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16. ABSTRACT

I. Introduction

The results of a recent investigation of the causes of corrosion of reinforcing steel indicated that the more imminent cause of corrosion was the presence of the chloride rather than the sulfate-ion in the environment. 1

This conclusion was based upon the result of a mathematical and graphical solution of data that reflected the incidence of corrosion of bridge substructures which were exposed to variable concentrations of both the chloride and the sulfate ions in the natural soils and waters.

Normally, mathematical solutions of data are considered to be accurate. But the solutions are only as accurate as the original data and the analytical method. Therefore, in order to verify whether the chloride-ion is more aggressive to the reinforcing than the sulfate-ion, a direct test of this mathematically inferred hypothesis was considered necessary.

The verification test was accomplished by the placing of reinforced concrete specimens into saturated solutions of either sodium chloride or sodium sulfate.

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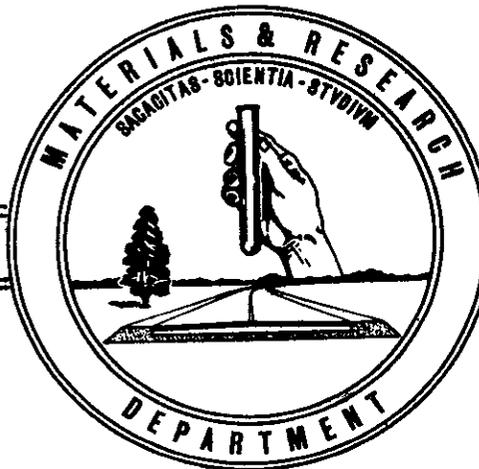
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STATE OF CALIFORNIA
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS



LABORATORY CORROSION TESTS
OF
REINFORCED CONCRETE EXPOSED TO SOLUTIONS
OF SODIUM CHLORIDE AND SODIUM SULFATE

FEBRUARY 1964



64-06

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

February 20, 1964
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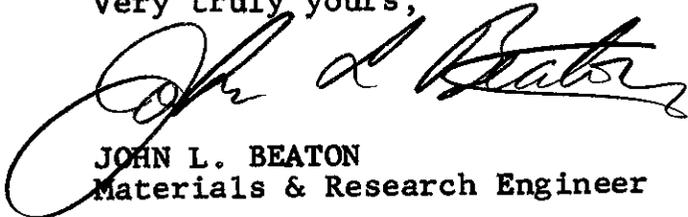
Dear Sir:

Submitted for your consideration is a report
on:

Laboratory Corrosion Tests of
Reinforced Concrete Exposed to Solutions
of Sodium Chloride and Sodium Sulfate

Study made by Technical Section
Under general direction of D. L. Spellman
Work supervised by R. F. Stratfull
Report written by R. F. Stratfull

Very truly yours,



JOHN L. BEATON
Materials & Research Engineer

cc:LRGillis
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LABORATORY CORROSION TESTS OF REINFORCED CONCRETE EXPOSED TO SOLUTIONS OF SODIUM CHLORIDE AND SODIUM SULFATE

I. Introduction

The results of a recent investigation of the causes of corrosion of reinforcing steel indicated that the more imminent cause of corrosion was the presence of the chloride rather than the sulfate-ion in the environment.¹

This conclusion was based upon the result of a mathematical and graphical solution of data that reflected the incidence of corrosion of bridge substructures which were exposed to variable concentrations of both the chloride and the sulfate ions in the natural soils and waters.

Normally, mathematical solutions of data are considered to be accurate. But the solutions are only as accurate as the original data and the analytical method. Therefore, in order to verify whether the chloride-ion is more aggressive to the reinforcing than the sulfate-ion, a direct test of this mathematically inferred hypothesis was considered necessary.

The verification test was accomplished by the placing of reinforced concrete specimens into saturated solutions of either sodium chloride or sodium sulfate.

II. Summary and Conclusions

Test results verify that chlorides are a more imminent cause of reinforcing steel corrosion than sulfates. However, the test results do not verify or infer that steel embedded in concrete will not ultimately corrode when subjected to a sulfate or other aggressive environment.

With other variables assumed to be constant, the data indicate that the time to the visual evidence of corrosion of the reinforcing will progressively decrease in the same order as the following test methods:

1. Alternate immersion (longest time to corrosion)
2. Partial immersion with the air at a high relative humidity (next longest time)
3. Partial immersion with the air at a low (50% +) relative humidity (shortest time)

Comparatively by weight, the concrete was found to absorb approximately seven times as much chlorides as sulfates.

Air-entrained concrete seems to have a higher ambient moisture level and absorbs more water than non-air-entrained concrete. Generally, steel corroded sooner in air-entrained concrete. However, the amount of corrosion as measured by length of cracks in the air-entrained concrete was found to be less in all partial immersion tests.

Electrical surface potential measurements are a valid means for determining the locations of steel corrosion. The magnitude of the voltage measurements is greatly influenced by the type of exposure. Therefore, this type of measurement can only be relied upon as a qualitative means of investigation.

The data show that once corrosion becomes localized, it will continue in the same place until the concrete is

ruptured or is stained by the corrosion products of the steel. The locations of new areas of corrosion or the expansion of this corrosion area will then be controlled by environmental and other concrete variables.

The time to corrosion of embedded steel depends upon concrete quality, moisture content, the thickness of cover over the embedded steel, and the kind of chemicals in the environment.

III. Test Method

It is most desirable to perform any test in a standardized manner so that the results may be reproduced. Therefore, the published literature was searched for a standardized method for conducting corrosion tests of reinforced concrete.

The following is a list of some of the more common types of corrosion testing:

1. Exposure to tidal water (alternate submersion)^{2,14}
2. Normal outdoors^{3,9,10,14,15,17}
3. Laboratory, high humidity^{3,4,10,13}
4. Laboratory, low humidity^{3,11}
5. Alternate immersion^{3,13}
6. Alternate but partial immersion^{5,7}
7. Variable salt, moisture, temperature⁶
8. Salt spray cabinet⁸
9. Covered with wet towel¹¹
10. Flow of water vapor¹²
11. Periodic spraying with salt water¹²
12. Immersed in water¹³
13. Partial immersion¹⁵
14. Dry cellar¹⁶
15. Impressed voltages¹⁸

From this list of test methods, it is apparent that there are many different methods used for corrosion testing of reinforced concrete.

In this study, two methods of testing were considered. They were:

1. Alternate immersion of a specimen in a test solution followed by forced air drying in the laboratory. The test solutions used in alternate immersion tests were:
 - A. Water saturated with sodium sulfate
 - B. Water saturated with sodium chloride
2. The creation of a differential concentration cell on the steel by placing a bottomless container that is filled with a saturated test solution on the center of a concrete block.

The first experiment with the second procedure started on six of the blocks, was discontinued after approximately 16 hours of test because the containers could not be prevented from leaking. The surfaces of these latter specimens were washed with distilled water, and then were partially immersed in the test solution as a satisfactory alternate to the second method. More will be said about these six blocks under Section VI, Test Results.

The first test of these reinforced concrete specimens was performed in a cabinet maintained at 95°F and at approximately 95-97% relative humidity. Another partial immersion test was performed with the relative humidity of the air in the test cabinet at an average of 54%. In this latter test, no solutions were allowed to contact the center of the reinforced concrete blocks.

The test solutions used in the partial immersion or differential concentration cell test were:

- A. Potable water
- B. Sodium sulfate solution consisting of 84,000 ppm as SO₄ in water
- C. Saturated sodium chloride solution consisting of 170,000 ppm of chloride-ion in water.

All of the test solutions were maintained in chemical saturation by the continual presence of an excess amount of salt in the bottom of the container.

IV. Test Specimens

The 20 test specimens were constructed using type II cement as cubes measuring 2"x5"x18" and contained two each of 1/2-inch diameter by fifteen 1/2-inch long sand-blasted reinforcing steel bars. The design depth of steel embedment was 3/4-inch all around from the outer surfaces of the concrete.

The proportions and other details of the concrete mixes used in the testing are shown in Table 1.

All of the reinforced concrete specimens were cured in a fog room at 73.4°F for 14 days, then air dried in the laboratory for approximately 60 days before testing.

The air entrainment of the concrete was accomplished by means of the addition of a neutralized Vinsol resin to the mix.

In addition to the usual concrete specimens, it was decided to explore the effect of certain coatings on corrosion of steel. Therefore, two each, of 7-sack, 4.5% air-entrained, reinforced concrete blocks were coated with a mixture of 95% raw linseed oil and 5% paraffin by volume. The coating was heated to approximately 350°F, then brushed on the concrete surface. One each of these coated blocks was placed into the saturated sulfate and chloride solutions of the alternate immersion test to determine if the coating had any effect on the corrosion of the steel.

V. Measurements

Electrical surface potential measurements and weights of the reinforced concrete specimens were periodically recorded.

The electrical surface potential measurements were performed by using two saturated calomel reference cells. One cell was left stationary at a marked and repeated location on the surface of the block. The other cell was moved to various permanently marked locations on the surface of the concrete. The voltage differences and polarity between the two cells were recorded by means of a 100-million ohm input impedance voltmeter. The purpose of the surface potential measurements was to determine their reliability in predetermining the locations where cracking caused by steel corrosion would likely occur.

The weighing measurements were originally considered as a means for determining when the greatest, and also reasonably duplicate quantities of test solution would be absorbed and evaporated by the concrete during the alternate immersion test.

VI. Test Results

A. Corrosion

Figure 1, "Some Variables that Affect the Deterioration of Reinforcing in Concrete when Subjected to Saturated Sodium Chloride Solutions", shows that corrosion of the reinforcing occurred only when the concrete was exposed to a chloride environment. Steel in concrete subjected to sodium sulfate solutions did not corrode up to the time the tests were terminated which was 214 days.

The data shown on Figure 1 and Table 2, indicate that corrosion occurs more rapidly when testing by partially rather than alternately immersing the reinforced concrete specimens.

The two blocks that had the chloride solution in contact with the center of the concrete for approximately 16 hours just prior to the partial immersion test, had evidence of corrosion of the steel in these same locations. The specimens which had the sulfate solution in contact with the center of the block for approximately 16 hours did not show any evidence of steel corrosion at the conclusion of the test.

The test results shown on Figure 1 and Table 2 indicate that the steel embedded in concrete which contains 6 sacks of cement per cubic yard corroded sooner and produced a greater amount of concrete cracking than occurred in the 7-sack concrete.

The criteria used in this test as evidence of corrosion was the visual observation of a rust stain, or a crack on the surface of the concrete.

As shown on Figure 1, the visual evidence of corrosion of steel occurs earlier in air-entrained concrete. However, Table 2 shows that the amount of corrosion-caused concrete cracking in the partial immersion test was less for air-entrained concrete than it was for non-air-entrained concrete.

B. Electrical Potential Measurements

Figure 2, "Average Surface Potential Difference Versus Days of Exposure of Reinforced Concrete Blocks Immersed at One End", is a typical plot of these measurements for the 7-sack, 4.5% air-entrained concrete exposed to 95-97% relative humidity.

Figure 3, "Average Surface Potential Difference Versus Days of Exposure of Reinforced Concrete Blocks Subjected to Alternate Immersion", shows the typical maximum surface potential difference values that were measured for a part of the test period for the 7-sack, 4.5% air-entrained concrete specimens.

In the chloride solution of the alternate immersion test, the maximum potential differences were generally found on the concrete surface after the first 2 to 4 days of the air drying cycle. Conversely, in this same test, the minimum potential difference values were found after approximately 14 days of the soaking cycle.

The first evidence of concrete deterioration was found to be at the locations where the maximum anodic surface potential differences were observed. As a result, these potential measurements were subjected to mathematical scrutiny, and the results are shown in Table 3. The data in this table indicate that in all cases, a potential gradient or voltage difference was found on the surface of the concrete irrespective of whether or not there was corrosion of the steel.

In order to determine if the measured maximum surface potentials were found at consistent locations, the data were subjected to further mathematical analysis.

Table 4, "Maximum Anodic Potential Locations as Percent of the Times Measured for Corroded Specimens (for Chloride Solutions)", shows that the locations on the concrete surface where the maximum anodic surface potential difference was measured could be repeated with a high degree of reliability. Also, once an anodic location is created, it tends to remain anodic at that location even though additional anodic locations may form.

It was also observed that the immersion of one end of a specimen into a saturated chloride solution for more than 30 days did not change the location of the maximum anodic surface potential difference which was initiated by approximately 16 hours of salt contact in the center section of the concrete.

In the alternate immersion tests, after the surface of the concrete was cracked or stained by the corrosion products of the steel, the locations of the maximum anodic surface potential difference shifted to a new anodic area.

Table 5, "Maximum Anodic Potential Locations as Percent of the Times Measured for Non-corroded Specimens (Tap Water and Sulfate Solution)", shows how often a particular location was found to be the area of a maximum anodic surface potential difference on the concrete in which the steel did not corrode. As shown on Table 5, the partial immersion of a relatively chloride-free concrete into a salt-free environment did not necessarily result in the presence of an aggressive corrosion cell at the submerged end of the block. Therefore, the findings of Schaschl and Marsh²⁰ regarding the corrosion of steel in soils of differential permeability and moisture content do not seem to be directly related to the outstanding durability of steel contained in salt-free concrete. However, this observation does not preclude the fact that a differential moisture content in the concrete could cause aggressive corrosion of the steel embedded in salt contaminated concrete.

Anodic, or corroding, locations of reinforcing steel have been found where the concrete has a low electrical resistivity. Also, concrete with a low resistivity is normally an area of concrete which has a greater moisture content.¹⁸ Therefore, it seems that the differential aeration or moisture content type of corrosion mechanism can be a very important factor in regulating the rate of corrosion of steel in salt contaminated, or perhaps chemically attacked concrete.

As shown by the potentials listed in Table 3, the presence of a variable amount of corrosion-causing electrical current was found in all of the concrete specimens for reasons that were not obvious and do not seem to be a matter for immediate concern.

It has been reported that corrosion of steel in salt-free concrete will not be likely until the alkalinity of the concrete at the steel interface is exhausted or becomes contaminated with salt.¹⁹

Based upon the results of previous tests,¹⁸ of applying various voltages to steel embedded in concrete, it is not likely that corrosion of the steel will occur within the normally expected service life of the structure if the concrete remains relatively chloride-free. This assumption is based upon competent concrete construction practice such as exercised by the California Division of Highways for reinforced concrete structures, and also the

lack of an external influence, such as a stray electrical current.

C. Weighing Measurements

As inferred by the data shown in Table 6, air-entrained concrete generally absorbs a greater quantity of moisture than non-air-entrained concrete. It is recognized that the concrete absorption values shown on Table 6 can only be considered as qualitative; the reason being that the weight ratios are based upon some unknown moisture content of the concrete at the beginning of the test. Therefore, the measurements cannot be regarded as quantitative as they were not related to a basic measure, such as the saturated or oven-dry weight of concrete.

It is believed that the greater weight gain of the concrete exposed to a chloride solution as inferred by the data in Table 6, could be the result of an accumulation of the chloride salt in the concrete.

D. Chemical Analysis of Concrete

At the conclusion of the test, fragments of concrete were obtained from various parts of the test specimens and chemically analyzed for their chloride and sulfate content. The result of the analyses is shown in Figure 4.

As one might generally assume, the 6-sack concrete absorbed a greater quantity of chlorides than did the 7-sack concrete. In the partial immersion test, the air-entrained concrete absorbed less chlorides than the non-air-entrained concrete; the reverse was true in the case of alternate immersion!

The chemical analysis also showed that the submerged portion of the concrete in the partial immersion test absorbed the approximate same quantity of chloride and also sulfate ions as when the concrete was alternately immersed and then air dried. Also, the weight of sulfate retained in the concrete was approximately 7% of the quantity of

chloride. This great variation of retained salt was found in the concrete irrespective of the fact that there was about a 50% differential in the quantity of the salts in the test solution.

These data indicate that there is a chemical and/or a mechanical restraining mechanism which inhibits the penetration of the sulfate-ion into the interior of the concrete. From this observation, one could speculate that the time to initial corrosion of embedded steel could be a direct function of the ability of the concrete to prevent the penetration of a sufficient quantity of the sulfate-ion to the surface of the steel either by virtue of its quality or thickness of cover over the steel.

VII. Discussion

The data obtained during this test indicate that the phenomenon of corrosion of steel in concrete is not completely understood. Even so, concrete quality is one of the major factors which controls the time to corrosion of the embedded steel.

The reader should be aware that there are a number of variables which can influence the corrosion test results of reinforced concrete. For instance, a recent report¹ indicates that the following variables should be considered as to their relative influence on the time to corrosion in this test. These are:

1. The variations in the quantity of mixing water used in the concrete mix could result in a 14% variation in testing time.
2. A change in 1 sack of cement could result in a 25% variation of testing time.
3. A difference of 0.1-inch from the design thickness of the concrete cover used in this test could result in approximately a 16% variation in test result time.

Tests of full sized reinforced concrete piles have shown that air-entrained concrete does not have a much corrosion-caused concrete cracking as does non-air-entrained concrete.²¹ The test results which are shown in Table 2, appear to correlate with the field experience regarding the smaller amount of cracking of air-entrained concrete. However, a report²¹ on the field performance of the piling did not say when corrosion was observed, it just told how much concrete was cracked. The results of the field tests definitely demonstrated that concrete quality is a primary control on the corrosion of the reinforcing.

The testing verifies that the more imminent cause of corrosion is the chloride-ion and not the sulfate-ion. However, it has been reported that hydrogen sulfide is a factor in the corrosion of prestressed concrete tanks.²²

Therefore, it should not be considered that steel embedded in concrete will indefinitely remain corrosion free if salts other than chloride eventually penetrate to the surface of the steel.

The reader should take care in interpreting these data quantitatively because of the number and the combined effect of the variables that can influence the numerical test results.

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January 1964

**SOME VARIABLES THAT AFFECT THE DETERIORATION OF REINFORCING IN CONCRETE
WHEN SUBJECTED TO SATURATED SODIUM CHLORIDE SOLUTIONS**

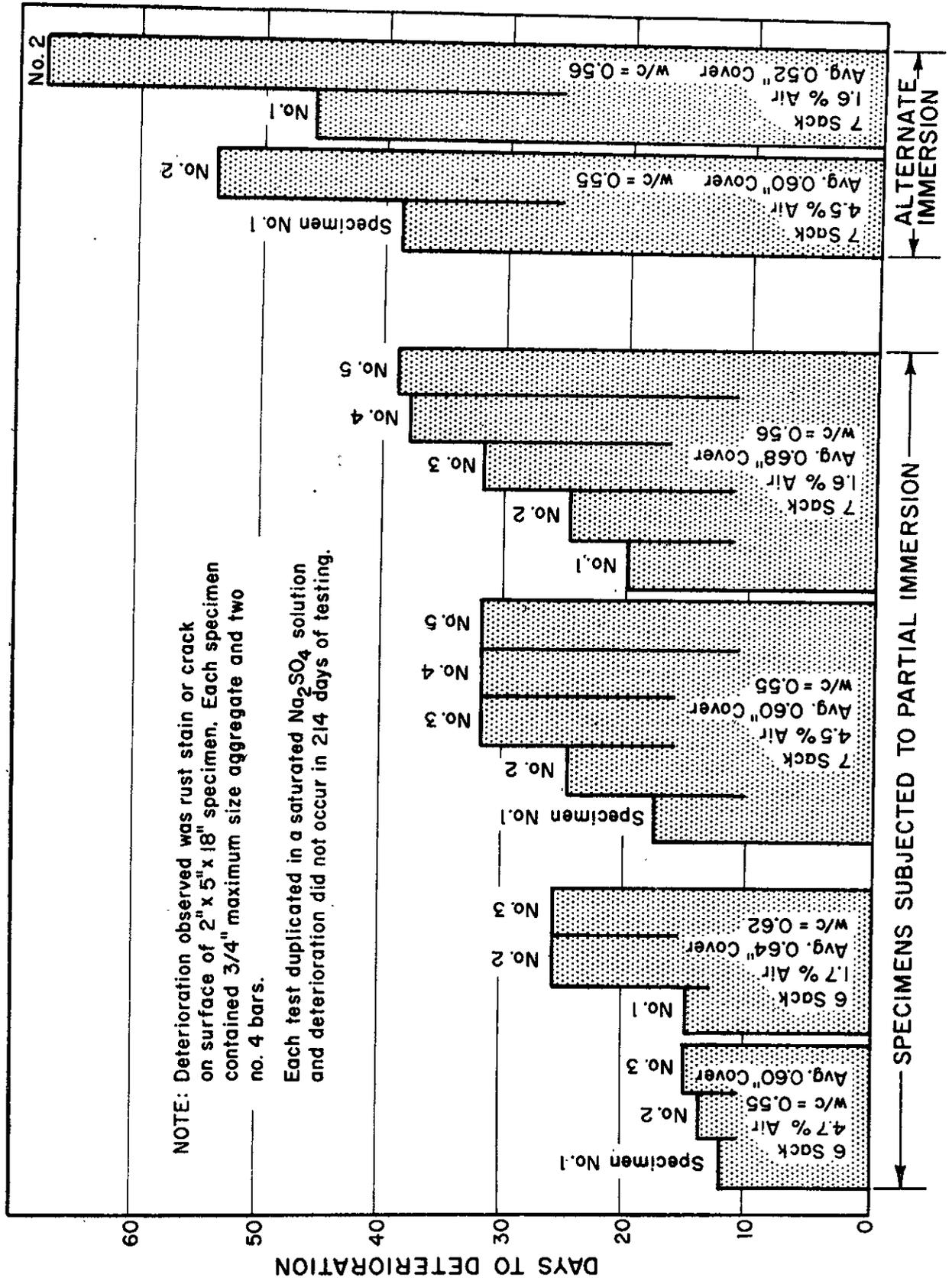


FIGURE 1

AVERAGE SURFACE POTENTIAL DIFFERENCE VERSUS DAYS OF EXPOSURE OF REINFORCED CONCRETE BLOCKS IMMERSED AT ONE END

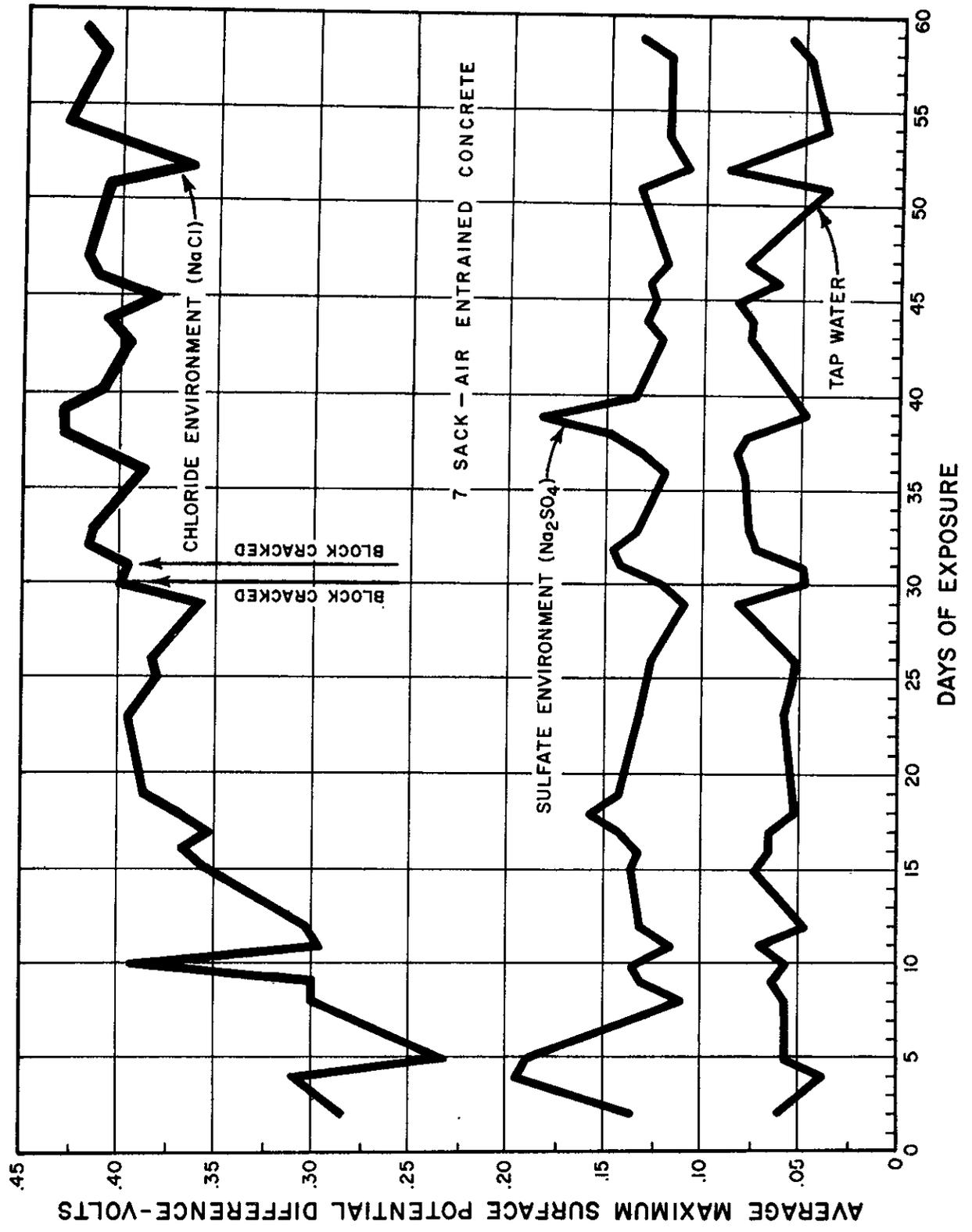
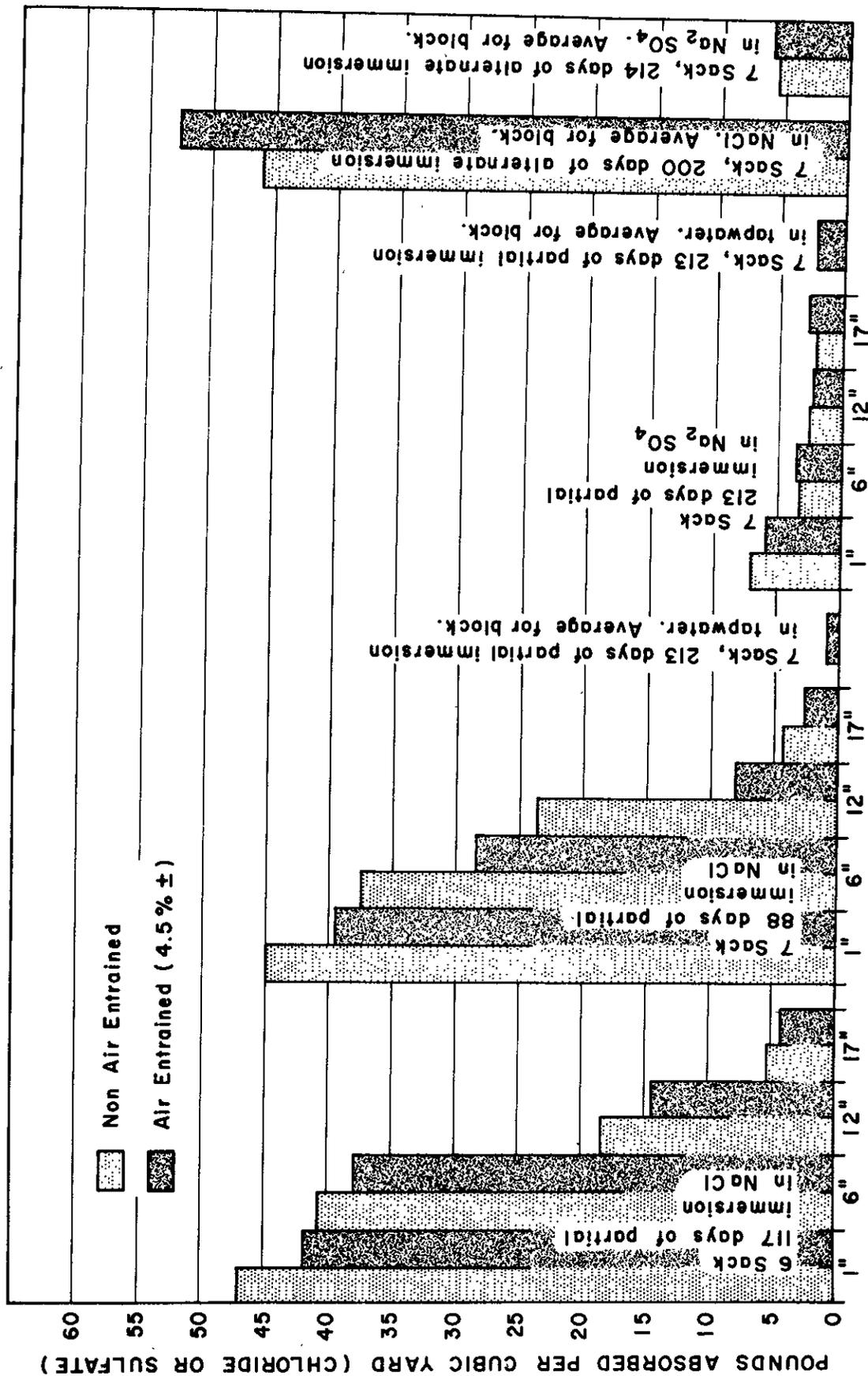


FIGURE 2

ABSORPTION OF CHLORIDES AND SULFATES BY CONCRETE



NOTE: Inches denote location above bottom of specimen where samples were taken for chemical analysis. The 2" x 5" x 18" reinforced concrete specimens were partially immersed to a depth of 2" of their length.

FIGURE 4

TABLE 1

CONCRETE MIX DATA

	Air Entrained		Air Entrained
	Plain		
Unit Weight, lbs./Cu. Ft.	150.1	145.8	145.9
Slump, Inches	4	4	3-1/2
Percent Air	1.6	4.5	4.7
Cement Factor, Sks./Cu. Yd.	7.02	6.95	5.92
W/C Net, Lbs./Sk. Cement	47.7	47.2	51.5
W/C Gross*, Lbs./Sk. Cement	54.2	53.1	59.2

*Including water absorbed by aggregates

Note: Air-entrained concrete was made with aggregate mixture ratio of 52% aggregate and 48% sand, and non-air-entrained was 50% and 50% respectively by volume.

TABLE 2

CRACKING OF 18-INCH LONG, REINFORCED
CONCRETE BLOCKS

		% Air in Concrete	Inches of Cracking	Days to Cracking
Partial Immersion:				
54% R.H. 6-sack concrete	Non-AE	1.7	5.8	22.3
	AE	4.7	3.3	13.7
54% R.H. 7-sack concrete	Non-AE	1.6	3.7	25.7
	AE	4.5	0.5	25
95-97% R.H. 7-sack concrete	Non-AE	1.6	---	38.5
	AE	4.5	---	32
Alternate Immersion:				
7-sack concrete	Non-AE	1.6	7	52
	AE	4.5	8	47

Note: Inches of concrete cracking is average length.
Days to deterioration is average time to observed
concrete cracking or rust stain. Length of crack-
ing not measured on 95-97% relative humidity blocks.

TABLE 3

AVERAGE ANODIC SURFACE POTENTIALS (VOLTS)

Exposure	Air Content of Concrete	Solution		
		Water	Sulfates	Chlorides
54% R.H.	1.6%	0.14	0.14	0.38
	4.5%	0.07	0.14	0.40
95-97% R.H.	1.6%	0.10	0.13	0.20
	4.5%	0.05	0.13	0.39
Alternate Immersion	1.6%	----	0.03	0.06
	4.5%	----	0.02	0.11
	4.5% (Coated)	----	0.07	0.17

Note: All concrete contained 7 sacks of cement per cubic yard.

R.H. - Relative Humidity

"Coated" was a block that was brush coated with raw linseed oil and paraffin.

TABLE 4

Maximum Anodic Potential Locations as Percent of the Times Measured for Corroded Specimens (for Chloride Solutions)

Immersion	Air Content of Concrete	Time	Times Measured	Days Test	Percent of Times Measured					
					Anodic location - inches from end of block					
					0	4.5	9	13.5	18	
Partial Immersion	1.6% - #1	At Deterioration	23	38	0	0	100	0	0	18
		End Test	38	63	3	3	94	0	0	0
	4.5% - #1	At Deterioration	24	32	100	0	0	0	0	0
		End Test	39	63	100	0	0	0	0	0
	1.6%	At Deterioration	12	25	100	0	0	0	0	0
		End Test	34	88	100	0	0	0	0	0
4.5%	At Deterioration	10	18	100	0	0	0	0	0	
	End Test	34	88	100	0	0	0	0	0	
Alternate Immersion	1.6%	At Deterioration	40	68	0	100	0	0	0	0
		End Test	94	200	9	87	3	1	0	0
	4.5%	At Deterioration	33	54	0	0	10	90	0	0
		End Test	97	200	1	0	31	67	1	1
	4.5% Coated	At Deterioration	22	39	0	0	0	41	59	26
		End Test	97	200	59	0	0	15	0	0

All concrete was 7-sack mix.

#1 - Samples had chloride solution in contact with center (9") block for approximately 16 hours prior to partial immersion to a 2-in. depth from "0" inch end of block.

Coated specimens were brush coated with hot mixture of raw linseed oil and paraffin.

At Deterioration - when first rust stain or concrete crack was observed.

TABLE 5

Maximum Anodic Potential Locations as Percent of the Times Measured for Non-corroded Specimens (Tap Water and Sulfate Solution)

	Exposure	Air Content of Concrete	Times Measured	Days Test	Percent of Times Measured				
					Anodic location - inches from end of block				
					0	4.5	9	13.5	18
Partial Immersion	Tap Water	1.6%	106	213	1	6	2	5	86
		4.5%	106	213	16	44	5	14	21
	Sulfate	1.6%	107	213	2	1	96	0	1
		4.5%	104	213	16	3	79	1	1
Alternate Immersion	Sulfate	1.6%	106	214	22	30	3	3	42
		4.5%	108	214	30	41	14	3	12
		4.5% (coated)	105	214	69	22	6	3	0

Note: Only sulfate partial immersion specimens had sulfate solution in contact with center (9") of block for approximately 16 hours prior to their partial immersion at a 2" depth from "0" inch end of block.

All concrete contained 7 sacks of cement per cubic yard. Coated specimen was brush coated with hot mixture of raw linseed oil and paraffin.

TABLE 6
WEIGHT GAIN OF CONCRETE

Exposure	Air Content of Concrete	Average Weight Increase, %			Total Moisture Absorbed, Percent	
		Tap Water Only	Sulfate Exposure	Chloride Exposure	Sulfate Exposure	Chloride Exposure
54% R.H.	1.6%	0.90	0.83	1.64	-----	-----
	4.5%	0.87	1.09	1.00	-----	-----
95-97% R.H.	1.6%	1.24	1.13	1.21	-----	-----
	4.5%	1.21	1.23	1.62	-----	-----
Alternate Immersion	1.6%	-----	3.37	3.92	9.30	9.61
	4.5%	-----	3.71	4.33	10.22	11.00
	4.5% Coated	-----	2.87	2.42	6.74	5.60

Note: All concrete contained 7 sacks of cement per cubic yard. The moisture gain of the concrete is based upon the weight of the concrete after air drying in the laboratory for approximately 60 days prior to the actual testing. Therefore, all data shown in this table should only be considered as qualitative.

Total moisture absorbed is a ratio of the total weight absorbed and the original weight of concrete at the end of 60 days of laboratory drying.

R. H. - Relative Humidity