

REPORT #74-014-7732

PROJECT 7732

---

ANTI-SCALING AGENTS FOR CONCRETE

INTERIM REPORT #2

by

Richard M. Weed  
Principal Engineer, Transportation Research

July 8, 1974

## ABSTRACT

This report describes the progress made to date on a project designed to (1) screen a large number of candidate anti-scaling agents, (2) field test those found most promising, and (3) investigate the scaling mechanism by means of laboratory freeze-thaw testing. The first phase is essentially complete and four out of a total of 41 treatments have been judged to be significantly beneficial on air-entrained concrete. The second phase was begun ahead of schedule with the field testing of two materials which were reported to be beneficial by other states. Future plans for this part of the program are discussed. The third phase has been plagued with many operational difficulties and no significant results have been obtained. Perhaps the most valuable outcome of the study has been the conclusion that adequate air-entrainment appears to be more beneficial than any of the treatments which were tested. As a result of this indication, an experiment was run to determine how much the specified air level could be increased without risking too great a loss of strength. As a result of this study, it was recommended that the air-entrainment specification be increased from  $4.5 \pm 1.5$  percent to  $6.0 \pm 1.5$  percent. This has been approved and is now in effect. This report also contains a section explaining how statistical principles can be applied to subjective rating data to determine the level at which a rating can be judged to be significantly beneficial or detrimental.

## PROJECT 7732

ANTI-SCALING AGENTS FOR CONCRETE

## Interim Report #2

This report should be read in conjunction with the first report dated September, 1971. Although the scope of this project and certain pertinent details will be reviewed in the present report, much will be omitted that was covered in detail in the first report.

1.0 REVIEW OF SCOPE OF PROJECT

A study of New Jersey bridges has indicated that scaling affects approximately one-third of the concrete decks. In order to explore the possibilities for remedying this demonstrated scaling problem, a long-range research project was undertaken.

The work of this project is being conducted in the following three phases:

Phase I

Test a relatively large number of anti-scaling agents by means of accelerated outdoor exposure tests in order to isolate a few that are promising.

Phase II

Field test on actual bridges the most promising materials found during Phase I.

Phase III

Continue testing with the aid of a laboratory freeze-thaw chamber to attempt to learn more about the actual mechanism of scaling.

Phase I was begun in the summer of 1970 with the casting of nearly 200 test slabs and the application of 41 treatments of various types. This testing has continued throughout the last three winters with water and de-icer applications timed to take advantage of the natural freeze-thaw cycles.

Phase II was begun ahead of schedule with the application of ~~two curing/sealer materials found promising by researchers in other states.~~ These treatments (designated C-1 and C-2) are based on linseed oil and a chlorinated rubber epoxy, respectively.

Phase III testing with the laboratory freeze-thaw chamber was just being initiated at the time of the first report. Numerous problems have plagued this part of the program and the results continue to be disappointing. This will be discussed in greater detail later in this report.

## 2.0 OUTDOOR EXPOSURE TESTING

The outdoor exposure test slabs are inspected each spring by a team of raters. The results are statistically analyzed to determine which treatments can be judged significantly beneficial. The most promising of these treatments will then be field tested on actual bridge decks.

### 2.1 RATING SYSTEM

The rating system shown below, which has been employed by several other investigators, was used to obtain a numerical deterioration rating for each test and control slab in the outdoor exposure phase of the study.

<u>Condition (Scaling Rating)</u>	<u>Surface Appearance (Severity)</u>
0	No scaling
1	Very slight scaling, 1/16" deep or less, virtually no coarse aggregate visible, includes laitance scaling.
2	Slight to moderate scaling, between Condition 1 and Condition 3.
3	Moderate scaling, approximately 1/8" to 1/4" deep, <del>some coarse aggregate visible.</del>
4	Moderate to severe scaling, between Condition 3 and Condition 5.
5	Severe scaling, approximately 1/2" or deeper, coarse aggregate completely exposed.

To determine a rating for any particular slab, a rater is required to estimate the percentage of the area of the slab displaying each level of severity. In actual practice, many slabs exhibited only one level of severity while very few had as many as three. For example, a slab which has 30 percent of its area in Condition 3, 20 percent in Condition 1, and 50 percent with no scaling, would be rated as follows:

<u>Extent</u>		<u>Severity</u>	=	
.30	x	3	=	.90
.20	x	1	=	.20
.50	x	0	=	<u>0</u>
		Total		1.10 = Rating

The Phase I test slabs have been evaluated at the end of each of the last three winters. Each slab was rated independently by seven engineers, and the results averaged to obtain a scaling rating for each slab. The net effectiveness of each treatment is estimated by the difference in scaling ratings between each test slab and its control. This is termed the "durability rating" and is negative when an apparently effective treatment prevents a test slab from deteriorating as rapidly as its control.

## 2.2 EVALUATION OF DURABILITY RATINGS

To properly evaluate the results, it must be determined at what level the ratings may be interpreted to indicate a significantly beneficial or detrimental effect. The theory is very basic. Every measurement has associated with it a certain amount of variability. This variability is made up of components of variance from different sources. In this case, the primary components are the variability associated with the rating system and the variability inherent in the performance of the treatments themselves. The combination of these two will be used to determine what magnitude a durability rating must be in order for it to be attributed to something other than chance effect (i.e., the magnitude necessary to demonstrate a real beneficial or detrimental effect of the treatment).

The derivation of these critical levels is described in Appendix III. It was found that a single test must achieve a durability rating of at least + 1.18 to be judged significant whereas a durability rating of only + 0.83 is required when the average of two tests is available.

### 2.3 RESULTS OF OUTDOOR EXPOSURE TESTING

There are certain trends apparent in the data in Appendix I which should be noted and discussed. In many cases, the measured effect, favorable or unfavorable, is seen to increase as the number of freeze-thaw cycles increases. This is probably the normal case and is to be expected. In some cases, however, the trend seems to reverse itself while, in others, there is no obvious pattern. This could be the result of the variability of the rating system but may also indicate a change in the rate at which particular test slabs (or their controls) are weathering. It should also be realized that, in an accelerated test of this type, the trend for all durability ratings (T-C) is to tend toward zero as the freeze-thaw cycles increase indefinitely. This is so because both the test slab and its control eventually will be completely deteriorated, resulting in a difference of zero. For this reason, it was decided to terminate any individual test when either the test or the control reached the maximum deterioration rating of 5.00.

The results obtained on air-entrained (AE) concrete and non-air-entrained (NAE) concrete have been listed separately in Appendix I for two reasons. First, since most exposed concrete nowadays is specified to be air-entrained, it was felt that the test results would be more useful if this factor were isolated. Second, there may be certain treatments which have different effects on the two types of concrete and their results would be obscured if they were averaged. In this study, slabs designated as AE had 3-6 percent air as measured by the pressure method. Those designated NAE received no air-entraining agent and usually contained approximately one percent entrapped air.

Table I lists the average durability ratings for the four general classes of materials tested. No attempt was made to analyze these results statistically but, because each rating in this table is the average for several test slabs, they can be assumed to be significant. In this case, all general classes appear to be beneficial as groups except for sealers and curing compounds on non-air-entrained concrete.

A slight problem was encountered in categorizing certain of the materials since the manufacturers sometimes referred to them as "penetrating-sealers" or "curing-sealers" which would constitute a cross between two of our classifications. Our general approach was to call any treatment (a) a curing compound if it was used as a curing compound, (i.e., if used on wet concrete) (b) a penetrating agent if it appeared to soak into the concrete, and (c) a sealer if it appeared to remain on the surface of the concrete. The remaining category, admixtures, was an obvious classification.

TABLE I

AVERAGE DURABILITY RATINGS FOR GENERAL CLASSES OF TREATMENTS

(Negative Values Indicate Beneficial Results)

<u>Type</u>	<u>Air-Entrained Concrete</u>	<u>Non-Air-Entrained Concrete</u>
Sealers (S)	-0.74	POOR*
Penetrating Agents (PA)	-0.45	-0.28
Curing Compounds (C)	-0.34	+0.68
Admixtures (A)	-0.24	NOT TESTED**

\*Some test slabs reached total failure prior to the last rating and, therefore, were excluded. Had they been included, they would have contributed positive values which reflect poor performance.

\*\*All admixtures tested caused the concrete to be air-entrained.

Perhaps the most interesting thing to note in Table I is the performance of the sealers and the curing compounds. A curing compound is, by necessity, a sealer which is applied immediately after the concrete is poured whereas the sealers in this study were applied about one month later. The average effects for both these classes of treatments were very similar in that good results were obtained with air-entrained concrete and poor results were obtained with non-air-entrained concrete. It should be re-emphasized that these effects are comparisons (T-C) and are isolated from any effects attributable to air-entrainment itself. Based on this study, the sealers and curing compounds appear to improve the scaling resistance of air-entrained concrete but reduce the scaling resistance of non-air-entrained concrete.

A possible explanation can be offered for this. It has long been recognized that air-entrainment is advantageous because it provides room for expansion as the free water in the concrete freezes. It remains effective as long as the air voids are not filled with water. Both the curing compounds and the sealers were applied before the water and de-icer applications were begun. Therefore, these treatments tended to keep the water out of the void systems and maintained their effectiveness. The relatively better performance of the sealers over the curing compounds may be the result of additional drying time before the sealers were applied, thereby reducing the free water content of these slabs still further.

In the case of the non-air-entrained slabs, there are insufficient air voids in the concrete to accommodate the increasing volume of water as it freezes. These slabs have very little frost resistance in any case but, what resistance they have can be improved by a period of drying which would tend to empty whatever air voids are present. Since the slabs were cast during the summer, those that did not receive curing or sealing compounds did experience a substantial period of drying. Those that received curing and sealing compounds would have remained fairly saturated which may account for their poorer performance.

In Table II-A, the treatments are listed in the order of effectiveness on air-entrained concrete and several interesting observations may be made. The two treatments, C-1 and C-2, which were field tested as a result of favorable reports by other researchers are seen to rank 16th and 12th, respectively, out of a total of 41 treatments. Although their average durability ratings of  $-.46$  and  $-.74$  are favorable (negative), neither achieved the level of  $-.83$  considered necessary to be confident that they are beneficial. However, as discussed in Appendix III, it is worthwhile to check the individual ratings in Appendix I to see if the variability of treatment effectiveness ( $\sigma_{TE}$ ) is more or less than average. Although the two values for each test do not permit the calculation of a reliable standard deviation, they do give an indication of the variability of the treatment in question. In this case, the individual ratings are  $-.40$  and  $-.51$  for treatment C-1 and  $-.86$  and  $-.61$  for treatment C-2. These are more

TABLE II-A  
AIR-ENTRAINED CONCRETE

ORDER OF EFFECTIVENESS OF TREATMENTS AFTER THREE YEARS EXPOSURE

Rank	Test Designation	Durability Rating	Significant at 95% Level	Rank	Test Designation	Durability Rating	Significant at 95% Level
1	A-5	-1.51	YES*	21	PA-4	-.32	NO*
2	S-6	-1.34	YES	22	C-0	-.29	NO
3	S-4	-1.30	YES	23	A-4b	-.20	NO*
4	PA-12a	-1.13	NO	24	C-4	-.20	NO*
5	S-5	-1.11	NO	25	PA-3	-.16	NO*
6	PA-13a	-1.06	NO	26	PA-12b	-.14	NO
7	PA-8	-.99	NO	27	C-8	-.13	NO*
8	PA-14	-.89	NO	28	PA-17	-.12	NO
9	C-5	-.86	YES*	29	PA-18	-.09	NO
10	S-1	-.85	NO	30	PA-5 on C-0	-.09	NO
11	PA-15	-.80	NO*	31	C-6	-.08	NO*
12	C-2	-.74	NO*	32	PA-10	-.06	NO*
13	PA-7	-.60	NO*	33	A-4a	-.06	NO*
14	PA-5	-.56	NO*	34	C-7	+.10	NO*
15	PA-1	-.50	NO*	35	PA-2	+.10	NO*
16	C-1	-.46	NO*	36	PA-13b	+.15	NO
17	PA-6	-.42	NO*	37	S-2	+.20	NO*
18	S-8	-.40	NO	38	A-3	+.22	NO*
19	C-3	-.38	NO*	39	A-1	+.35	NO*
20	S-3	-.38	NO	40	PA-4 on C-0	+1.06	NO
				41	LO-MS on C-0	+2.03	YES

\*Average of two tests in which case a durability rating of  $\pm 0.83$  or more is considered significant at the 95 percent level of confidence. Others are single tests for which a durability rating of  $\pm 1.18$  or more demonstrates significance.

TABLE II-B  
NON-AIR-ENTRAINED CONCRETE

ORDER OF EFFECTIVENESS OF TREATMENTS AFTER THREE YEARS EXPOSURE

Rank	Test Designation	Durability Rating	Significant at 95% Level	Rank	Test Designation	Durability Rating	Significant at 95% Level
1	S-1	-2.03	YES	16	S-2	0	NO*
2	PA-13a	-1.23	YES	17	PA-12b	+0.03	NO
3	PA-12a	-.99	NO	18	C-1	+0.13	NO
4	PA-6	-.96	YES*	19	C-1	+0.13	NO
5	C-5	-.70	NO*	20	S-8	+0.14	NO*
6	C-3	-.64	NO	21	PA-7	+0.62	NO*
7	PA-10	-.62	NO*	22	PA-14	+0.67	NO
8	PA-18	-.57	NO	23	C-7	+0.76	NO*
9	PA-8	-.52	NO	24	PA-1	+0.80	NO
10	PA-15	-.50	NO*	25	C-8	+0.88	YES*
11	PA-2	-.37	NO*	26	C-6	+1.10	YES*
12	PA-13b	-.34	NO	27	C-2	+1.27	YES*
13	PA-4	-.33	NO*	28	C-4	+2.65	YES
14	PA-5	-.30	NO*	29	S-3	POOR	YES**
15	PA-3	-.25	NO*	30	S-4	POOR	YES**

\*Average of two tests in which case a durability rating of  $\pm 0.83$  or more is considered significant at the 95 percent level of confidence. Others are single tests for which a durability rating of  $\pm 1.18$  or more demonstrates significance.

\*\*Some test slabs reached total failure prior to the last rating and, therefore, were excluded. Had they been included, they would have received high positive ratings.

consistent than most of the treatment pairs which indicates that the acceptance requirement of  $\pm .83$  may be too high for these two treatments. Although there is not enough data to estimate  $\sigma_{TE}$  (variability of treatment effectiveness) for individual treatments, it is at least possible to determine lower limits for the statistical acceptance criteria by assuming that  $\sigma_{TE} = 0$ . In this case, the only remaining component of variability is that due to the rating system ( $\sigma_{RS} = 0.27$ ). The 95 percent significance level is then  $\pm 1.96 \sigma_{RS} / \sqrt{2} = \pm 0.37$  for an average of two tests. The average rating for treatment C-1 (-.46) is only slightly larger than -.37 and, therefore, its significance is doubtful. The average for treatment C-2 (-.74) is considerably larger than -.37 and it is possible that it could be significantly beneficial. The field installations of these two treatments will continue to be observed periodically in a further attempt to evaluate their performance.

As described in the first report, treatment C-1 is a linseed oil emulsion and treatment C-2 is a chlorinated rubber epoxy, both of which are used as curing compounds. Based on past experience with treatment C-2, one contractor has requested permission to use it again on a project during the 1974 construction season. Its field performance to date on the test bridge appears satisfactory and it is believed that this request will be approved. It is expected that the material will be used for approximately half of the project, thereby providing both test and control sections.

Another interesting observation in Table II-A is that all four general types of treatments have individual ratings near the top of the list, although none of the penetrating agents quite achieved the level judged to indicate significance. The tests for two of the penetrating agents (PA-12, soybean oil, and PA-13, castor oil) were split to include both cut (combined with an equal amount of mineral spirits) and uncut (pure) versions of these treatments. For this reason, no replicate tests were available from which to judge within-treatment variability. However, it is interesting to note that the cut versions appear near the top of the list for both air-entrained and non-air-entrained concrete while the uncut versions appear substantially farther down in both cases. It seems unlikely that this could happen due only to chance factors and it is assumed to indicate that the effectiveness of these treatments is improved by the addition of mineral spirits. It is believed that the thinner (cut) material penetrates deeper and remains for a longer time in the surface layer of the concrete.

Of the four treatments that rank as significantly beneficial on air-entrained concrete in Table II-A, the one that tops the list (treatment A-5, a water-reducing admixture) has several very desirable features. First, since it is an admixture, it is simpler to use than the other three types of treatments, requiring only that the correct amount be added to the mix water during batching. Second, since it is not a surface coating, there is no surface slipperiness problem that is a concern with some of the other treatments. Third, although the two test slabs are not as large a sample as we would like, the durability ratings for this

treatment appear to be remarkably consistent (-1.46 and -1.56). Fourth, auxiliary benefits to be expected from this treatment are an increase in strength and a decrease in permeability due to a lower water-cement ratio. Because this treatment appears to be beneficial in every respect with apparently no undesirable characteristics, it will receive the major emphasis for future field testing.

~~The next two ranking beneficial treatments in Table II-A (S-6, an epoxy resin, and S-4, a urethane) have certain undesirable features and will not be pursued further at this time. Both are somewhat difficult chemicals to work with and both require a sand topping to prevent a slipperiness problem. Although the sand topping appeared adequate by our test method (British Portable Tester), it is not known how it would hold up under traffic.~~

The remaining treatment which passed the test of being significantly beneficial on air-entrained concrete is treatment C-5, a two component epoxy curing compound which is also comparatively difficult to use. A sand topping was not used and an undesirably low skid value was obtained (38 whereas 45 or below is judged potentially slippery). A further drawback was its failure to pass the moisture retention test for curing compounds. For these reasons, this treatment will not receive further consideration.

### 3.0 AIR-ENTRAINMENT STUDY

Although it was not specifically planned to confirm the beneficial effect of air-entrainment, this was easily accomplished by comparing the air-entrained and non-air-entrained control slabs. In this case, the

scaling ratings (C) must be used since durability ratings (T-C) cannot be calculated for the control slabs. Since there were 26 control slabs each of air-entrained and non-air-entrained concrete, a reasonably good estimate of the overall standard deviation ( $\sigma_{AM}$ ) can be made for both types of concrete. The individual scaling ratings for the control slabs are listed in Appendix II and the histograms plotted from these data showed both distributions to be nearly normal with standard deviations of 0.45 and 0.91, respectively, for AE and NAE concrete. Since the standard deviations are reasonably well determined and quite different, it was decided not to pool them but, instead, to use the "t" test of means assuming known standard deviations. With mean scaling ratings of 1.18 and 2.40, respectively, for AE and NAE concrete, the "t" statistic is calculated to be 6.13. This far exceeds the one-tailed table value of  $z = 2.33$  for a confidence level of 99 percent, indicating a highly significant benefit derived from an average air-entrainment level of 4.5 percent.

Because of this very beneficial effect of air-entrainment, plus the fact that most states specified levels of air-entrainment higher than ours, it was decided to investigate the feasibility of increasing the current specification of  $4.5 \pm 1.5$  percent to some higher level. Since it was recognized that additional air-entrainment would be accompanied by an attendant loss of strength, it was necessary to determine to what extent the air level could be increased without seriously jeopardizing the structural strength of the concrete.

It was decided to cast a series of test cylinders with varying amounts of entrained air in order to determine the loss of strength to be expected. Several laboratory batches of structural grade concrete

were prepared with a cement factor of 6.7 sacks/c.y. and the water-cement ratio controlled as closely as possible at 5.25 gallons/sack. The air content of the fresh concrete was measured by the pressure method and was varied from 3 percent to 12 percent. This resulted in a total of 67 cylinders to be used for the analysis.

Because these batches were carefully controlled and the cylinders were cured in an optimum manner, the resulting strengths represent the potential of field concrete under the best conditions. Strengths achieved in the field would be expected to be lower. However, the rate of decline of compressive strength with increasing air-entrainment is assumed to be approximately the same for both laboratory and field concrete.

The solid line in Figure 1 shows the linear regression line obtained from the laboratory cylinder strengths. In order to approximate the line for field concrete, the dashed line in Figure 1 is drawn through the known mean strength for field concrete (4750 psi. at an average air level of 4.5 percent) parallel to the laboratory line. To determine the maximum amount the air-entrainment specification can be increased without an unacceptable loss of strength requires some fairly complicated statistical calculations which have been described in a separate report<sup>1</sup>. As a result of this study, it was recommended that a specification of  $6.0 \pm 1.5$  percent be adopted. This is now in effect and it is expected that a substantial improvement in concrete durability will be realized as a result of this study.

---

<sup>1</sup>Weed, R. M., Statistical Analysis of Concrete Strength versus Air Entrainment, Highway Research Record 433, 1973.

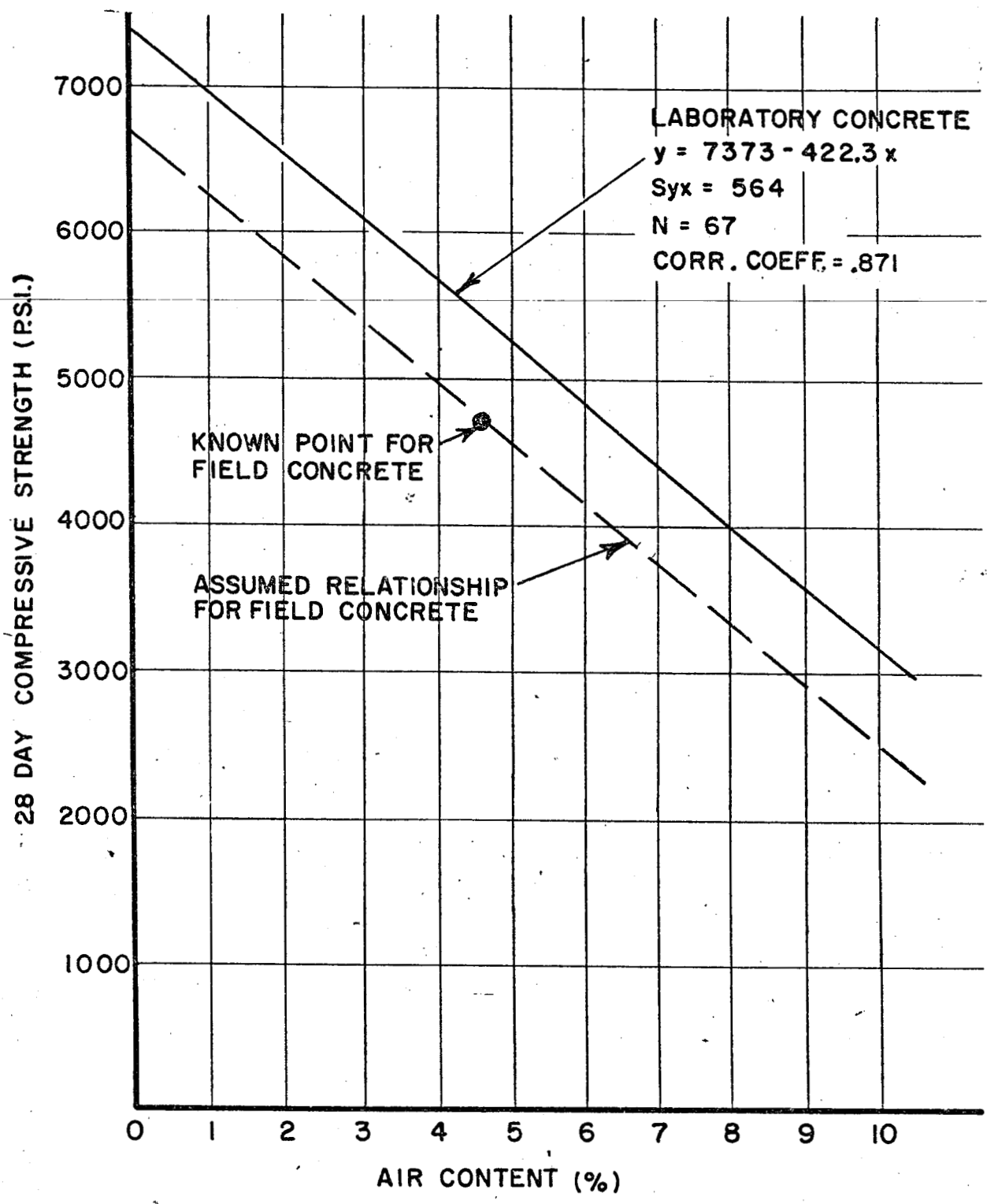


Figure 1. Regression Analysis Of Compressive Strength Vs. Air Content

#### 4.0 LABORATORY FREEZE-THAW STUDY

In addition to the accelerated outdoor exposure tests and the field tests on actual bridges, it was also planned to study the scaling phenomenon by means of a laboratory freeze-thaw chamber in an attempt to learn more about the actual physical scaling mechanism. A detailed specification was prepared and a laboratory freeze-thaw chamber was built which was similar to others in use. It consisted of a sealed chamber filled with water-saturated kerosene into which the test specimens were placed. Automatic controls were capable of continuously cycling the temperature from +50°F to 0°F and back at a preset rate. The tests were monitored by (a) several thermocouples placed at varying depths below the surface of each specimen, (b) strain gauges contacting special metal plugs mounted in the specimens to measure dilation, and (c) a thermocouple placed in the kerosene bath to measure its temperature. The outputs of all these devices were recorded by multipoint recorders.

As described in more detail in the original work plan, it was thought that isotherms (steps) in the temperature plots for various depths in the specimens could be related to hydraulic pressure within the concrete. At atmospheric pressure, water freezes at 32°F and remains at that temperature (isotherm) until all the available water is frozen. If isotherms were found at temperatures below 32°F, a phase diagram of water could be utilized to estimate the hydraulic pressure existing within the concrete. The strain gauges would monitor length changes and would indicate when the concrete no longer returned to its original length. Any "permanent set" could be interpreted to indicate some degree of physical damage.

In order to provide the greatest opportunity for scaling to occur, the test specimens were placed in the freeze-thaw chamber in a saturated state. After casting, they were stored either in a fog room or a salt water bath until they were used.

The freeze-thaw chamber was put into operation in the fall of 1971 and, although it performed according to specification, it soon became a source of continual maintenance problems which interrupted the continuity of the testing. However, even during the periods of normal operation, the expected results were not achieved. The thermocouples inside the test specimens indicated that the specimens underwent over 200 freeze-thaw cycles but no isotherms were detected.

Although the large volume of kerosene in the freeze-thaw chamber provided a large thermal inertia which was expected to damp out the cycling of both the refrigeration and heating units, the thermocouple in the bath showed that a slight step function existed. In order to determine if these steps were obscuring the isotherms, the unit was put into a constant cooling mode so that the temperature of the kerosene (and, therefore, the test specimens) would follow a smooth descending curve. This, too, proved fruitless because nothing resembling an isotherm was observed.

An additional attempt was made with smaller specimens, two of which were sealed with paraffin. The idea was to test whether the size of the specimen or the possible intrusion of kerosene could somehow affect the detection of the isotherms. However, as in all the other cases, no isotherms were observed.

At this point it was thought necessary to re-evaluate this phase of the project. Since the freeze-thaw chamber was not producing meaningful results and was becoming increasingly more difficult to maintain, plus the fact that the other phases of the project have already yielded implementable results, it was decided to terminate the laboratory freeze-thaw testing part of the program.

#### SUMMARY AND CONCLUSIONS

Phase I, the outdoor screening of a large number of treatments, is essentially complete and has isolated four apparently beneficial treatments. Since three of these four present potential problems (difficult to apply and/or slippery when applied), they are not being considered for further testing under Phase II (field testing). The remaining beneficial treatment, an admixture, appears to be superior in every respect and will be further tested by actual field applications.

The Phase I study demonstrated such a significant benefit of entrained air that a satellite study was conducted which led to the recommendation that the air-entrainment specification be increased from  $4.5 \pm 1.5$  percent to  $6.0 \pm 1.5$  percent. This has since been implemented.

Phase III, laboratory freeze-thaw testing, has been unproductive and has been discontinued.

#### INTERIM RECOMMENDATION

The single most effective treatment found by Phase I testing is Pozzolith 200-N, manufactured by Master Builders Company. This is described by the manufacturer as a "multi-component, chloride-free, water-reducing admixture." It is recommended that this product be included in the plans for a limited number of bridges soon to be constructed. Both test and control sections should be provided and both should receive the standard membrane curing compound.

APPENDIX I

DURABILITY RATINGS

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
PA-1	Linseed Oil (50% Mineral Spirits)	-.28 +.26 (-.01)	-.63 -.22 (-.42)	-.71 -.28 (-.50)		NO*	+.06	+.75	+.80		NO
PA-2	Fish Oil (50% Mineral Spirits)	+.07 +.08 (+.08)	-.12 +.67 (+.28)	-.43 +.63 (+.10)		NO*	-1.23 +.04 (-.60)	-.90 -.17 (-.53)	-.93 +.19 (-.37)		NO*
PA-3	Linseed and Fish Oils (50% Mineral Spirits)	-.20 +.01 (-.10)	+.41 -.11 (+.15)	+.18 -.50 (-.16)		NO*	-1.01 -.18 (-.60)	-.65 -.32 (-.48)	-.52 +.02 (-.25)		NO*
PA-4	Tall Oil (50% Mineral Spirits)	-.78 0 (-.39)	-.92 +.42 (-.25)	-1.03 +.38 (-.32)		NO*	-.74 +.29 (-.22)	-.86 +.01 (-.42)	-.87 +.21 (-.33)		NO*
PA-5	Tung Oil (50% Mineral Spirits)	-.49 -.17 (-.33)	-1.03 -.13 (-.58)	-1.02 -.09 (-.56)		NO*	-.88 +.19 (-.34)	-.38 +.01 (-.18)	-.40 -.21 (-.30)		NO*
PA-6	Emulsifiable Linseed Oil	-.37 -.06 (-.22)	-.79 -.08 (-.44)	-.70 -.14 (-.42)		NO*	-.44 -.58 (-.51)	-.68 -.83 (-.76)	-.44 -1.47 (-.96)		YES*

APPENDIX I

DURABILITY RATINGS

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
PA-7	Linseed Oil "Special Solvent"	-.37 -.38 (-.38)	-.66 -.22 (-.44)	-.99 -.21 (-.60)		NO*	+.35 +.25 (+.30)	+.57 +.32 (+.44)	+.99 +.25 (+.62)		NO*
PA-8	Polysulfide Epoxy	-.10	-.60	-.99		NO	-.39	-.57	-.52		NO
PA-10	Polymerized Solution of Metal Organic Compounds	+.10 -.13 (-.02)	-.26 +.08 (-.09)	-.37 +.26 (-.06)		NO*	-.52 -.12 (-.32)	-.85 -.50 (-.68)	-.88 -.36 (-.62)		NO*
PA-12a	Soybean Oil (50% Mineral Spirits)	-.25	-.82	-1.13		NO	-.51	-.94	-.99		NO
PA-12b	Soybean Oil (Uncut)	-.09	-.10	-.14		NO	+.09	-.38	+.03		NO
PA-13a	Castor Oil (50% Mineral Spirits)	-.37	-.62	-1.06		NO	-1.22	-1.16	-1.23		YES
PA-13b	Castor Oil (Uncut)	-.16	+.12	+.15		NO	-.06	-.40	-.34		NO

APPENDIX I

DURABILITY RATINGS

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
PA-14	Linseed Oil (Uncut)	-.22	-.67	-.89		NO	+.07	+.35	+.67		NO
PA-15	Chlorinated Rubber Epoxy	-.02 +.35 (+.16)	-.28 -.74 (-.51)	-.49 -1.12 (-.80)		NO*	+.09 -.50 (-.20)	-.29 -.45 (-.37)	-.32 -.69 (-.50)		NO*
PA-17	Waterproofing Compound	-.02 +.03 (0)	-.06 +.01 (-.02)	-.13 -.10 (-.12)		NO*	+.33 -.18 (+.08)	+.31 -.29 (+.01)	+.22 -.09 (+.06)		NO*
PA-18	Penetrating Epoxy	+.08	+.02	-.09		NO	-.37	-.68	-.57		NO
S-1	Tar-Based Sealer	-.17	-.58	-.85		NO	-1.43	-1.60	-2.03		YES
S-2	Resin-Based Sealer	-.14 +.17 (+.02)	-.08 +.25 (+.08)	+.09 +.31 (+.20)		NO*	-.34 -.25 (-.30)	-.33 -.32 (-.32)	+.05 -.06 (0)		NO*
S-3	Styrene Butadiene	-.17	-.42	-.38		NO	+2.37	+2.86	----		YES
S-4	Urethane	-.21	-.89	-1.30		YES	+1.74	+2.86	----		YES

**APPENDIX I**

**DURABILITY RATINGS**

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
S-5	Furan Resin	+0.01	-.76	-1.11		NO	----	----	----		---
S-6	Epoxy Resin	-.29	-.82	-1.34		YES	----	----	----		---
S-8	Butyrate Polymer	-.20	-.42	-.40		NO	-.41 +.14 (-.14)	-.18 -.14 (-.16)	+.12 +.16 (+.14)		NO*
A-1	Reactive Silane	-.27	+.05	+.35		NO	----	----	----		---
A-3	Polymerized Solution of Metal Organic Compounds	-.05	+.07	+.22		NO	----	----	----		---
A-4a	Calcium Chloride Based Waterproofing Agent	-.39 +.28 (-.06)	-.55 +.27 (-.14)	-.76 +.65 (-.06)		NO*	----	----	----		---
A-4b**	Calcium Chloride Based Waterproofing Agent	-.15 -.08 (-.12)	-1.10 +.10 (-.50)	-1.23 +.82 (-.20)		NO*	----	----	----		---
A-5	Multi-component, Chloride Free, Water Reducing Admixture	-.15 -.08 (-.12)	-1.21 -1.33 (-1.27)	-1.46 -1.56 (-1.51)		YES*	----	----	----		---

\*\*These slabs were dry cured because the manufacturer claims no special curing procedure is necessary.

**APPENDIX I**

**DURABILITY RATINGS**

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
C-1	Linseed Oil Emulsion (Diluted with 1/3 Water)	+ .10 + .58 (+ .34)	- .44 - .68 (- .56)	- .40 - .51 (- .46)		NO*	- .12 - .26 (- .19)	+ .03 + .93 (+ .48)	+ .14 + .12 (+ .13)		NO*
C-2	Chlorinated Rubber Epoxy	- .07 - .21 (- .14)	- .55 - .88 (- .72)	- .86 - .61 (- .74)		NO*	+ .70 + .48 (+ .59)	+ 2.06 + .17 (+ 1.16)	+ 1.99 + .55 (+ 1.27)		YES*
C-3	Reactive Silane	+ .10 - .04 (+ .03)	+ .01 - .73 (- .36)	+ .11 - .87 (- .38)		NO*	- .35	- .50	- .64		NO
C-4	Commercial Curing Compound	- .06 - .08 (- .07)	+ .08 - .60 (- .26)	0 - .41 (- .20)		NO*	+ 3.27	+ 3.58	+ 2.65		YES
C-5	Two Component Epoxy	- .21 - .31 (- .26)	- .60 - .61 (- .60)	- .87 - .85 (- .86)		YES*	- .69 - 1.17 (- .93)	- .25 - 1.43 (- 1.34)	+ .11 - 1.52 (- .70)		NO*
C-6	Epoxy Modified Acrylic Polymer	- .10 + .46 (+ .18)	- .16 + .10 (- .03)	- .33 + .16 (- .08)		NO*	- .37 + 1.10 (+ .36)	+ .83 + 1.93 (+ 1.38)	+ .43 + 1.77 (+ 1.10)		YES*

**APPENDIX I**

**DURABILITY RATINGS**

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
C-7	Chlorinated Rubber Curing Compound (Higher Solids Content)	+0.10 -0.07 (+0.02)	+0.26 -0.28 (-0.01)	+1.12 +0.40 (+0.76)		NO*	-1.05 +0.02 (-0.52)	+1.21 +0.77 (+0.99)	+0.29 -0.10 (+0.10)		NO*
C-8	Chlorinated Rubber Curing Compound (Lower Solids Content)	-0.17 +0.09 (-0.04)	-0.16 -0.28 (-0.22)	-0.33 +0.07 (-0.13)		NO*	0 +0.56 (+0.28)	+0.81 +2.42 (+1.62)	+0.39 +1.37 (+0.88)		YES*
C-0	N.J. Specification Quality White Pigmented Curing Compound	-0.07	+0.02	-0.29		NO	----	----	----		---
PA-4 on C-0	Tall Oil and Mineral Spirits Applied Over Curing Compound	+1.46	+1.72	+1.06		NO	----	----	----		---
PA-5 on C-0	Tung Oil and Mineral Spirits Applied Over Curing Compound	-0.04	-0.14	-0.09		NO	----	----	----		---

**APPENDIX I**

**DURABILITY RATINGS**

(Negative Values Indicate Beneficial Results)

Test Designation	Description of Material	FIRST TEST SLAB RATING SECOND TEST SLAB RATING (AVERAGE IN PARENTHESES) WHERE APPLICABLE									
		Air-Entrained Concrete					Non-Air-Entrained Concrete				
		Freeze-Thaw Cycles					Freeze-Thaw Cycles				
		46	102	147		Significant at 95%	46	102	147		Significant at 95%
LO-MS on C-0	Linseed Oil and Mineral Spirits Applied Over Curing Compound	-.04	-.06	+2.03		YES	---	---	---		---

\*Average of two tests in which case a durability rating of + 0.83 is considered significant at the 95 percent level of confidence. Others are single tests for which a durability rating of + 1.18 demonstrates significance.

APPENDIX IIAIR-ENTRAINED CONCRETE

<u>Slab Number</u>	<u>Air Level</u>	<u>Scaling Rating</u>
3	5.4	1.25
6	4.2	.35
9	3.9	1.42
12	4.6	1.03
14	4.8	1.64
32	4.0	1.35
36	4.8	1.60
38	4.8	1.52
45	4.8	.82
61	3.8	1.82
65	5.2	.71
69	4.9	.81
70	4.8	1.29
74	4.6	1.64
97	5.2	2.03
101	4.6	.75
103	4.8	1.32
152	4.8	1.20
157	5.7	.81
159	5.4	.44
164	4.9	.98
167	4.8	1.12
169	6.0	.76
172	4.6	.75
174	4.8	1.32
176	4.5	1.98

$$\bar{x}_{AE} = 1.18$$

$$\sigma_{AE} = 0.45$$

NON-AIR-ENTRAINED CONCRETE

<u>Slab Number</u>	<u>Air Level</u>	<u>Scaling Rating</u>
17	1.3	2.08
21	1.3	1.98
23	0.6	1.62
27	2.2	1.38
30	0.6	.69
48	1.4	1.92
50	1.8	2.29
54	1.0	2.30
57	1.2	2.28
60	2.3	1.94
80	1.6	1.95
82	1.6	2.04
87	2.3	1.61
90	0.8	2.57
107	1.5	3.24
111	1.1	2.45
112	1.5	2.14
121	0.6	1.75
127	1.0	1.82
130	1.0	3.02
132	1.1	3.65
134	0.7	4.86
137	1.1	3.51
142	1.4	2.80
147	1.0	2.35
148	1.0	4.28

$$\bar{x}_{NAE} = 2.40$$

$$\sigma_{NAE} = 0.91$$

APPENDIX IIIDEVELOPMENT OF STATISTICAL CRITERIA

The variability associated with the rating system was relatively easy to determine by having the group make replicate ratings. This was done within a short enough period of time so that no real changes in the slabs occurred, and in a manner which made it unlikely that any rater would be biased by remembering previous ratings. Since the durability rating (test minus control, hereafter designated T-C) is the parameter of interest, the corresponding durability ratings were calculated for the replicate evaluation. In order to apply the techniques of statistical analysis, a variable of the form  $\bar{X}_i = [(T-C)_1 - (T-C)_2]_i$  was used in which the subscripts 1 and 2 represent the replicate evaluations and the subscript "i" represents particular treatments. It was theorized, and later confirmed by chi-square and "t" tests, that this variable is approximately normally distributed with a mean of zero. Since the durability ratings are averages which are reported to the second decimal place, the distribution will be considered continuous.

This particular variable ( $\bar{X}_i$ ) was chosen because it furnishes two very important bits of information. First, the mean of zero confirmed the intuitive belief that, even though the rating group might be generally more severe on one day than on some other, there will (on the average) be no effect on the durability ratings which are the differences between two scaling ratings (T-C). That is, if both the T and C values are rated higher (or lower) by about the same amount, the difference (durability rating) will remain about the same. Thus, it is assumed that any possible rating group bias can be ignored.

Second, the principle of additive variances enables us to calculate the standard deviation attributable to the rating system ( $\sigma_{RS}$ ). The distribution of the difference between two normally distributed variables is also normal with a variance equal to the sum of the variances of the two original variables. This can be expressed mathematically as follows:

$$\begin{aligned}\sigma_{\bar{X}}^2 &= \sigma_{(T-C)_1}^2 + \sigma_{(T-C)_2}^2 = 2\sigma_{(T-C)}^2 \quad \text{and} \\ \sigma_{(T-C)}^2 &= \sigma_{RS}^2 \quad \text{by definition so} \\ \sigma_{RS} &= \sigma_{\bar{X}} / \sqrt{2}\end{aligned}$$

was found to be 0.194 and, from this,  $\sigma_{RS} = 0.14$  rating units. This implies, for example, that if there is truly no difference between a test slab and its control, the rating group will indicate differences as great as  $\pm 1.96 \sigma_{RS} = \pm 0.27$  rating units 95 percent of the time. Therefore, a difference of at least  $\pm 0.27$  must be obtained for any particular test before it could be attributed to anything other than the variability of the rating system (at the 95 percent confidence level).

The component of variance associated with the effectiveness of the treatments is not so easy to estimate accurately. The large quantity of treatments being evaluated made it impractical to include enough replicate test slabs to calculate this parameter accurately for each individual treatment. Instead, it was necessary to use all treatment pairs to estimate an average standard deviation for treatment effectiveness ( $\sigma_{TE}$ ) which will be assumed to apply to all treatment for the Phase I screening test. Any treatment which barely fails to exhibit adequate

performance, but which appears to have a lower than average  $\sigma_{TE}$  value based on the two tests for that treatment, may warrant further consideration. Conversely, any treatment which barely demonstrates acceptable performance, but which appears to be more variable than average, probably should be subjected to further screening tests.

The variable to be used for the determination of  $\sigma_{TE}$  is  $\bar{X}_1 = [(T-C)_1 - (T-C)_2]_1$  in which the subscripts 1 and 2 represent the two test slabs upon which treatment "i" was applied. Theoretically, this variable should range both plus and minus. However, since there is no way to know which of the two test slabs should be considered first, absolute values were used and the distribution was analyzed as a "half-normal" curve. This was done by entering each data point twice, once as a positive value and once as a negative value. The resulting histograms appeared very normal for both air-entrained and non-air-entrained concrete so it was not deemed necessary to run chi-square tests to confirm this.

Using the additive variance principle as before, the standard deviation of the (T-C) values is again  $\sigma_x/\sqrt{2}$  except that, in this case, this term automatically includes the variability associated with the rating system and, therefore, is the overall variability of the measurement process. This will be designated  $\sigma_{AM}$  to represent the variability "as measured" and was calculated to be 0.58 and 0.62 rating units, respectively, for air-entrained and non-air-entrained concrete. These values are so close to each other that it was decided to pool them to arrive at a common value of  $\sigma_{AM} = 0.60$  to determine the level at which a treatment is to be considered significantly beneficial or

detrimental. If there is no real difference between a test and its control, the rating group can be expected to indicate differences as large as  $\pm 1.96 \sigma_{AM} = \pm 1.18$  rating units 95 percent of the time. Therefore, at the 95 percent level of confidence, a single test must have a durability rating of at least  $\pm 1.18$  rating units before it can be considered significant. In most cases, the durability ratings are the average of two tests (slabs) for which a level of  $\pm 1.96 \sigma_{AM}/\sqrt{2}$  =  $\pm 0.83$  is required to establish significance.

The standard deviation associated only with treatment effectiveness ( $\sigma_{TE}$ ) can be determined from the relationship

$$\sigma_{TE} = \sqrt{\sigma_{AM}^2 - \sigma_{RS}^2}$$

and is found to be  $\sigma_{TE} = 0.58$ , considerably larger than the value of  $\sigma_{RS} = 0.14$  associated with the rating system.