

**THE EFFECT OF CHEMICAL ADMIXTURES  
ON THE DRYING SHRINKAGE OF CONCRETE AND THE CONTROL  
OF CHEMICAL UNIFORMITY OF ADMIXTURES**

by

**Patrick H. Torrans**  
Research Assistant

**Don L. Ivey**  
Assistant Research Engineer

**Teddy J. Hirsch**  
Associate Research Engineer

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# Part I: The Effect of Chemical Admixtures on the Drying Shrinkage of Concrete Batched at 75° F

## Chapter I Introduction

### General

In recent years chemical admixtures for use in Portland cement concrete have come into prominent use. A chemical admixture is "a material other than water, aggregates, and portland cement (including air-entraining portland blast-furnace slag cement) that is used as an ingredient of concrete and is added to the batch immediately before or during its mixing."<sup>1\*</sup> These admixtures are employed to improve or modify certain properties of concrete, to compensate for some deficiency in a primary constituent, or effect a reduction in cost. Among the more important properties modified by the use of admixtures are workability, strength development, time of set, and adverse effect of high temperature. While these properties may be beneficially modified, it is pointed out that a specific admixture may affect more than one property of the plastic or hardened concrete, and not all such effects are necessarily beneficial. In particular, it has been found that many of the admixes in use at the present time cause an undesirable amount of drying shrinkage in the concrete.

This report is concerned with the effect that certain chemical admixes have on drying shrinkage, and data are presented which show that under current specifications<sup>2</sup> the acceptance or rejection of certain admixes on the basis of their contribution to shrinkage may be purely a matter of the constituents used in the test concrete.

### Phenomenon of Shrinkage and Swelling of Concrete

Powers<sup>3</sup> states that shrinkage and swelling of concrete are almost exclusively a property of the paste. It has been established that cement paste is an aggregation of colloidal particles so minute that the average amount of solid material in each particle of the aggregation could be contained in a sphere having a diameter of about four thousandths of a micro-inch. Electron-micrographs obtained by Grudemo in 1957 showed that these particles, referred to as gel particles, were in the shape of slender ribbons and crumpled sheets or foils. This aggregation of gel particles contains a characteristic amount of interstitial space, referred to as gel pores. A unit volume of gel (gel particles plus gel pores) is composed of about .65 units of solid, the rest being gel pores.

The structure of the paste may be such that gel does not fill all the space within the boundaries and those spaces not occupied by the gel are referred to as capillary pores or capillary cavities.

\*Superior numbers denote corresponding references cited in Bibliography.

It has been pointed out that the particles composing the cement gel are exceedingly small. The dimensions of the interstitial spaces are also minute; the average distance between gel pores being on the order of four times the diameter of a water molecule. It is known that when two bodies have such proximity they exert mutual forces known as the van der Waal forces.

When water molecules are allowed to invade dry paste, the mutual attraction between the invading molecules and the molecules or atoms composing the solid surfaces cause the invaders to spread over the gel particles. When this spreading is obstructed by adjacent solid surfaces, (surfaces of other gel particles) pressures develop which cause the gel particles to be displaced or spread apart. The magnitude of these swelling pressures depends on the intensity of interaction between the invading molecules and the surfaces. In the case of invading water molecules this interaction is very strong and thus the water molecules act against the cohesive van der Waal forces and produce swelling.

Shrinkage may be explained by reversing the above explanation. As the water evaporates, the forces producing displacements of gel particles are diminished and the van der Waal forces draw the gel particles together.

As dry gel takes on moisture the physical reaction results in a release of heat and the interfacial surface tension between gel particles is reduced. With surface tension reduced, the particle is allowed to expand. Thus, when water is withdrawn from the wet paste, distortions of particles caused by their own surface tensions will be reversed, accounting for the shrinkage of the drying paste.

As these explanations of shrinking and swelling point out, it is those water molecules within the range of the force field of the solid surfaces (i.e., within the range of mutual attraction) that contribute to volume change, and it would seem that those particles in direct contact with solid surfaces have by far the greatest effect. The water molecules in direct contact with solid surfaces are present as adsorbed water while those molecules having less effect are present as capillary water.

Consequently, shrinkage from loss of water depends on what portion of the water loss is adsorbed and what amount is capillary. Initial water loss constitutes the capillary water while secondary losses are adsorbed water. As a result the initial losses have a much different effect on shrinkage than do the losses occurring at a later stage.

This is illustrated in Figure 1, which was published by Menzel.<sup>4</sup> The curves show water loss versus shrink-

## SHRINKAGE VS. WATER LOSS BY MENZEL (4)

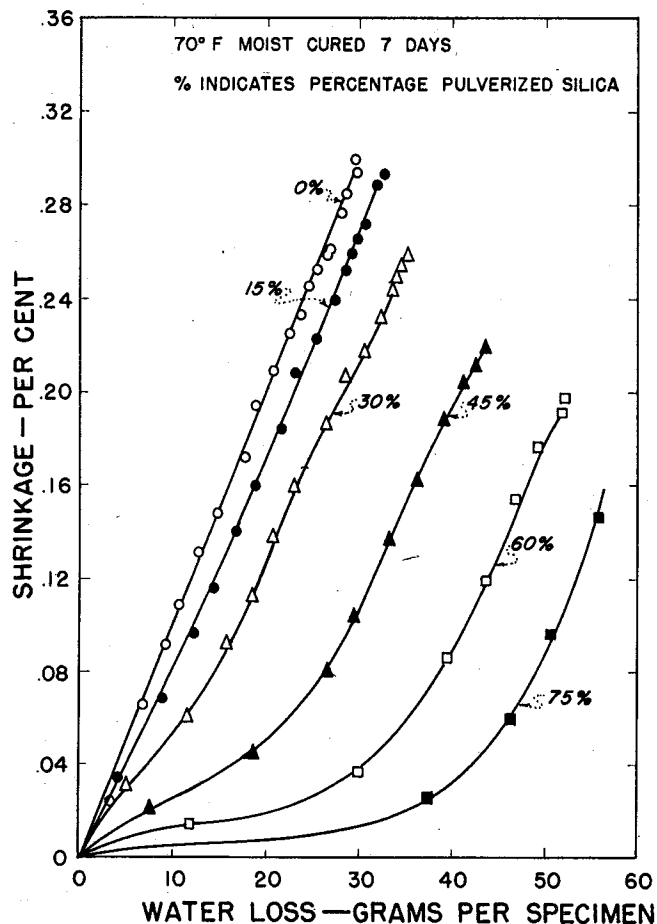


Figure 1.

age for specimens ranging in consistency from a neat cement (cement and water) with 0% pulverized silica to specimens containing 75% pulverized silica and 25% cement. The proportion of capillary cavities in the paste increased with the proportion of silica. Shrinkage of the densest specimen is directly proportional to the water loss, indicating little if any capillary water loss. Presence of capillary cavities is evident from the shape of the curves (deviation from a straight line variation) and it is noted that shrinkage versus water loss is not linear for these specimens. From these considerations it appears that a reduction in the water requirement of a given mix design would reduce the ultimate shrinkage. Carlson states that<sup>5</sup> "in concrete, as in neat cement, the water content is probably the most important single factor affecting shrinkage. Any condition which will permit the use of less mixing water will reduce inherent shrinkage greatly." Other research which confirms Carlson's findings has been done on concrete containing no chemical admixtures.<sup>6,7</sup>

This led to the assumption that any admix which reduces the water requirement of a given mix design would also reduce the ultimate shrinkage. However, work done by the Bureau of Public Roads and the California Division of Highways shows that the chemical composition of the admix may be such that the shrinkage is increased even though the water requirement is reduced.<sup>8,9</sup> Trem-

per and Spellman state that<sup>10</sup> "the effect on shrinkage of members known as chemical admixtures is varied and cannot be correlated with a possible reduction in unit water content, which often is substantial in amount. Many, but not all, chemical admixtures of the set-retarding type reduce shrinkage or increase it only slightly. On the other hand, some set-retarding admixtures and most lignin base admixtures that have been treated to destroy set-retarding properties, when used at manufacturer's recommended dosage, produce substantial increases in shrinkage, up to 60 percent when measured in concrete specimens dried for 14 days." They further point out that "there is evidence that many chemical admixtures react with the cement, thus forming compounds that are not otherwise present. This is a probable reason that many of them increase shrinkage while also reducing the unit water content."

Work done in the concrete laboratory of the Civil Engineering Department, Texas A&M University, has shown that in many instances, dealers' recommended dosage was not sufficient to meet the specifications for initial set retardation as stated in the Texas Highway Department Standard Specifications for Road and Bridge Construction. Thus, the 60% increase in shrinkage of concrete containing admix at dealer's recommended dosage would not seem a reasonable figure when the dosage must be increased considerably to meet these specifications.

### Present Method of Test

Because of the increased use of commercial admixtures in the field, and the wide range of effects caused by their use, the American Society for Testing Materials has published a tentative specification for the acceptance or rejection of chemical admixtures.<sup>2</sup> The specification separates the admixtures into five types according to their use in the field and defined the types as follows:

Type A: Water-reducing admixtures — an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency.

Type B: Retarding admixtures — an admixture that retards the setting of concrete.

Type C: Accelerating admixture — an admixture that accelerates the setting and early strength development of concrete.

Type D: Water-reducing and retarding admixtures — an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency and retards the setting of concrete.

Type E: Water-reducing and accelerating admixture — an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency and accelerates the setting and early strength development of concrete.

The specification<sup>2</sup> states allowable values for water reduction, time of setting, compressive, flexural, and bond strength, volume change, loss of water by bleeding, and durability relative to a control concrete containing no admixture.

Aside from being lengthy and time consuming some researchers feel that the test for volume change as required by specifications<sup>2</sup> is unrealistic because it fails to take into account the effect of the aggregate.

The specification<sup>2</sup> allows shrinkage of a 4" x 4" x 10" concrete specimen, moist cured for 14 days and containing any of the above admixes, to be 100 micro-inches greater than the control concrete (containing no admixture) at 28 days, six months, and one year.

Bailey Tremper of the California Division of Highways maintains<sup>11</sup> that there is "an inconsistency in the method of specifying the effect on volume change as a numerical increase with relation to the control. It is noted that the effect of the admixture on most other properties is specified as a percentage of the control, not as an absolute difference. The reason for specifying changes in properties in terms of percentage of control is because differences in test cements and test aggregates may affect absolute differences and thus not express the effect of the admixture in a consistent manner. The same reasoning applies equally well to volume change."

Concerning the effect of aggregate type, Carlson<sup>5</sup> states that "large differences in concrete shrinkage are obtained from different aggregates, especially in lean mixes. Some difference results from the fact that one aggregate requires more mixing water than does another, but the greater difference is probably due to the physical properties of the aggregate itself."

The most important factor contributing to the ability of aggregate particles to restrain the cement paste is the non-compressibility of the aggregate itself. That is, if the aggregate were readily compressible, the concrete would shrink as much as the paste. Of the aggregates tested by Carlson, it was found that sandstone and slate produce high shrinkage. Among the pure mineral aggregates that were tested, hornblends and pyroxene produced the highest shrinkage. Quartz, feldspar, dolomite and calcite produce low shrinkage. Pickett<sup>12</sup> was able to show theoretically and confirm experimentally that in practical concrete mixes, the size and gradation of

aggregate affected shrinkage only slightly and that shrinkage was a function of the total quantity of aggregate per unit volume of concrete.

The cement paste in concrete may be partly restrained by unhydrated cement or clinker, just as it is by aggregates. With an increase in duration of moist curing, it may be expected that the capacity of the paste for shrinkage is increased by eliminating clinker. Powers<sup>3</sup> states that prolonged curing makes paste more prone to crack when severely restrained and that if cracking of paste relieves stresses around aggregate particles, the over-all shrinkage may thereby be diminished.

From these considerations it appears that if the present specification<sup>2</sup> is to limit volume change satisfactorily, some modification is needed.

### **Objectives**

As indicated in the foregoing discussion there is not now a generally satisfactory method of determining the effect of chemical admixes on the shrinkage of concrete.

Accordingly, the objectives of this project were as follows:

1. Determine the effect that varying periods of moist curing have on volume change.
2. Determine the effect of different type admixtures when used with different aggregate types.
3. Determine the effect of drying temperature and humidity on volume change in order to investigate the possibility of a more rapid test procedure.
4. Investigate the length of drying period on shrinkage of concrete containing an admix relative to a control, to determine if a reduction in time required for the test can be realized.
5. Using this information, evaluate the method of test proposed by the American Society for Testing Materials for determining the effect of chemical admixtures on volume change of concrete.

## **Chapter II**

### **Testing Program**

Fourteen batches of concrete were poured in this study, each containing about two cubic feet of concrete. The batch proportions of all batches are given in Table 1. Batches S-1 through S-7 were made from a siliceous gravel and sand, and batches L-1 through L-7 were made with a crushed limestone coarse aggregate and sand fine. The unit weight, specific gravity, absorption, and sieve analysis of these aggregates are presented in Table 2. All coarse aggregate was washed to insure that no deleterious material coated it, then sieved and recombined to give a constant gradation.

Table 3 gives the specimen schedule for batches S-1 and L-1; Table 4 for S-2 through S-7 and L-2 through L-7.

#### **Design and Fabrication of Batches**

The concrete batches were designed by the absolute volume method with a coarse aggregate factor of 0.7 and

a cement factor of 5½ sacks of cement per cubic yard. Type I Atlas portland cement was used.

Prior to mixing, samples of the aggregate were taken for moisture content determination. The prescribed amounts of fine and coarse aggregate were weighed and placed in a vertical drum mixer with a rated two cubic foot capacity.

The aggregate was mixed thoroughly before the other constituents were added. Then the air-entraining admixture was mixed with one-fourth of the total water and added to the batch prior to the addition of cement. If an admixture other than an air-entraining admix was to be used it was mixed with another one-fourth of the estimated water and added to the batch. The cement was then added and the time was recorded in order that setting time determinations could be made later. Finally, sufficient water was added to cause the slump to be 4 ± ½". When the slump was in this range, air content,

Table 1  
**CONCRETE MIX DATA**  
 Batches S-1 through 7 and L-1 through 7  
 Quantities per C. Y. Concrete

Batch Designation	Aggregate Type	Cement, Sacks	Aggregate (SSD)		Mixing Water, lbs.	Air* Content, %	Slump, in.	Admix**
			Coarse, lbs.	Fine, lbs.				
S-1	Siliceous	5.55	1795	1404	361	4.1	3.5	None
S-2	Siliceous	5.67	1810	1418	322	5.2	4	AL
S-3	Siliceous	5.44	1802	1407	304	7.0	4	DL <sub>1</sub>
S-4	Siliceous	5.40	1797	1406	353	4.5	4	CC
S-5	Siliceous	5.50	1815	1413	295	4.0	4.5	DL <sub>2</sub>
S-6	Siliceous	5.50	1800	1409	330	2.9	4	DO <sub>1</sub>
S-7	Siliceous	5.54	1812	1416	322	5.5	4	DO <sub>2</sub>
L-1	Limestone	5.40	1796	1405	360	4.5	4	None
L-2	Limestone	5.44	1800	1408	332	4.9	4	AL
L-3	Limestone	5.39	1795	1404	311	3.5	3.5	DL <sub>1</sub>
L-4	Limestone	5.35	1797	1405	348	5.0	3.5	CC
L-5	Limestone	5.55	1814	1413	320	5.6	3.5	DL <sub>2</sub>
L-6	Limestone	5.38	1801	1409	310	4.8	4	DO <sub>1</sub>
L-7	Limestone	5.45	1802	1407	314	4.0	3.5	DO <sub>2</sub>

\*Sika-Aer Air-entraining agent was used in all batches to give air content.

\*\*The first letter of the admix designation is the ASTM designation given in ASTM C494-63T (A-Water reducing, C-Accelerating, D-Water reducing and retarding). The second letter refers to chemical type (L-Lignosulfonate, O-Organic Acid, C-Calcium Chloride). The subscript which may occur differentiates between different commercial products of the same ASTM designation and chemical type.

unit weight, and batch temperature were determined. Mixing time after the addition of cement was between 8 and 10 minutes.

The air content was determined with a "Press-ur-meter" manufactured by the Concrete Specialties Company, Spokane, Washington. The method of test conforms with ASTM Designation: C231-56T rather closely except that in the preparation of the air content sample it was vibrated instead of hand-tamped. This subjects

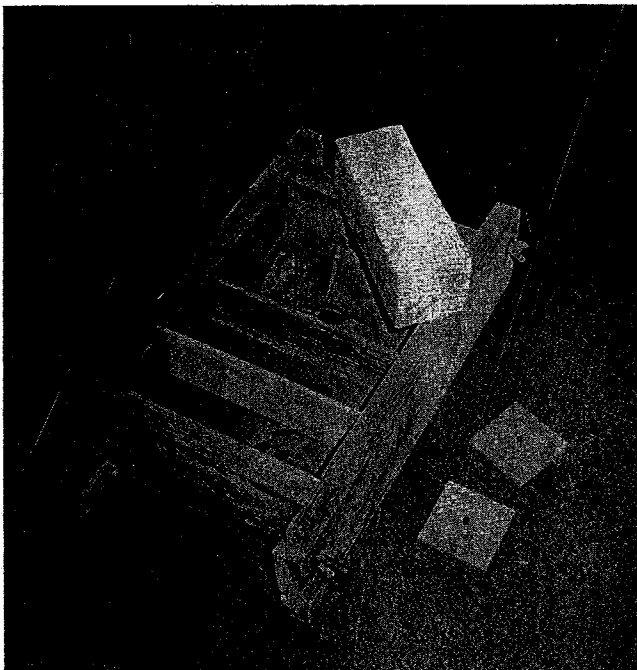


Figure 2. Mold and gage studs for shrinkage specimens.

Table 2  
 AGGREGATE PROPERTIES

	Limestone used in batches L-1 thru 7, coarse	Siliceous used in batches S-1 thru 7, coarse
Unit weight in lb/c.f. (dry loose)	88	93
Specific gravity (SSD)	2.64	2.61
Absorption (% of dry wt.)	1.44	1.24

Sieve analysis,  
% passing

1	100	96.70
3/4	100	88.85
1/2	71.15	61.14
3/8	18.27	24.14
#4	1.40	4.57
#8	1.40	1.39
#16	1.40	.78
#30	1.40	.56
#50	.14	.38
#100	.14	.28
#200	.08	.16

Natural sand fine aggregate used in S-1 thru 7 and L-1 thru 7

Unit weight in lb./cu.ft. (dry loose)	98.5
Specific gravity (SSD)	2.62
Absorption (% of dry wt.)	.81

Sieve analysis,  
% passing

#4	99.74
#8	92.96
#16	87.01
#30	70.16
#50	18.40
#100	2.38
#200	.31



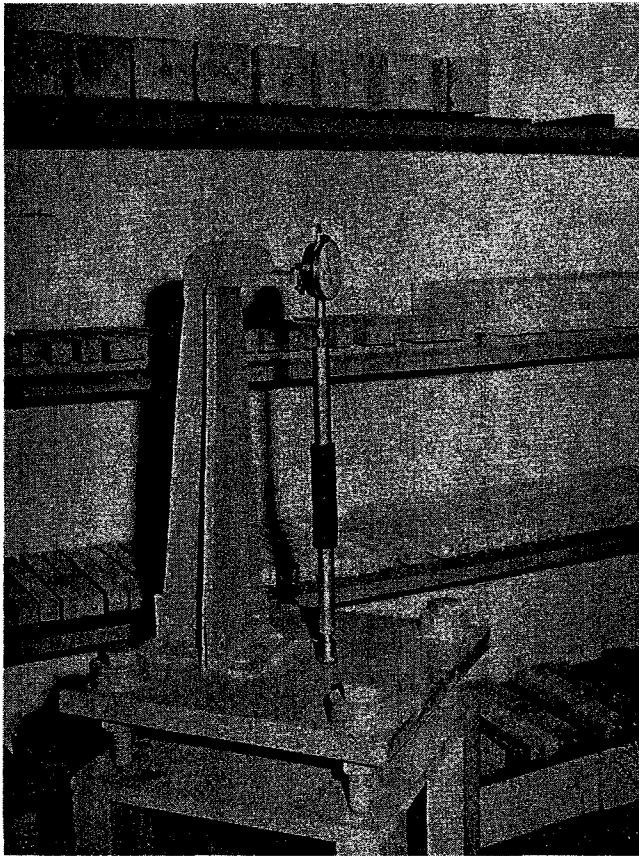


Figure 3. Length comparator and standard.

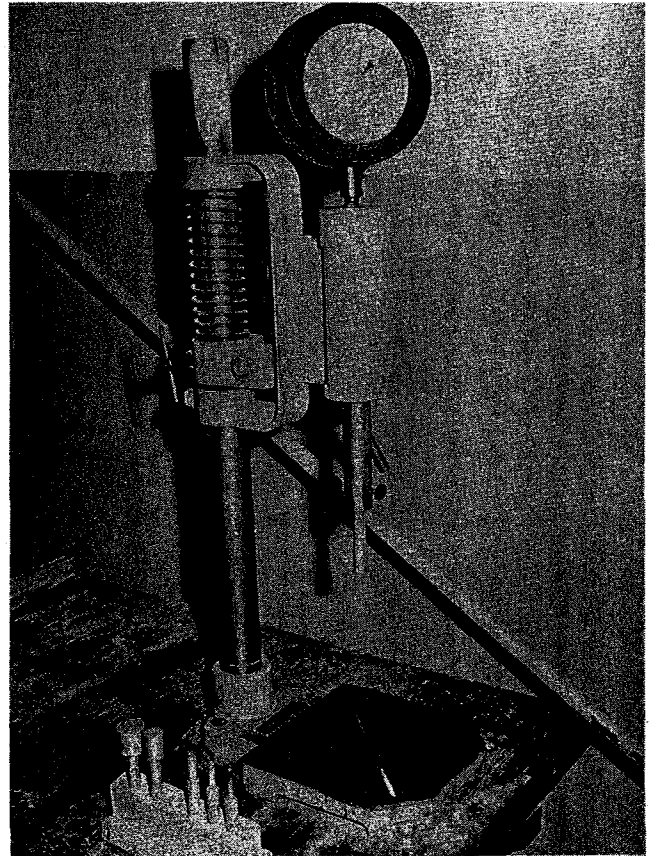


Figure 4. Proctor penetrometer.

the sample to the same casting method as the test specimens and minimizes the quantity of entrapped air.

*Molds.* Molds for the shrinkage specimens (4" x 4" x 11" prismatic specimens) were made of wood of sufficient thickness to prevent distortion during placing of the concrete. The device for holding the gage studs

in position was so arranged that it could be released after the mixture had been compacted in order to prevent restraint of the gage studs during initial shrinkage of the specimen. The molds were so designed that the distance between the innermost ends of the gage studs (used as the gage length in computations of unit length change) were  $10 \pm 0.1$  in. Prior to molding the speci-

Table 3  
SPECIMEN SCHEDULE  
Batches S-1 and L-1

Specimen Number	Specimen Type	Curing	Type of Test
1	4" x 4" x 11"	Placed in $60 \pm 5\%$ * Relative humidity and 72° F after removal from molds at 36 hours	All specimens listed were measured for shrinkage
2	4" x 4" x 11" Prism		
3	4" x 4" x 11"		
4	4" x 4" x 11"		
5	4" x 4" x 11"	Placed in $60 \pm 5\%$ Relative humidity and 72° F after 7 days moist curing	"
6	4" x 4" x 11" Prism		
7	4" x 4" x 11"		
8	4" x 4" x 11"		
9	4" x 4" x 11"	Placed in $60 \pm 5\%$ Relative humidity and 72° F after 14 days moist curing	"
10	4" x 4" x 11" Prism		
11	4" x 4" x 11"		
12	4" x 4" x 11"		
13	4" x 4" x 11"	Placed in $60 \pm 5\%$ Relative humidity and 72° F after 28 days moist curing	"
14	4" x 4" x 11" Prism		
15	4" x 4" x 11"		
16	4" x 4" x 11"		
17	6" x 6" cylinder	None (Test on wet mortar)	Penetration Resistance ASTM: C403
18	6" x 6" cylinder		

\*The relative humidity was maintained at  $60 \pm 5\%$  by a salt solution contained in trays and the room was air-conditioned to maintain  $72^\circ \pm 2^\circ$  F temperature.

Table 4  
SPECIMEN SCHEDULE  
Batches S-2 thru S-7 and L-2 thru L-7

Specimen Number	Specimen Type	Curing	Type of Test
1	4" x 4" x 11" Prism	Moist cured for 7 days and stored in 60 ± 5% Relative humidity and 72° F	Shrinkage measurements
2			
3			
4	4" x 4" x 11" Prism	Moist cured 7 days and stored in 17% Relative humidity and 120° F	Shrinkage measurements
5			
6			
7	3" x 4" x 16" Prism	Moist cured for 28 days	Weight and sonic modulus determined at 3, 7, 14, 28 days. Flexure and compressive strength at 28 days
8			
9			
10	3" x 4" x 16" Prism	Moist cured for 7 days, then subjected to ASTM freeze-thaw test C310-57	Weight and sonic modulus determined every 5 freeze-thaw cycles
11			
12			
13	6" x 6" Cylinder	None (Test on wet mortar)	Penetration Resistance ASTM: C403
14	6" x 6" Cylinder		

mens, the joints of the molds were sealed with wax to prevent leakage of water from the concrete. The concrete was placed in the molds in three layers and after each layer was placed it was vibrated. Figure 2 shows the gage studs and molds used.

*Length Comparator.* The comparator used for measuring linear shrinkage was designed to provide a positive means of contact with the gage studs. The comparator utilized an Ames dial graduated to .0001 in. A 0.5 in. range in the measuring device allowed for variations in the gage length of specimens. A bar of Invar steel was used for the reference length measurements. Figure 3 shows the length comparator and the standard reference bar used for measuring shrinkage.

### Test Procedures

*Shrinkage Tests.* Initial length measurements were made on shrinkage specimens after they had been subjected to their designated moist curing periods. The

specimens were then allowed to dry, as shown in Tables 3 and 4. Comparator readings were taken at frequent intervals as long as the specimens underwent rapid shrinkage. The reading intervals gradually increased to once a month as the shrinkage rate became small.

*Time of Setting by Proctor Penetrometer.* The method of test used here is in accordance with ASTM Designation: C403-61T. A Proctor penetration apparatus with a hydraulic indicating dial was used. The apparatus is shown in Figure 4.

*Modulus of Rupture.* The modulus of rupture values were obtained by testing the 3" x 4" x 16" specimens with a center point load applied perpendicular to the 3" side over a span of 14". Except for the span length, this test was conducted in accordance with ASTM Designation: C293-54T.

*Compressive Strength.* The modified cube compressive test was used in this series of tests and was performed in accordance with ASTM Designation: C116-49.

## Chapter III

### Test Results and Discussion

#### Water Reduction and Set Retardation

The water reduction and set retardation obtained from the six admixtures used in batches S-2 through S-7 and L-2 through L-7 are given in Table 5. Type D admixes (see page 7) are set-retarding water reducing admixtures and it should be noted that the dealers' recommended dosage and the actual amount used differ significantly in many instances; this was done in order to meet Texas Highway Department specifications which require that the initial set must be retarded by a minimum of two hours over the same concrete containing no admix. A laboratory dosage to meet this specification was determined by successive trials. The increased dosage also caused a greater water reduction than was expected. Since water requirement is dependent to a degree upon temperature, and admixture dosage must be increased with a rise in temperature, it was necessary

that the concrete ingredients reach equilibrium with room temperature ( $73 \pm 2^\circ\text{F}$ ) before being mixed. The batch temperature recorded in Table 5 was taken about 10 minutes after the first contact of water and cement. The higher temperatures are attributed to the chemical reactions between the cement, water and admixture. This rise is especially noticeable in the case of calcium chloride and admix AL which also contained calcium chloride.

Typical curves for retardation of mixes S-7 and L-7 are shown in Figure 5.

#### Moist Curing Period

Figures 6 and 7 illustrate the effect that varying periods of moist curing have on 60-day shrinkage. Specimens that were cured 28 days expanded as much as 50 micro inches per inch during their curing period and

required as long as three days to shrink back to their initial length at the time of removal from the molds. Specimens of the same aggregate type cured 7 and 14 days exhibited essentially the same amount of shrinkage. Specimens moist cured for 28 days shrink less after 60 days of drying than specimens with less curing. A 28-day curing period is believed to be unrealistic for field cured concrete. The California Division of Highways (10) found "Substantially constant shrinkage in concrete that was moist cured for 7, 14, and 28 days before drying was started" and reported that "given a period of moist curing adequate to develop reasonable strength, additional moist curing is relatively unimportant in the control of shrinkage." Data presented in this report will substantiate this statement.

### Admixture and Aggregate Effect

It is known that concrete shrinkage is influenced by the choice of aggregate and admixtures used. In many cases the cumulative effect of these two factors may be very large. As pointed out by Powers<sup>3</sup> the combined effect may be such that the resulting shrinkage is the product, rather than the sum, of the individual effects and therefore is much larger than if they were simply additive.

Data have been obtained in this report that supports Powers' theory and are illustrated in Table 6. The data from Table 13 (see Appendix) are used to compute the shrinkage for limestone aggregate first on the assumption that the effects are additive (Item 4) and second, that they are factors to be multiplied together (Item 7). It will be noted that the simple addition consistently underestimates the shrinkage, whereas multiplication yields a close estimate of the observed shrinkage except in the case of seven-day shrinkage.

These data show the large effect that aggregate type has on the shrinkage of concrete containing an admix-

### TYPICAL PENETROMETER CURVES

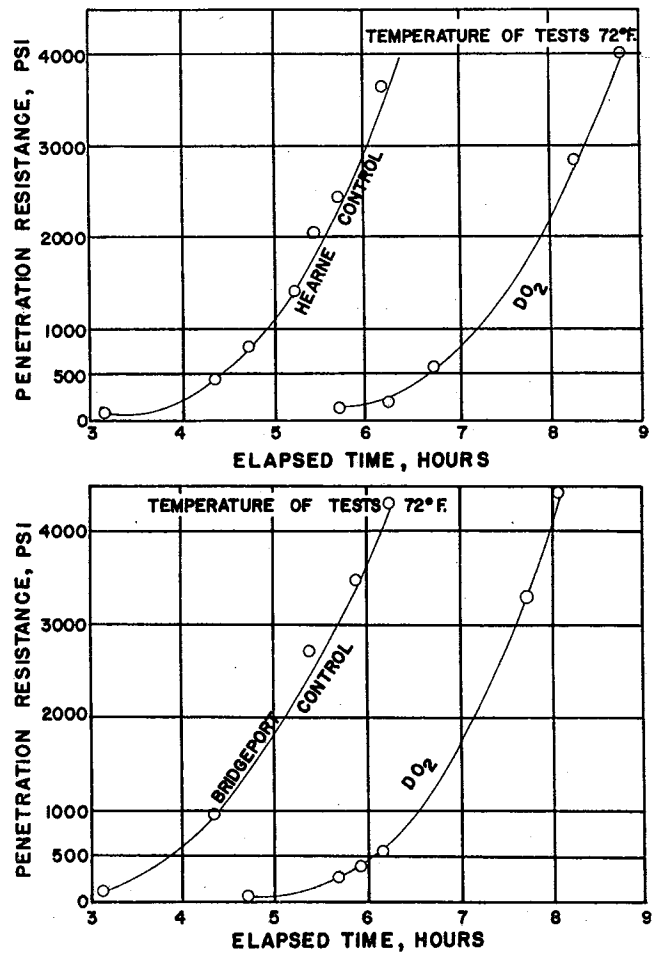


Figure 5.

Table 5  
ADMIXTURE DATA

Batch Designation	Admix Designation	% Water Reduction	Initial Set Retardation (hrs.)	Chemical Type	Dealers Recommended Dosage (65°F to 85°F)	Lab. Dosage	Batch** Temp.
S-1	No Admix						77
S-2	AL*	10.7%	-00:30***	Lignosulfonate	.25 lb./sk.	0.25 lb./sk.	77
S-3	DL <sub>1</sub>	16.0%	+ 3:12	Lignosulfonate	8.0 oz./sk.	10.5 oz./sk.	77
S-4	CC	2.2%	- 2:28	Calcium Chloride	2.0 lb./sk.	2.0 lb./sk.	86
S-5	DL <sub>2</sub>	8.5%	+ 3:12	Lignosulfonate	0.25 lb./sk.	0.25 lb./sk.	77
S-6	DO <sub>1</sub>	8.7%	+ 2:00	Organic Acid	3.0 oz./sk.	4.0 oz./sk.	81
S-7	DO <sub>2</sub>	10.8%	+ 2:27	Organic Acid	3.0 oz./sk.	4.5 oz./sk.	82
L-1	No Admix						77
L-2	AL	7.2%	0:00	Lignosulfonate	0.25 lb./sk.	0.25 lb./sk.	77
L-3	DL <sub>1</sub>	13.0%	+ 2:09	Lignosulfonate	8.0 oz./sk.	7.5 oz./sk.	81
L-4	CC	3.3%	- 2:18	Calcium Chloride	2.0 lb./sk.	2.0 lb./sk.	79
L-5	DL <sub>2</sub>	12.4%	+ 2:42	Lignosulfonate	0.25 lb./sk.	0.25 lb./sk.	77
L-6	DO <sub>1</sub>	10.4%	+ 2:42	Organic Acid	3.0 oz./sk.	4.0 oz./sk.	77
L-7	DO <sub>2</sub>	12.8%	+ 2:15	Organic Acid	3.0 oz./sk.	4.5 oz./sk.	81

\*The first letter of the admix designation is the ASTM designation given in ASTM C494-63T (A-water reducing, C-accelerating, D-water reducing and retarding). The second letter refers to chemical type (L-lignosulfonate, O-organic acid, C-calcium chloride). The subscript which may occur differentiates between different commercial products of the same ASTM designation and chemical type.

\*\*All ingredients of the batch were allowed to reach room temperature (73 ± 2°F) before being mixed: higher batch temperatures are due to hydration of the cement.

\*\*\*(+ ) Indicates retardation; (-) indicates acceleration.

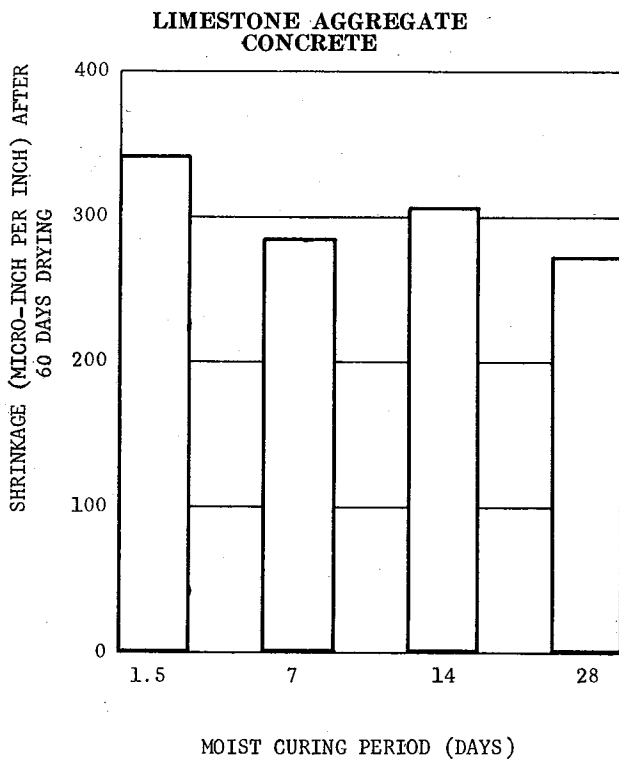


Figure 6.

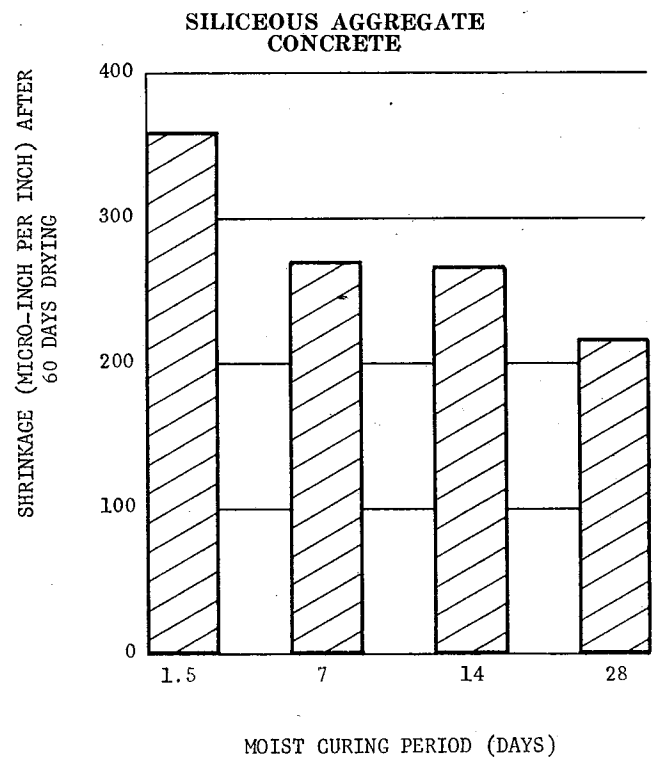


Figure 7.

ture. This is further illustrated in Figures 15 and 16 (see Appendix). The three admixes consistently causing the greatest amount of shrinkage were calcium chloride, admix AL, and admix DL<sub>2</sub>. The magnitude of the shrinkage after 40 days of drying was much larger with the limestone aggregate than with the siliceous aggregate. Admix DL<sub>1</sub> and admix DO<sub>2</sub> gave consistently lower values of shrinkage and produced essentially the same shrinkage regardless of the aggregate used.

#### **Drying Temperature and Humidity Effect**

Comparison of the curves in Figures 13 and 14 (see Appendix) with the respective curves in Figures 15 and 16 (see Appendix) shows that the shrinkage of those specimens subjected to drying conditions of 120°F and 17% relative humidity (oven dried) varied considerably

Table 6  
EFFECT OF INDIVIDUAL SHRINKAGE FACTORS ON  
CUMULATIVE SHRINKAGE

	Days of Drying		
	7	14	28
(1) Shrinkage siliceous aggregate	0.0095	0.0138	0.0210
(2) Increased shrinkage due to admixture, siliceous aggregate	0.0105	0.0167	0.0195
(3) Shrinkage, limestone aggregate	0.0104	0.0180	0.0247
(4) Computed shrinkage of limestone aggregate with admixture (2) + (3)	0.0209	0.0347	0.0442
(5) Relative shrinkage, limestone to siliceous aggregate	1.1	1.3	1.2
(6) Contribution of admixture (5) x (2)	0.0116	0.0217	0.0234
(7) Computed shrinkage limestone aggregate with admix (3) + (6)	0.0220	0.0397	0.0481
(8) Measured shrinkage	0.0303	0.0428	0.0487

with respect to the variability of the air dried specimens. It was found that length measurements between the three individual specimens from a common batch varied radically.

It was not expected that different drying conditions would produce identical shrinkage, but the curves presented in Figures 13 and 14 are not believed to be representative of typical concrete shrinkage. For this reason, the use of higher drying temperatures in order to develop a more rapid test for evaluation of admixtures was not indicated by this test series.

#### **Effect of Drying Period**

As illustrated in Figures 15 and 16, (see Appendix) the two control batches, S-1 and L-1, showed essentially the same 40 day shrinkage with more of the shrinkage taking place in the first 28 days of drying. Concrete made with siliceous aggregate and containing admixtures showed little gain in shrinkage after 30 days of drying, while limestone aggregate concrete continued to shrink after 40 to 45 days of drying.

Regardless of this continued shrinkage after 45 days of drying, it is noted that throughout the drying period, the curves in Figures 15 and 16 maintain their relative positions reasonably well. For this reason, it appears that a great reduction in drying time may be realized over that specified by the American Society for Testing Materials (ASTM Designation: C494-62T) by specifying the shrinkage of a concrete containing an admixture to be no more than a certain percentage of the control concrete at a specified age. The California Division of Highways has adopted a 14-day drying period for specimens 3" x 3" x 11", moist cured seven days. They found excellent correlation between their method of test (see Appendix) and beams exposed out-of-doors for 69 weeks.<sup>10</sup>

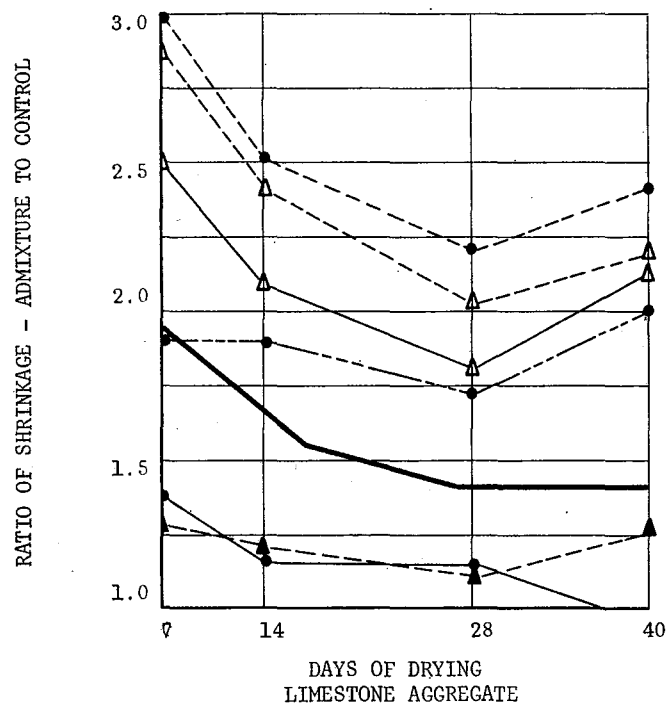
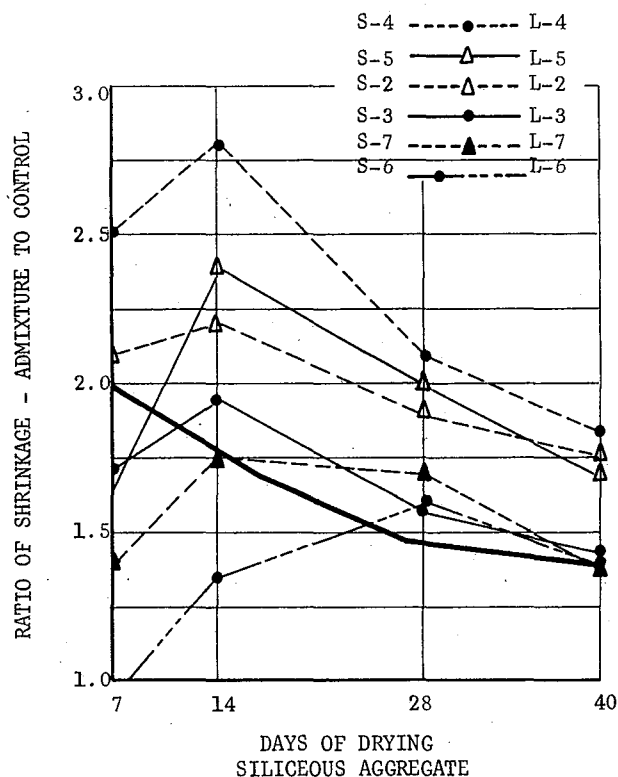


Figure 8. Change in relative shrinkage with drying time—air dried specimens.

This has reduced their time for determining the effect of admixtures on drying shrinkage from a period of one year as proposed by the American Society for Testing Materials (ASTM Designation: C494-62T) to 21 days.

Tremper and Spellman<sup>10</sup> have found that if 4" x 4" x 11" specimens are used rather than 3" x 3" x 11" specimens, a drying period of 28 days should be required rather than 14 days. This increased drying period has been found to yield the same results as the smaller specimens and shorter drying period.

#### Evaluation of the Proposed ASTM Test

Figure 8 shows the proposed test (ASTM: C494-62T) to be inconsistent in its method of determining the

effect of an admixture on the drying shrinkage of concrete. The heavy line drawn on the figures represents the cut-off line as determined by the specifications of the test. Those admixtures which fall above this line do not meet the requirements for shrinkage and must be rejected, while those below may be accepted on the basis of shrinkage.

It is shown that while the admix used in batches 6 and 7 is questionable when used with the siliceous aggregate (S-6 and S-7), it is acceptable at all drying ages to 40 days when used with the limestone aggregate. This illustrates the inadequacy of the proposed specification.

This inconsistency is further illustrated in Table 7. Both aggregate A and aggregate B complied in all respects with ASTM Designation: C33 and were selected

Table 7  
Data Taken by Tremper<sup>11</sup>  
DRYING SHRINKAGE AFTER NUMBER OF DAYS SHOWN  
Each value is the average of 12 specimens from 4 batches; values are in percent

	Control			Aggregate A Admixture 1			Admixture 2		
	7	14	28	7	14	28	7	14	28
Shrinkage, %	.0170	.0230	.0293	.0163	.0217	.0277	.0240	.0319	.0397
Numerical Difference				-.0007	-.0013	-.0016	+.0070	+.0089	+.0104
% of Control	100	100	100	96	93	95	141	137	135
	Control			Aggregate B Admixture 1			Admixture 2		
	7	14	28	7	14	28	7	14	28
Shrinkage, %	.0305	.0443	.0594	.0298	.0418	.0551	.0455	.0611	.0760
Numerical Difference				-.0007	-.0025	-.0043	+.0150	+.0150	+.0166
% of Control	100	100	100	98	94	93	149	138	128

Table 8  
PHYSICAL PROPERTIES

Batch Designation	Compressive Strength (psi), 28 Days	% of Control	Flexural Strength (psi), 28 Days	% of Control	28-Day Dynamic Modulus of Elasticity (psi x 10 <sup>6</sup> )
S-1	3470	100	810	100	6.43
S-2	3310	95	830	102	6.42
S-3	4200	121	835	103	6.31
S-4	3450	100	670	83	6.08
S-5	4250	123	965	120	6.30
S-6	3570	104	950	115	6.21
S-7	3710	106	820	101	7.09
L-1	4090	100	840	100	5.83
L-2	4080	100	851	101	5.96
L-3	4300	105	840	100	6.11
L-4	3160	77	720	86	5.87
L-5	4380	107	964	115	6.45
L-6	4520	110	951	113	6.60
L-7	3680	90	950	113	6.03

to give a wide range in drying shrinkage so as to bring the differences in effect of admixtures into clear focus. It is noted that admixture 1 complies with the specification when tested with either aggregate. On the other hand, admixture 2, when used with aggregate A produced increases in drying shrinkage which were less than 0.010 by a considerable amount.

These data therefore show that when rated in accordance with the method prescribed in the proposed ASTM specifications, the acceptance or rejection of admixture 2 is purely a matter of choice of the aggregates used.

If the effect of the admixture is rated on the basis of drying shrinkage relative to that of the control, it is

seen that the values found for each admixture with each aggregate are quite consistent. For example, if the specifications required that the drying shrinkage of the concrete containing the admixture shall not be more than 140% of that of the reference concrete at 14 or 28 days of drying, admixture 2 would have been found to meet the requirements regardless of the aggregate used.

#### *Strength Properties*

Table 8 shows the effect of the admixtures on the flexural and compressive strength of all batches. Calcium chloride at a dosage of 2% by weight of cement was used in batches S-4 and L-4 and seemed to have a detrimental effect on both flexural and compressive strength.

## *Chapter IV*

### *Conclusions*

The main points brought out by this investigation of the effect of admixtures on the drying shrinkage of concrete are as follows:

1. Substantially constant shrinkage after 60 days of air drying was found for 4" x 4" x 11" specimens moist cured 7 and 14 days while specimens cured 1.5 days exhibited greater shrinkage. Slightly less shrinkage took place in specimens that were cured for 28 days before drying.

2. A given admixture used in concrete made with a particular aggregate type may result in an increase of 50% or more (Figure 12) in shrinkage over another concrete containing the same admixture but made with a different aggregate type.

3. Increased dosages over those recommended by the dealer were required in some batches to obtain two hours of initial set retardation.

4. Current specifications<sup>2</sup> fail to take into account the effect of different aggregate types when the shrinkage of concrete containing an admixture is limited to an absolute increase over the shrinkage of a control concrete.

5. An attempt to arrive at a more rapid test procedure by using high drying temperature was not successful because of wide variation in shrinkage of individual specimens.

From the data contained in this report the following recommendations are presented in order that a more rapid and efficient method for determining the effect of admixtures on drying shrinkage of concrete may be obtained.

1. The cement, admixture, and aggregate type used in the laboratory test should be that proposed for use on the job. The cement content used for batching test specimens should also be that which is proposed for job use.

2. Specimens should be 3" x 3" x 11" prism specimens, moist cured 7 days and air dried (72°F, 50% relative humidity) 14 days or 4" x 4" x 11" prism specimens, moist cured 7 days and air dried 28 days.

3. A minimum number of 5 shrinkage specimens per batch should be used.

## *Chapter V*

### *Recommendations for Future Research*

This investigation has shown a need for determining the effect of the use of admixtures under actual job conditions. The magnitude of the shrinkage on test specimens has been found to be as high or higher than 2.5 times that for concrete containing no admixture and this amount of shrinkage could be critical in bridge decks and pavements if it causes cracking of the concrete.

Some admixture dealers feel that the use of admix-

tures improves the extensibility of concrete, thus allowing much larger shrinkage without cracking. The tentative findings of research now underway at the Structural Research Laboratory, Texas A&M University tend to refute this opinion. Before a limiting value of shrinkage may be stated, extensive research should be done to determine what effect chemical admixtures have on the extensibility of concrete.

# Part II: The Effect of Chemical Admixtures on the Drying Shrinkage of Concrete Batched at 95° F

## Chapter I Introduction

### General

It is known that higher batching and pouring temperatures require an increase in the amount of mixing water and adversely affect strength properties and setting time. Because of the increased mixing water, it would seem that drying shrinkage would also be increased. It was found in Part I that the use of certain chemical admixes greatly increased drying shrinkage when used at a dosage sufficient to give a two-hour retardation of initial set (as measured by Proctor Penetration) at temperatures of 75°F. If higher tempera-

tures cause a further increase in admixture dosage required for a two-hour retardation, this factor, coupled with increased mixing water could magnify shrinkage greatly.

### Objective

The objective of this part of the project was to determine the effect of an elevated temperature, (i.e., batching, pouring, and curing temperatures) on the drying shrinkage of concrete containing admixture dosages sufficient to give two-hour initial set retardation.

## Chapter II Testing Program

Seven batches of siliceous aggregate concrete were poured in this study, at a temperature of 95°F. The batch proportions are given in Table 9. The unit weight, specific gravity, absorption, and sieve analysis of these aggregates are the same as that given in Table 2, Part I, for siliceous aggregate and natural sand fines.

### Design and Fabrication of Batches

The concrete batches were designed in exactly the same way as given in Part I. The same procedures were followed in mixing and testing the plastic concrete except that the laboratory was maintained at a temperature of 95 ± 3°F throughout the tests. The concrete was

placed in molds according to the procedure given previously. The specimen schedule for the seven batches poured is given in Table 10.

The high temperature curing was achieved using 95°F water circulated around the specimens which were contained in barrels.

### Test Procedures

The shrinkage tests, time of setting by Proctor Penetrometer, modulus of rupture, and compressive strengths were all determined in the same manner given under "Testing Procedures," Part I.

Table 9  
CONCRETE MIX DATA  
Batches SF1 through SF7  
Quantities per C. Y. Concrete

Batch Designation	Aggregate, Type	Cement, Sacks	Aggregate (SSD)		Mixing Water, lbs.	Air Content, %	Slump, in.	Admix*
			Coarse, lbs.	Fine, lbs.				
SF-1	Siliceous	5.46	1772	1356	373	5.0	3.5	None
SF-2	Siliceous	5.54	1801	1396	330	3.5**	3.5	AL
SF-3	Siliceous	5.54	1799	1363	324	5.0	3.5	DL <sub>1</sub>
SF-4	Siliceous	5.43	1764	1358	364	2.0**	4.5	CC
SF-5	Siliceous	5.60	1831	1392	311	5.0	4.0	DL <sub>2</sub>
SF-6	Siliceous	5.35	1818	1407	343	5.5	4.0	DO <sub>1</sub>
SF-7	Siliceous	5.51	1800	1375	332	5.0	4.0	DO <sub>2</sub>

\*Sika-Aer air-entraining agent was used in all batches to obtain nominal 5% entrained air.

\*\*The presence of calcium chloride apparently did not allow the air entraining agent to entrain the desired 5% air although additional Sika-Aer was added.



Table 10  
SPECIMEN SCHEDULE  
Batches SF-1 through SF-7

Specimen Number	Specimen Type	Curing	Type of Test
1	4" x 4" x 11"	Immersed in water for 7 days at 95° ± 3°F and stored at 95 ± 3°F	Shrinkage measurements
2	4" x 4" x 11" Prism		
3	4" x 4" x 11"		
4	4" x 4" x 11"	Immersed in water for 7 days at 95 ± 3°F and stored in 17% relative humidity and 120°F	Shrinkage measurements
5	4" x 4" x 11" Prism		
6	4" x 4" x 11"		
7	3" x 4" x 16"	Immersed in water for 28 days at 95 ± 3°F	Weight and sonic modulus determined at 3, 7, 14, 28 days. Flexure and compressive strength at 28 days
8	3" x 4" x 16" Prism		
9	3" x 4" x 16"		
10	3" x 4" x 16"	Immersed in water for 7 days at 95 ± 3°F then subjected to ASTM freeze-thaw test C310-57	Weight and sonic modulus determined every 5 freeze-thaw cycles
11	3" x 4" x 16" Prism		
12	3" x 4" x 16"		
13	6" x 6" cylinder	None (Test on wet mortar)	Penetration Resistance ASTM: C408
14	6" x 6" cylinder		

## Chapter III

### Tests Results and Discussion

#### Water Reduction and Set Retardation

The water reduction and set retardation values obtained from concretes utilizing the six admixtures are given in Table 11.

Comparison of the percent water reduction in Table 11 with that in Table 5 shows that in the case of each admixture, the respective water reduction remained relatively constant. This is true regardless of the fact that due to the higher temperature, more mixing water was required for the SF batches (95°F) than the S batches (75°F). The increase in mixing water is evident in comparing the control batches (S-1 and SF-1) in Tables 1 and 9. Batch S-1, mixed at 75°F, required 361 lbs./cubic yd. while batch SF-1 mixed at 95°F required 373 lbs./cubic yd.

No overdosage (i.e., dosage above dealers recommended) of admixture was required to obtain a two-hour initial set retardation (Type D admixtures) at this

higher temperature. As given in Table 11, dealers recommended dosage for admix DL<sub>1</sub> is 8 oz./sk., although no definite limit has been placed on the dosage of this product at elevated temperature by the manufacturer. Its use in field work at a dosage of 12 oz./sk. has been reported.

By comparing the dosage of set retarding-water reducing admixtures required for two hours of initial retardation at a temperature of 75°F with that dosage required for the same retardation at 95°F, it appears that type D admixtures increase retardation at higher temperatures and no appreciable change in percent water reduction may be expected.

#### Shrinkage

Figures 17 and 18 (see Appendix) are the shrinkage curves for the seven batches of concrete poured at high temperatures. It appears that the calcium chloride content of admix AL definitely increases the shrinkage of

Table 11  
ADMIXTURE DATA

Batch Designation	Admix Designation	% Water Reduction	Initial Set Retardation (hrs.)	Chemical Type	Dealers Recommended Dosage above 85°F	Lab. Dosage	Batch** Temp.
SF-1	None						95
SF-2	AL*	10%	+00:28***	Lignosulfonate	.25 lb./sk.	0.25 lb./sk.	95
SF-3	DL <sub>1</sub>	15%	+ 2:40	Lignosulfonate	8.0 oz./sk.	10.0 oz./sk.	93
SF-4	CC	3%	- 1:45	Calcium Chloride	2.0 lb./sk.	2.0 lb./sk.	99
SF-5	DL <sub>2</sub>	12%	+ 2:00	Lignosulfonate	.25-.35 lb./sk.	0.25 lb./sk.	93
SF-6	DO <sub>1</sub>	9%	+ 1:55	Organic Acid	4.0 oz./sk.	4.0 oz./sk.	92
SF-7	DO <sub>1</sub>	12%	+ 2:10	Organic Acid	4.0 oz./sk.	4.0 oz./sk.	95

\*Same as page 21.

\*\*All ingredients of the batch were allowed to reach room temperature (95°F) before being mixed: higher batch temperatures are due to hydration of the cement.

\*\*\*(+ )Indicates retardation; (-) indicates acceleration.

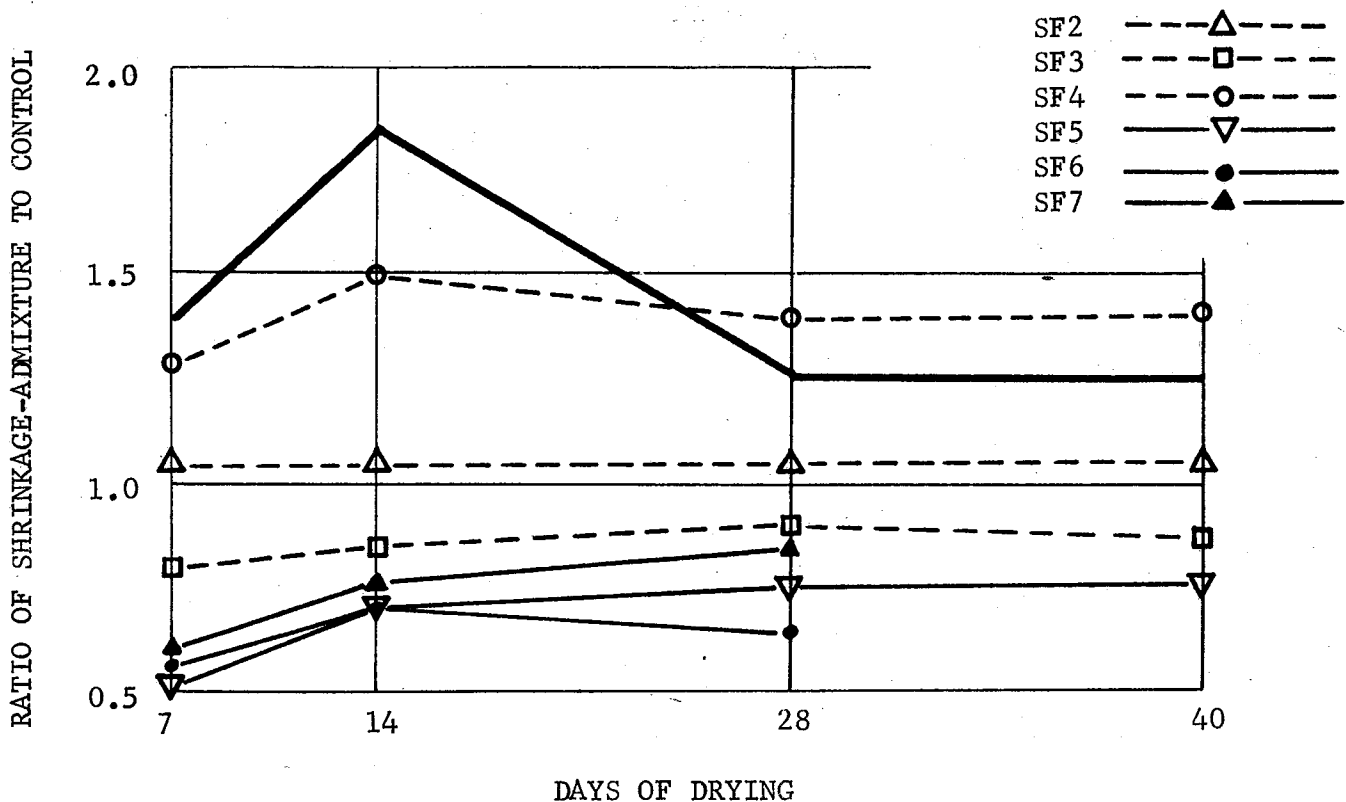


Figure 9. Change in relative shrinkage with drying time—specimens cured and batched at 95°F.

concrete as is also the case if calcium chloride is used at a dosage of 2% by weight of cement. The control batch, which contained no admixture, showed an increase in 40-day shrinkage of about 150 micro inches per inch when poured at 95°F over the same batch poured at 75°F. However, the concrete containing water-reducing set-retarding admixture and batched at 95°F shows essentially the same or a decrease in shrinkage over the batches poured at 75°F. This effect is illustrated by comparing the curves of Figure 15 with those of Figure 17 and 18 (see Appendix).

While the shrinkage of the concrete containing no admixture is increased with the increase in batching temperature, the shrinkage of the concretes containing water-reducing, set-retarding, admixtures is either unaffected by batching temperature or produces a decrease in shrinkage when used at higher temperatures.

Figures 17 and 18 show that with the exception of batches SF-2 and SF-4, all batches exhibited essentially the same or less shrinkage than the control after 40 days of drying.

Figure 9 shows that under the proposed shrinkage test, (ASTM: C494-62T) all admixtures reported, with the exception of calcium chloride, are acceptable when used in concrete batched and cured at 95°F (the heavy line represents the cut-off line as determined by the specification).

### Strength Properties

Table 12 shows the effect of the admixtures on the compressive strength, flexural strength, and dynamic modulus of elasticity of concrete batched at 95°F and immersed in water at 95°F.

Table 12  
PHYSICAL PROPERTIES

Batch Designation	Compressive Strength (psi), 28 Days	% of Control	Flexural Strength (psi), 28 Days	% of Control	28-Day Dynamic Modulus of Elasticity (psi x 10 <sup>6</sup> )
SF-1	3680	100	820	100	6.50
SF-2*	3870	105	690	85	6.50
SF-3	2860	80	540	65	5.30
SF-4*	3680	100	670	80	6.50
SF-5	3020	85	530	65	6.20
SF-6	3150	85	680	85	6.10
SF-7	3280	90	650	80	6.20

\*Properties determined at age 31 days.

The compressive strengths are decreased in all batches except those containing admix AL (SF-2) and calcium chloride (SF-4). In one case this decrease was 20%. It appears that the calcium chloride content of batches SF-2 and SF-4 (admix AL contains calcium chloride) improved the compressive strength of the concrete slightly.

By comparing the compressive strengths of batches S-1 through S-7 (Table 8) with batches SF-1 through SF-7 (Table 12) it is apparent that the addition of these chemical admixtures to concrete batched at high temperature does little to improve the strength of the concrete and in some cases is harmful.

## *Chapter IV*

### *Conclusions*

From the data presented in Part I and Part II, the following conclusions and comparisons may be made concerning concrete batched, poured, cured, and dried at 95°F.

1. At 75°F the dosage necessary for two hour initial set retardation was usually more than recommended by the dealer for that particular temperature. At 95°F it was found that the dealers' recommended dosage was sufficient to give the required retardation.

2. Though the water requirement for the concrete batched at 95°F was higher than for the concrete batched at 75°F, the water reduction in percent remained reasonably constant for essentially the same admixture dosage.

3. Concrete which contained no admixture and was batched at 95°F showed an increase in shrinkage over that batched at 75°F of about 150 microinches per inch after 40 days of drying.

4. Concrete batched at 95°F containing type D admixtures showed essentially the same or a decrease in drying shrinkage when compared with concrete containing the same admixture but batched at 75°F.

5. The shrinkage of concrete batched at 95°F and containing type D admixtures was essentially the same or less than the control concrete batched at the same temperature.

# Part III: Control of Uniformity of Chemical Admixes

## Chapter I Introduction

Uniformity of the chemical composition of admixtures may be difficult to regulate and little is known about the constituents in the admixtures which cause an

increase in shrinkage. It is therefore extremely important that the chemical formulation of an admix remain reasonably constant after it has been tested and approved.

## Chapter II Objectives

Because of the importance of uniformity in chemical composition of admixtures the objective of this phase of the research was to investigate the use of activation

analysis methods and infrared spectrographic methods in order to arrive at a simple solution to the complex problem of checking chemical formulation.

## Chapter III Results

Samples of the six admixtures used in this research were submitted to Pedro Jimenez of the Activation Analysis Laboratory, Texas A&M University and to Jack D. Price of the Agricultural Analytical Services, Texas A&M University.

Jimenez reported that no satisfactory solution to the problem could be obtained through activation analysis due to the large amount of activity induced in sodium. A longer activation period would make it possible to analyze K, Zn, and Na after a 40-hour wait time. How-

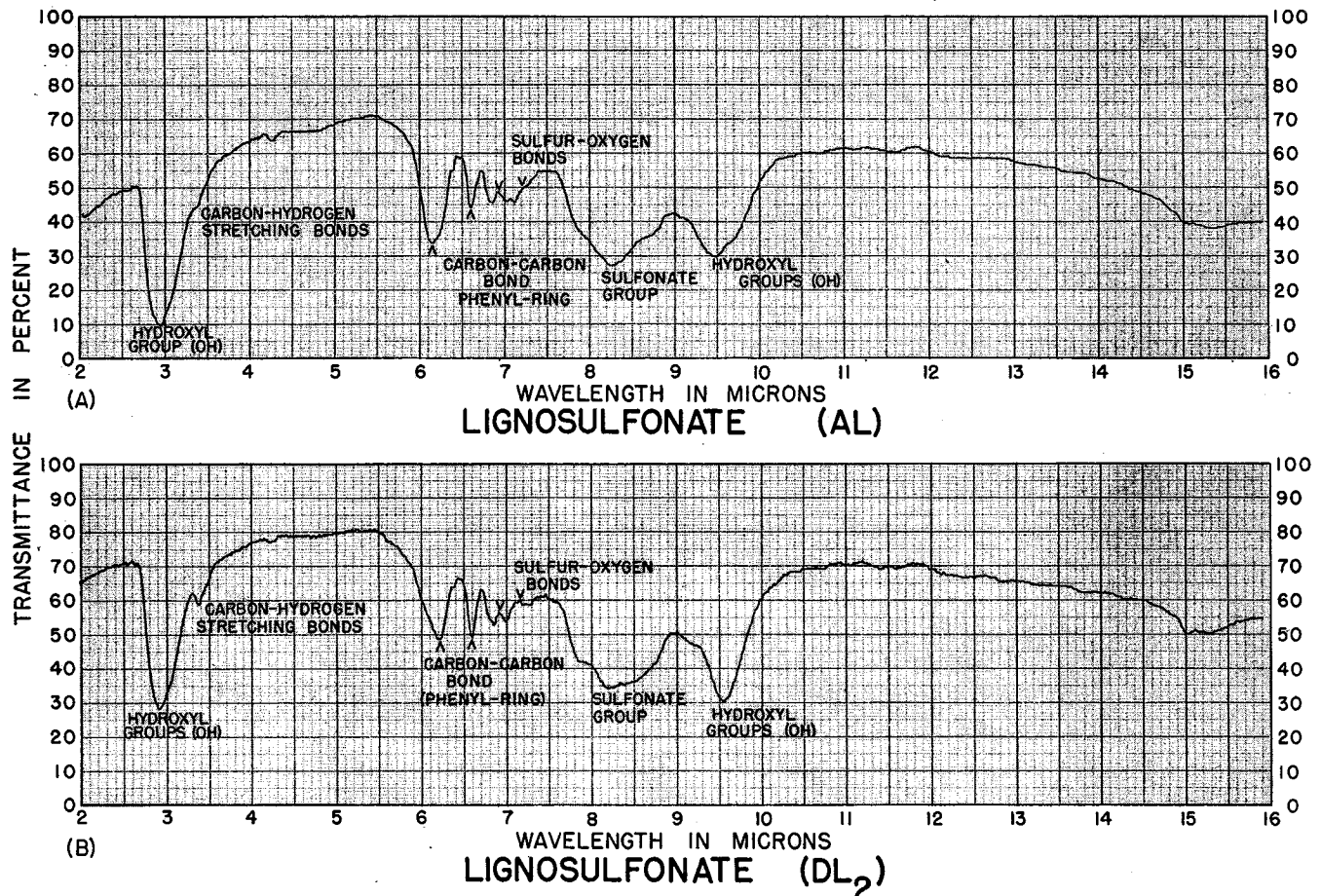


Figure 10. Infrared spectrographic scans of concrete admixtures.

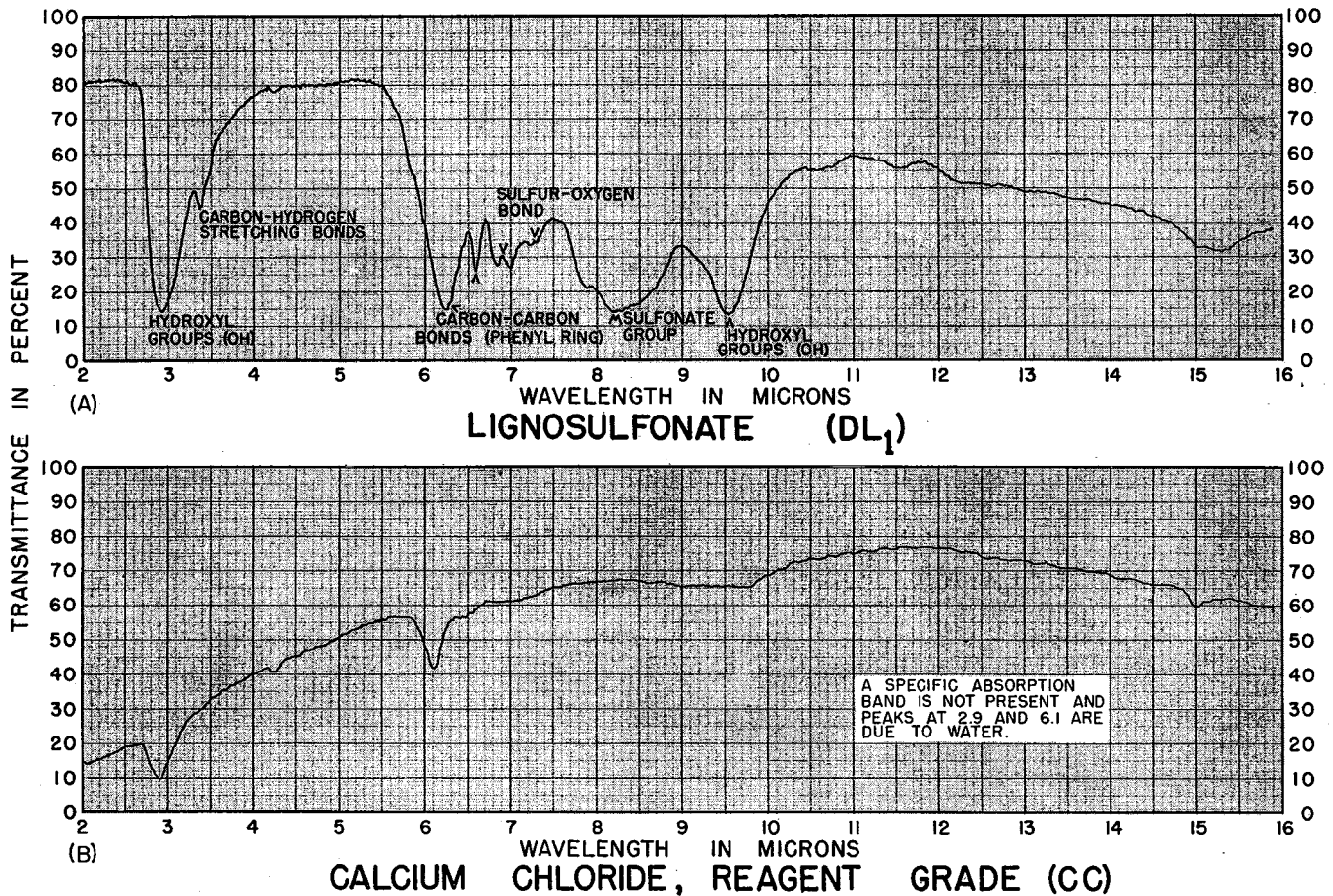


Figure 11. Infrared spectrographic scans of concrete admixtures.

ever, at the present time this would be impractical because of the expense and the limitations of the process (see letter dated August 18, 1964, Appendix).

Price, however, reported that infrared spectrographic analysis was a satisfactory method of checking chemical formulation of the admixes. The pressed disk technique was used to prepare samples of the retarder for spectrographic analysis. As stated by Price, (see letter dated August 13, 1964, Appendix) the procedure followed in preparing the samples is the same as that given by Halstead and Chaiken<sup>8</sup> with the exception of the equipment employed. The scans obtained by Price are shown in Figures 10, 11, and 12.

The distinctive peaks at different wave lengths on the scans are produced by the chemical groups shown on the scans and it is noticed that the peaks are characteristic of each retarder group, i.e., lignosulfonates have characteristic peaks at 2.9 microns, 6.1 microns, etc. This is seen by comparing Figure 10, scans (A) and (B) and Figure 11, scan (A). Organic acid retarders have other characteristic peaks, as shown in Figure 12. It is seen that scan (A) and scan (B) of Figure 12 are in no way similar as was the case with the lignosulfonates. This is because scan (B) is a complexed organic acid whereas scan (A) is not. No attempt was made to identify the peaks on scan (A), the

organic acid, because this scan is so unique. Thus, from these peaks, their intensity and location, a "fingerprint" of a particular retarder may be made and compared to another "fingerprint" of the same product made later to determine if the chemical makeup has changed noticeably.

As a result of the large shrinkage of concrete containing calcium chloride as an admix, it was desirable to investigate the possibility of identifying calcium chloride by spectrographic methods. As reported by Price, (see letter dated May 27, 1964, Appendix) however, "it appears doubtful that we can demonstrate the presence of calcium chloride as an additive with our instrument." This is illustrated by comparing scans (A) and (B) of Figure 10. It was known that admix AL contained calcium chloride whereas admix DL<sub>2</sub> did not; however, there is no appreciable difference in the scans of the two admixtures and therefore no way of distinguishing calcium chloride.

Since admix AL was known to contain calcium chloride, the admixture was submitted to Price for conventional chemical analysis in order to determine the percent by weight of calcium chloride. He reported (see letter dated August 13, 1964, Appendix) that on the basis of the calcium and chloride content the calcium chloride content of admix AL is approximately 30%.

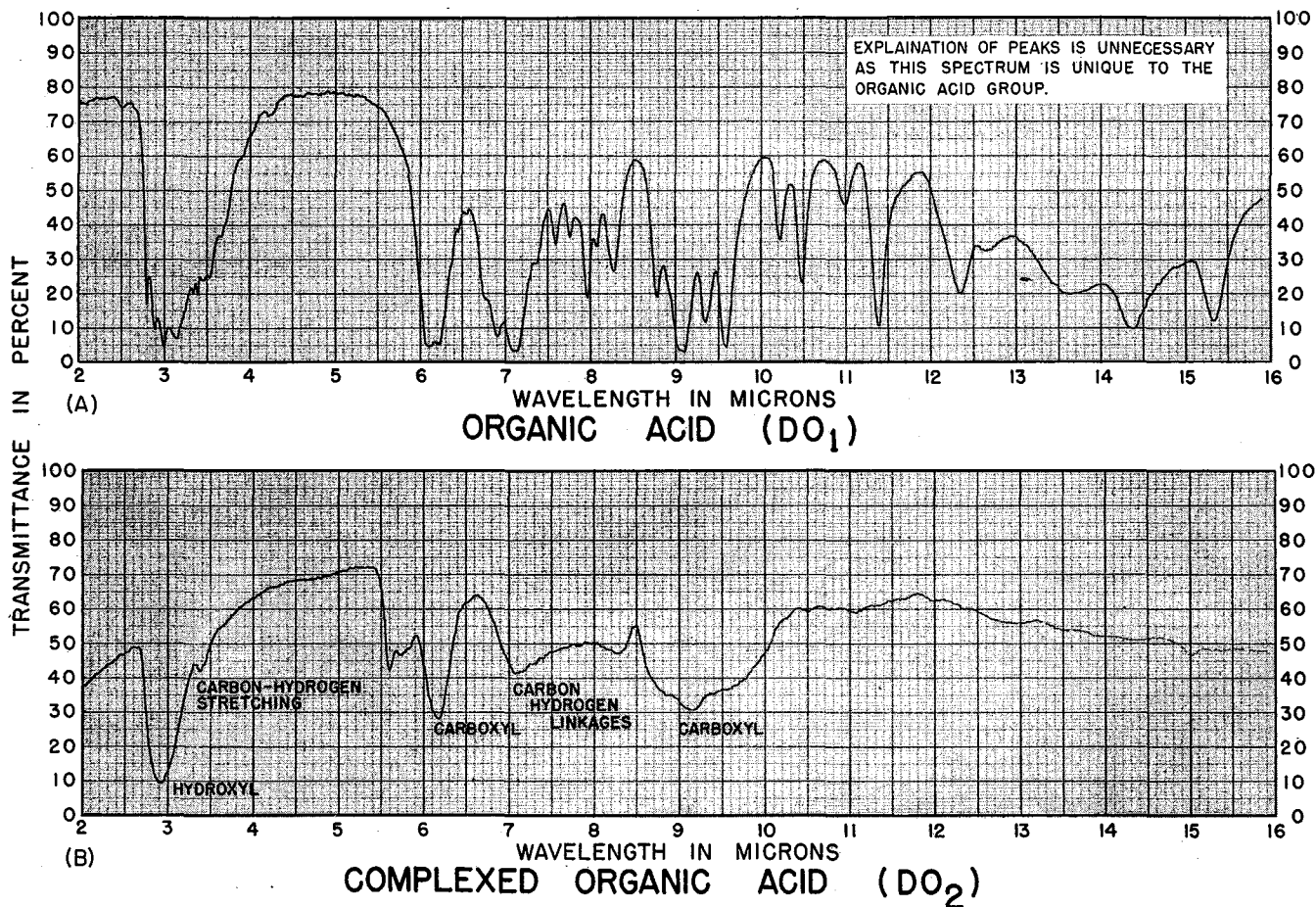


Figure 12. Infrared spectrographic scans of concrete admixtures.

## Chapter IV

### Summary

Each retarder tested gave a characteristic infrared spectrum which could be used both to identify the material and establish the concentration of major active constituents.

Though no attempt was made in this research to investigate the uniformity of admixes, infrared spectro-

graphic analysis has satisfactorily shown its ability to do so. By obtaining new samples of the admixes used in this project and having a spectrographic analysis run on each, their "fingerprints" may be compared with those obtained earlier. Locations of peaks and intensity of peaks will determine if the new admix is consistent with that previously tested.

## Part IV

### Selected References

1. "Tentative Method of Test for Volume Change of Cement Mortar and Concrete," A.S.T.M. Standards, Part 4, 1961.
2. "Tentative Specifications for Chemical Admixtures for Concrete," Supplement to Book of A.S.T.M. Standards, Part 4.
3. Powers, T. C., "Causes and Control of Volume Change," Journal of Research and Development Laboratories, Volume 1, Number 1, January, 1959.
4. Menzel, Carl A., "Strength and Volume Change of Steam Cured Portland Cement Mortar and Concrete," Proceedings, American Concrete Institute, Volume 31, 1935.
5. Carlson, Roy W., "Drying Shrinkage of Concrete as Affected by Many Factors," Proceedings of American Society for Testing Materials, Volume 38, 1938.
6. Wallace, George B., Ore, Elwood L., "Structural and Lean Mass Concrete as Affected by Water-Reducing, Set-Retarding Agents," A.S.T.M. Technical Publication Number 266, 1959.
7. Morgan, W. T., Jackson, F. H., Bruce, Foster E., Powers, T. C., "Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete," Journal of American Concrete Institute, Vol. 22, Number 1, September, 1950.
8. Halstead, Woodrew J., Chaiken, Bernard, "Water-Reducing, Retarders for Concrete-Chemical and Spectral Analysis," Public Roads Journal, Volume 31, Number 6, February, 1961.
9. Schutz, Raymond J., "Admixtures for Concrete," a paper presented to the Thirty-seventh Annual Highway Short Course, (unpublished), Texas A&M University, December, 1963.
10. Tremper, Bailey, Spellman, D. S., "Shrinkage of Concrete, Comparison of Laboratory and Field Performance," a report presented to the Forty-second Annual Meeting of the Highway Research Board, January, 1963.
11. Tremper, Bailey, "Method of Test for Determining the Effect of Water-Reducing and Set-Retarding Admixtures on the Drying Shrinkage of Concrete," a letter to T. M. Kelly, (unpublished), August, 1960.
12. Pickett, Gerald, "Effect of Aggregate of Shrinkage and a Hypothesis Concerning Shrinkage," Proceedings, American Concrete Institute, Vol. 52, 1956.
13. Beaton, John L., a letter to D. L. Ivey, (unpublished), March 20, 1964.
14. "Method of Test for Determining the Effect of Water-Reducing and Set-Retarding Admixtures on the Drying Shrinkage of Concrete," Test Method Number California 530-C, July, 1963.

## Part V

### Appendix

Shrinkage curves for all batches poured in this project are included in this appendix along with information received from the California Division of Highways concerning their research in the field of concrete shrinkage.<sup>13,14</sup>

Letters received from Mr. Pedro Jimenez and Dr. J. D. Price concerning Activation analysis and Spectrographic Analysis are also included.



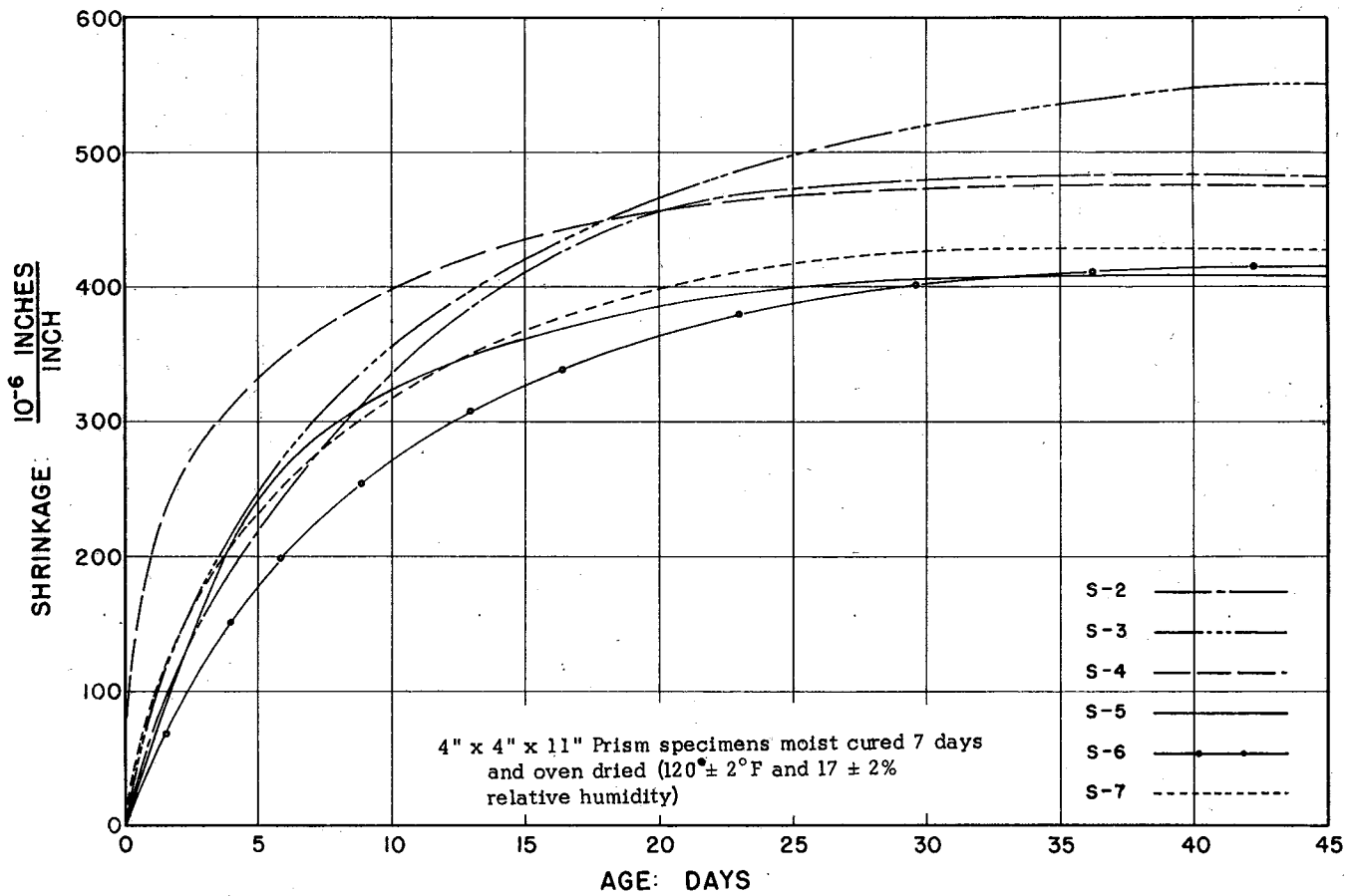


Figure 13. Shrinkage of siliceous aggregate concrete.

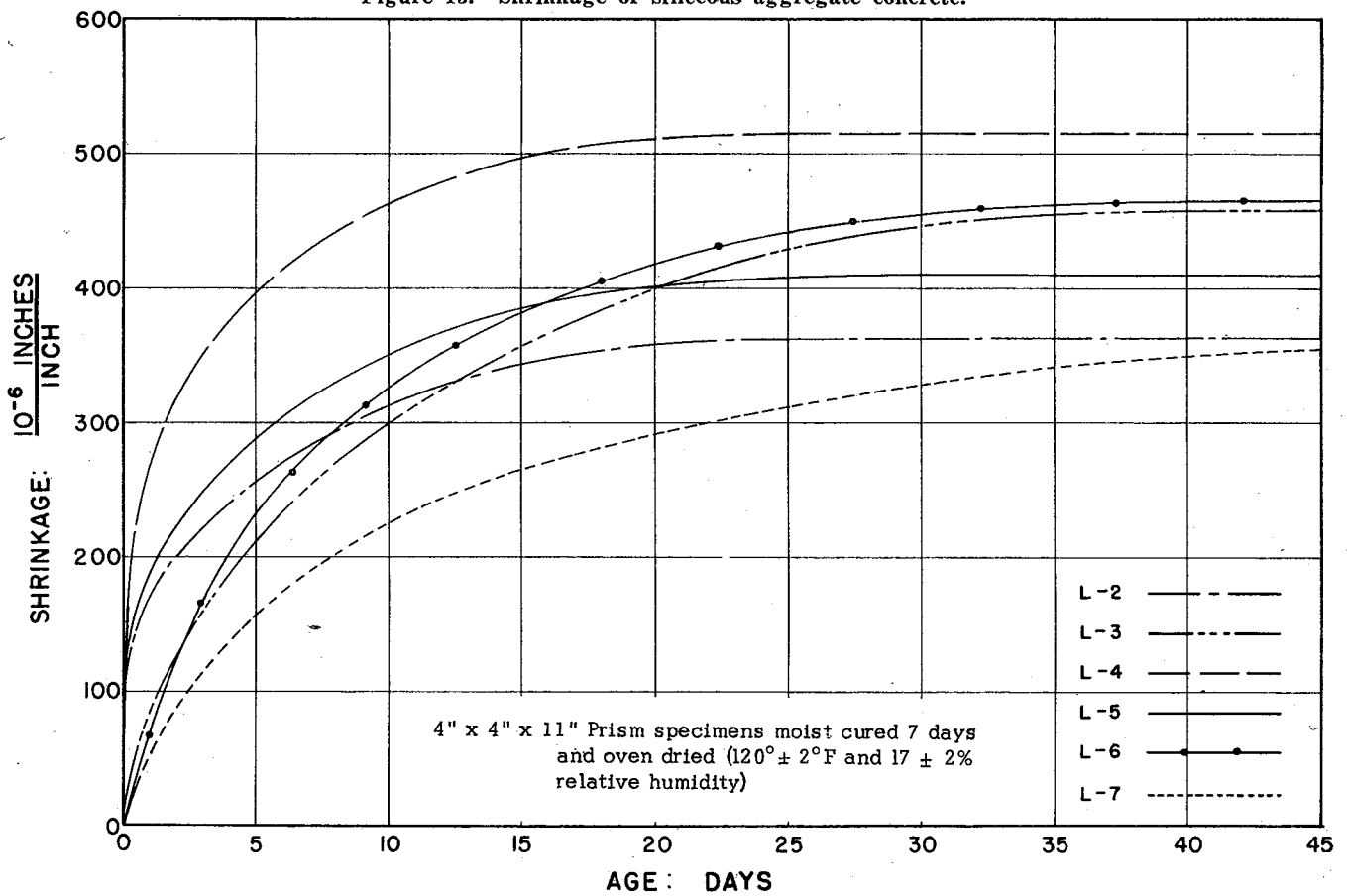


Figure 14. Shrinkage of limestone aggregate concrete.

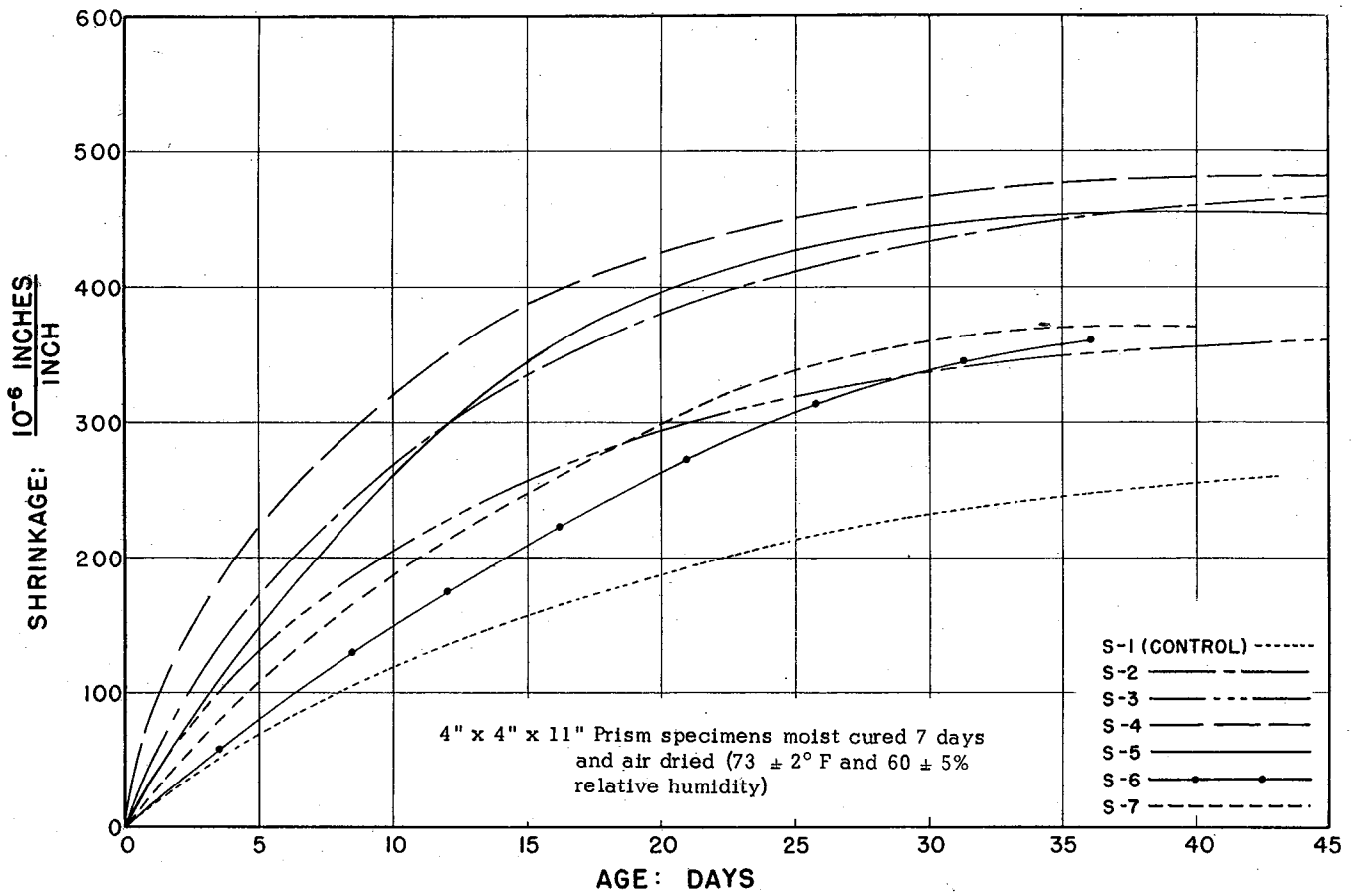


Figure 15. Shrinkage of siliceous aggregate concrete.

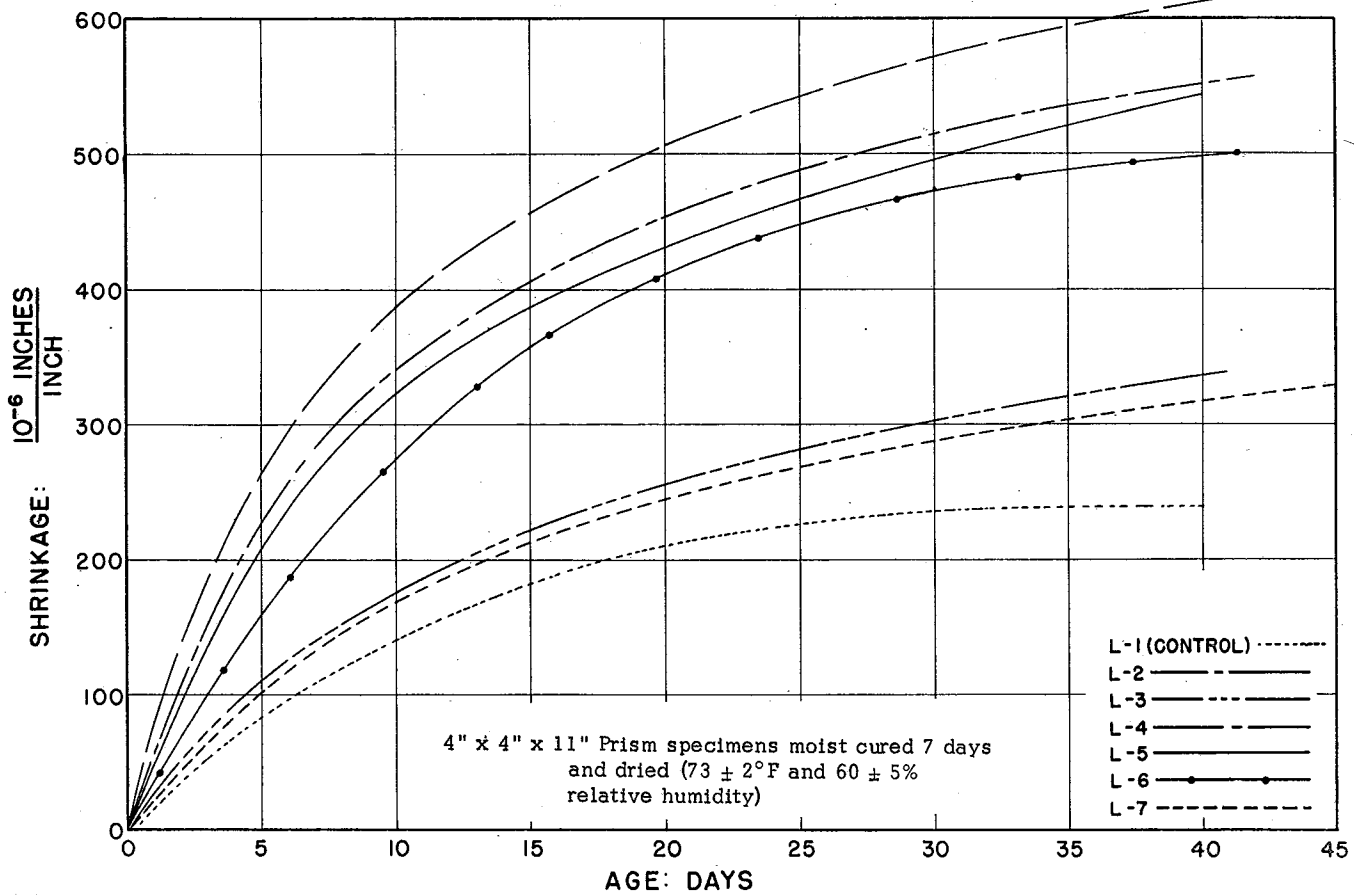


Figure 16. Shrinkage of limestone aggregate concrete.

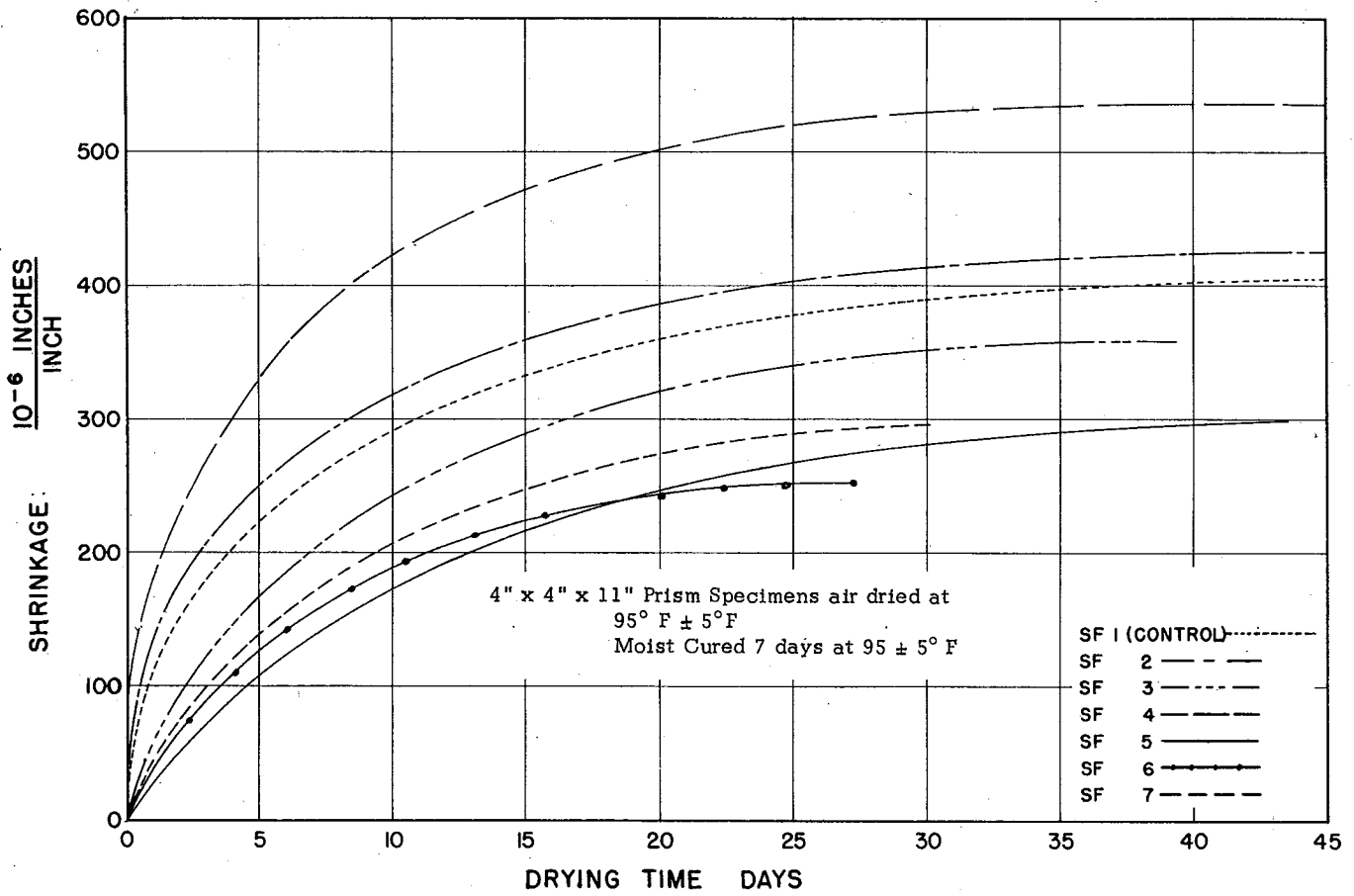


Figure 17.

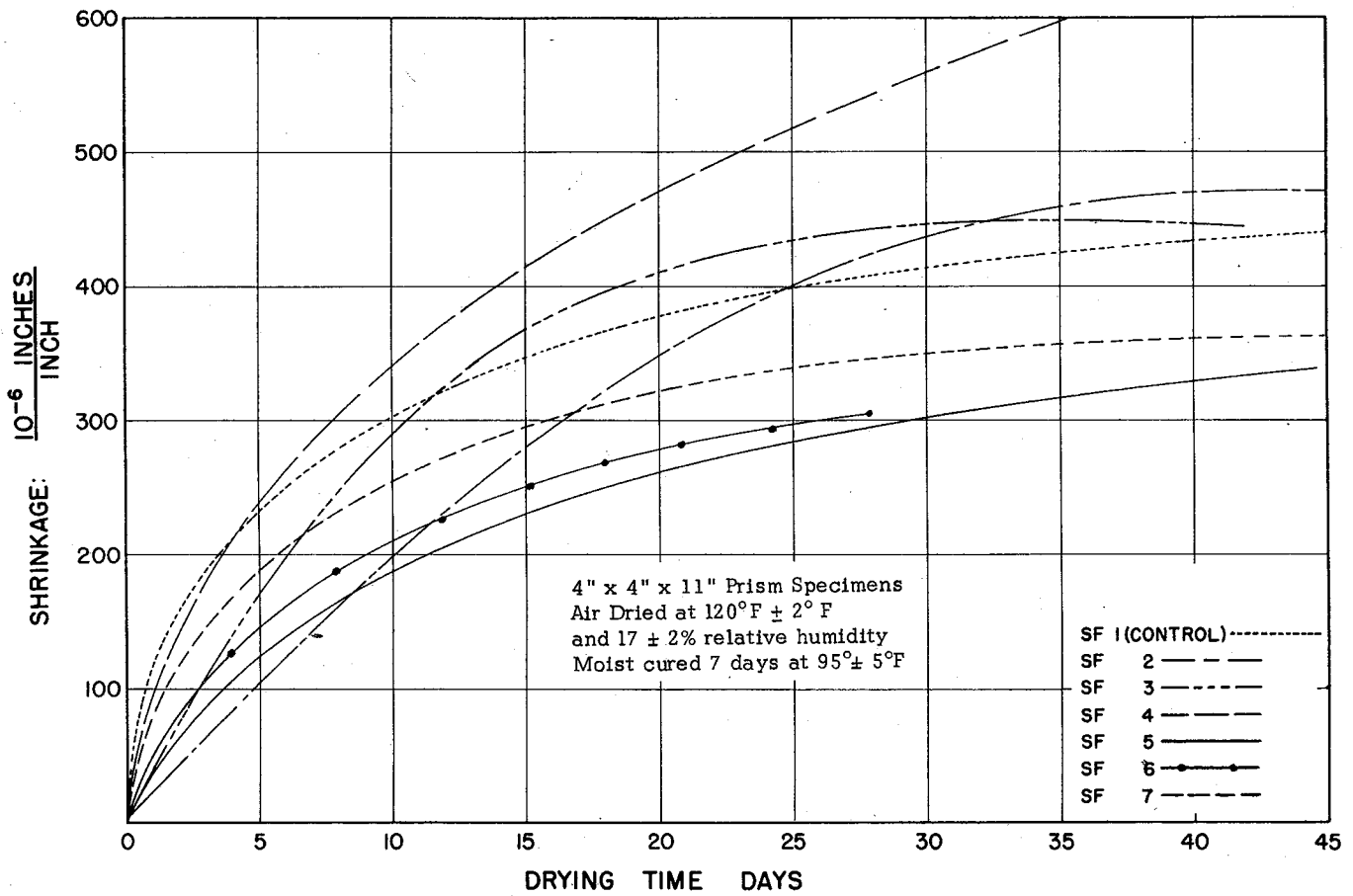


Figure 18.

Table 13  
 TABULATION OF SHRINKAGE DATA

Batch	Shrinkage in Microinches per Inch, Air Dried Specimens			Shrinkage in Microinches per Inch, Oven Dried Specimens			
	AGE	7	14	28	7	14	28
S-1 (Control)		95	138	210			
S-2		200	305	405	270	400	476
S-3		162	270	330	300	410	510
S-4		234	380	448	360	430	470
S-5		159	330	420	300	355	409
S-6		85	186	335	220	320	400
S-7		130	245	358	270	360	423
L-1 (Control)		104	180	247			
L-2		303	428	487	283	340	363
L-3		142	207	274	250	340	430
L-4		315	444	564	430	490	513
L-5		258	382	453	320	375	410
L-6		195	338	420	278	373	450
L-7		130	217	268	140	259	322

State of California  
Department of Public Works  
DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT  
5900 Folsom Blvd.  
Sacramento 19, California  
95819  
March 20, 1964

Please Refer To  
File No.

Mr. Don L. Ivey  
Assistant Research Engineer  
Structural Research Department  
Texas Transportation Institute  
Texas A&M University  
College Station, Texas

Dear Mr. Ivey:

We have received your letter addressed to Mr. Bailey Tremper, dated March 10, 1964, concerning shrinkage of concrete. Mr. Tremper has retired from the Division of Highways and is now engaged in private practice. As a matter of information, Mr. Hveem who was formerly the Materials and Research Engineer, has also retired and has been succeeded by Mr. John L. Beaton.

It is interesting to find others investigating the effects of admixtures on the drying shrinkage of concrete. There has been much written on the subject and to aid you in finding reference material, we are enclosing a copy of our report on the subject which includes a list of references. It is by no means complete. Many recent articles have appeared in technical publications dealing with concrete, such as the Journal of the American Concrete Institute.

We believe any test for drying shrinkage should include control over evaporation rates of the drying room, particularly if shrinkage of concrete or mortar is to be determined on an absolute basis. To eliminate variables of aggregates and cements, we believe shrinkage tests to evaluate effects of admixtures should be on a comparative basis, that is, compare shrinkage of plain concrete with similar concrete containing the admixture. If done on an absolute basis, the results may not mean much as they can be greatly influenced by the choice of aggregate. To get a low value, one could select a quartz aggregate. There are other aggregates which give shrinkage values over twice that of the quartz. We have recently tested some aggregate from Mississippi and Spain which exhibit drying shrinkage in concrete less any of that found in California.

Our current specifications limit the use of admixtures in concrete based on their effect on drying shrinkage. Of course there are other requirements also, but we use Test Method No. Calif. 530 to evaluate these effects. A copy of the test method is also enclosed. In general, the dosage of an admixture that can be used is limited to that which does not increase drying shrinkage more than 20% for precast, prestressed concrete, nor more than 10% for cast-in-place reinforced concrete.

We hope this information will be of help to you in your work.

Very truly yours,  
JOHN L. BEATON  
Materials & Research Engineer

By/S/ D. L. Spellman

D. L. Spellman  
Assistant Materials and  
Research Engineer — Technical

DLS:fp

Enclosures—"Shrinkage of Concrete, Comparison of Laboratory and Field Performance" by B. Tremper and D. L. Spellman

Test method No. Calif. 530-C "Method of Test for Determining the Effect of Water-Reducing and Set-Retarding Admixtures on the Drying Shrinkage of Concrete"

## METHOD OF TEST FOR DETERMINING THE EFFECT OF WATER-REDUCING AND SET-RETARDING ADMIXTURES ON THE DRYING SHRINKAGE OF CONCRETE

### SCOPE

This method is a modification of A.S.T.M. Designation: C 157 and is intended to provide a standardized procedure for determining the effect of water-reducing and set-retarding admixtures on the drying shrinkage of concrete.

### PROCEDURE

#### A. Apparatus

1. The measuring instrument, tamper, tamping rod, and molds shall conform to A.S.T.M. Designation: C 157, except that the width and depth of the molds shall be 3 inches.

2. Drying Room. A room equipped with suitable racks for storing test specimens during drying. The racks shall be designed to permit free circulation of air around specimens except for necessary supports. The room shall be large enough to permit the use of a manually operated sling psychrometer. The temperature of the room shall be maintained at  $73.4 \pm 2$  F and the relative humidity at  $50 \pm 4$  percent. Conditioned air shall be circulated continuously through the room at such a rate that evaporation of water from an atmometer<sup>1</sup> is maintained at  $3 \pm 0.5$  ml. per hour. Circulation of air shall be controlled by deflectors, if necessary, to result in equal rates of evaporation from the atmometer placed adjacent to specimens at different locations on the storage racks. The temperature and relative humidity of the room shall be measured with a sling psychrometer at least twice daily. The room shall be equipped with means of measuring and recording wet and dry bulb temperatures continuously. The recorded data shall be checked against the sling psychrometer and the results of the latter shall govern.

#### B. Concrete Materials

1. Cement. The cement used in the test shall conform to the requirements of the Standard Specifications of the California Division of Highways and shall be composed of a mixture of equal parts produced by the following mills.

Permanente Cement Company  
Permanente, California  
Calaveras Cement Company  
San Andreas, California  
Southwestern Portland Cement Company  
Victorville, California  
Riverside Cement Company  
Oro Grande, California  
California Portland Cement Company  
Colton, California

2. Aggregates. The aggregates used in the test shall consist substantially of uncrushed sand and gravel obtained from washed stocks produced by Fair Oaks

<sup>1</sup>A working drawing of an atmometer used by the Materials and Research Department is available on request.

Gravel Company operations in the American River in the vicinity of Fair Oaks, California. The aggregates shall be separated on sieves and recombined to the following grading:

Percent Passing 1 inch sieve.....	100
Percent Passing $\frac{3}{4}$ inch sieve.....	90
Percent Passing $\frac{3}{8}$ inch sieve.....	63
Percent Passing No. 4 sieve.....	48
Percent Passing No. 8 sieve.....	39
Percent Passing No. 16 sieve.....	27
Percent Passing No. 30 sieve.....	18
Percent Passing No. 50 sieve.....	7

#### C. Mixing Concrete

1. Proportion the concrete to contain  $7.0 \pm 0.1$  sacks of cement per cubic yard.

2. Mix the concrete in batches of 0.3 cu. ft. or larger.

3. Bring the materials used in the concrete to room temperature. After mixing, the concrete shall have a temperature of  $73 \pm 3$  F.

4. Prepare the molds by coating their interior surfaces with a release agent. (A commercial agent known as "Powerfilm" is used by the Materials and Research Laboratory for this purpose, but a satisfactory substitute can be made by mixing a 12% solution, by weight, of white paraffin and a low aniline petroleum solvent of the intermediate grade, such as "Socul No. 2," available from the Standard Oil Company). After coating the molds with the release agent, place the measuring studs in the end plates, using care to avoid getting any release agent on the studs.

5. Mix the concrete in an open tub type mixer for 2 minutes followed by a rest period of 2 minutes, after which continue mixing for 2 minutes.

6. All aggregates and about two-thirds of the mixing water should be in the mixer and mixed briefly before the addition of the cement. The admixture, if used, should be added as a diluted solution made with part of the remaining mixing water. Finally, add the quantity of water required to produce a slump of  $3\frac{1}{2} \pm \frac{1}{2}$  inches.

7. After mixing, determine the slump, unit weight, and air content. Take up excess water from surface of concrete in air meter with a sponge.

8. Return the concrete used in the above tests to the mixer and remix briefly.

9. Molding of Test Specimens:

a. Mold at least 3 test specimens from each batch in accordance with the procedure described in A.S.T.M. Designation: C 157, except that in lieu of rodding and spading the mix into the molds, an external vibrator such as a vibratory packer may be used to compact the mix in two layers.

Test Method No. Calif. 530-C  
July, 1963

b. The first layer of concrete should just cover the top of the gage studs. While vibrating briefly, work the concrete into the corners and around the gage studs with the fingers; then fill the mold to slightly overflowing and vibrate again.

c. Avoid over vibration.

d. When compaction is complete, strike off the top surface and finish with a steel straightedge.

e. Immediately after molding, release gage plug holders.

#### D. Curing and Drying Specimens

1. After molding, cover specimens with wet cloth for 3 to 4 hours before placing them in a fog room maintained at  $73.4 \pm 3$  F. The fog room shall be so operated that free water is maintained on the surface of the specimens at all times.

2. When specimens are  $24 \pm 4$  hours old, remove them from the molds and return to the fog room.

3. At the age of 7 days after molding, measure the specimens for length.

4. Store on racks in the drying room, maintained at  $73.3 \pm 2$  F. and  $50 \pm 4$  percent relative humidity, for 14 days. Store them with at least 1 inch clearance on all sides except for the necessary supports.

5. At the age of 21 days after molding (14 days of drying), measure the specimens again for length.

#### E. Number of Batches of Concrete

1. Mix at least 3 rounds of concrete, each on a different day.

2. Each round shall consist of one batch of concrete containing each dosage of admixture plus one batch of control concrete containing no admixture.

3. Use at least two dosages of admixture, one of which shall be the maximum recommended by the manufacturer, and one at  $\frac{1}{2}$  the maximum dosage recommended.

#### F. Calculations

1. Compute the drying shrinkage for each condition (no admixture, and at least 2 dosages of admixture) to the nearest 0.001 percentage point, based on the gage length of 10 inches. The computed shrinkage must be the average of at least 7 specimens, the individual values of which do not depart from the average by more than 0.004 percentage point.

2. Compute the relative drying shrinkage of the concrete for each of the dosages, making the plain concrete shrinkage equal to 100%. Plot the relative shrinkages on a graph to be used in determining the maximum permissible dosage of the admixture, depending on the class of work proposed.

#### G. Reporting of Results

1. Report drying shrinkage for each specimen as the length at 7 days minus the length at 21 days.

2. Include in the report, the average cement factor, the average slump, and the average air content of the control mixture and similar data for concrete containing the admixture. Also, include the name and designation of the admixture under test and the dosages used per sack of cement.

#### REFERENCES

A.S.T.M. Designation: C 157  
Standard Specifications of the California  
Division of Highways

End of Text on Calif. 530-C

TEXAS A&M UNIVERSITY  
COLLEGE OF AGRICULTURE  
College Station, Texas

TEXAS AGRICULTURAL  
EXPERIMENT STATION  
Agricultural Analytical Services

May 27, 1964

Dr. T. J. Hirsch  
Associate Professor  
Texas Transportation Institute  
Campus

Dear Dr. Hirsch,

I am enclosing infrared scans of the six materials which you submitted for analysis. The spectra were compared with those appearing in the Journal of Public Roads and identification was made on this basis.

There appears to be little doubt the basic material can be readily identified by this procedure. A considerable amount of time was spent in an attempt to resolve the calcium chloride present in the sample identified as Pozzolith 4L. A scan of reagent grade calcium chloride is included. As you will note, a specific absorption band is not present in the range of our instrument (2 to 16 microns). The absorption bands present are due to water even though the material had been dried in a vacuum oven for 24 hours. As you know calcium chloride is extremely hygroscopic and is commonly used as a drying agent.

Ionic solids of the type composed of monatomic ions produce only so-called "lattice" vibrations resulting in very broad spectral bands. It appears doubtful therefore that we can demonstrate the presence of calcium chloride as an additive with our particular instrument, however conventional chemical means can readily be employed for this purpose.

Invoice number 4045-4050 is enclosed in the amount of \$60.00 covering the cost of analysis. Should you have any questions concerning the procedure please let me know.

Sincerely yours,  
Jack D. Price  
Assistant Professor

JDP/ct



TEXAS A&M UNIVERSITY  
COLLEGE OF AGRICULTURE  
College Station, Texas

TEXAS AGRICULTURAL  
EXPERIMENT STATION  
Agricultural Analytical Services

August 13, 1964

Mr. Pat Torrans  
Texas Transportation Institute  
Campus

Dear Mr. Torrans,

The sample of material identified as Admixture AL\* has been analyzed and found to contain 10.5% calcium.

Empirically related, the calcium content is the equivalent of 29.1 percent anhydrous calcium chloride. The material was also analyzed for chlorine and found to contain 19.5 percent. Empirically related, the chlorine content is the equivalent of 30.4 percent calcium chloride. The difference in values based on the analysis of the two elements may be explained on the basis of slight error inherent to each method magnified by the factors involved in relating the elements to the compound. The average value (29.7) is suggested as the estimated anhydrous calcium chloride content of the sample.

I am not aware at the present time of any reason why the Agricultural Analytical Service could not conduct infrared analysis of water-reducing retarders, under a contractual arrangement, for the Texas Highway Department.

The procedure employed for the infrared analysis of the materials in that of Halstead and Chaiken (Public Roads, 1961; pp. 126-135) with the exception of the equipment employed. The Service employs a micro-disk die and a beam condenser for preparation of a micro-disk rather than the 13 millimeter disk referenced in the article. The instrument used is a Beckman IR-5 double beam Infrared Spectrophotometer.

I estimate the cost of infrared analysis to be approximately \$20.00 per sample.

If I can be of further service, please let me know.

Sincerely yours,  
JACK D. PRICE  
Assistant Professor

JDP:ct

\*Original code designation was altered on July 28, 1965 by D. L. Ivey.

TEXAS A&M UNIVERSITY  
College Station, Texas

ACTIVATION ANALYSIS RESEARCH LABORATORY

August 18, 1964

Dr. T. J. Hirsch  
Structural Research Department  
Texas Transportation Institute  
Campus

Dear Sir:

The six samples of chemical admixes for concrete which you submitted for activation analysis of Na, Mg, Cl, K, Ca, and Zn were run on May 14, 1964. The samples were activated for 30 seconds in a neutron flux of  $5 \times 10^{11}$  neutrons/cm<sup>2</sup>-sec. Due to the large amount of activity induced in sodium, it was not possible to analyze for any other constituents. Because of other pending experiments, no further attempts were made to optimize for the analysis the elements under consideration. A six-hour activation would induce sufficient activity that K, Zn, and Na could be analyzed for after a 40-hour wait time. This increases the time for analysis to 3 to 4 days for a purely instrumental technique.

The table enclosed lists the nuclear properties for the isotopes being considered.

Sincerely yours,  
S/ PETE JIMENEZ

PJ:ah  
Enclosure: Table 1