Technical Report **130**

WATER VAPOR TRANSMISSION OF PLAIN CONCRETE

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U. S. NAVAL CIVIL ENGINEERING LABORATORY

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WATER VAPOR TRANSMISSION OF PLAIN CONCRETE

Y-R011-01-025

Type C

by

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OBJECT OF TASK

To measure water vapor permeability of concrete and relate it to corrosion of embedded steel; to investigate salt whisker crystal growth on concrete; and to measure electrical resistivity of concrete.

ABSTRACT

The effects of water-cement ratio, relative humidity, aggregate size, concrete slice position, and certain admixtures such as oleic acid and sodium chloride on the permeability of plain concrete were investigated. Included is a quarter replicate statistical experiment for two levels of each of six factors to permit an analysis of variance of different variables in the permeability study. Salt whisker crystal growth on specimens with sodium chloride as an admixture, believed to be reported for the first time, is discussed.

Water vapor transmission values were found to be significantly higher for higher water-cement ratios, smaller aggregate, and the absence of sodium chloride. Concrete slice position, oleic acid, and relative humidity were found to have no significant effect when compared with experimental variability.

The study demonstrates that water vapor transmission is not directly proportional to water vapor pressure differentials between the ends of the flow path.

The investigation of water vapor transmission in plain concrete will continue, together with studies of the electrical restivity of concrete and the relation of water vapor transmission to the corrosion of steel in reinforced concrete.

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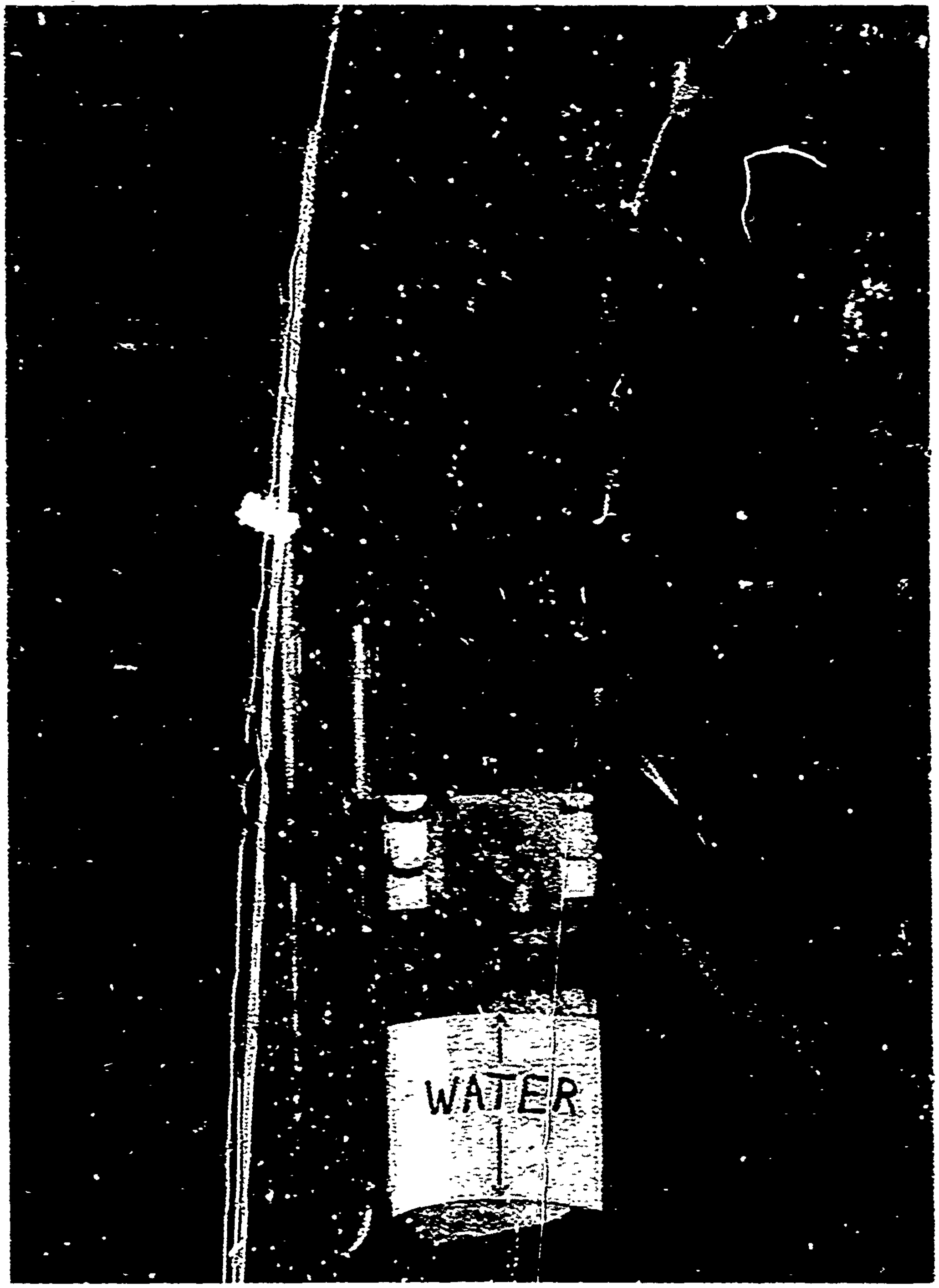

Figure 1. Galvanized steel wet cup and manometer.

BACKGROUND

In connection with a field study of reinforced coral concrete structures in tropical islands conducted by Professor C. H. Scholer under Contract NBy-3171 of 15 March 1959, and in conjunction with Task Y-R007-05-012, core samples were drilled from these structures in order to determine their water vapor permeability. The wet cup method was deemed most suitable for this purpose.

Twenty-six samples, approximately 3.5 inches in diameter and 1.5 inches thick, were sealed in galvanized steel cups by using alternating layers of plaster of Paris and 3M sealer (EC879), as shown in the cross section of a cup in Figure 1.

A few days after being placed in a 20-percent relative humidity room at 73.4 F, several of the specimens showed a fuzzy growth on the exposed surface (Figure 2). An X-ray diffraction analysis revealed this material to be primarily sodium chloride. A semiquantitative spectrographic analysis indicated the principal cation to be Na; traces of Ca, Al, Mg, Si, Fe, and Cu were also detected.

There was no direct correlation between the apparent quantity of salt whisker growth and the amount of salt present in the concrete. A chemical analysis of the concrete revealed that all chlorides expressed as a percentage of NaCl by weight of concrete ranged from 0.16 to 1.95. Vigorous whisker growth as well as traces of whisker growth were observed on specimens containing high as well as low salt contents, indicating that other factors influencing whisker growth were present.

The quantitative values of permeability were inconclusive because over a period of time many of the specimens were dislodged in the cups by internal pressures (Figure 3). To investigate this phenomenon, a mercury manometer was placed on an undisturbed cup, as pictured in Figure 1. It behaved in a repeatable manner; during the first week 4 inches of mercury vacuum pressure developed and during the following six weeks a positive pressure of up to 24 inches of mercury developed. It was concluded by the investigators that such pressures could dislodge a specimen from its seal.

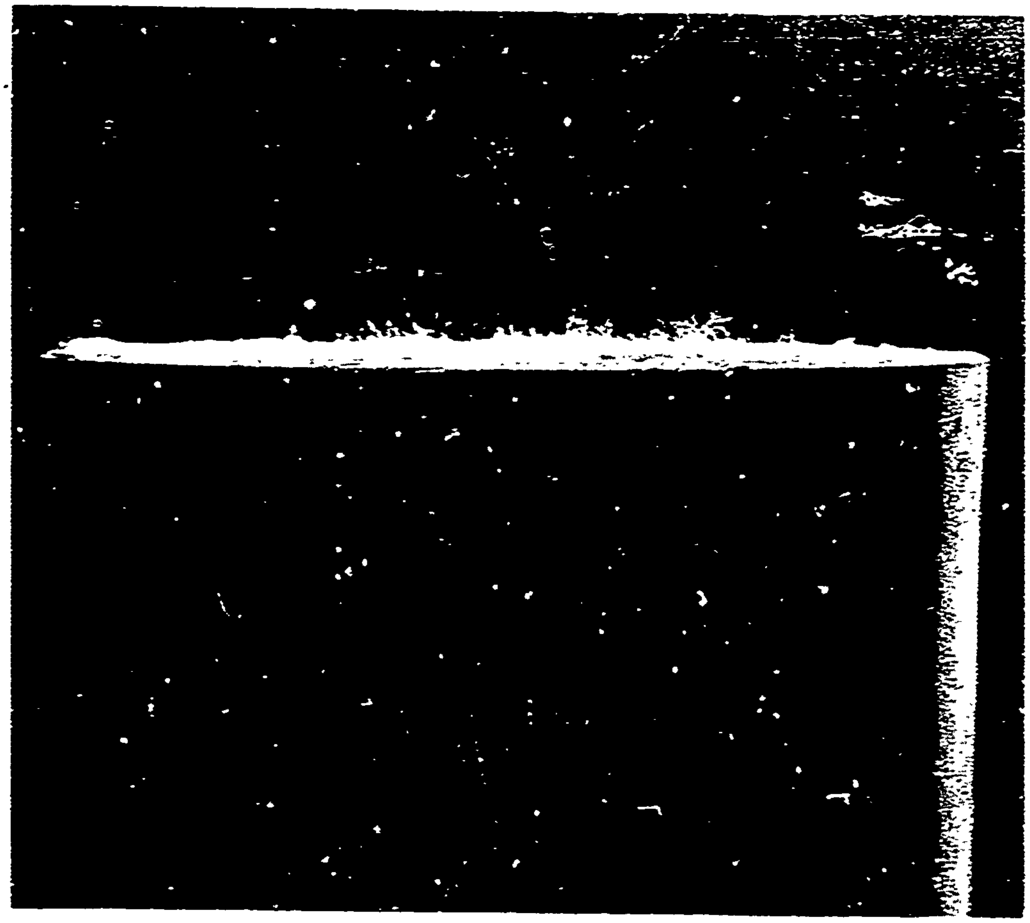

Figure 2. Sodium chloride whisker crystal growth on coral concrete.

A mass spectrometric analysis of the gas in the cup indicated that approximately 20 percent by volume was hydrogen; no nonatomospheric gas was detected. One possible explanation for the presence of hydrogen is that the zinc and the iron in the galvanized steel cup created an electrolytic couple, causing a release of hydrogen gas.

Figure 3. Effects of internal pressure caused by generation of hydrogen gas in galvanized steel cup.

STATEMENT OF THE PROBLEM

As an outgrowth of the above study, it was desired to explore the subject further. A literature search revealed that very little information exists about water vapor permeability of portland cement concrete. Therefore, a Laboratory Selected Research Task was established to determine whether or not significant relationships exist between the spalling and cracking of reinforced concrete, the effects of sodium chloride incorporated in the concrete, electrical conductivity of concrete, and water vapor permeability of the concrete. The corrosion of reinforcing steels associated with concrete structures, spalling and cracking of concrete, and electrical conductivity of concrete will be subjects of separate reports.

APPROACH TO THE PROBLEM

An inert material was needed to replace the steel cup; therefore, acrylic resin tubes and sheets were chosen as the most economical inert materials for fabrication of the wet cups.

Concrete cylinders 4 inches in diameter and 8 inches long were cast, from which 1.5-inch-thick specimens were cut. Thirty cylinders were cast from each 2.25-cubic-foot batch of concrete mixed in a Lancaster mixer.

Since the sealer used for previous specimens was inadequate, another material had to be found. A sealer was required that would (1) bond to the concrete as well as the acrylic tube, (2) be inert, (3) be impervious to vapor transmission, and (4) be able to withstand unpredictable pressures inside the cups. Shell Epon Resin No. 815 combined with Shell Epon Curing Agent T-1 was found to meet the requirements.

RESEARCH PROGRAM

Phase I

The first study, hereafter referred to as Phase I, was as follows:

1. The first factor in the experiment to be varied with respect to the permeability was strength of concrete — defined by water-cement ratio. All three concretes — high, medium, and low strengths — were designed for equal water

contents and a 3/4-inch maximum aggregate particle size. The strength was varied by varying the cement content and adjusting the proportions of fine and coarse aggregate. All concrete mixes were designed for equal consistencies without additives or admixtures; a 3-inch slump was used as the measure of consistency. See Table I for the complete mix designs. The cylinders not cut for wet cups were tested for compressive strength after fourteen days of fog curing at 73.4 F to obtain basic information about the standard deviations of compressive strengths.

2. The second factor to be varied was the absence or presence of sodium chloride as an admixture. The amount of NaCl to be added was 1.5 percent by weight of plastic concrete, based on the NaCl content of the coral concrete samples. The NaCl was dissolved in the mixing water before the water was added to the batch.

3. The third factor to be varied was the ambient condition in which the permeability would be studied. The cups were placed in either a 50-percent or a 20-percent relative humidity (RH) room at 73.4 F.

4. The fourth factor to be varied was the location of the specimen (disk) within the length of the cylinder. On the basis of information obtained informally from the Waterways Experiment Station that the upper half of a concrete cylinder as cast is more permeable than the lower half, it was decided to investigate slices taken from the upper, middle, and lower portions of the cylinder.

To summarize, all the variables involved in Phase I of this experiment were as follows:

1. Low (L), medium (M), or high (H) strength of concrete.

2. Absence (A) or presence (P) of NaCl.

3. 20 percent (L) or 50 percent (M) RH at 73.4 F.

4. Lower (L), middle (M), or upper (U) slice from the test cylinder of concrete.

Thus, there were four factors, two with two levels and two with three levels, making a total of 36 combinations in all. The design variables identified by batch number appear in Table II.

Table I. Summary of Concrete Mix Design Data

A. Characteristics of Materials

Cement: Victor, Type III
Aggregate: San Gabriel

Coarse: sp gr = 2.66; 24-hr abs = 1.6 percent
Fine: sp gr = 2.63; 24-hr abs = 1.8 percent
Grading:

Pounds Retained on Each Sieve

Sieve	3/4-inch max			3/8-inch max	
	H	M	L	H	L
3/4	7.4	7.8	6.2	0	0
3/8	74.1	75.5	73.7	0	0
No. 4	46.9	44.2	45.4	103.9	106.2
No. 8	27.2	28.6	32.2	38.3	39.8
No. 16	24.7	31.2	31.1	27.0	31.8
No. 30	27.2	28.6	31.3	23.6	23.7
No. 50	22.2	23.4	28.6	18.7	24.0
No. 100	12.4	13.0	14.6	18.4	21.0
Pan	5.0	7.8	7.0	17.2	19.2
Total	247.1	260.1	270.1	247.1	265.7

Water: Port Hueneme tap water at 73.4 F

Chemical analysis (ppm): hydroxide (0.0); carbonate (0.0); bicarbonate (137.0); chlorides (62.0); calcium (38.0); magnesium (14.6); sulphate (465.0); sodium and potash (219.0)

Slump: 3 in. (without additives)
Additives: Sodium chloride, U.S.P. granular; F.W. = 58.45 (1.5 percent by weight of plastic concrete unless otherwise noted); oleic acid, U.S.P. (0.25 percent by weight of cement)

Table I. Summary of Concrete Mix Design Data (Cont'd)

B. Batch of 2.25 cu ft consisted of the following: (Phases I and II, large aggregate)

Gravel: 3/4-in. max. particle size
Sand: FM = 3.16

Mix	Cement (lb)	Water[a] (lb)	Cement Factor (sacks per cu yd)	W/C
H	59.50	26.4	7.62	0.444
M	46.25	26.4	5.92	0.571
L	37.60	26.4	4.81	0.702

C. Batch of 2.25 cu ft consisted of the following: (Phase II, small aggregate)

Gravel: 3/8-in. max. particle size
Sand: FM = 2.95

Mix	Cement (lb)	Water[a] (lb)	Cement Factor (sacks per cu yd)	W/C
H	61.9	27.5	7.92	0.444
L	39.2	27.5	5.02	0.702

a/The quantity of water added at the mixer was corrected for moisture present in the aggregate and moisture required for absorption.

Table II. Compressive Strengths, Standard Deviations, and Design Variables - Phase I
(Wet Cups Washed With Helium)

Batch No.	No. of Specimens	Compressive Strength, psi a/ (avg)	Standard Deviation, psi	Strength	Aggregate	Sodium Chloride b/	Oleic Acid a/	Slice	Relative Humidity
1 L	28	4280	180	L	L	A	A	L, M, U	L, M
1 LS	28	3380	95	L	L	P	A	L, M, U	L, M
1 M	28	5370	145	M	L	A	A	L, M, U	L, M
1 MS	28	4260	85	M	L	P	A	L, M, U	L, M
1 H	28	6970	265	H	L	A	A	L, M, U	L, M
1 HS	28	6620	105	H	L	P	A	L, M, U	L, M

a/ At age 14 days, cured in 100 percent RH at 73.4 F.
b/ 1.5 percent by weight of plastic concrete.
c/ 0.25 percent by weight of cement.

Note: L, M, H: Low, medium, or high strength.
L, M, U: Lower, middle, or upper slice of 4 by 8 cylinder (as cast).
L : Large (3/4-inch) maximum size aggregate.
A, P : Absence or presence of sodium chloride or oleic acid.
L, M : 20-percent or 50-percent RH at 73.4 F.

A batch design sheet was made to include identifications, amounts of materials, including admixtures, to be used, and the mixing procedure. Mixing instructions on the design sheet were as follows:

1. Add gravel, sand, cement.

2. Mix dry for 30 seconds.

3. Add water (73.4 F) in shortest time possible with mixer running.

4. Mix for 150 seconds, or a total of 3 minutes including items 2 and 3.

5. Take slump test: designed for 3 inches; actual slump ______ inches.

6. If slump is too low, add water and mix for 30 seconds.

7. Repeat slump test. Final slump _____ inches.

8. Place concrete in molds and vibrate with a 1-inch stud vibrator.

9. Smooth off tops with wooden float.

10. Cover with metal plates and place in fog room.

11. After 24 hours, strip molds, number specimens, and place them in fog room on shelves.

Although the mix was designed for a 3-inch slump, the NaCl increased the slump to approximately 7 inches. After the hardened cylinders were numbered, a table of random numbers[1] was used to designate two cylinders from each batch for permeability tests.

After 14 days in the fog room at 73.4 F, the 28 cylinders not chosen for the permeability experiment were tested in compression. The compressive strengths as well as their standard deviations were computed and appear in Table II. Disks were sawed from the two cylinders chosen randomly, then placed in wet cups.

Before being sawed, each cylinder was embedded in plaster of Paris to avoid chipping the edges of the disk at the breakthrough of the saw. A 3/4-inch slice was removed from either end of the embedded cylinder, then a 1-1/2-inch slice was cut from each end and one from the middle of the cylinder. After sawing, the plaster of Paris was broken away from the disk (Figure 4). The disk was then quickly prepared for sealing in the cup by washing, surface drying, and stamping with an identification number.

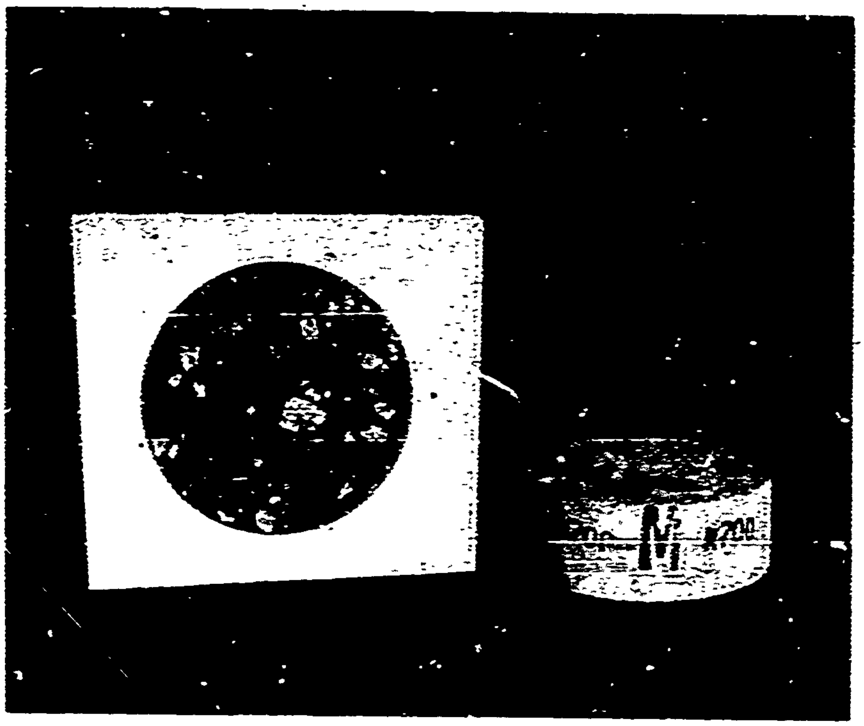

Figure 4. Plaster of Paris casting around concrete cylinder to prevent chipping of concrete at breakthrough of stone saw.

A detailed description of the plastic cup and component parts (Figure 5) is given in Appendix A. The cup was prepared for assembly by cleaning with ethyl alcohol and checking all the parts for a right fit. The disk was then sealed in the wet cup in the upright position with respect to the as-cast position, and the cup was assembled according to the procedure described in Appendix B. After the cup was assembled, it was half filled with distilled water. The water inside the cup was considered to be an infinite source for water vapor at 100-percent RH.

A mercury manometer was attached to the top outlet. Before placing the mercury, the air inside the cup was washed out with helium in order to eliminate unknown effects of any active gases that might be present. Tygon flexible plastic tubing was used on the cup system — a short piece about 2 inches long on the bottom outlet and a longer piece about 12 inches long connecting the top outlet to the manometer (Figure 6). All connections were wrap-sealed with Scotch Plastic Electrical Tape No. 33 to prevent leakage, and a short length of masking tape was applied over the electrical tape to prevent its own tension from causing it to loosen.

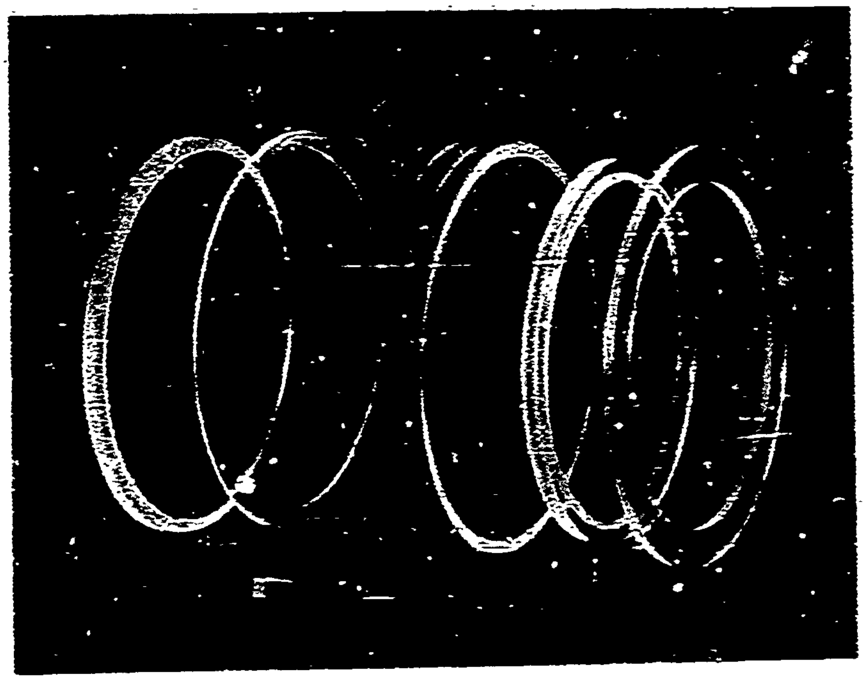

Figure 5. Component parts of acrylic wet cup.

The wet cup was then placed in the appropriate RH room. Five to six hours elapsed from the time the disk was sawed to the time the wet cup was placed in the RH room. Another twelve hours or so were allowed for the cup and contents to reach the 73.4 F temperature of the RH room before the initial weight was taken and the mercury in the manometer was leveled.

The wet cup was weighed in the RH room to the nearest gram (estimated to the nearest half gram). All cups were weighed weekly. Weight losses in grams versus time in days were plotted on a graph like the typical one in Figure 7.

After the first day each cup developed a few millimeters of vacuum, as indicated on the manometer. The vacuum increased at a different rate for each cup; after a month some cups developed a vacuum of over 200 millimeters of mercury. The vacuum continued to increase, but as the mercury reached its limit of travel, clamps were placed on the Tygon tubing and the manometers were removed without disturbing the interior of the cup. A likely explanation for the vacuum in the wet cups is that the helium has a diffusion coefficient through most substances greater than that of the atmosphere; therefore, a drop in internal pressure developed in each cup as the helium diffused through the assembled cup system.

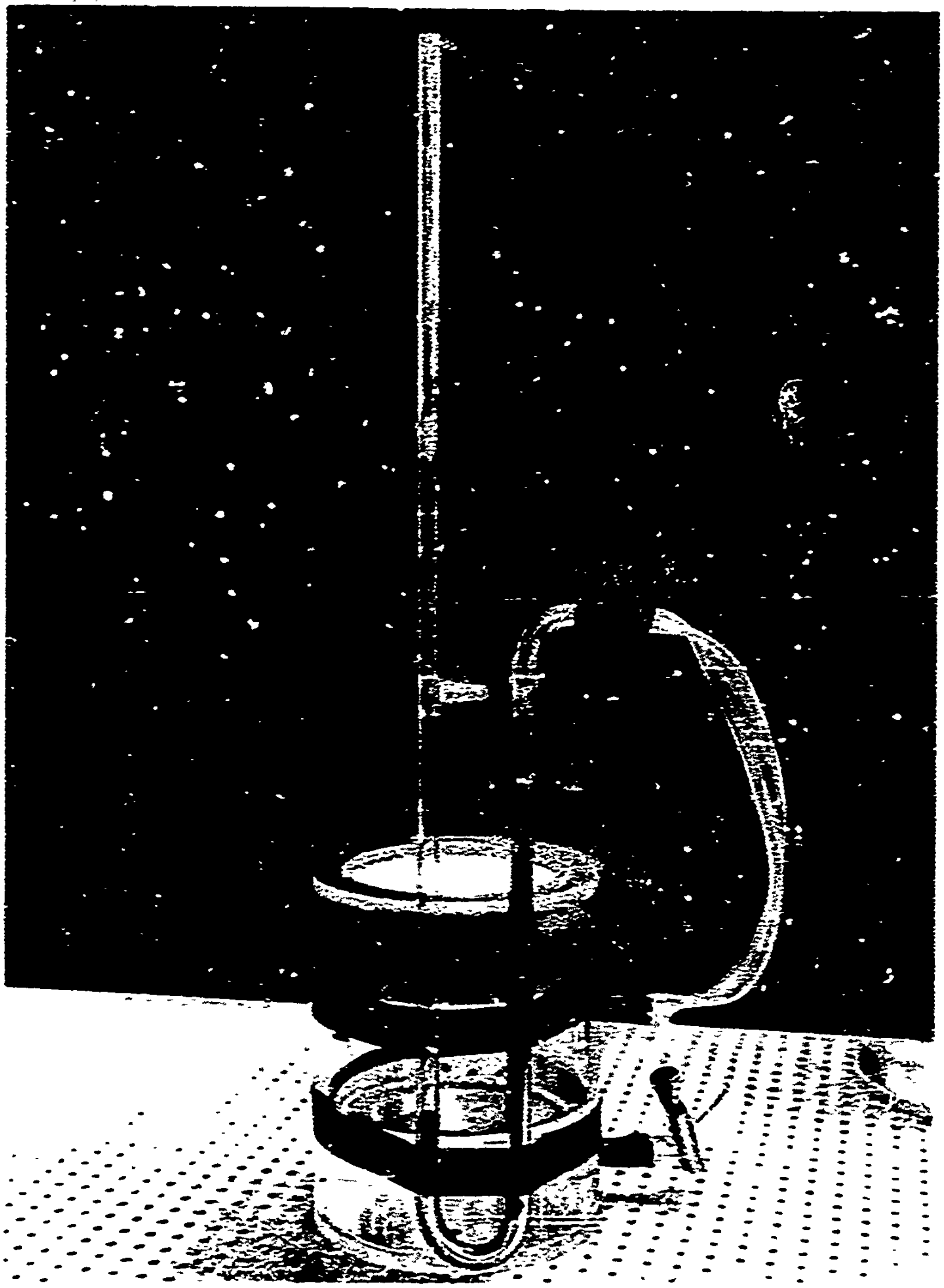

Figure 6. Acrylic wet cup assembled with specimen and manometer.

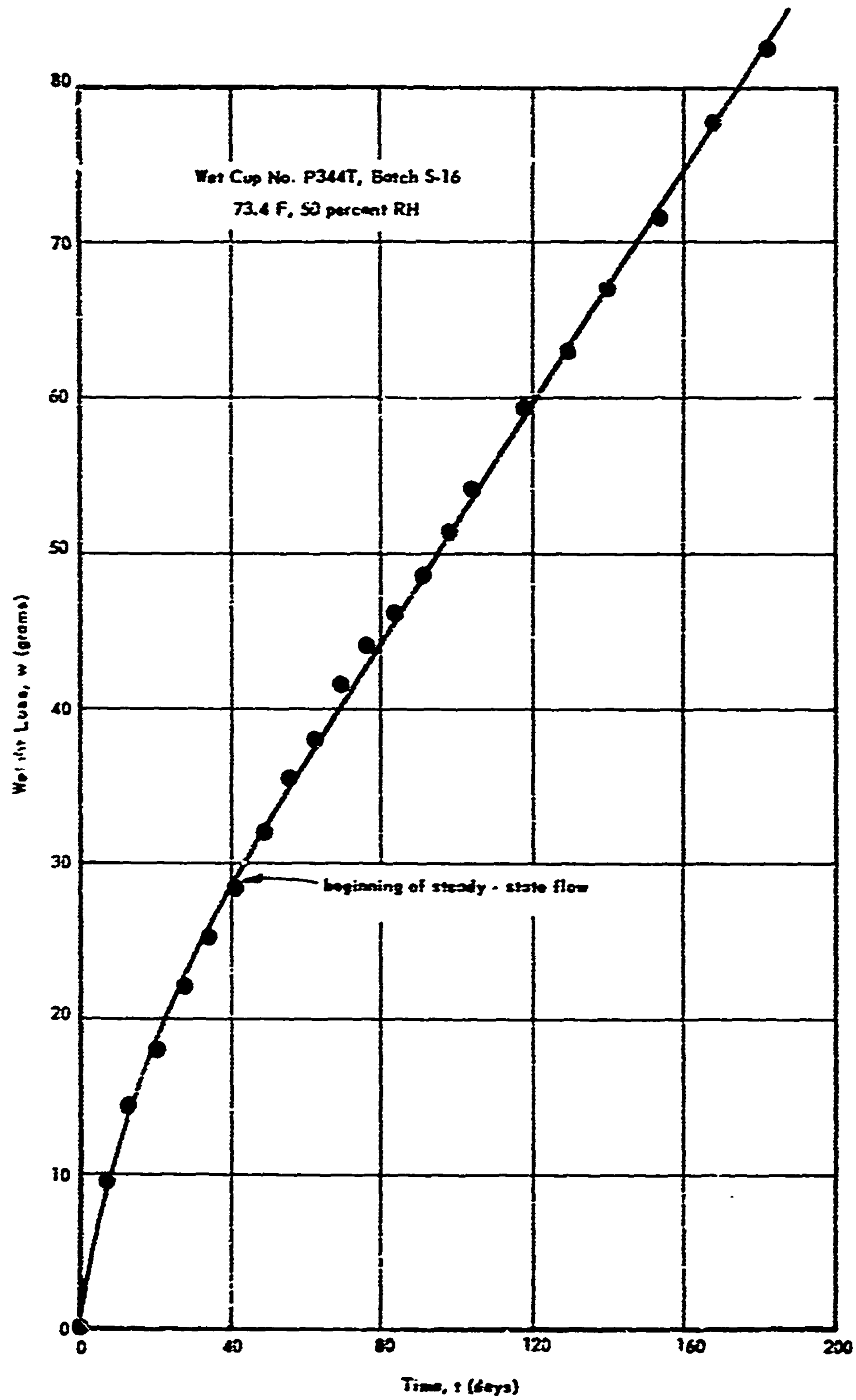

Figure 7. Typical weight loss versus time for wet cup.

Discussion of Phase I (Results)

Compressive Strength. The curves in Figure 8 were plotted from Table II. The top two curves, representing compressive strength plotted with respect to water-cement ratio, relate concrete without NaCl to concrete with 1.5 percent NaCl by weight of plastic concrete. These curves show that concrete without NaCl has a higher 14-day compressive strength than concrete with 1.5 percent NaCl. This relation held true for all the water-cement ratios used — 0.444, 0.571, and 0.702 for high-, medium-, and low-strength concretes, respectively. This relation was also verified later in Phases II and III.

The bottom two curves of Figure 8, representing standard deviation of compressive strength versus water-cement ratio, relate concrete with 1.5 percent NaCl and without NaCl. The standard deviation for concrete with 1.5 percent NaCl is definitely lower than the standard deviation for concrete without NaCl. There also seems to be a minimum standard deviation at a certain water-cement ratio; in these plots the minimum standard deviation occurs at a water-cement ratio of 0.61 for both concretes. At each of these water-cement ratios there is the least deviation or variation of compressive strength among samples.

Permeability. The equation, based on a form of Fick's law, that generally has been used to calculate water vapor permeability (WVP) coefficients for concrete is:[2]

$$WVP = \frac{W \ell}{A\, t\, \Delta P} \qquad (1)$$

where: W = total weight of vapor transmitted, grains (1 gram = 15.43 grains)

ℓ = length of flow path (or thickness of specimen), inches

A = area of cross section of flow path, square feet

t = time during which the vapor transmission occurred, hours

ΔP = difference of vapor pressure between ends of flow path, inches of mercury (at 73.4 F: 100-0 percent RH, $\Delta P = 0.82948$; for 100-20 percent RH, $\Delta P = 0.8\ (0.82948) = 0.66358$; for 100-50 percent RH, $\Delta P = 0.5\ (0.82948) = 0.41474$)

WVP = permeability coefficient, grain-inches per square foot per hour per inch of mercury vapor pressure difference

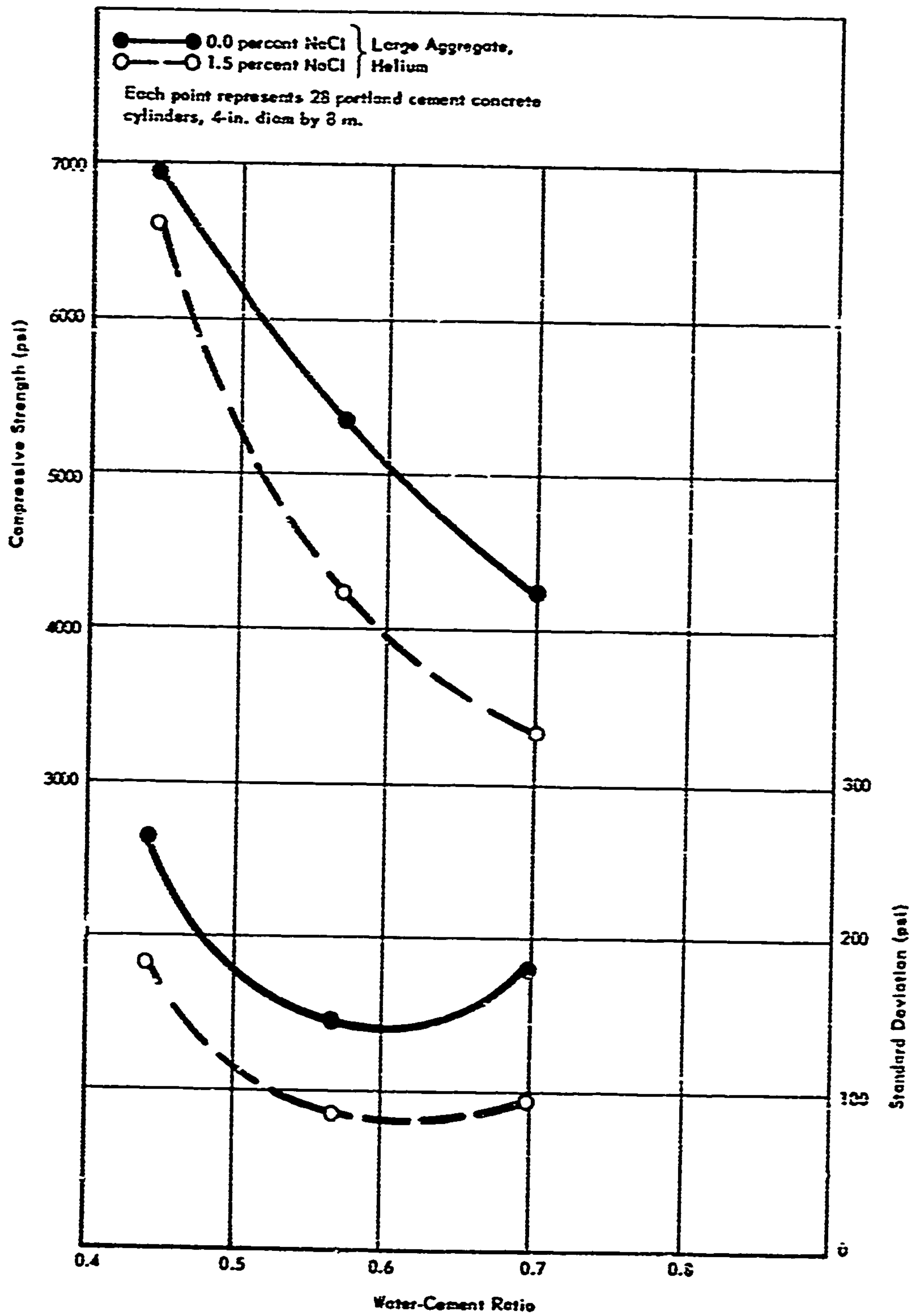

Figure 8. Compressive strength and its standard deviation versus water-cement ratio - Phase I.

Using Equation 1 to calculate WVP gave higher values for wet cups in the 50-percent RH room than for companion cups in the 20-percent RH room. After examination and comparison, it was decided that such calculated WVP values were incompatible. For specimens of identical mix design, A was constant and ℓ was virtually constant; for equal values of t, W was measured and found to be nearly constant; therefore, the only variable thought to be significant was ΔP, the motivating force.

The Building Research Advisory Board Report No. 14 to the Federal Housing Administration[3] has summarized the current professional thinking about the way in which water moves in concrete as follows:

> "The Committee (Advisory Committee of BRAB) believes that moisture is transferred through partially dry concrete in the absorbed or condensed state by surface diffusion and does not move as vapor through concrete. Also, where good practice has been followed in the design and curing of concrete, it is believed that moisture does not move through the concrete by capillarity as it is usually understood. The transfer process is considered to be the same where either liquid water or water vapor are present directly beneath a slab-on-ground. However, other conditions being similar, there is believed to be a much slower transfer to the absorbed state when vapor rather than liquid water is in contact with the slab. This results in a slower rate of moisture transfer through the slab where only vapor is present."
>
> Further, "Due to the hygroscopic nature of concrete it is believed incorrect to consider the rate of moisture transfer proportional to the vapor pressure differential between a moist and dry side of a slab. This is because moisture moves through a mature concrete in an absorbed or condensed state and not in a vapor phase. For water moving through concrete in a condensed state, the driving force is not computed from the difference between the two pressures but from the ratio between the two relative humidities."
>
> Moreover, "The Committee recommends against expressing results in terms of permeance as defined in ASTM Designation: E96-53T, Appendix 2. Computation of permeance is based on the assumption that moisture travels through partially dry concrete as vapor, whereas, under the conditions of these tests, it is transferred in the absorbed state by surface diffusion. Motive force is proportional to the logarithm of the ratio between the vapor pressure on the moist side and that on the dry side; it is not equal to the difference between the two vapor pressures as assumed in the ASTM definition."

According to Babbitt,[4] Fick's law states that the concentration gradient is the driving force for diffusion and, since concentration is independent of the physical state of the molecules, Fick's law does not distinguish absorbed moisture from moisture in the gaseous or liquid phases, and therefore is not applicable to hygroscopic materials such as concrete.

Babbitt[4] further points out that "...movement of moisture through porous or granular solids is a complicated phenomenon that cannot be solved completely by any single approach." Moreover, "...any satisfactory explanation of the movement of moisture not only must consider the physical state of the water molecules within the solid but also must include all possible modes by which the molecules might move." He distinguishes six possible states: vapor, liquid, ice, chemical compound, absorbed, and dissolved; and adds: "In each of these states, the water molecules, under certain conditions, are capable of movement, and all six states may contribute to the migration of the moisture." He then presents a form of the Stokes-Navier equation as a generalized equation of flow, since "...any movement of a fluid must obey an equation of transport of the form of the Stokes-Navier equation":

$$\frac{\delta P}{\delta x} - A_x = 0$$

In the absence of a numerical evaluation for concrete of the pressure gradient $\delta P/\delta x$, and the resistive force A_x, the following expression for water vapor transmission (WVT) was arbitrarily adopted for this study:

$$WVT = \frac{\ell W}{A t} \qquad (2)$$

where: ℓ = length of flow path (or thickness of specimen), inches

W = weight of vapor transmitted, grains (1 gram = 15.43 grains)

A = area of cross section of flow path, square inches (11.12 square inches for specimens reported herein)

t = time during which the vapor transmission occurred, days (100 days for this study)

WVT = water vapor transmission, inch grains per square inch per day

Equation 2 can be reduced to the following simple working form:

$$WVT = \ell WC \qquad (3)$$

where C = 0.13876, and ℓ and W are used in units of inches and grams, respectively.

The weight of water loss for each cup for a period of 100 days was interpolated from the straight-line portion of a graph like the typical one in Figure 7. Substituting values of W and ℓ from Table III into Equation 3, the WVT values were computed for each wet cup. All three cups (top, middle, and bottom slice) of the same design were averaged because there was no appreciable difference in their values (Table III).

It is interesting to note that the WVT values are almost equal for identical specimens in the different RH rooms. It must be remembered that all specimens had nearly equal dimensions; therefore, W is the only significant variable in Equation 3 for this study. Since measured values of W were almost equal for identical specimens, it is apparent that ambient relative humidity had no significant effect with reference to WVT. Therefore, ambient relative humidities used in this study seem to have little or no effect on the transmission of moisture through a concrete disk. This condition can be better understood by examining Figure 9. This figure shows that the upper two curves representing concrete without NaCl lie very close to each other, as do the lower two curves for concrete with 1.5 percent NaCl. The WVT increases with an increase in water-cement ratio (lower strength concrete).

It is most important to note that the WVT for concrete without NaCl is about twice that for concrete with 1.5 percent NaCl. The following is a summary of the effect of 1.5 percent NaCl in concrete with a high water-cement ratio:

1. The slump is increased from 3 inches to 7 inches.

2. NaCl whiskers develop on the wet cup specimens.

3. The compressive strength is reduced.

4. The WVT is reduced.

A faint growth of whiskers was first detected after the low-strength specimens containing NaCl were in the RH room for three days; the higher the concrete strength (the lower the water-cement ratio), the longer the time that was required for the initial growth of whiskers to begin. A discussion appears later in this report under the heading "Sodium Chloride Whisker Crystals."

Table III. Water Vapor Transmission - Phase I
(Wet Cups Washed With Helium)

Batch No.	Cup No.[a]	RH at 73.4 F, percent	W,[b] grams	ℓ,[c] in.	Water Vapor Transmission,[d] avg	Batch No.	Cup No.[a]	RH at 73.4 F, percent	W,[b] grams	ℓ,[c] in.	Water Vapor Transmission,[d] avg
1 L	N572T	50	25.6	1.566	-	1 L	N579T	20	29.6	1.595	-
1 L	N572M	50	25.1	1.487	0.537	1 L	N579M	20	25.8	1.517	0.617
1 L	N572B	50	26.4	1.468	-	1 L	N579B	20	31.4	1.499	-
1 LS	N610T	50	13.7	1.496	-	1 LS	N599T	20	13.4	1.467	-
1 LS	N610M	50	14.6	1.472	0.298	1 LS	N599M	20	15.0	1.439	0.280
1 LS	N610B	50	14.2	1.580	-	1 LS	N599B	20	12.5	1.549	-
1 M	N671T	50	16.6	1.511	-	1 M	N660T	20	15.7	1.466	-
1 M	N671M	50	17.2	1.468	0.356	1 M	N660M	20	18.0	1.493	0.362
1 M	N671B	50	17.5	1.524	-	1 M	N660B	20	18.9	1.502	-
1 MS	N683T	50	9.7	1.508	-	1 MS	N700T	20	8.2	1.502	-
1 MS	N683M	50	9.3	1.546	0.190	1 MS	N700M	20	8.4	1.470	0.172
1 MS	N683B	50	8.1	1.507	-	1 MS	N700B	20	8.2	1.518	-
1 H	N734T	50	9.6	1.504	-	1 H	N719T	20	11.9	1.526	-
1 H	N734M	50	11.7	1.514	0.226	1 H	N719M	20	12.4	1.456	0.259
1 H	N734B	50	11.4	1.470	-	1 H	N719B	20	13.1	1.507	-
1 HS	N759T	50	5.4	1.560	-	1 HS	N755T	20	6.4	1.536	-
1 HS	N759M	50	5.8	1.459	0.121	1 HS	N755M	20	7.1	1.526	0.133
1 HS	N759B	50	6.3	1.481	-	1 HS	N755B	20	5.5	1.479	-

a/ T, M, B equals U, M, L on Table II.
b/ Weight of water loss for 100 days on straight-line portion of graph.
c/ Length of flow path (or thickness of specimen).
d/ Inch-grains per square inch per day.

Note: Area of cross section of flow path = 11.12 square inches.

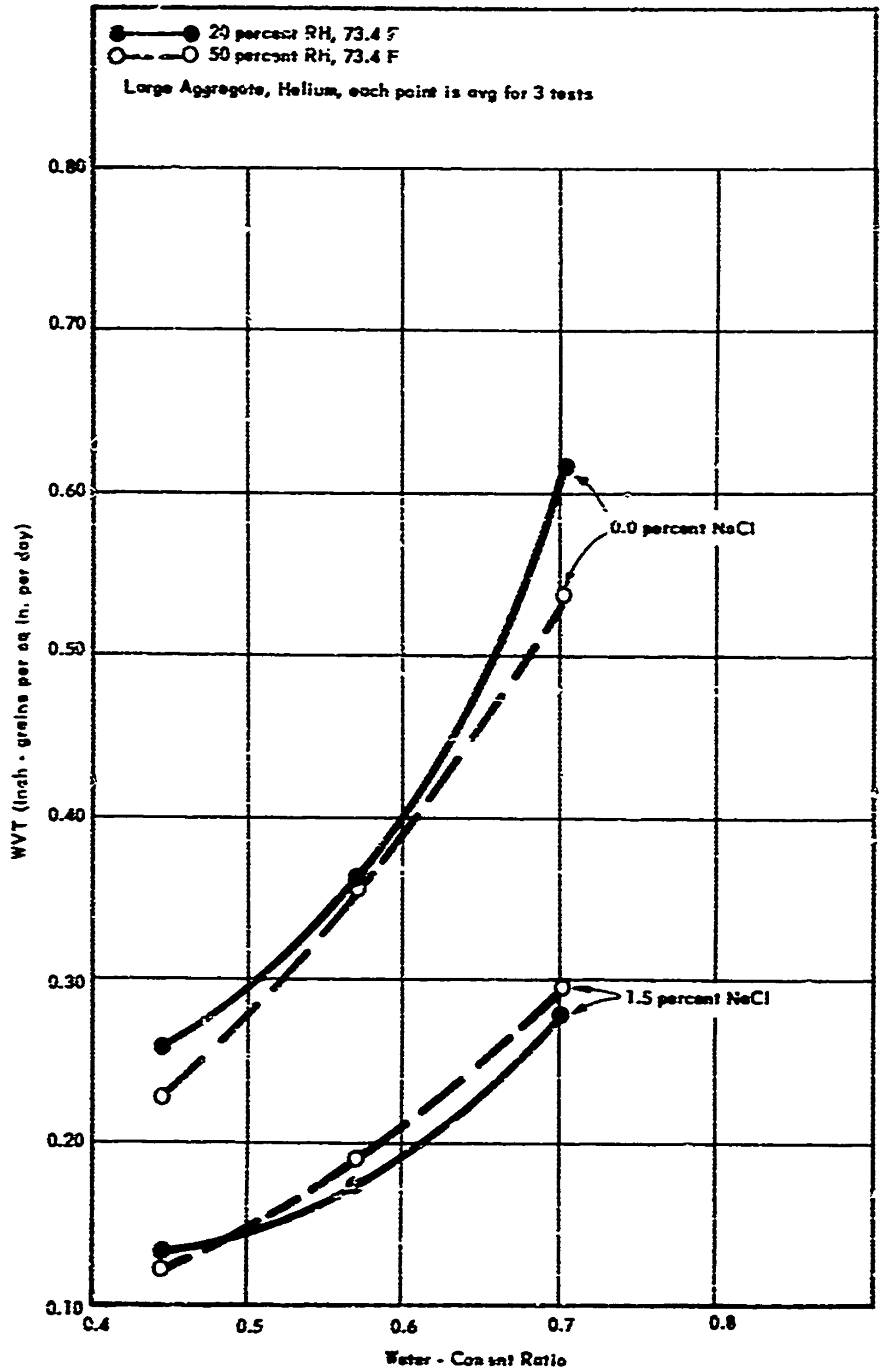

Figure 9. Water vapor transmission versus water-cement ratio - Phase I.

Phase II

Phase II was initiated to consider two additional factors. In connection with the Laboratory's "Hydrophobic Cement" Task No. Y-R007-05-026, oleic acid is used as an additive to make cement resistant to sack-hardening, particularly in tropical locations; therefore, it was decided to include oleic acid as a variable. In addition, the effect of maximum aggregate size was of interest. It was decided from the results of Phase I that the medium strength of concrete and the middle slice from the test cylinder were not necessary in the Phase II study. The variables involved in Phase II of this experiment were as follows:

1. Low (L) or high (H) strength of concrete.

2. 3/8-inch (S) or 3/4-inch (L) maximum aggregate size.

3. Absence (A) or presence (P) of NaCl.

4. Absence (A) or presence (P) of oleic acid.

5. Lower (L) or upper (U) slice from the test cylinder of concrete.

6. 20 percent (L) or 50 percent (M) RH at 73.4 F.

Thus there were two levels of each of six factors, making a total of 64 combinations in all. A quarter replicate experiment — that is, one having 16 combinations — was used as Phase II. This phase had more variables than Phase I but took less than half the number of wet cups, although it did not reveal the shapes of the trend curves as defined by three points. The design variables identified by batch number appear in Table IV.

The procedures for Phase II were the same as those for Phase I except that nitrogen gas was used in place of helium gas to avoid development of a vacuum. Oleic acid and dry cement were mixed in a pebble mill (without the pebbles) for 25,000 revolutions.

After a wet cup was assembled, it was placed in its designated RH room and periodic weight and manometer readings were taken as in Phase I. The mercury manometers revealed little or no vacuum. After six weeks it was decided that the manometers were of no further consequence and they were removed from all the wet cups.

The NaCl whiskers developed, as previously described, on all those specimens which contained 1.5 percent NaCl.

Table IV. Compressive Strengths, Standard Deviations, and Design Variables – Phase II
(Wet Cups Washed With Nitrogen)

Batch No.	No. of Specimens	Compressive Strength, psi a/ (avg)	Standard Deviation, psi	Strength	Aggregate	Sodium Chloride b/	Oleic Acid c/	Slice	Relative Humidity
S 1	28	3880	85	L	S	A	A	L	L
S 2	29	6640	450	H	L	A	P	U	L
S 3	29	5700	210	H	S	P	A	U	M
S 4	29	2640	40	L	L	P	P	L	M
S 5	29	4760	80	H	L	P	A	L	L
S 6	29	5750	195	H	S	A	P	L	M
S 7	28	3800	340	L	L	A	A	U	M
S 8	29	2870	115	L	S	P	P	U	L
S 9	29	7210	245	H	S	A	A	U	L
S 10	29	3150	160	L	L	A	P	L	L
S 11	29	3230	80	L	S	P	A	L	M
S 12	29	5200	150	H	L	P	P	U	M
S 13	29	6060	245	H	L	A	A	L	M
S 14	29	5630	210	H	S	P	P	L	L
S 15	29	3490	50	L	L	P	A	U	L
S 16	29	3540	105	L	S	A	P	U	M

a/ At age 14 days, cured in 100 percent RH at 73.4 F.
b/ 1.5 percent by weight of plastic concrete.
c/ 0.25 percent by weight of cement.

Note: L, H: Low or high strength.
L, U: Lower or upper slice of 4 by 8 cylinder (as cast).
S, L: Small (3/8-inch) or large (3/4-inch) maximum size aggregate.
A, P: Absence or presence of sodium chloride or oleic acid.
L, M: 20 percent or 50 percent RH at 73.4 F.

To ascertain if the cup assembly system was impervious to moisture, three types of control cups were assembled as follows:

1. In place of a concrete disk, an acrylic disk was cemented to the top of the cup, the cup half-filled with water, the air flushed out with helium, and the small access tubes clamped tight.

2. Same as (1), but flushed out with nitrogen.

3. A cup with a concrete disk was sealed across the top with a 1/4-inch layer of epoxy.

One of each of the above assemblies was placed in the 50-percent RH room and one each was placed in the 20-percent RH room. After six months there was no detectable weight loss, thus showing that the system was impervious to moisture.

Discussion of Phase II (Results)

The curves for small and large aggregate with and without NaCl were plotted from Table IV and appear as the top four curves in Figure 10. Here again, it is shown that the presence of 1.5 percent NaCl in concrete lowers the compressive strength. Figure 10 also relates the effect of aggregate size and strength. With and without NaCl, the concrete containing small aggregate had a higher compressive strength than the concrete containing large aggregate. (The designation of 3/4-inch aggregate as "large aggregate" is a relative term within this experiment.)

Figure 10 also contains curves for standard deviation of compressive strength versus water-cement ratio for concrete containing small as well as large aggregate with and without NaCl. Here again, the standard deviation for concrete with 1.5 percent NaCl is less than concrete without NaCl. For some unexplainable reason the spread of the curves is greater for large aggregate with and without NaCl than for small aggregate with and without NaCl. The NaCl seemed to have slightly less effect on the small than on the large aggregate concrete.

As in Phase I the weight of vapor loss for a period of 100 days was taken from the straight line portion of the weight-time graphs for each cup. Computed values of water vapor transmission appear in Table V.

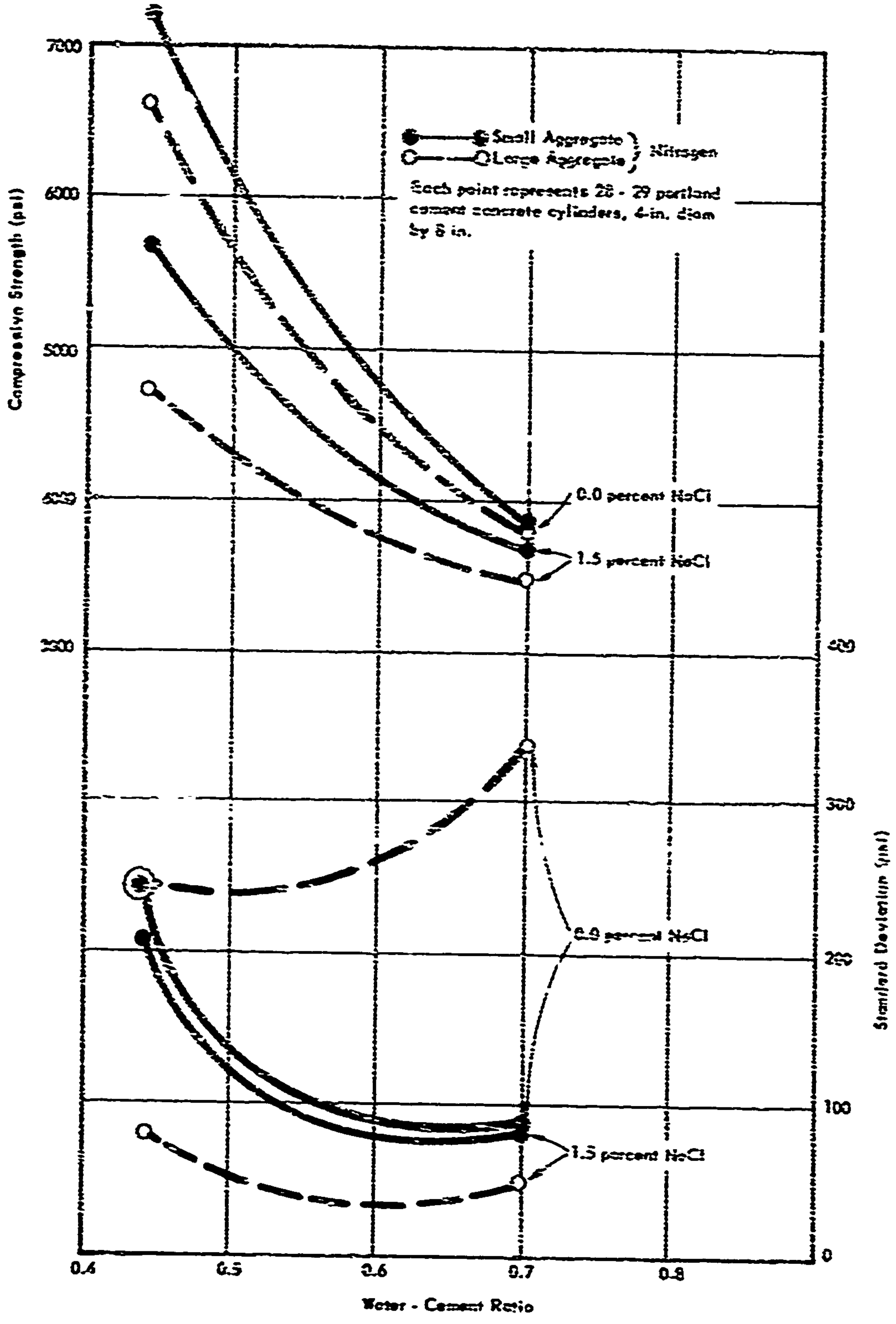

Figure 10. Compressive strength and its standard deviation versus water-cement ratio - Phase II.

Table V. Water Vapor Transmission - Phase II
(Wet Cups Washed With Nitrogen)

Batch No.	Cup No.[a]	RH at 73.4 F, percent	W,[b] grams	ℓ,[c] in.	WVT[d]
S 1	N872B	20	34.4	1.494	0.714
S 2	P124T	20	14.0	1.503	0.292
S 3	N990T	50	13.2	1.491	0.274
S 4	P190B	50	15.5	1.527	0.329
S 5	P66B	20	9.0	1.482	0.185
S 6	P248B	50	18.3	1.498	0.381
S 7	N852T	50	27.1	1.481	0.558
S 8	P385T	20	20.7	1.539	0.443
S 9	N947T	20	18.6	1.503	0.389
S 10	P214B	20	28.3	1.476	0.581
S 11	N919B	50	17.9	1.513	0.376
S 12	P147T	50	7.7	1.497	0.160
S 13	P35B	50	13.4	1.509	0.281
S 14	P296B	20	10.8	1.520	0.228
S 15	P17T	20	19.6	1.499	0.408
S 16	P344T	50	38.6	1.522	0.817

a/ T, M, B equals U, M, L on Table II.
b/ Weight of water loss for 100 days on straight-line portion of graph.
c/ Length of flow path (or thickness of specimen).
d/ ~~Inch-grains~~ per square inch per day.

Note: Area of cross section of flow path = 11.12 square inches.

These values of WVT were plotted with respect to water-cement ratio for small and large aggregate with and without NaCl (Figure 11). The shapes of these two-point curves were based on the three-point curves of Figure 10. Again the fact that the presence of 1.5 percent NaCl lowers the WVT value is demonstrated. The top two curves, representing concrete without NaCl, clearly show that the small aggregate concrete has a higher WVT value than the large aggregate concrete. Unfortunately, because of a condition referred to as a "random deviate" by statisticians, the curves representing small and large aggregate concrete with 1.5 percent NaCl intersect, and therefore do not allow any direct conclusion to be drawn, without benefit of analysis of variance. The "expected" rates of WVT, plotted on Figure 11, demonstrate the probable relationships. These two curves, identified by a triangular symbol, help to explain these variables in that they follow the same pattern as the other comparative curves. All the curves show that higher water-cement-ratio concrete has a much higher WVT value than low water-cement-ratio concrete.

It may be noted on Figure 11 that there is an apparent inconsistency in data sources for ambient RH, NaCl, and aggregate curves. Because of the quarter replicate chosen to be investigated, there was neither a specimen with small aggregate and without NaCl in the 50-percent RH room nor a specimen with large aggregate and without NaCl in the 20-percent RH room. The opposite situation existed for specimens with 1.5 percent NaCl. But since it has been concluded that RH has no significant effect on WVT, the curves can be compared on a sound basis.

The resulting values of WVT, as found in Table V, were subjected to an analysis of variance. Results of this analysis are presented in Appendix C. Water vapor transmission values were found to be significantly higher for higher water-cement ratios (lower concrete strengths), the absence of NaCl, and, to a lesser extent, the smaller aggregate size. Concrete slice position, oleic acid, and RH were found to have no significant effect.

Comparison of Phase I and Phase II

Phases I and II were similarly conducted except for the gas used to flush the inside of the wet cups and the kind and number of variables studied. The curves in Figure 12 represent WVT values from both phases plotted with respect to water-cement ratio for nitrogen and helium gases for concretes with and without NaCl. The top two curves, representing concrete without NaCl, show the effect of nitrogen relative to helium to give a higher WVT. The same relation holds true for concrete with 1.5 percent NaCl. This is shown further by the following example. By taking the WVT value for a wet cup containing helium from Phase I and adding to it the correction factor taken from Figure 12 (The ordinate value between two curves with similar variables), it was found that the new point falls on or very near the curve representing a wet cup with similar variables containing nitrogen from Phase II. The effect of different gases was not intended to be a variable in this experiment, but interesting developments have come from their use.

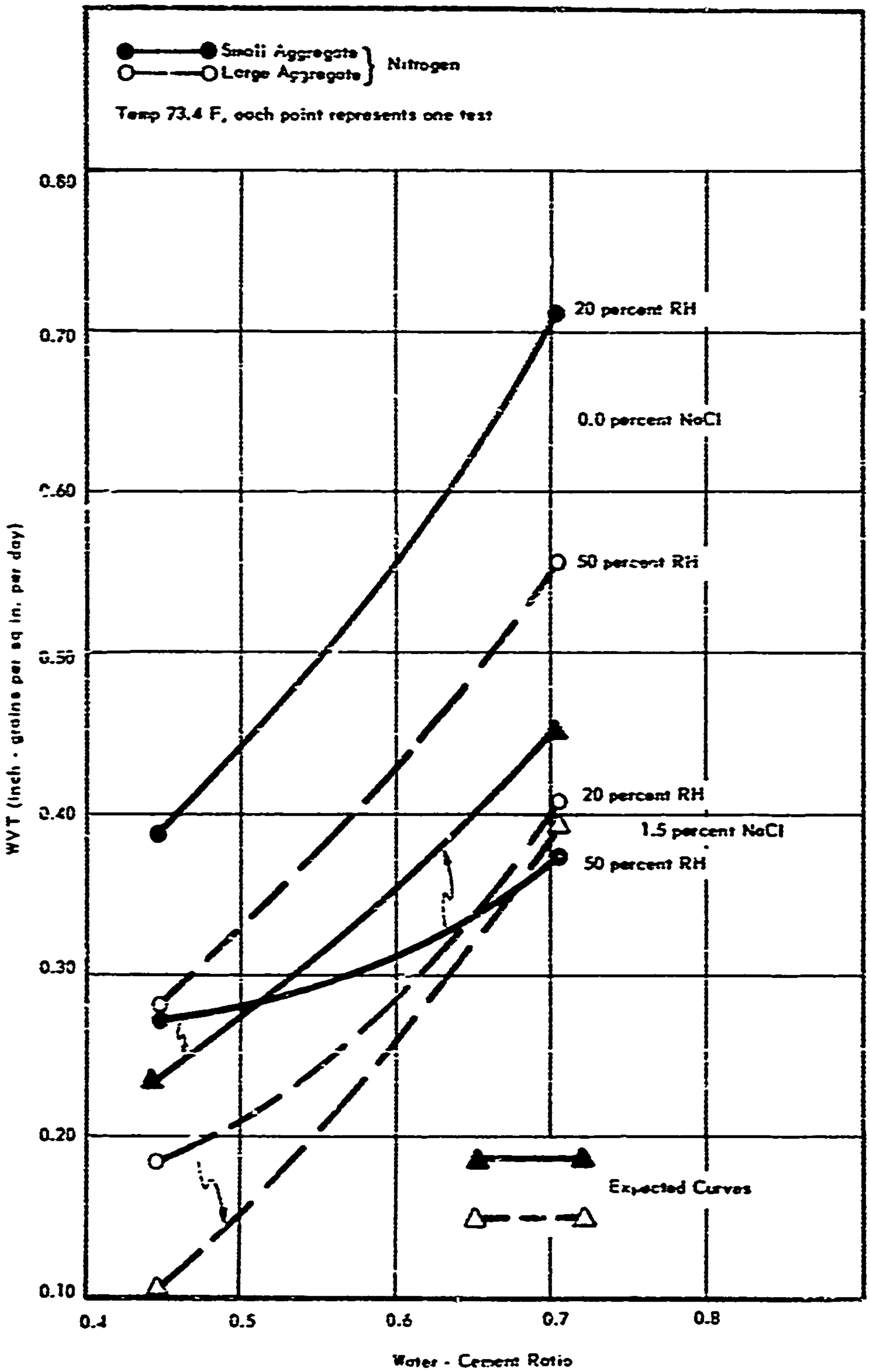

Figure 11. Water vapor transmission versus water-cement ratio - Aggregate size effect, Phase II.

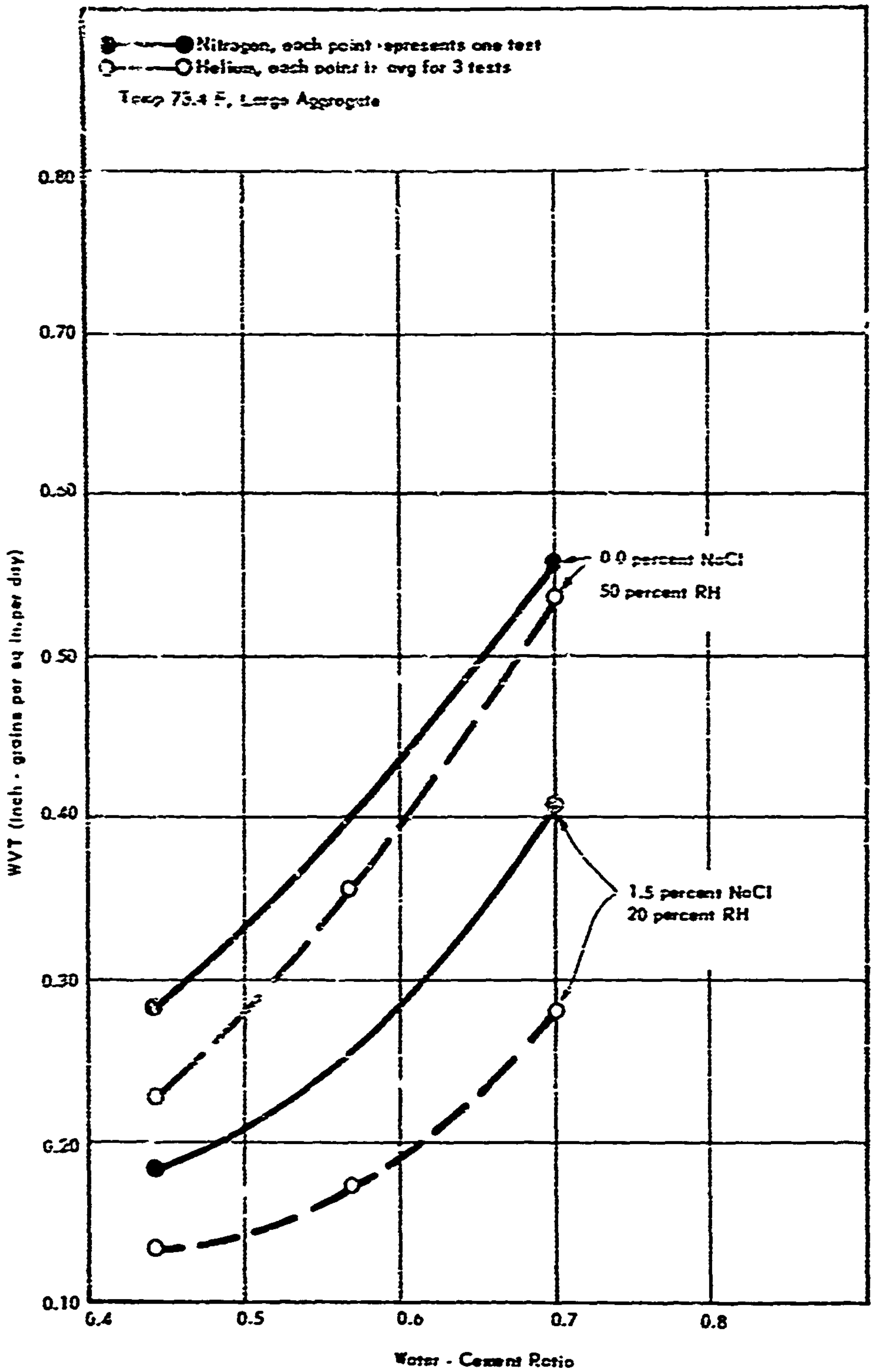

Figure 12. Water vapor transmission versus water-cement ratio - Gas effect.

Sodium Chloride Study

Apart from Phases I and II, but as an outgrowth of the effect of NaCl on the compressive strength of the concrete of those studies, an investigation was conducted varying the percent of NaCl in the concrete. Additional specimens were made following the same procedure as in Phase II but varying the percent NaCl between 0 and 1.5. The design variables, compressive strengths, and standard deviations appear in Table VI. Some specimens from Phases I and II are listed in Table VI for comparison and convenience.

A graph of compressive strength plotted with respect to percent NaCl (Table VI) appears as the top curve in Figure 13. Note that with an increase of NaCl from 0 to 0.6 percent there is an increase of compressive strength; with additional salt the compressive strength decreases to the limit of the experiment. The bottom curve, though inconclusive, represents the standard deviation of compressive strength (Table VI) also plotted with respect to percent NaCl. Both curves in Figure 13 are for low-strength concrete (water-cement ratio, 0.702). Table VII summarizes the NaCl study data and is included for informational and comparative purposes.

As small an amount of sodium chloride as 0.075 percent by weight of plastic concrete increased the slump from 3 to 6 inches. Increasing the amounts of salt up to 1.5 percent further increased the slump to 7 inches. The data in Figure 13 are for a constant water-cement ratio of 0.702. If the water-cement ratios were reduced to provide equal consistencies (slumps), the curve should show significantly higher compressive strength values.

Nothing was found in the literature to either verify or nullify the finding of an optimum salt content for maximum strength; however, Kleinlogel[5] reported that experiments made by the building department of the city of Berlin-Charlottenburg and by Dieckmann resulted in an increase in strength in most cases when sodium chloride was added to concrete. He further added that American publications reported a loss in strength of from 7 to 40 percent when 1 to 20 percent sodium chloride solutions were added to the concrete mix. He also made the following interesting observations: Sodium chloride may cause efflorescence;* it will cause reinforcing steel to rust; and concrete made with chloride absorbs much moisture from the air.

The results of the present study point out the need for further investigation about the effects of NaCl on concrete. At present a detailed investigation of salt-water concrete is being conducted under Task Number Y-R007-05-012.

* This may have been salt whisker crystals.

Table VI. Compressive Strengths, Standard Deviations, and Design Variables - Sodium Chloride Study (Wet Cups Washed With Nitrogen)

Batch No.	No. of Specimens	Compressive Strength, psi[a] (avg)	Standard Deviation, psi	Strength	Aggregate	Sodium Chloride[b]	Oleic Acid[c]	Slice	Relative Humidity
S 7	28	3800	340	L	L	0.00	A	U	M
1 L	28	4280	180	L	L	0.00	A	L, M, U	L, M
1 L 0.5 S	29	4050	95	L	L	0.15	A	M	L
1 L 1 S	29	4130	80	L	L	0.30	A	M	L
1 L 2 S	29	4160	125	L	L	0.60	A	M	L
1 L 3 S	29	3820	140	L	L	0.90	A	M	L
1 L 4 S	29	3860	60	L	L	1.20	A	M	L
1 L S	28	3380	95	L	L	1.50	A	L, M, U	L, M
1 L S (R)	29	3570	50	L	L	1.50	A	-	-
S 15	29	3490	50	L	L	1.50	A	U	L

a/ At age 14 days, cured in 100 percent RH at 73.4 F.
b/ Percent by weight of plastic concrete.
c/ 0.25 percent by weight of cement.

Note: L : Low strength.
L, M, U: Lower, middle, or upper slice of 4 by 8 cylinder (as cast).
L : Large (3/4-inch) maximum size aggregate.
A, P : Absence or presence of oleic acid.
L, M : 20 percent or 50 percent RH at 73.4 F.

Table VII. Water Vapor Transmission - Sodium Chloride Study
(Wet Cups Washed With Nitrogen)

Batch No.	Cup No.[a]	RH at 73.4 F, percent	W,[b] grams	ℓ,[c] in.	WVT[d]	Sodium Chloride percent[e]
S 7	N852T	50	27.1	1.481	0.558	0.00
1 L 0.5 S	P788M	20	29.2	1.550	0.628	0.15
1 L 1 S	P736M	20	24.6	1.524	0.520	0.30
1 L 2 S	P683M	20	24.1	1.509	0.505	0.60
1 L 3 S	P671M	20	17.1	1.526	0.362	0.90
1 L 4 S (R)	P859M	20	27.9	1.555	0.602	1.20
S 15	P17T	20	19.6	1.499	0.408	1.50

a/ T, M, B equals U, M, L on Table II.
b/ Weight of water loss measured for 100 days on straight-line portion of graph.
c/ Length of flow path (or thickness of specimen).
d/ Inch-grains per square inch per day.
e/ By weight of plastic concrete.

Note: Area of cross section of flow path = 11.12 square inches.

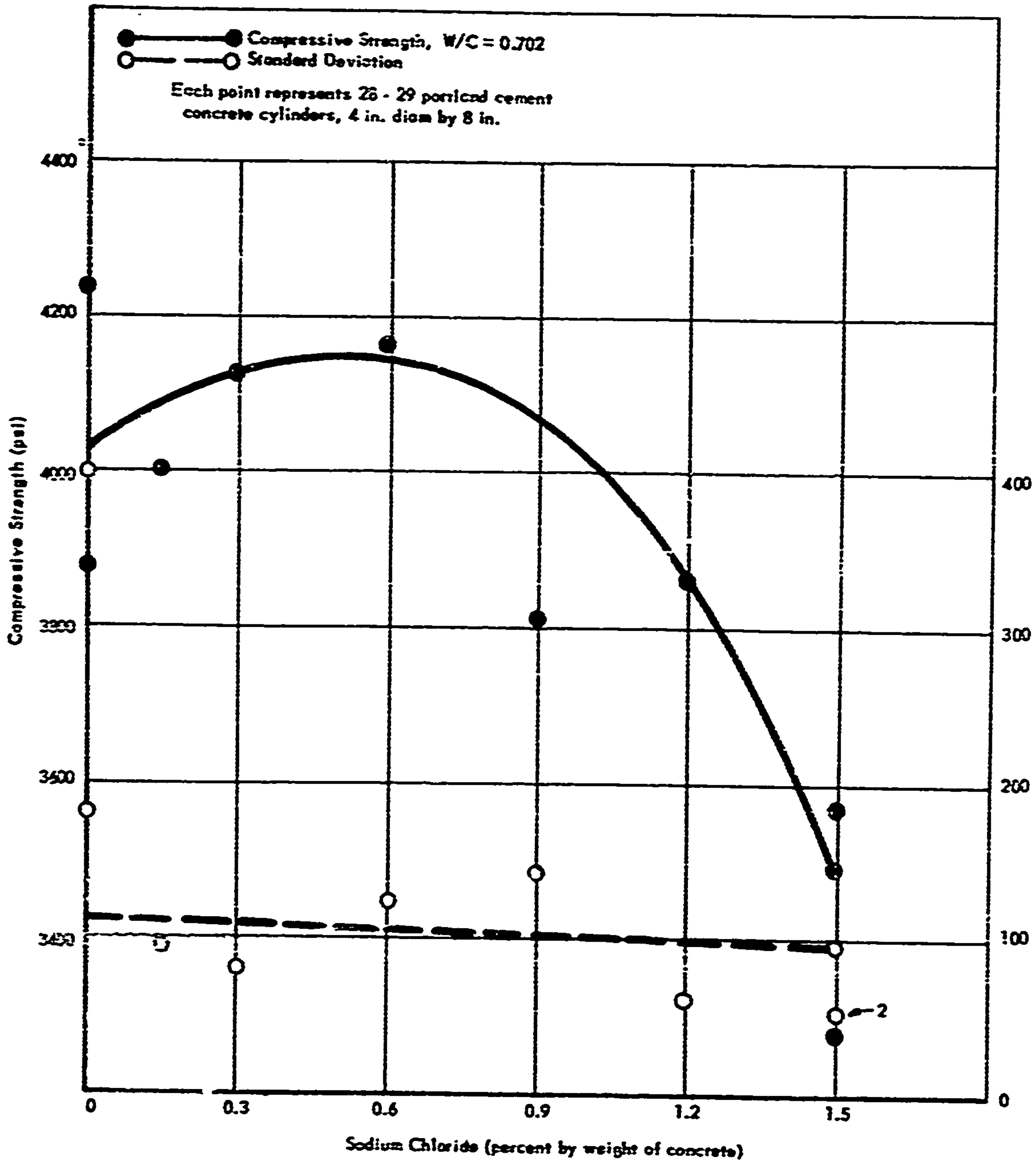

Figure 13. Compressive strength and its standard deviation versus sodium chloride content.

Sodium Chloride Whisker Crystals

Whisker crystal growth on portland cement concrete incorporating sodium chloride is believed to have been first reported in this study. Within three days after being placed in either of the test environments, the high water-cement ratio or low-strength concrete containing NaCl developed visible whiskers. The rate of growth appeared to be somewhat more rapid in 20 percent than in 50 percent RH; in general, however, the ultimate amount of growth over three or four months appeared to be about the same.

One of the authors (Griffin) observed the same type of growth in an air-conditioned building on Midway, where the external temperature ranged between 70 and 100 F and the relative humidity ranged between 100 and 50 percent. On 6 July 1960, in an air-conditioned room inside the building the temperature was 69 F and the RH was 51 percent. In a non air-conditioned room, where whiskers were also growing, the temperature was 80 F and the RH was 54.5. These whiskers emerged through a layer of paint and the surface of the walls had flaked off to a depth of 1/32 inch. The whiskers were fine enough to move about in the room on air currents and thus damage the large amount of electronic equipment that was housed in the building. It is not known whether the whiskers had the same ultimate deteriorating effect on this equipment as do metal whiskers that develop short circuits on microminiature electronic devices.

Many articles about whisker crystals have appeared in various journals and magazines in recent years. Reference 6, an authoritative and comprehensive single volume dealing with the growth and perfection of crystals, describes whiskers of many substances, including sodium chloride. The description of these whiskers corresponds very closely with observations made by the authors in this study.

On some of the Laboratory's specimens the whiskers wer like lamb's wool, on others they were stubby and coarse. Figure 14 shows the whisker growth on low-strength concrete after 5 months. Figure 15 shows growths magnified 15 times. Individual whiskers were observed to be as long as 4 centimeters and as small as 0.025 millimeter in diameter. Geometric configurations on a single concrete disk included spirals, helices, and noncurvilinear shapes. In addition to individual whiskers, dense mats of closely packed parallel whiskers emerged, usually separated from each other near the tips. Individual whiskers were striated longitudinally and had the appearance of having been extruded from a die as they continued to grow from just under the surface of the concrete. They definitely appeared to have grown from the base. In fact, if viewed at the proper time under a low-powered microscope, they may be observed breaking through a crust in the matrix that has been lifted above its original position by the formation of the whiskers.

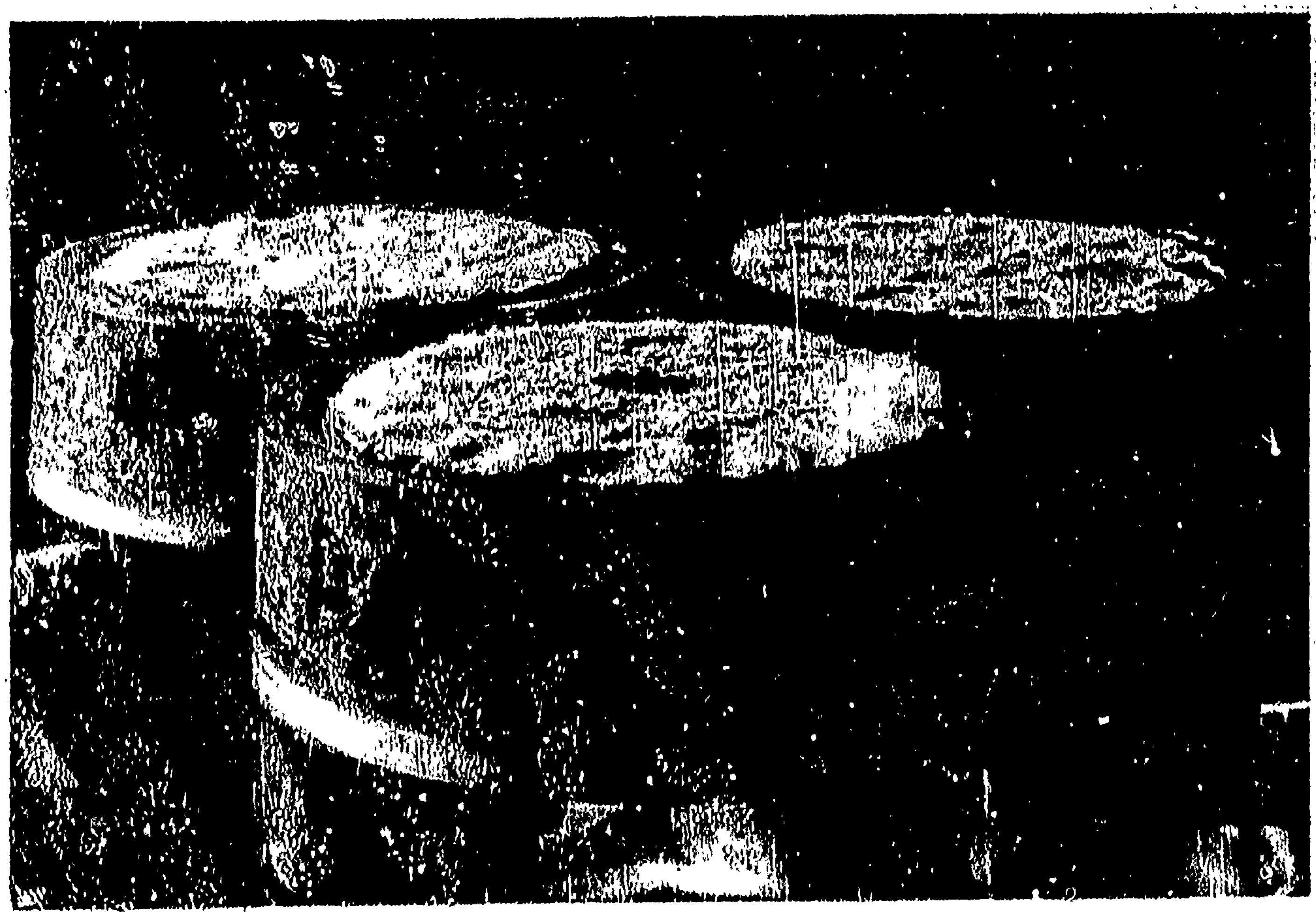

Figure 14. Salt whisker crystal growth on low strength concrete after exposure to 73.4 F, at 20 percent RH for 5 months.

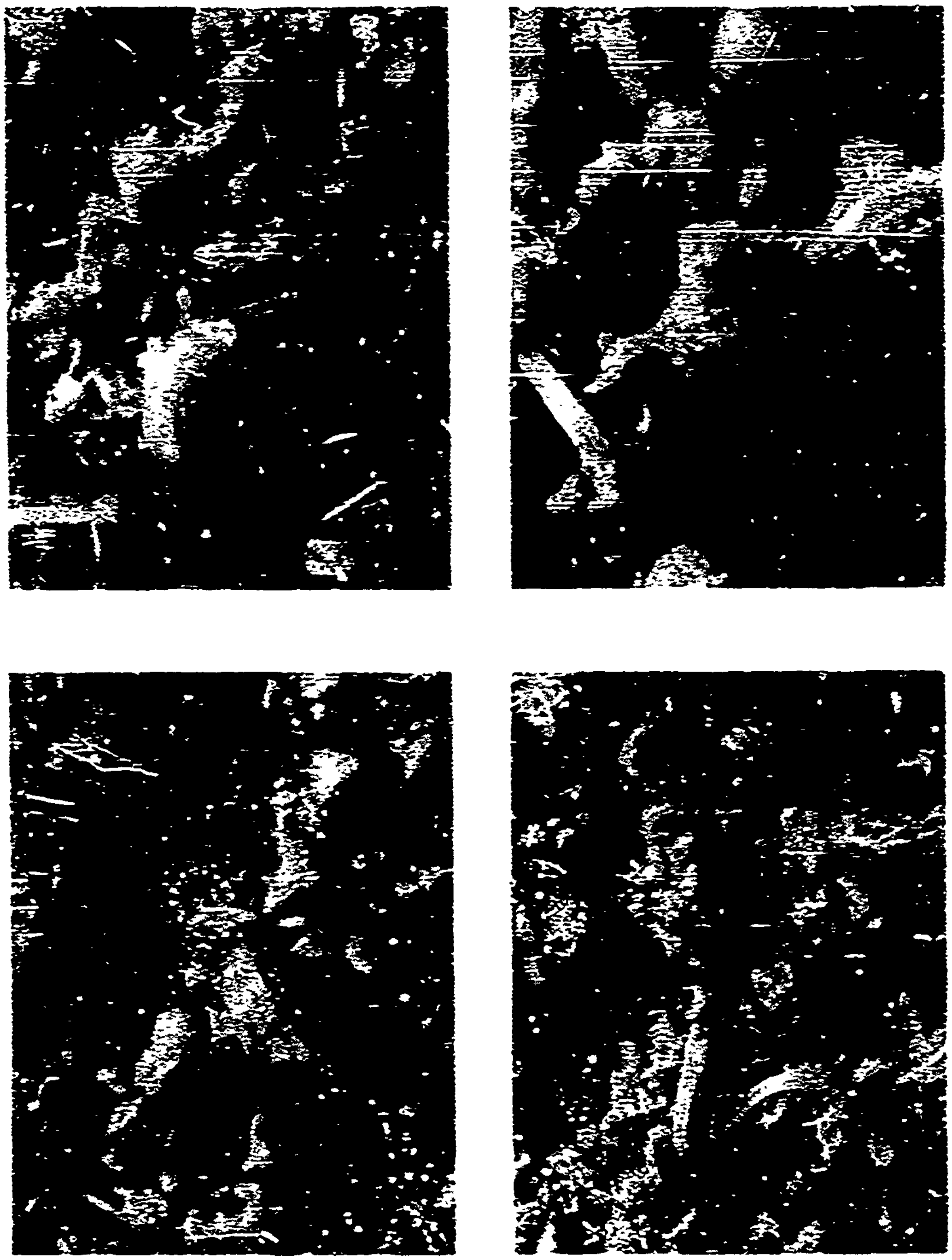

Figure 15. Examples of salt whisker growth (15x).

FINDINGS

Water Vapor Permeability

1. The form of Fick's law which is commonly used to determine water vapor permeability for concrete was not verified.

2. An equation for a WVT (water vapor transmission) quantity equal to $\frac{\Sigma W}{At}$ is more suitable for the observed phenomenon than Fick's law.

Strength of Concrete

1. There is an optimum concentration of NaCl for maximum compressive strength of concrete with a given high water-cement ratio.

2. Concrete with 1.5 percent NaCl has less strength than concrete without NaCl, other factors being equal.

3. Concrete containing small aggregate is stronger than concrete containing large aggregate for identical water-cement ratios.

Water Vapor Transmission

1. WVT increases with an increase in water-cement ratio.

2. WVT increases with a decrease in maximum particle size of aggregate.

3. WVT decreases with the presence of 1.5 percent NaCl in the concrete.

4. WVT decreases with the presence of helium inside wet cups.

5. WVT is independent of the location of segments in a concrete cylinder.

6. WVT is independent of the presence or absence of oleic acid.

7. WVT rate appears to increase slightly in a majority of cases as ambient RH decreases (partial vapor pressure increases), but for the range of the tests, 20 and 50 percent RH at 73.4 F, the trend is not significant when compared with the experimental variability.

General

1. Salt whisker crystal gr deteriorates the surface of the concrete where the whiskers form.

2. Acrylic cups, which are inert, proved to be impervious to vapor leak as indicated by the control cup tests.

3. The results of Phase II (quarter replicate) were verified by results of Phase I (full replicate).

CONCLUSIONS

1. Acrylic cups, as used in this investigation, are completely satisfactory for studying WVT.

2. A fractional factorial design for estimating WVT for several variables is not only reliable but also is economical.

3. A well-founded mathematical expression is yet to be established for predicting WVT for a given concrete. In the meantime, straight-line portions of the weight-loss-time curve provide a practical solution.

FUTURE PLANS

All the wet cups will continue to be weighed for an indefinite time, and additional information will be sought.

A Phase III study is planned to investigate the effect of water vapor transmission on steel embedded in concrete disks placed in 6-inch-diameter wet cups. This will provide at least 48 additional wet cups from which water vapor transmission and the effect of specimen size can be studied. It is also planned to study the electrical resistivity of concrete.

ACKNOWLEDGMENT

The authors gratefully acknowledge the contributions of Samuel H. Brooks, Sc. D., the Laboratory's statistical consultant on this work.

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Appendix A

PLASTIC WET CUP DESCRIPTION

The following is a detailed description of the plastic wet cup and component parts. Reference should be made to Figures 5 and 6 in the main text of the report.

The main body of the cup, which is cut from Cadco polished colorless and transparent cast-acrylic-resin tubing, is 5-1/2 inches long, with a 5-inch outside diameter and a 4-1/2-inch inside diameter. The tubing is machined smooth on both ends, cut with three circumferential gaging grooves on the inside near the top within the dimension of the thickness of the specimen, and drilled for the access tubes. The purpose of the grooves is to offer the maximum seal with the epoxy.

The top ring is cut and machined to size from 1/4-inch Plexiglas "G" sheets. The outside diameter is 5 inches and the inside diameter is 3-3/4 inches ±1/64 inch.

The annular ring with the counterbore is cut and machined from 3/8-inch Plexiglas to fit tightly inside the main tubing body. The outside diameter is approximately 4-1/2 inches and the inside diameter is 3-3/4 inches ±1/64 inch; the counterbore is 4 inches +1/16 inch in diameter and 3/16 inch deep. It is important that this ring fits tightly in order to have a perfect seal.

The bottom plate is cut and machined to size from 3/8-inch Plexiglas, with a diameter of 5 inches ±1/32 inch. A shoulder 1/8 inch deep and approximately 1/4 inch wide is machined to fit the bottom of each section of the main tubing.

The access tubes are cut 1-1/2 inches long from extruded acrylic tubing with a 3/8-inch outer diameter and a 1/4-inch inner diameter. The tubes and holes in the main body are tapered slightly to provide a tight fit.

Appendix B

WET CUP ASSEMBLY

The following is a detailed description of the step-by-step procedure for assembling the wet cup. Reference should be made to Figures 4, 5, and 6 in the main body of the text.

1. Measure the thickness of the concrete disk in four different places with a micrometer and record the average thickness.

2. Clean all plastic parts of cup with ethyl alcohol and allow to dry.

3. Fit counterbore of annular ring onto bottom of concrete disk.

4. Mix the desired quantity of epoxy in the proportion of four parts Shell Epon Resin 815 with one part Shell Curing Agent T-1 by weight and thicken to consistency of paste with a thickening agent, "Cab-O-Sil," and seal the ring to the disk by constructing a fillet of paste on the top surface of the ring adjacent to the concrete.

5. Place the main body of the cup (4-1/2-inch id tubing) over the inverted disk-ring component and press it down so that the concrete disk is flush with the top of the plastic main body in the inverted position.

6. Seal the disk-ring component in the main body with Cadco-94 General Purpose Acrylic Cement (consistency of water). At the same time, seal the access tubes in place. Allow to dry 15 minutes.

7. Mix the desired quantity of epoxy in the same proportions as in step 4 without thickener. Set the cup upright on a level surface and fill the annular space.

8. Allow the epoxy to set until hardened. This may take as much as five hours.

9. Seal the top ring onto the cup with a thin layer of epoxy. Place a pressure plate and weight (12-15 pounds) on top of the ring to hold it in position. Allow to set.

10. Seal the bottom plate to the cup with a thick acrylic cement. Place a pressure plate and weight (12-15 pounds) on top of the cup to hold it in place. Allow 30 to 45 minutes to set.

11. Attach a 2-inch length of Tygon flexible plastic tubing (Formulation R-3603; 3/8-inch id, 1/2-inch od) securely to the bottom access tube.

The cup assembly is now completed and the cup is ready to be filled with water, washed with gas, and to have the manometer attached to the upper access tube, if one is desired. If a manometer is not required the upper access tube is not needed.

Water vapor transmission values were found to be significantly higher for the high water-cement ratio, the absence of sodium chloride, and, to a lesser extent, the smaller aggregate size. Concrete slice position, oleic acid, and relative humidity were factors found to have no significant effects.

It is interesting to note that the small residual error indicates that much of the variability has been accounted for in the experimental procedures and in this analysis.

Implied in the above analysis is this linear statistical model for the "expected" rate of the water vapor transmission:

$$\text{"Expected" rate} = 0.401 - 0.127A + 0.017B - 0.052C - 0.101D + 0.003E - 0.004F$$

The following table shows the experimental design, the resulting observations of the rates of water vapor transmission for the indicated combination of factors, the "expected" rate as computed above, and a check column to show that:

$$\Sigma\ (\text{observed} - \text{"expected"}) = 0$$

Table IX. Experimental Design and Resulting Observed and "Expected" Rates of Water Vapor Transmission

Factor Combination	A	B	C	D	E	F	Observed Rate[a/]	"Expected" Rate[a/]	(Observed- "Expected")
S 1	-1	-1	-1	-1	-1	-1	0.714	0.665	0.049
S 2	+1	+1	+1	-1	+1	-1	0.292	0.347	-0.055
S 3	+1	+1	-1	+1	-1	+1	0.274	0.235	0.039
S 4	-1	-1	+1	+1	+1	+1	0.329	0.357	-0.028
S 5	+1	-1	+1	+1	-1	-1	0.185	0.105	0.080
S 6	+1	-1	-1	-1	+1	+1	0.381	0.409	-0.028
S 7	-1	+1	+1	-1	-1	+1	0.558	0.587	-0.029
S 8	-1	+1	-1	+1	+1	-1	0.443	0.503	-0.060
S 9	+1	+1	-1	-1	-1	-1	0.389	0.445	-0.056
S 10	-1	-1	+1	-1	+1	-1	0.581	0.567	0.014
S 11	-1	-1	-1	+1	-1	+1	0.376	0.455	-0.079
S 12	+1	+1	+1	+1	+1	+1	0.160	0.137	0.023
S 13	+1	-1	+1	-1	-1	+1	0.281	0.299	-0.018
S 14	+1	-1	-1	+1	+1	-1	0.228	0.215	0.013
S 15	-1	+1	+1	+1	-1	-1	0.408	0.393	0.015
S 16	-1	+1	-1	-1	+1	+1	0.817	0.697	0.120
								Σ (observed - "expected") =	0.000

a/ Inch-grains per square inch per day.

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