standard Portland Cement," Report of Committee 202, ACI Journal, Vol. 26, 1930, pp. 65-100.

15. Carlson, R. W., "The Significance of Early Heat Liberation of Cement Paste," Proc., Highway Research Board, Vol. 17, 1937, pp. 360-367.

16. Forbrich, L. R., "The Effect of Various Reagents on the Heat Liberation Characteristics of Portland Cement," Journal, American Concrete Institute, Vol. 37, 1940, pp. 161-184.

17. Lerch, William, "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," Proc., ASTM, Vol. 46, 1946, pp. 1252-1297.

18. Kuntze, R. A., and Hawkins, P., "Determination of the False Set of Portland Cement," Journal of Materials, ASTM, Vol. 5, No. 4, December 1970, pp. 763-766.

19. Berger, E. E., "Significant Time-of-Set Studies and Their Possible Relation to Current Time-of-Set Problems," ASTM, Proc., Vol. 63, 1963, pp. 886-903.

20. Tamas, Ferenc D., "Acceleration and Retardation of Portland Cement Hydration by Additives," Highway Research Board Special Report No. 90, 1966, pp. 392-397.

21. Bruere, G. M., "Effects of Mixing Sequence on Mortar Consistencies When Using Water-Reducing Agents," Highway Research Board Special Report No. 90, 1966, pp. 26-35.

22. Gaynor, R. D., "Tests of Water-Reducing Retarders," Publication No. 12, Joint Research Laboratory National Sand and Gravel Association and National Ready Mixed Concrete Association, August 1962, pp. 1-14.

APPENDIX A

METHODS OF PREPARATION OF PASTES AND CONCRETE

Paste Preparation

MIXING SEQUENCE

Time, Min.	Operation
0	add weighed cement to water plus admixture in bowl of Hobart N-50 mixer
1/2	start mixing on speed 1
1	stop mixer; scrape down sides of bowl for 15 sec
1-1/4	start mixing on speed 2
2-1/4	stop mixer; scrape down sides of bowl (cool entire bowl in icewater if necessary)
12-1/4	start mixing on speed 2
13-1/4	stop mixing; place sample into numbered cup and metal penetration containers lined with plastic wrap

BATCH WEIGHTS FOR PASTE

Admixture per 100 lb cement	Cement, g	Admixture, ml	H_2O, ml
0	850	0	298
3 oz	850	51	247
5 oz	850	85	213
7 oz	850	119	179

(65.2 ml admixture diluted with distilled water to make 2000 ml of solution)

Preparation of Concrete

MIXING SEQUENCE

Time, Min.	Operation
0	start mixer and simultaneously add water and undiluted admixture to weighed sand, gravel, and cement
3	stop mixer and cover with moist burlap to prevent evaporation
8	start mixer and make final addition of water to correct slump if too dry
11	stop mixer, run slump test and sieve out mortar over No. 4 sieve (using vibrating nest of 3/8 and No. 4 sieve)

BATCH WEIGHTS FOR CONCRETE, DRY

Cement	- 10.0 lb	
Sand	- 25.4 lb	Corrected for moist aggregate at time of use
Gravel (6A)	- 33.0 lb	
Water (Total)	- 4.9 lb	
Admixture	- 3 oz/100 lb - 8.9 ml	
	5 oz/100 lb - 14.8 ml	
	7 oz/100 lb - 20.7 ml	

APPENDIX B

TESTS OF PORTLAND CEMENT

		Cement No. 1	Cement No. 2	Cement No. 3
PHYSICAL TESTS				
Normal consistency, percent		25.0	25.0	26.8
Time of set, Gillmore				
initial, hr min		3:10	2:00	2:55
final, hr min		4:55	3:45	4:55
Air in mortar, percent		11.8	11.3	11.7
Specific surface, air permeability, sq cm/g		4,102	4,339	3,481
Autoclave expansion, percent		0.07	0.08	0.11
Compressive strength, mortar, psi				
7-days		3,410	3,980	3,740
28-days		4,570	5,020	5,330
False set, mortar, C 359				
Penetration at 5 min, mm		2	36	50+
Penetration at 8 min, mm		0	21	50+
Penetration at 11 min, mm		1	12	50+
CHEMICAL COMPOSITION (percent by weight)				
Silicon dioxide,	SiO_2	20.1	20.1	21.1
Aluminum oxide,	Al_2O_3	6.1	5.5	5.6
Ferric oxide,	Fe_2O_3	2.7	2.6	2.8
Calcium oxide,	CaO	61.9	63.0	62.4
Magnesium oxide,	MgO	2.3	2.5	3.5
Sulfur trioxide,	SO_3	3.2	3.0	2.5
Loss on ignition		2.5	2.2	1.0
Sodium oxide,	Na_2O	0.22	0.27	0.18
Potassium oxide,	K_2O	0.71	0.62	0.69
Total alkali as	Na_2O	0.69	0.68	0.63
Insoluble residue		0.3	0.3	0.2
Calculated compounds				
tricalcium silicate,	C_3S	45	54	45
dicalcium silicate,	C_2S	24	17	27
tricalcium aluminate,	C_3A	11.5	10.3	10.0
tetracalcium aluminoferrite,	C_4AF	8.2	7.9	8.5

APPENDIX C

CHARACTERISTICS OF AGGREGATES USED IN MAKING CONCRETE

Aggregates

FINE AGGREGATE

Natural Sand, 2NS Gradation

Specific Gravity (dry) - 2.59

Absorption, percent - 1.48

COARSE AGGREGATE

Natural Gravel (6A gradation, 1-in max. size)

Specific Gravity (dry) - 2.68

Absorption, percent - 1.05

wt/cu ft, dry, loose, lb - 98

APPENDIX D

DETAILED TESTS OF PASTES

DETAILED TESTS OF PASTES

CEMENT NO.	ADMIXTURE	NOMINAL TEMP. °F	ADMIXTURE DOSAGE OZ/100 lb	HEAT GENERATION: TIME TO REACH MAXIMUM TEMP MIN	HEAT GENERATION: PEAK TEMP °F	SETTING TIME - MIN: VICAT 25 mm PEN	SETTING TIME - MIN: VICAT 5 mm PEN	SETTING TIME - MIN: CONE 30 mm PEN	SETTING TIME - MIN: CONE 5 mm PEN	REMARKS
1	1	60	0	500	116	370	420	170	300	
			3	1200	155	~1320	~1370	300	~1230	
			5	1500	156	~1860	~1890	120	1620	
			7	2040	130	1980+	1980+	30	~1800	
		75	0	530	114	250	270	100	220	
			3	950	155	720	760	160	670	
			5	1380	139	735+	735+	60	720+	
			7	1740	112	~700	~760	45	220	
		90	0	370	145	130	160	50	120	
			3	550	142	320	340	65	290	
			5	810	121	440	490	110	340	
			7	N.D.	N.D.	210	250	20	100	No peak at 31 hr.
2	1	60	0	435	121	300	340	155	255	
			3	615	163	515	600	270	435	
			5	960	166	1005	1140	450	900	
			7	1410	150	1665	~1800	240	1560	
		75	0	360	135	195	225	80	165	
			3	600	175	370	420	210	345	
			5	960	176	~790	~850	330	~765	
			7	1290	158	750+	750+	195	750+	
		90	0	360	143	120	140	60	105	
			3	600	174	330	350	165	315	
			5	900	168	600	615	165	570	
			7	1200	138	660	750	110	570	
	2	60	0	405	122	285	340	135	240	
			3	510	166	385	450	225	345	
			5	630	182	500	620	320	510	
			7	675	184	690	795	240	875	
		75	0	360	122	180	200	85	150	
			3	510	165	280	310	165	250	
			5	615	176	375	430	240	370	
			7	750	170	525	585	260	525	
		90	0	315	140	120	140	60	95	
			3	420	192	220	240	120	195	
			5	540	190	320	345	145	310	
			7	600	165	370	420	75	330	
	3	60	0	420	125	295	385	105	195	
			3	570	161	440	540	195	345	
			5	825	172	765	840	390	720	
			7	1020	166	N.D.	N.D.	120	~1020	N.D. - Not determined.
		75	0	345	133	215	230	105	165	
			3	510	171	360	405	190	310	
			5	735	180	600	630	260	585	
			7	990	169	~735	~790	~30	795	
		90	0	360	140	120	135	50	100	
			3	480	176	225	250	130	210	
			5	540	180	320	345	130	300	
			7	855	175	585	610	75	550	
	4	60	0	410	126	270	345	135	240	
			3	555	158	400	495	210	365	
			5	690	172	570	690	290	540	
			7	900	163	855	960	330	840	
		75	0	360	131	170	210	85	150	
			3	480	167	270	345	150	255	
			5	630	176	420	495	275	410	
			7	795	169	615	690	320	580	
		90	0	345	135	145	165	75	120	
			3	450	175	225	270	135	215	
			5	525	185	295	350	165	300	
			7	750	176	510	615	120	480	
3	4	60	0	510	129	345	420	135	300	
			3	660	168	525	600	255	450	
			5	840	185	630	735	255	570	
			7	1260	172	1840	1950	255	1740	
		75	0	450	129	195	265	95	195	
			3	615	171	345	405	165	330	
			5	780	182	520	575	285	495	
			7	1200	175	855	945	240	870	
		90	0	390	136	160	180	80	135	
			3	570	172	290	345	150	300	
			5	810	185	470	540	210	470	
			7	1440	163	~940	1035	140	900	
	3	60	0	480	129	360	450	150	330	
			3	660	175	555	630	255	480	
			5	930	187	780	930	300	780	
			7	N.D.	N.D.	NO SET	NO SET	270	NO SET	No peak, Vicat set, or 5 mm conc set at 24 hrs.
		75	0	420	134	210	270	105	180	
			3	600	179	370	420	195	350	
			5	810	188	575	660	285	525	
			7	1420	179	1150	1220	930	1125	
		90	0	405	130	135	175	75	135	
			3	600	180	300	355	150	355	
			5	900	185	530	600	225	525	
			7	N.D.	N.D.	~2000	~2000	150	~2000	N.D. - No heat peak at 26 hrs.
	2	60	0	510	130	345	435	135	300	
			3	600	175	450	510	225	390	
			5	690	190	585	705	300	540	
			7	210	100	780	840	225	660	
		75	0	465	129	215	255	105	180	
			3	570	175	315	375	180	290	
			5	690	186	450	505	250	420	
			7	180	101	~250	~260	165	330	
		90	0	405	126	150	175	80	150	
			3	435	149	175	210	45	150	
			5	NO PEAK						Severe set in first 10 min.
			7	NO PEAK						Severe set in first 10 min.
	1	60	0	450	135	345	435	135	270	
			3	690	173	600	720	175	555	
			5	1230	173	N.D.	N.D.	315	1050	No Vicat set in 17 hrs.
			7	N.D.	N.D.	N.D.	N.D.	210	1050	No Vicat set in 17 hrs. (No peak at 28 hrs.)
		75	0	450	127	210	250	90	180	
			3	765	169	470	540	180	435	
			5	1440	163	900+	900+	240	765	
			7	N.D.	N.D.	600	900	135	480	No peak at 24 hrs.
		90	0	390	131	145	180	75	120	
			3	780	171	420	480	180	390	
			5	1320	155	675	795	165	405	
			7	N.D.	N.D.	405	615	75	300	No peak at 23 hrs.