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Procedures for Evaluating Corrosion-Inhibiting Admixtures for Structural Concrete

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DISCLAIMER

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SUMMARY

PROCEDURES FOR EVALUATING CORROSION-INHIBITING ADMIXTURES FOR STRUCTURAL CONCRETE

During the past fifteen years, corrosion-inhibiting admixtures (CIAs) have become increasingly popular for long-term protection of reinforced and prestressed concrete components of highway bridges and other structures. However, there remains considerable debate about the benefits of CIAs in concrete. The objectives of this research were to (1) develop procedures for evaluating and qualifying corrosion-inhibiting admixtures and (2) recommend performance criteria for their acceptance.

Phase I work included a literature review of CIAs, the review of test procedures presently used for evaluating CIAs, and the development of a laboratory test plan for evaluating CIAs. In Phase II, the laboratory test plan was executed and performance criteria for qualifying an admixture as a CIA were developed.

The following corrosion inhibiting admixtures (CIAs), which are available commercially, were identified. Based on proposed mechanism of protection, they were combined into four groups.

1. Ferrogard 901, a modified version of Armatec 2000, (SIKA) and MCI 2000 (Cortec). A blend of surfactants and amine salts (specifically, dimethyl ethanolamine [DMEA], also referred to as alkanolamines or amino alcohols [AMA]) in a water carrier. Both Armatec and MCI were manufactured by Cortec; the Armatec version (until recently marketed by Sika) purportedly has proportions and concentrations of the ingredients slightly different from those for the MCI version. Ferrogard is manufactured by Sika.

2. Rheocrete 222 and Rheocrete 222+ (Master Builders). A water-based combination of amines and esters. Rheocrete 222+ is a new and, supposedly, improved version.

3. DCI and DCI-S (W.R. Grace). Calcium nitrite-based admixture (about 30% concentration of the active ingredient). DCI-S contains a set-retarding admixture.

4. Catexol 1000 CI (Axim Concrete Technologies). According to the manufacturer, a water-based solution of amine derivatives. No specific performance data was available for this admixture at the time of the review.

The active ingredient of the DCI corrosion inhibitor, calcium nitrite, provides protection of reinforcing steel by facilitating the formation of a passive oxide film on the steel surface. DCI, therefore, falls into the category of anodic inhibitor.

Rheocrete, Ferrogard/Armatec/MCI, and Catexol are all organic film forming type inhibitors. These inhibitors would all be classified as “mixed-type” inhibitors, since they inhibit both the anodic and cathodic reactions. Also, these inhibitors claim to retard chloride penetration into concrete to some degree.

No data were available on Catexol beyond the manufacturer’s data sheets. Therefore, the detailed performance analysis provided in the report focused on three primary commercially available CIAs: DCI, Rheocrete, and Ferrogard/Armatec/ MCI. The vast majority of performance data involved laboratory testing. This testing indicated that the three CIAs can inhibit corrosion of reinforcing steel in concrete for at least some of the conditions tested; the mechanisms of protection were apparently different, and the effectiveness depended on test conditions.

CIAs have been used in a wide range of concrete structures, but there is very limited field performance information on any of the CIAs. This is due to the long time required to assess the performance and the lack of follow-up studies once a structure is in place.

The manufacturers have produced the bulk of the performance data available for the CIAs. Although several CIAs have been used or are being evaluated by one or more states, the most widely tested CIA is calcium nitrite-based DCI, which has been on the market much longer than other commercial CIAs. Some data were available from other independent sources, such as Federal Highway Administration (FHWA) projects and studies by the state departments of transportation (DOTs). These data suggest that, in general, CIAs provided a reduction in the time-to-corrosion initiation and/or a reduction of corrosion rate following initiation versus control specimens. The degree of benefits was dependent on the specific CIA, concentration of CIA, and specific test conditions.

Based on the available performance data, it was not possible to accurately define the benefits of the different CIAs, other than to say that reduction in corrosion is possible through their use. At the present time, there is no standard testing regimen that can evaluate and compare performance with respect to claims to extended life; additionally, "improved" formulations make prior testing results invalid. Phase II of this project executed a laboratory-testing program designed to establish a standard testing protocol for evaluating CIAs and to establish performance criteria for qualifying an admixture as a CIA.

Laboratory tests included concrete mechanical property tests, corrosion rate tests for both small short-term specimens and long-term slabs, tests of simulated cracked minibeam specimens, and chloride penetration tests. All of these tests were performed for a range of CIA concentrations for each CIA.

A life prediction model was presented which serves as the basis of the performance criteria for acceptance of a CIA. The prediction model is relatively simple and is not intended to predict life of an actual structure exposed to a specific set of environmental conditions.

It is shown that all three tested CIAs gave beneficial results, as determined by the prediction model and the long-term slab tests. However, some discrepancies between the short-term test results and long-term slab tests are discussed in light of the different exposure conditions for the different methods.

A draft recommended practice, titled “Proposed Method For Qualifying Corrosion Inhibiting Admixtures That Mitigate Corrosion Of Reinforcing Steel In Concrete,” was developed. In this practice, a testing protocol is presented that generates all of the pertinent data required for the prediction model, as well as data to define certain concrete properties and simulated cracked concrete behavior. The practice, as written, presently has not been validated through testing to show its general applicability. The proposed performance criteria for qualifying an admixture as a CIA are as follows:

Criterion 1. The CIA should provide an improvement over the base (no-CIA) condition with respect to the predicted life by a minimum of 25 percent.

Criterion 2. Increase in life must be due to one or both of the following: (1) increased chloride threshold for initiation of corrosion or (2) a decrease in the slope of the linear regression fit of corrosion rate versus chloride concentration.

Criterion 3. The CIA should provide some improvement in corrosion performance for cracked concrete.

Criterion 4. The CIA should not adversely affect the concrete properties in such a manner that pertinent specifications are not met. At a minimum, these should include compressive strength, flexural strength, modulus of elasticity, slump, time-to-set, and air content. Other properties that were not specifically studied in this project but could have a significant effect on concrete performance are air distribution and shrinkage.

All four of these criteria should be met for an admixture can be qualified as a CIA.

CHAPTER **1**

INTRODUCTION AND RESEARCH APPROACH

PROBLEM STATEMENT

During the past fifteen years, corrosion-inhibiting admixtures (CIAs) have become increasingly popular for long-term protection of reinforced and prestressed concrete components of highway bridges and other structures. However, there has been considerable debate recently about the benefits of CIAs in concrete.

In 1992, a Joint Committee of the American Association of State Highway and Transportation Officials (AASHTO), the Associated General Contractors (ACG), and the American Road and Transportation Builders Association (ARTBA), through Task Force Number 32, prepared a Manual for Corrosion Protection of Concrete Components in Bridges (see Special Note A). The manual addresses the various systems, including CIAs available to provide corrosion protection for bridge components. However, neither this manual nor other publications provide specifications or guidelines to help engineers evaluate and compare CIAs. In the absence of such information, engineers frequently rely on information provided by product manufacturers when making evaluations and recommendations. This information, in many cases, is not based on well-defined, consistent procedures; therefore, research was needed to develop a set of tests for evaluating CIAs to enable engineers to make more rational product comparisons and recommendations.

RESEARCH OBJECTIVES

The objectives of this research were to (1) develop procedures for evaluating and qualifying corrosion-inhibiting admixtures and (2) recommend performance criteria for their acceptance. This research was limited to CIAs as defined in the American Concrete Institute Manual of Concrete Practice, i.e. the research was limited to chemical admixtures to be added to the portland cement concrete mixtures, usually in very small concentrations, for the primary purpose of corrosion protection. While other materials, such as microsilica, fly ash, and ground- granulated blast-furnace slag may provide corrosion protection, they were not regarded, for the purpose of this research, as CIAs. These materials, however, may be included in concrete mixtures containing CIAs.

RESEARCH APPROACH

The work plan for NCHRP Project 10-45 was divided into the following two Phases.

PHASE I – PLAN DEVELOPMENT
  Task 1 – State-of-the-Art of Corrosion Inhibiting Admixtures.
  Task 2 – Test Methods.
  Task 3 – Research Plan.
  Task 4 – Interim Report.

PHASE II – PLAN IMPLEMENTATION
  Task 5 – Laboratory Test Program.
  Task 6 –Test Protocol.

In Task 1, the following activities were performed: (1) collect and review relevant domestic and foreign literature, research findings, performance data, and current practices relative to the use, testing, and evaluation of CIAs, (2) compile a list of available CIAs, (3) delineate the mechanism by which each admixture works, and (4) summarize the effects of each admixture on the properties of the fresh and hardened concrete.

Task 2 identified and evaluated (with consideration to performance predictability, practicality, cost, and other pertinent factors) both screening and long-term verification test procedures currently used in the United States and other countries for evaluating the effectiveness of CIAs. Special consideration was given to the duration of the tests, the quality of the concrete used in bridge components, and the performance in cracked concrete.

A research plan was finalized that encompassed a laboratory investigation to evaluate and validate test procedures for testing the performance of CIAs (Task 3). An interim report was issued that documented the research performed in Tasks 1 through 3 and provided a work plan for the Phase II of the project (Task 4). Details of the experimental work plan and test matrixes performed are provided in Appendix A. The plan consisted of the following:

- Prediction of corrosion rate as a function of chloride concentration and CIA concentration.

- Prediction of chloride threshold concentration.
- Prediction of chloride penetration rate through concrete as a function of CIA concentration.
- Measurement of concrete property data as a function of CIA concentration.
- Measurement of CIA performance in the presence of preformed cracks in the concrete extending down to the steel bar surface.

In Task 5, the laboratory test program was performed. The test procedures addressed the corrosion inhibiting effectiveness of the admixture and the effect of the admixture on the properties of the fresh and hardened concrete.

In Task 6, a draft standard practice titled “Proposed Method For Qualifying Corrosion Inhibiting Admixtures That Mitigate Corrosion Of Reinforcing Steel In Concrete” was developed. This practice defined a laboratory testing protocol that would provide the required data to evaluate a proposed CIA based on a set of performance criteria given in the standard.

CHAPTER **2**

RESEARCH FINDINGS

REVIEW OF COMMERCIALLY AVAILABLE CIAS

A detailed review of the literature on the corrosion inhibiting admixtures (CIAs) is given in Appendix B. Five commercially available CIAs were identified in this review: DCI (W.R. Grace), Rheocrete 222 (Master Builders), Ferrogard 901 (an improved version of Armatec 2000 by Sika), MCI 2000 (Cortec), and Catexol 1000 CI (Axim Concrete Technologies). Ferrogard, Armatec, and MCI have the same basic active ingredients and were grouped together in the analysis. The following briefly describes the four identified CIAs.

1. Rheocrete 222 and Rheocrete 222+ (Master Builders). A water-based combination of amines and esters. Rheocrete 222+ is a new and, supposedly, improved version.

2. Ferrogard 901, a modified version of Armatec 2000, (SIKA) and MCI 2000 (Cortec). A blend of surfactants and amine salts (specifically, dimethyl ethanolamine [DMEA], also referred to as alkanolamine or amino alcohol [AMA]) in a water carrier. Both Armatec and MCI were, at some time, manufactured by Cortec; the Armatec version (until recently marketed by Sika) purportedly has proportions and concentrations of the ingredients slightly different from those for the MCI version. Ferrogard is manufactured by Sika.

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No data were available on Catexol beyond the manufacturer's data sheets. Therefore, the detailed performance analysis provided in the report focuses on Rheocrete, DCI, and Ferrogard/ Armatec/MCI. The vast majority of performance data to date on these compounds involves laboratory testing. This testing indicated that the three CIAs can inhibit corrosion of reinforcing steel in concrete for at least some of the conditions tested; the mechanisms of protection were apparently different, and the effectiveness depended on test conditions.

The manufacturers have produced the bulk of the performance data available for the CIAs. Although several CIAs have been used or are being evaluated by one or more states, the most widely tested CIA is calcium nitrite-based DCI. DCI has been on the market for a much longer time than other commercially available CIAs. Some data were available from other sources, such as Federal Highway Administration (FHWA) projects and studies by state departments of transportation (DOTs). These data suggest that, in general, CIAs provided a reduction in the time to corrosion initiation and/or a reduction of corrosion rate following initiation versus control specimens. The degree of benefits was dependent on the specific CIA, concentration of CIA, and specific test conditions. Based on the available performance data, it was not possible to accurately define the benefits of the different CIAs, other than to say that reduction in corrosion was possible through their use.

Appendix C presents the industry survey and the results of the limited field studies (performed or underway). CIAs have been used in a wide range of concrete structures, but there was very limited field performance information on any of the CIAs. This was due to the time required to assess the performance and the lack of follow-up studies once a structure was in place.

LABORATORY TESTING PROGRAM

The laboratory testing program included tests to examine the corrosion rate of steel in concrete, the chloride penetration of chloride into concrete, and the mechanical properties of concrete (see Appendix A for details of experimental plan). Each of these types of testing was performed for each CIA for a range of concentrations.

Two concretes were used in the tests. Concrete 0 was a Type I portland cement based concrete with a 0.40 w/c, 30% (by volume) cementitious material paste, silica coarse aggregate, quartz sand, and a 6% air content. Concrete 1 had the same basic design, with 7% silica fume added to the cementitious component.

The testing program included the following: (1) fixed-chloride tests as a relatively short-term test to establish the corrosion rate as a function of chloride concentration, (2) chloride threshold tests to determine the threshold level of chloride to initiate corrosion, (3) simulated cracked minibeam tests to establish the benefit of CIA for the of cracked concrete condition, (4) long-term concrete slab tests to determine the effect of CIA on corrosion in more realistic concrete slab tests, (5) chloride penetration tests to establish an effective diffusion coefficient for chloride in concrete, and (6) concrete property tests to examine the effect of CIA on specific parameters (compressive strength, flexural strength, modulus of elasticity, slump, setting time, and air void content).

The three CIAs tested in this program were designated as CIA-A, CIA-B, and CIA-C. The purpose of this project was to develop a general test protocol and associated performance criteria for evaluating CIAs, not a ranking of available CIAs. Therefore, the identity of CIAs is not disclosed. The CIAs were tested at four concentrations, specifically, at 10, 50, 75, and 100% of the manufacturer's maximum recommended dosage.

Fixed-Chloride Tests

The purpose the fixed chloride test was to establish the corrosion rate of steel as a function of chloride and CIA concentration. Appendix D contains the individual corrosion rate and potential maps for each CIA in Concrete 0 as a function of the targeted chloride concentration (targeted concentrations were 3, 6, and 9 kg/m^3 (5, 10, and 15 lb/yd^3) of chloride). For Concrete 0, the targeted and measured chloride concentrations were relatively close (see Appendix E for the tabulated data). The targeted chloride concentrations were not achieved for the Concrete 1 tests, thus preventing plots similar to those in Appendix D to be made for Concrete 1.

To summarize significant effects, the mean corrosion rate for each CIA concentration was determined. This analysis averages the results for all of the specimens and different chloride levels to provide a mean corrosion rate at each CIA concentration. Figures 1 and 2 give the comparison of the CIA corrosion performance in

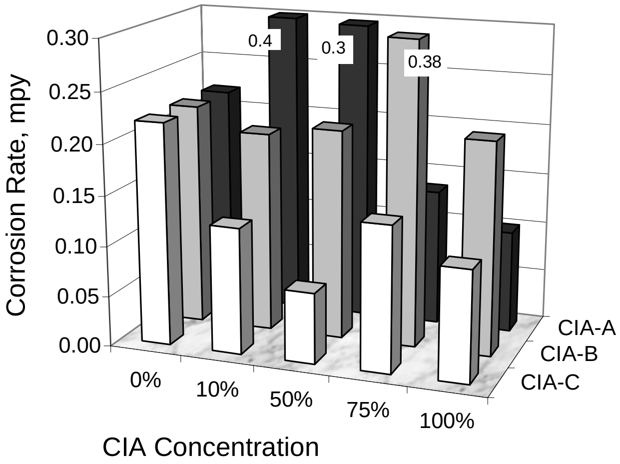


Figure 1. Comparison of CIA corrosion performance in Concrete 0 (average for all chloride concentrations). [1 mm/yr = 39 mpy]

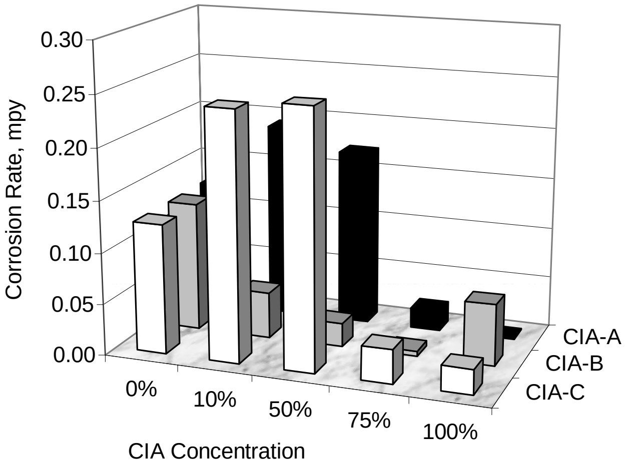


Figure 2. Comparison of CIA corrosion performance in Concrete 1 (average for all chloride concentrations). [1 mm/yr = 39 mpy]

Concretes 0 and 1 respectively. Figure 1 indicates some beneficial effect of CIA-A and CIA-C, but no effect of CIA-B tested in Concrete 0 (no silica fume). However, Figure 2 indicates that CIA-B decreased corrosion rate at each CIA concentration tested in Concrete 1 (silica fume added).

In general, the effect of the CIAs on the corrosion of steel was more pronounced for steel in Concrete 1 (Figure 2) than for Concrete 0 (Figure 1). The effect of each of the CIAs in Concrete 1 was to significantly reduce the corrosion of the steel. At 100 percent of the manufacturer’s recommended maximum dose, CIA-A exhibited the best performance. However, all three CIA-containing specimens exhibited improvement over the control (no-CIA) specimens. From Appendix E, it is seen that the targeted chloride concentrations were not achieved for the Concrete 1 tests. Therefore, some care is required in analyzing the corrosion rate, as a function of inhibitor concentrations, since the chloride achieved concentrations may not have been the same for all CIAs. For example, the analysis shown in Figure 2 indicates a very low corrosion rate for 75% dosage for CIA-B; however, close analysis shows that there were no high chloride concentrations achieved for that specific condition. The same is true for 100% dosage of CIA-A. Although there are some problems with certain concentrations, Figures 1 and 2 provide a reasonable overview of the data obtained.

As discussed above, the targeted chloride concentrations were not always achieved. Therefore, the following analysis utilizes the measured chloride concentrations. Figures 3 through 5 show corrosion rates versus chloride concentration in Concrete 0 for CIA-A, CIA-B, and CIA-C, respectively. There is significant scatter in the data; however, a regression analysis provided a comparison of the CIA data to the control (no CIA). According to Figure 3, CIA-A produces a decrease in the corrosion rate upon increasing the CIA concentration from 50 to 75 to 100% of the maximum recommended dosage. Figure 5 shows a beneficial effect of CIA-C, with 50 and 100% of the recommended maximum dosage giving similar results. No beneficial effect of CIA-B was found in these tests for Concrete 0 (Figure 4).

Figures 6 through 8 show corrosion rates versus chloride concentration in Concrete 1 for CIA-A, CIA-B, and CIA-C, respectively. It is seen that only relatively few had high chloride concentrations making the conclusions for the Concrete 1 tests be based on fewer data points than originally planned. CIA-A (Figure 6) shows an increasing beneficial effect upon going from 50 to 75% of maximum recommended

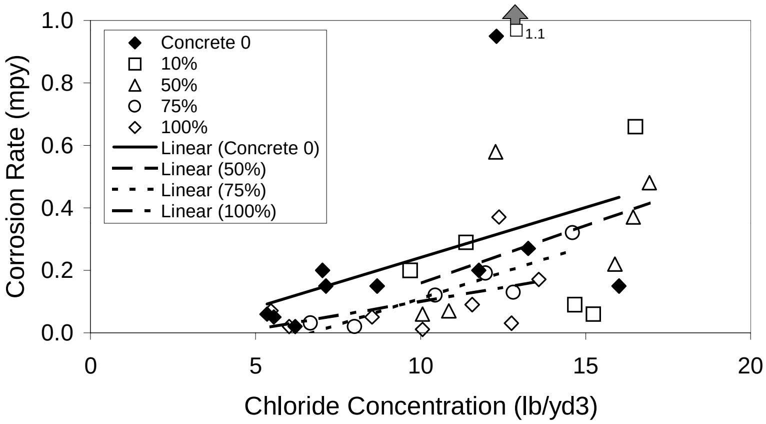


Figure 3. Corrosion rate versus chloride concentration for different CIA-A levels in concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

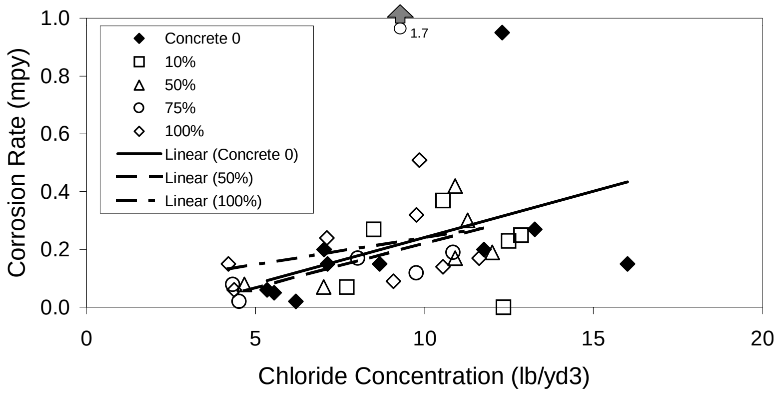


Figure 4. Corrosion rate versus chloride concentration for different CIA-B levels in Concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

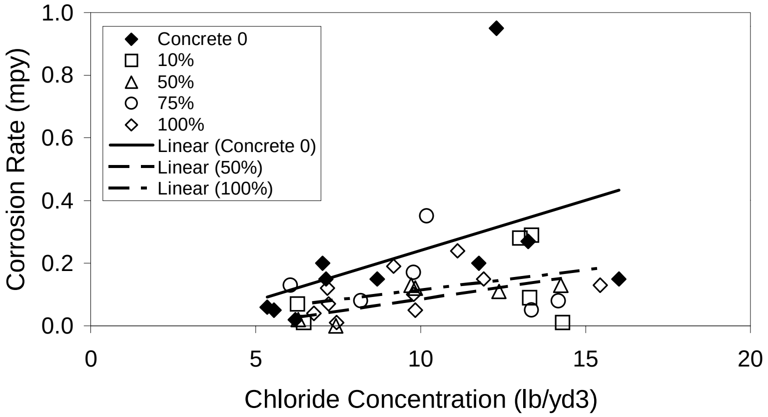


Figure 5. Corrosion rate versus chloride concentration for different CIA-C levels in concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

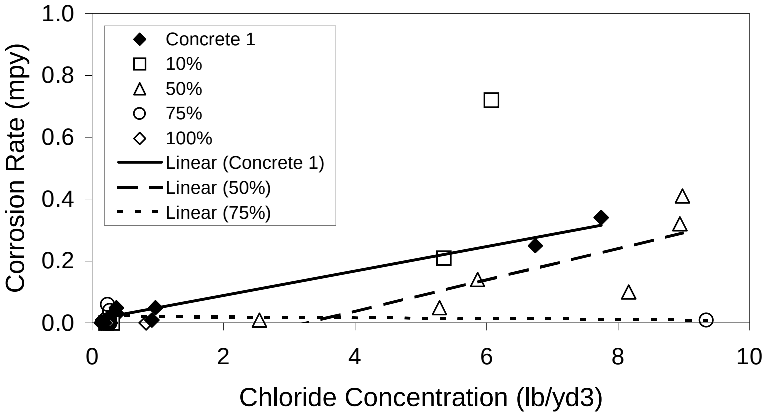


Figure 6. Corrosion rate versus chloride concentration for different CIA-A levels in concrete 1. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

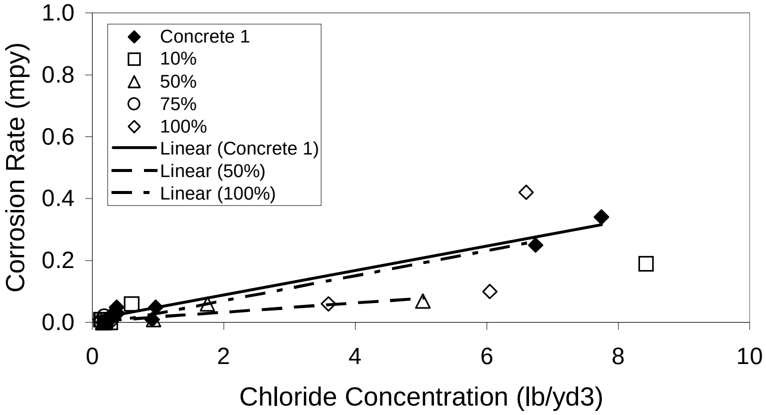


Figure 7. Corrosion rate versus chloride concentration for different CIA-B levels in concrete 1. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

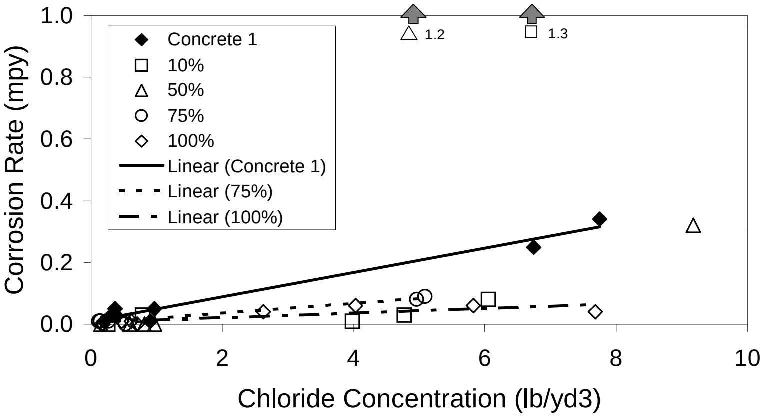


Figure 8. Corrosion rate versus chloride concentration for different CIA-C levels in concrete 1. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

dosage (at 100%, no high chloride concentrations were achieved, making conclusions for this dosage level impossible). Figure 7 shows a benefit at a 50% dosage, but no benefit at 100% dosage for CIA-B. For CIA-C (Figure 8), 75 and 100% dosage provided similar improvements in corrosion mitigation.

It is of interest to note that, for a given CIA, similar trends were observed for both Concretes 0 and 1. For CIA-A, increases in CIA concentrations produced progressively improved corrosion resistance. For CIA-C, the presence of the CIA resulted in improved corrosion resistance with similar behavior for the different CIA concentrations between 50 and 100%. For CIA-B, intermediate CIA concentration produced better corrosion performance than 100% CIA dosage.

Chloride Threshold Tests

The purpose of the chloride threshold tests was to determine the threshold concentration of chloride required to initiate corrosion. The test specimens were identical to the fixed chloride test specimens. The specimen preparation did not include a drying/vacuum/ponding cycle to force chlorides into the concrete, as did the fixed chloride tests. Instead, the specimens were ponded with a 15% NaCl solution and chlorides were permitted to diffuse into the concrete. Corrosion rate and potentials were periodically monitored until corrosion was considered to have initiated.

The criteria for initiation of corrosion were set as (1) a corrosion rate greater than 0.1 mpy (0.0025 mm/yr) and (2) a corrosion potential more negative than -0.350 V (copper/copper sulfate electrode [CSE]). These criteria were used as a guideline only. Occasionally, the two indicators (corrosion rate and potential) did not meet the above-established criteria at the same time. If the corrosion rate criterion was achieved, the tests were terminated. If the potential criterion was achieved (potentials exceeded -0.350 V) and the corrosion rate increased but did not exceed the established criterion (e.g., corrosion rate was 0.07 mpy [0.0018 mm/yr]), the test would be terminated and corrosion was assumed to have initiated.

The chloride threshold data for each individual test cell are given in Appendix F. Note that multiple chloride analyses were performed. This was necessary due to problems with collecting the concrete for chloride analysis. The desire was to have a sample of the concrete very close to the steel surface. Therefore, the quantity of collected concrete was kept to the minimum required for analysis. Upon re-examination of the collected sample, it was concluded that the original concrete sample was likely high in cement paste and low in aggregate phase. Since most of the chlorides were in the cement paste phase (aggregate had very low absorption coefficient), the samples collected in the first trial could be higher in chloride than it would be based on total concrete weight. Furthermore, because chloride sampling was performed at various times, the time lag could produce inconsistent results. Since the second sample (5-g) was farther away from the steel surface, (in the threshold tests, chloride diffused into the concrete, so that their concentration should increase with distance from the steel surface), the interpretation was as follows:

- A higher chloride concentration in the ‘5-g Sample’ may reflect a higher chloride concentration away from the steel surface due to the expected chloride gradient. The hypothesis that the procedure used for the initial (Previous) sample caused artificially high readings can not be verified. Therefore, the initial chloride sample (Previous) is used.
- A lower (or similar) chloride concentration in the 5-g Sample may reflect a more realistic value, since it fits the hypothesis that the initial (Previous) measurement was artificially high. Furthermore, there is no condition that would produce a lower chloride concentration at a greater distance from the steel surface.

The averages of the chloride threshold data are given in Table 1. Note that none of the Concrete 1 data initiated corrosion during the 185-day exposure period. Also, none of the CIA-B tests for Concrete 0 showed signs of corrosion initiation over the 209-day exposure.

The value for chloride threshold concentration for the control specimens in Concrete 0 was relatively high (3.8 kg/m^3 [6.4 lb/yd^3]). This value is higher than the typical value mentioned in the literature (approximately 1.2 kg/m^3 [2 lb/yd^3]). A few possible explanations for this result are provided below:

1. Continuous ponding in conjunction with a sealed specimen design limited oxygen diffusion to the steel surface (oxygen only available through the ponding solution; with the specimen bottom and sides completely sealed with epoxy) and made initiation of corrosion more difficult than in other specimen configurations or in real service.

Table 1. Summary of chloride threshold data. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA (%)	Chloride (lbs /yd^3)	Corrosion Initiated	Exposure (days)
0	None	None	6.4	yes	74
0	A	10	10.1	yes	166
0	A	50	15.2	no	209
0	A	75	12.5	yes	201
0	A	100	9.5	yes	82
0	B	10	8.2	no	209
0	B	50	6.8	no	209
0	B	75	6.1	no	209
0	B	100	4.7	no	209
0	C	10	3.0	yes	20
0	C	50	2.2	yes	38
0	C	75	2.3	yes	53
0	C	100	6.8	yes	207
1	None	None	1.6	no	209
1	A	10	1.9	no	185
1	A	50	1.2	no	185
1	A	75	1.4	no	185
1	A	100	3.5	no	185
1	B	10	1.1	no	185
1	B	50	0.6	no	185
1	B	75	1.3	no	185
1	B	100	1.1	no	185
1	C	10	0.7	no	185
1	C	50	0.7	no	185
1	C	75	0.5	no	185
1	C	100	0.6	no	185

2. The concrete selected for use in this concrete was a high quality (low w/c with silica and quartz aggregate) concrete, thus making the chloride threshold for corrosion initiation greater than generally expected.

3. The chloride ponding solution was 15% NaCl solution, and sufficient time was not provided for the initiation process to occur at lower chloride concentrations (i.e. if specimens were exposed for longer times at lower chloride concentrations, corrosion may have initiated at the lower chloride levels).

4. Finally, it is possible that for these specimens, the criteria for corrosion initiation were too severe and by the time the tests were terminated, corrosion had been initiated for some time and chloride concentrations had continued to increase. Possibly, different less stringent criteria for corrosion initiation (e.g., corrosion rate ≥0.05 mpy [≥0.0013 mm/yr] and a potential of ≤–0.250 V [CSE]) would have given different results. For the test specimen design, the resolution of corrosion rate is greater than that for a typical slab test specimen.

Although (i) the chloride threshold concentration was somewhat greater than expected for the control concrete and (ii) specimens for only two CIAs initiated corrosion for Concrete 0 and none of the Concrete 1 specimens showed corrosion, it is still worthwhile to examine the results. CIA-A exhibited a much higher chloride threshold for corrosion initiation than the no-CIA control specimens. This was consistent at each CIA-A concentration examined. CIA-C exhibited a lower chloride threshold for corrosion initiation than the control test for each concentration below 100%, but similar to the controls for the 100% of the maximum recommended dosage. For CIA-B, none of the tests initiated corrosion, even though the chlorides measured for these tests were in the range of the chloride threshold for the control Concrete 0 test. It can be concluded that CIA-B has a similar or greater chloride threshold than the control test.

For the Concrete 1 (silica fume) tests, the chloride concentrations remained relatively low even for the control tests, and no corrosion initiated.

Simulated Crack Minibeam Tests

The purpose of the simulated crack minibeam tests was to provide an assessment of the performance of the CIAs in the presence of cracks extending down to the steel surface. All three CIAs were tested in the simulated crack minibeam tests at 50 and

100% of the manufacturers maximum recommended dose. Concrete 0 was used in these tests; no tests were performed using Concrete 1 (silica fume).

Figures 9 – 11 compare the results for each of the CIAs to the control specimens (no CIA) for the 100% CIA dose conditions. Figure 9 shows that a significant reduction in coupled current magnitude and an increased time-to-initiation was observed for CIA-A. The next best performance was observed for CIA-B; two out of the three CIA-B specimens displayed behavior similar to the CIA-A specimens (Figure 10). CIA-C (Figure 11) did not show as good a performance as CIA-A or CIA-B. For CIA-C, the time-to-corrosion was longer than for the control specimens, but the magnitude of current, once corrosion initiated, was only marginally less than the control.

The performance of the CIAs as a function of CIA concentration was dependent on the individual CIA. All data are presented in Appendix G. For CIA-A, the 100% concentration slightly improved performance (lower coupled currents and/or longer time to initiation) as compared to the 50% concentration. For CIA-B, little difference was observed for the two concentrations. For CIA-C, the 50% concentration performed better than the 100% concentration.

Long-Term Concrete Slab Tests

The purpose of the long-term concrete slab tests was to verify results of the fixed chloride tests with more realistic specimen design. In these tests, the slabs underwent a cyclic exposure (14 days wet / 7 days dry) ponded with a 15% NaCl solution. The concrete tested was Concrete 0. Each of the three CIAs was tested at 10 and 75% of the manufacturer's maximum recommended dose.

Appendix H gives the data for each slab. Table 2 gives average currents, times-to-initiation, corrosion coverage by post-test examination, and chlorides measured at the steel bar depth. It is seen that each of the three CIAs provided improvement in corrosion performance, as compared to the control (no CIA) for the long-term slab tests.

The average time-to-initiation and coupled current data indicate that only the control, 10% CIA-A, and 10% CIA-C initiated corrosion during these tests. Even at the 10% levels, the coupled current and time to initiation were improved over the control. CIA-B did not initiate corrosion even at the 10% level.

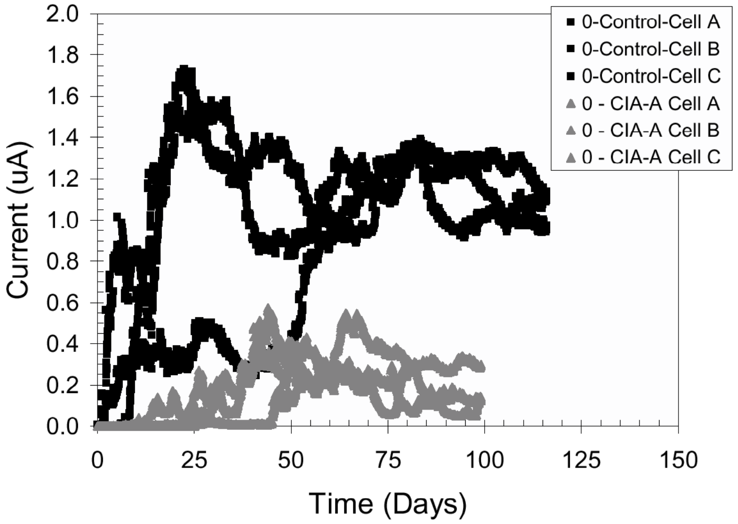


Figure 9. Performance of CIA-A in presence of a crack.

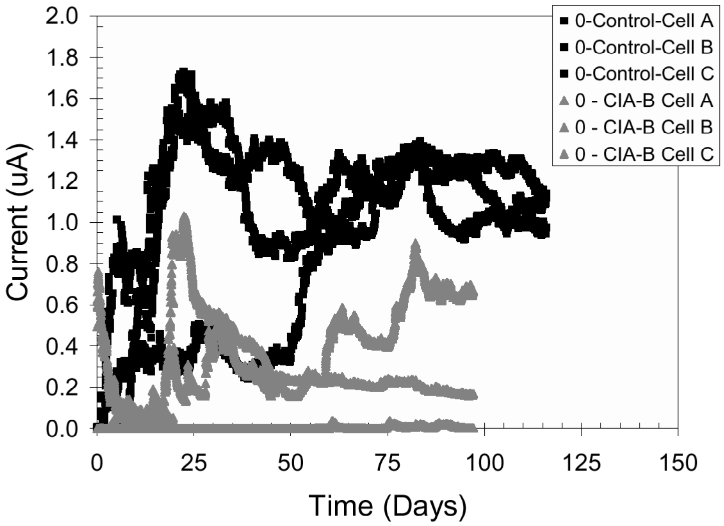


Figure 10. Performance of CIA-B in presence of a crack.

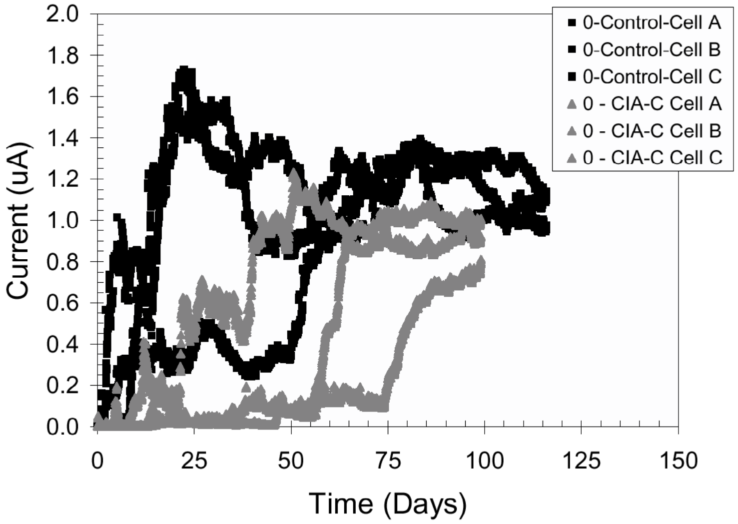


Figure 11. Performance of CIA-C in presence of a crack.

Table 2. Average data for long-term concrete slab tests. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA (%)	Time to Initiation (days)	Current (μA)	Corrosion Coverage[(b)] (%)	Chlorides (lb/yd^3)
0	None	None	265	31	25	14
0	A	10	354	9	10	13
0	A	75	(a)	1	<10	9
0	B	10	(a)	1	<10	11
0	B	75	(a)	<1	0	8
0	C	10	306	18	10	12
0	C	75	(a)	1	<10	6

(a): Did not initiate corrosion.

(b): Post test examination.

The total exposure period was approximately 450 days. For those slabs which initiated corrosion (Table 2), the length of time following corrosion initiation was only 100 to 200 days. The post test examination qualitatively confirmed the coupled current findings. Note that a coverage of <10% is negligible corrosion, but some very slight corrosion may have occurred. The coverage of 0% indicates that no signs of attack are present.

Figure 12 shows the plot of current versus time for 10% CIA-C Slab D. These data are shown as an example. The current reported for this condition was 20 μA, although the current was continuing to increase as a function of time. The cyclic fluctuation in the data corresponds to the wet-dry cycle period. The reported data are an average of the recorded current.

The chlorides measured at the steel level indicate that, at the high CIA concentrations, there was a measurable decrease in the chloride concentration for each CIA. For each of CIA concentration, the chlorides measured at the steel bar level at the end of the exposure period are quite high considering that corrosion was not initiated for several conditions, thus indicating a high threshold for corrosion initiation.

Chloride Penetration Tests

The purpose of the chloride penetration tests was to determine the effective diffusion coefficient for chloride penetration into the concrete with and without CIA. The test procedure used was AASHTO T-259-80 [1993]. Chloride measurements were made after both 90 and 365-day exposure periods. Both concretes and all three CIAs (each at four concentration levels) were tested.

The plots of chloride concentration versus distance into the concrete and the diffusion coefficient calculations are given in Appendix I. Effective diffusion coefficient was calculated by assuming a simplified model based on Fick's law for steady-state diffusion in a semi-infinite solid; it was further assumed that diffusion coefficient (D_{eff}) is independent of position x.

The boundary conditions for the equation are:

$C=C_0$ for $t>0$ at $x=0$
$C=0$ for $t=0$ at $x>0$

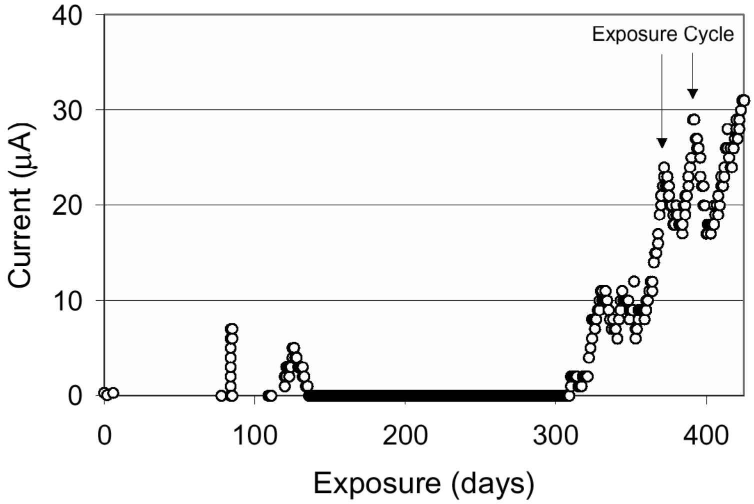


Figure 12. Coupled current versus time for 10% CIA-C Slab D.

The solution to the diffusion equation takes the form:

$$c(x,t) = c_o\left[1 - erf\left(\frac{x}{2\sqrt{D_{eff}\,t}}\right)\right] = c_o\left[1 - erf(z)\right]$$

The chloride measurements at different depths (x) were entered into an Excel spreadsheet. Then, a built-in Excel analysis tool (SOLVER) was employed for a simultaneous fitting of the two variables (D_{eff} and C_0) to the experimental curve. However, SOLVER experienced considerable difficulties in fitting an experimental curve with only 3-4 datapoints. The algorithm requires the ‘seed’ values for both variables; once the values were entered, SOLVER attempted to fit the experimental curve in such a way that the sum of the squares of the actual/fitted pair variances were minimized (see Sum (error)^2 entries in Table I3, Appendix I). Given the small number of datapoints, the program ran through the iteration process, but ‘froze’ one the parameters and changed only the other one. The message “SOLVER could not find a feasible solution” typically appeared, and the resulting fitted curve was clearly far from the ‘best fit’ one.

In view of the above, it was decided to make further improvements to the fit manually, using the combination of SOLVER solutions and GOAL SEEK function (works similarly to SOLVER, but changes only one specified variable at a time). The fitting was deemed finished when the sum of the squared variances was either at a certain value (typically 1 or less), or further changes to the fitting parameters did not lead to any marked improvements to the fit. The resulting values of C_0 and D_{eff} are shown in Table I3.

In Figures 13 through 15, the diffusion coefficients from the multiple slabs are averaged to provide a mean value for each condition for Concretes 0 and 1. Error bars are provided at plus and minus one standard deviation (typically 3 slabs per condition). Analysis shows that the addition of silica fume decreased the effective diffusion coefficient by 72%.

For CIA-A in Concrete 0, the diffusion coefficients increased as the CIA concentration increased. For CIA-A 100% maximum recommended dosage in Concrete 0, effective diffusion coefficient was 57% greater than the no-CIA condition. In Concrete 1, the diffusion coefficient remained slightly greater than the no-CIA condition at all CIA-A concentrations.

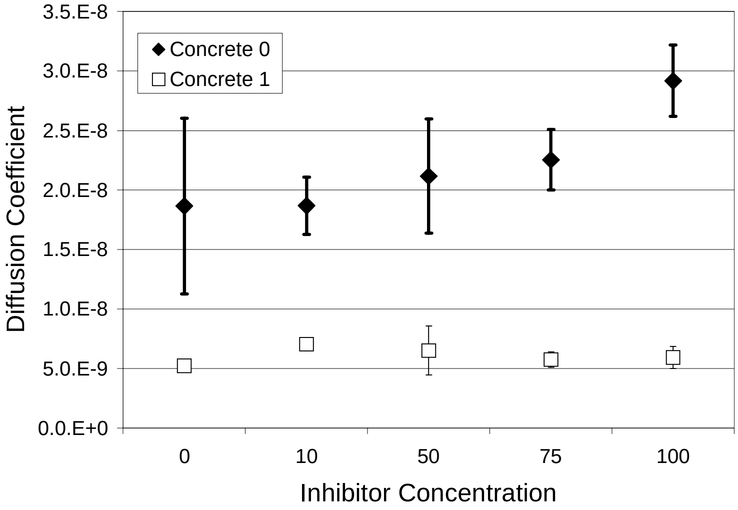


Figure 13. Average effective diffusion coefficients (cm^2/s) for CIA-A.

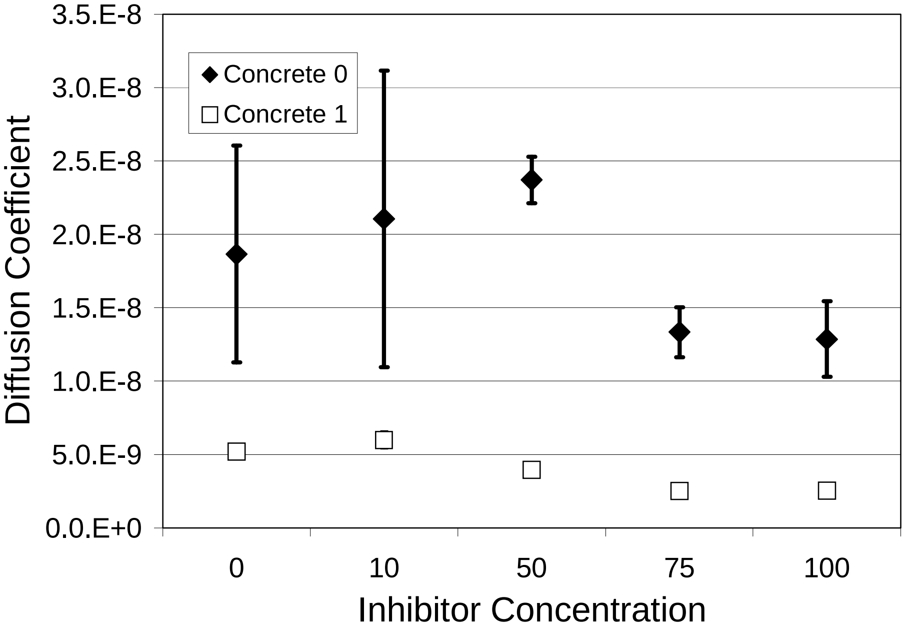


Figure 14. Average effective diffusion coefficients (cm^2/s) for CIA-B.

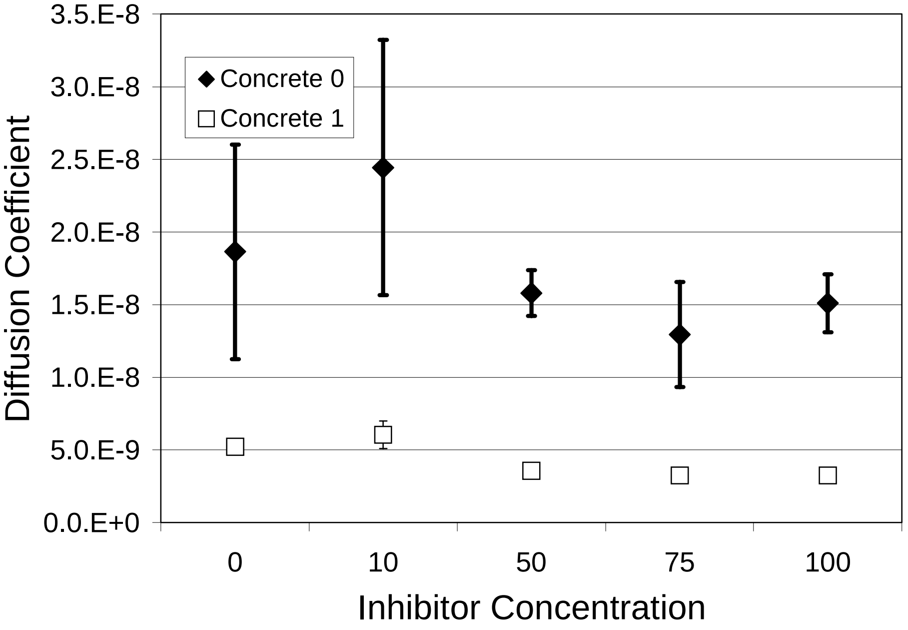


Figure 15. Average effective diffusion coefficients (cm^2/s) for CIA-C.

Figure 14 shows that a maximum in the effective diffusion coefficient was observed at intermediate CIA-B concentrations for Concrete 0. At 100% maximum recommended dosage, the diffusion coefficient is 30% less than the controls. For Concrete 1, CIA-B exhibited a decrease in diffusion coefficient as a function of CIA concentration (a decrease of 51% at 75 and 100% of recommended maximum dosage of CIA-B).

Figure 15 shows that CIA-C exhibited a maximum in the effective diffusion coefficient at intermediate CIA-C concentrations in Concrete 0. At 100% maximum recommended dosage, the decrease in diffusion coefficient for CIA-C was 19%. For Concrete 1, the diffusion coefficient was decreased by approximately 38% at higher CIA-C concentrations.

Figures 16 and 17 show the chloride concentrations as a function of depth in the concrete for the control slabs for Concrete 0 and 1, respectively. The difference in the diffusion coefficient is evident in the chloride-achieved concentrations. The tabulated data for all conditions tested are given in Appendix I.

Concrete Properties

The purpose of the concrete property tests was to determine whether CIA additions cause a detrimental effect on concrete properties. Properties in both the fresh and hardened concrete states were examined.

Compressive Strength

Compressive strength tests were performed according to ASTM C39. Tests were performed following 28-day (50 and 100% CIA concentrations) curing and after 360-days (100% CIA concentration). Appendix J contains all of the concrete property data.

Tables 3 and 4 give the mechanical property data for the 28-day tests for Concrete 0 and 1, respectively.

There were only minor effects of CIA additions on compressive strength at the CIA dosages tested. For Concrete 0, all corrosion inhibiting admixtures slightly increased strengths when a 100% of recommended dose of inhibitor was used as compared to 50% of the maximum dose (CIA-B containing specimens had compressive strengths slightly lower than that of the controls).

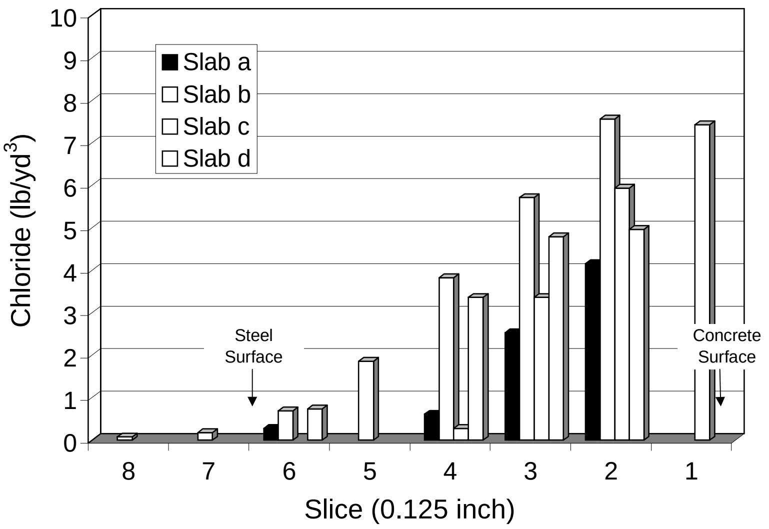


Figure 16. Chloride concentration as a function of depth for Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3], [1 mm = 0.039 in]

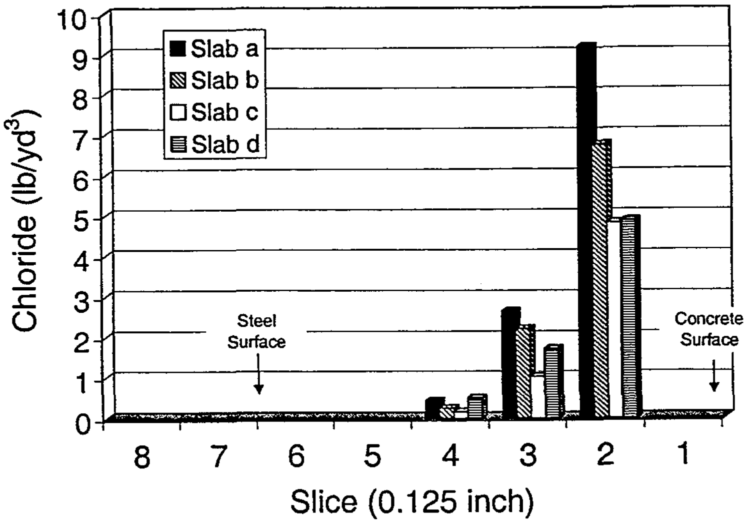


Figure 17. Chloride concentration as a function of depth for Concrete 1. [1 kg/m^3 = 1.67 lb/yd^3], [1 mm = 0.039 in]

Table 3. Mechanical strength properties for Concrete 0. [1 kPa = 0.145 psi]

Condition	Compressive Strength (psi) 28-Day	Modulus of Elasticity (psi x 10^6) 28-Day	Flexural Strength (psi) 28-Day
Control	6,540	5.18	820
CIA-A 50%	7,030	5.58	830
CIA-A 100%	7,280	4.94	680
CIA-B 50%	6,250	4.98	750
CIA-B 100%	6,450	5.47	820
CIA-C 50%	6,840	5.63	830
CIA-C 100%	7,030	5.79	820

Table 4. Mechanical strength properties for Concrete 1. [1 kPa = 0.145 psi]

Condition	Compressive Strength (psi) 28-Day	Modulus of Elasticity (psi x 10^6) 28-Day	Flexural Strength (psi) 28-Day
Control	8,090	5.79	1,040
CIA-A 50%	9,760	6.19	990
CIA-A 100%	10,000	5.72	950
CIA-B 50%	8,370	5.81	1,020
CIA-B 100%	8,500	5.90	1,040
CIA-C 50%	8,990	6.18	960
CIA-C 100%	8,920	5.97	1,015

As expected, the silica fume-containing Concrete 1 specimens had greater strength than the Concrete 0 specimens. A similar trend was observed for the Concrete 1 specimens as for the Concrete 0 specimens, i.e., the higher dosage additions of CIAs tended to increase the compressive strength readings (with an exception of CIA-C, for which the strength remained unchanged). The CIA-containing specimens had higher compressive strength than the control (in particular, the addition of CIA-A led to a 20% increase [10,000 versus 8,090 psi]).

Flexural Strength

Flexural strength tests were performed according to ASTM C78. Tests were performed following 28-day (50 and 100% CIA concentrations) curing and after 360-days (100% CIA concentration). Appendix J contains all of the concrete property data. Tables 3 and 4 give the mechanical property data for the 28-day tests for Concrete 0 and 1, respectively. Concrete 0 (no silica fume) had a flexural strength of 820 psi (5,655 kPa); silica fume containing concrete had a strength of 1,040 psi (7,172 kPa). CIA-A at a 100% dose seemed to slightly lower the test results for both concretes; other inhibitors added at a 100% dose level did not cause any notable change in the flexural strength.

Modulus of Elasticity

Modulus of elasticity tests were performed according to ASTM C469. Tests were performed following 28-day (50 and 100% CIA concentrations) curing and after 360-days (100% CIA concentration). Appendix J contains all of the concrete property data. Tables 3 and 4 give the mechanical property data for the 28-day tests for Concrete 0 and 1, respectively. Concrete 0 (no silica fume) had a modulus of 5.18×10^6 psi (3.57×10^7 kPa); the control concrete with silica fume addition had a modulus of 5.78×10^6 psi (3.99×10^7 kPa). The data suggest that none of the CIAs caused any significant change in the modulus when compared to the control specimens.

Electrical Resistivity

One-, 7-, 28-, 90-, 180-, and 360-day electrical resistivity tests were performed for all conditions. Appendix K contains the detailed resistivity data. Concrete 0 (no silica fume) with no CIA added had a 180-day resistivity of 8,368 ohm-cm; Concrete 1 (with silica fume) with no CIA added had a 180-day resistivity of 49,383 ohm-cm. Therefore, silica fume had a significant effect on concrete resistivity, as expected. The data for the CIA additions indicate that CIA-A slightly decreases resistivity, while CIA-B and CIA-C slightly increase resistivity. It is assumed that an increase in resistivity is a benefit for corrosion resistance.

Other Properties

Slump (ASTM C143), air content (ASTM C457), and time of set (ASTM C403) have been measured for all conditions. The results are given in Appendix L. Although some variation was observed, the CIAs had no significant effects on these concrete properties that could not be adjusted for in the final mix.

CHAPTER **3**

INTERPRETATION, APPRAISAL, APPLICATION

PREDICTIVE MODEL

A primary objective of this research was to develop a test procedure for evaluating CIA performance in relatively short-term laboratory tests and to develop a corresponding performance criterion (or criteria) that would serve as a quantitative means of qualifying CIAs for use in concrete structures.

The first step in this process was to develop a life prediction model. The sole purpose of this model was to facilitate the application of the performance criterion for a “typical” concrete structure and was not designed to include all the necessary variables that might be required to predict life of an actual structure in a specific location. In addition, the case of cracked structures was not addressed in this prediction model. The following description outlines the proposed model.

The following three rate processes govern life of a concrete structure:

1. Rate of chloride diffusion into the concrete.
2. Rate of corrosion, once initiated.
3. Rate of development of damage.

The life of a structure can be divided into the following three phases.

- Phase I – Corrosion Initiation.
- Phase II – Corrosion Propagation without Damage.
- Phase III – Damage to Structure.

The following information is required for the above model and can be determined by the testing protocol utilized in this research.

- Phase I – Corrosion Initiation.
  1. Effective diffusion coefficients for chloride penetration into concrete.
  2. Critical chloride concentration required to initiate corrosion.

- Phase II – Corrosion Propagation without Damage.

  1. Corrosion rate as a function of chloride concentration.
  2. Chloride concentration as a function of time.
  3. Cumulative corrosion necessary to initiate concrete damage.

- Phase III – Damage to Structure.
  1. Damage versus cumulative corrosion relationship (at a minimum, cumulative corrosion to cause cracking).

The following provides the predictive modeling analyses for the three CIAs studied in this research.

Model Justification

For actual structures, conditions are relatively complex and not easily specified, especially for non-marine applications. Salting occurs periodically, and the length of time the chloride solution remains on the concrete surface can vary greatly. The number of salt applications also varies. Therefore, on any given structure, there are multiple salt applications, each lasting an unspecified time, depending on the weather conditions. During these periodic intervals, additional salt (at some unspecified concentration) is available on the surface of the concrete to penetrate into the concrete. At all other times, the salt within the concrete can redistribute itself, but no additional salt is available at the concrete surface. It would appear that any model designed to provide the basis of a general performance criterion would require a “grossly average” condition to be established for applying the performance criterion. In addition, ease of calculation must be considered for the application of a performance criterion.

In this model, the following average scenario is used. It is assumed that salt applications occur at the beginning of the structure’s life. Following the first application and for all times thereafter, the concentration of salt at the structure’s surface is equivalent to 18 kg/m^3 (30 lb/yd^3). This significantly simplifies the calculation of chloride concentration as a function of time. One of the assumptions is that once cracking occurs, the corrosion rate is not affected by the presence of cracks.

Chloride Concentration

Effective diffusion coefficients for chloride penetration into concrete are used to predict the chloride concentration at the steel surface as a function of time for the specified conditions. This is required to establish the Phase I – Phase II transition and to calculate corrosion rates in Phase II and III.

Table 5 gives the diffusion coefficients for the CIAs at maximum recommended dosage and the controls (no CIA) for the two concretes used in this study. The chloride concentration at a given depth (x) and at a specified time (t) is given by:

$$c(x,t) = c_o\left[1 - erf\left(\frac{x}{2\sqrt{D_{eff}t}}\right)\right] = c_o[1 - erf(z)]$$

where C_o is the concentration of chlorides at the concrete surface (assumed to be 30 lb/yd^3), D_{eff} is the effective diffusion coefficient, and erf is the error function. It should be noted that the value of 18 kg/m^3 (30 lb/yd^3) is based on field experience and not on that achieved during laboratory testing. For this model, the chloride concentrations were calculated as the chloride concentration at a reinforcement bar depth of 64 mm (2.5 in).

Figures 18 through 20 show the predicted chloride concentrations versus time for the effective diffusion coefficients provided in Table 5.

Figure 18 shows the effect of the decrease in diffusion coefficient for the silica fume added Concrete 1. The chloride concentrations represent the chloride concentration at a reinforcement bar depth of 2.5 in (6.4 cm). The addition of silica fume has a significant impact on the chloride concentration at the steel reinforcement level.

Figure 19 shows the effect on the chloride concentration of CIA additions to Concrete 0. The data indicate that the addition of CIA-B and CIA-C decreases chloride at the steel level when compared to no-CIA Concrete 0. CIA-A had a greater diffusion coefficient and resulted in a higher chloride concentration at the steel level at a given time.

Figure 20 shows the effect on the chloride concentration of CIA additions to Concrete 1. CIA-B and CIA-C decreased the diffusion coefficient and resulted in a decrease in the chloride concentration at a given time. CIA-A had only a slight effect compared to no-CIA condition.

Phase I – Corrosion Initiation

The data input necessary to establish the time prior to corrosion initiation is (1) the chloride concentration as a function of time (see Figures 18 through 20, above) and (2)

Table 5. Effective diffusion coefficients for concretes with and without CIAs.

CIA	Diffusion Coefficients (cm^2/s)	
	Concrete 0	Concrete 1
No CIA	1.86E-08	5.21E-09
CIA-A	2.92E-08	5.92E-09
CIA-B	1.29E-08	2.54E-09
CIA-C	1.51E-08	3.25E-09

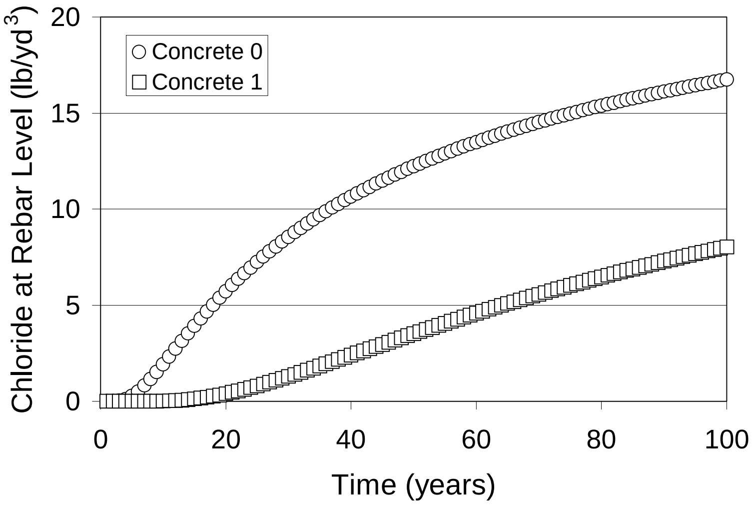


Figure 18. Chloride concentration versus time for Concretes 0 and 1 (no CIA). [1 kg/m^3 = 1.67 lb/yd^3]

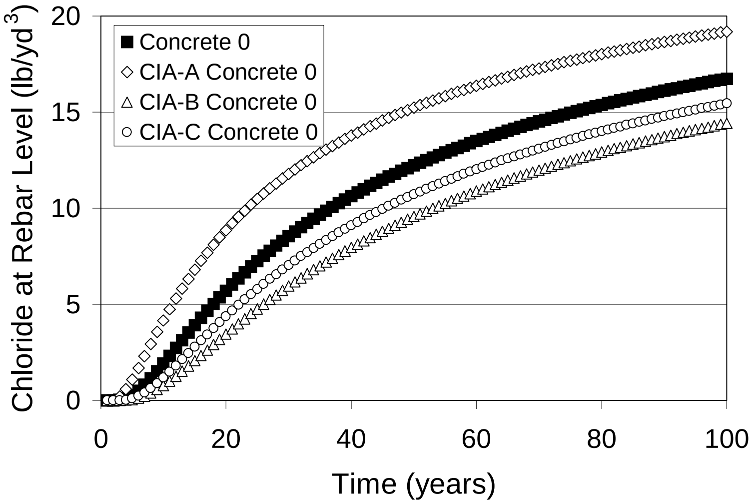


Figure 19. Chloride concentration versus time for the three CIAs in Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3]

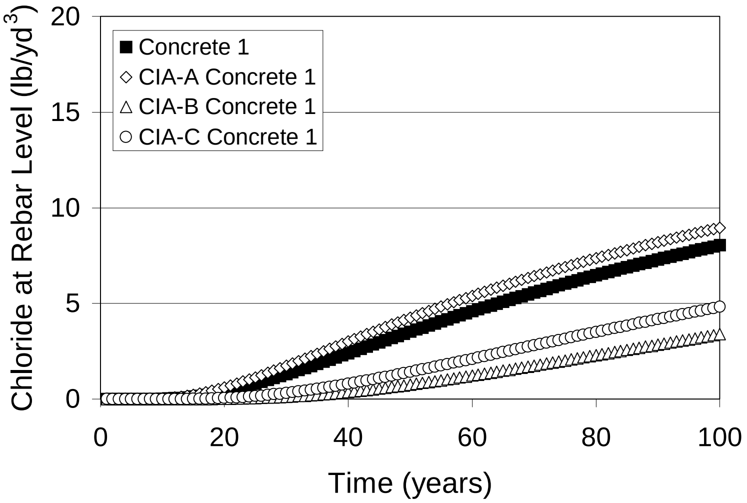


Figure 20. Chloride concentration versus time for the three CIAs in Concrete 1. [1 kg/m^3 = 1.67 lb/yd^3]

the chloride threshold for corrosion initiation. Chloride threshold tests were performed specifically for the purpose of establishing a chloride threshold for corrosion. These data were presented in Table 1. However, because chloride thresholds were not determined for all CIAs, these data were not used in the model.

Chloride threshold for corrosion initiation can also be estimated from the fixed chloride tests. In the fixed chloride tests, the linear regression fit to the data can be extrapolated to negligible corrosion rates to establish the threshold. It should be recalled that the fixed chloride tests were performed at 98% relative humidity (no ponding after chloride was introduced), while the threshold tests were continuously ponded with chloride. Table 6 gives the equations for corrosion rate as a function of chloride for the conditions tested in Concrete 0. Sufficient data were not available to calculate equations for Concrete 1.

The equations shown in Table 6 for the Control, CIA-A, and CIA-C were the linear regression fit to the Control, 100% CIA-A dosage, and 100% CIA-C dosage data, respectively (see Figures 3 and 5). The R^2 values for the linear regression fits were 0.20, 0.22, and 0.24, respectively. The R^2 value is a measure of the variance between the regression fit and the data. The larger the R^2 value the better the data fit ($R^2 = 1$ is perfect fit). From the R^2 values, it is seen that there was significant scatter in the data. The highest R^2 value (0.83) was observed for the 75% dosage CIA-A data. The CIA-B linear regression produced the following equation for the 100% CIA-B dosage: $y = 0.0190X + 0.0521$; $R^2 = 0.13$. For comparison, the 50% CIA-B data resulted in the following linear fit equation: $y = 0.0309X – 0.0873$; $R^2 = 0.45$ (sufficient range of chloride values was not achieved to use the 75% dosage data). The equation for the 100% CIA-B produces unrealistic chloride threshold predictions upon extrapolation to low corrosion rate values. Figure 4 indicates that the magnitudes of corrosion rate versus chloride concentration data for the 100% and 50% CIA-B data are similar at intermediate chloride levels (they are equal at approximately 7.2 kg/m^3 [12 lb/yd^3]). To permit analysis in a similar manner as the other CIAs, the equation given in Table 6 for CIA-B is based on the 50% CIA-B data. It should be noted that, overall, the 50% CIA-B data represented slightly improved corrosion performance compared to the 100% CIA-B data.

Using equations from Table 6, the chloride threshold can be estimated as the chloride concentration at negligible corrosion rate. Assuming the same value for

Table 6. Equations for linear fit to corrosion rate versus chloride concentration. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

			y = mx + b		
Concrete	CIA	% CIA	m	b	R^2
0	None	None	0.0319	-0.0776	0.20
0	CIA-A	100	0.0180	-0.0795	0.22
0	CIA-B	50*	0.0309	-0.0873	0.45
0	CIA-C	100	0.0127	-0.0118	0.24

y = Corrosion rate in mpy.
x = Chloride concentration in lb/yd^3.
m = slope
b = y intercept
R^2 = Percent of variance explained by regression fit.
*: 50% CIA data used due to scatter in 100% and 75% CIA data. 50% CIA data was similar in magnitude to 100% CIA data (Figure 4).

corrosion rate at initiation as the threshold tests (corrosion rate of 0.1 mpy [0.0025 mm/yr] was used to define when corrosion initiated), Table 7 presents the predicted chloride threshold from the “Fixed Chloride Tests” as compared to the “Chloride Threshold Tests” for Concrete 0 (no chloride threshold data was available for Concrete 1). Although these chloride threshold values are greater than typically stated in the literature, there is reasonably good agreement between the two test methods. The high chloride threshold values may, in part, be due to the chosen level of corrosion rate defined as the threshold for corrosion (i.e., 0.1 mpy [0.0025 mm/yr]). It is believed that the LPR measurements performed on these small test cells are significantly more sensitive than LPR measurements for concrete field applications or for large slab tests. If the level of corrosion rate for establishing the threshold is lowered (either 0.05 or 0.025 mpy [0.0013 or 0.00063 mm/yr]), the threshold concentration is closer to expected values (see Table 8).

For the life prediction model, the chloride threshold is determined by the “Fixed Chloride Tests” regression analyses as the chloride concentration necessary to give a corrosion rate of 0.05 mpy (0.0013 mm/yr). From Table 8, the no-CIA, CIA-B and CIA-C conditions gave chloride threshold values between 2.4 and 3.0 kg/m^3 (4 and 5 lb/yd^3), and CIA-A gave a chloride threshold value of 4.2 kg/m^3 (7.0 lb/yd^3). Therefore, CIA-A was the only inhibitor to significantly increase the chloride threshold for Concrete 0.

Appendix M contains the tabulated results of chloride versus time (plotted in Figures 18 through 20); estimated time-to-corrosion initiations are given in Table 9. The following life predictions are based on Concrete 0 data. The no-CIA condition and CIA-A have similar times-to-initiation (15 years). CIA-A had a greater chloride threshold than the no-CIA condition, which increased time-to-corrosion, but it also had a greater diffusion coefficient, which reduced time to corrosion initiation. These two factors cancelled each other for CIA-A when compared to no-CIA.

CIA-B and CIA-C had slightly greater chloride thresholds and smaller diffusion coefficients than the no-CIA condition, resulting in longer time-to-corrosion (23 and 22 years, respectively).

Phase II – Corrosion Propagation without Damage

The three parameters necessary to calculate Phase II life are (1) corrosion rate as a function of chloride concentration (equations given in Table 6), (2) chloride concentration as a function of time (Figures 18-20), and (3) cumulative corrosion

Table 7. Comparison of chloride threshold concentration based on fixed chloride tests versus chloride threshold tests. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	% CIA	Fixed Chloride (0.1 mpy) (lb/yd^3)	Threshold Test (lb/yd^3)
0	None	None	5.6	6
0	CIA-A	100	9.8	10
0	CIA-B	50*	6.1	>5
0	CIA-C	100	8.8	7

*: 50% CIA data used due to scatter in 100% and 75% CIA data. 50% CIA data were similar in magnitude to 100% CIA data (Figure 4).

Table 8. Comparison of chloride threshold for lower specified levels of corrosion rate. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	% CIA	Fixed Chloride (0.1 mpy) (lb/yd^3)	Fixed Chloride (0.05 mpy) (lb/yd^3)	Fixed Chloride (0.025 mpy) (lb/yd^3)
0	None	None	5.6	4.0	3.2
0	CIA-A	100	9.8	7.0	5.6
0	CIA-B	50*	6.1	4.4	3.6
0	CIA-C	100	8.8	4.9	2.9

*: 50% CIA data used due to scatter in 100% and 75% CIA data. 50% CIA data were similar in magnitude to 100% CIA data (Figure 4).

Table 9. Prediction of the time to initiation of corrosion (Phase I) [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Chloride Threshold (lb/yd^3)	Time to Corrosion Initiation (yr)
None	4.0	15
CIA-A	7.0	15
CIA-B	4.4	23
CIA-C	4.9	22

necessary to initiate damage to concrete. In a recently completed FHWA study, the cumulative corrosion to cause visible cracking the length of the reinforced steel was estimated to be 0.05 to 0.18 mm (2 to 7 mil) depending on the concrete mix. *(Thompson, N. G. and Lankard, D. R., "Optimization on Concretes and Repair Materials for Corrosion Resistance," FHWA, Final Report on DTFH61-93-C-00028, 1999.)* For this study, 0.05 mm (2 mil) of cumulative corrosion is assumed to initiate cracking.

The corrosion rate and cumulative corrosion as a function of time can be calculated by combining chloride concentration as a function of time and the corrosion rate as a function of chloride concentration. These data are tabulated in Appendix N. Figure 21 gives the plot of cumulative corrosion versus time for Concrete 0 conditions of no-CIA, CIA-A, CIA-B, and CIA-C.

The end of Phase II life (defined as 0.05 mm [2 mil] of cumulative corrosion) is indicated in Figure 21, and is 30 years for no-CIA, 33 years for CIA-A, 41 years for CIA-B, and 44 years for CIA-C.

Phase III – Damage to Structure

The two parameters necessary to calculate Phase III life are (1) cumulative corrosion versus time (Figure 21) and (2) cumulative corrosion to end life. The cumulative corrosion to end Phase III life is a difficult parameter to establish because it depends on many variables, including maintenance procedures during the life of a structure, concrete variables, structure loading, etc. Also, what constitutes "end of life" is important. In this scenario, it is assumed that no maintenance is performed and that Phase III life is the point in time when sufficient damage has occurred to require significant repairs. The value of 0.25 mm (10 mil) of cumulative corrosion over the entire structure is assumed to define the end of Phase III life.

From Figure 21 (and Appendix N), the Phase III life span is determined. Table 10 summarizes the life predictions based on the above model. The Phase III life is 58 years for no-CIA, 73 years for CIA-A, 74 years for CIA-B, and 96 years for CIA-C. Table 11 gives the percent improvement in predicted life extension by the CIAs for Concrete 0.

PERFORMANCE CRITERIA

One of the primary goals of this research was to provide performance criteria for qualifying prospective CIAs based on laboratory testing protocol. The final

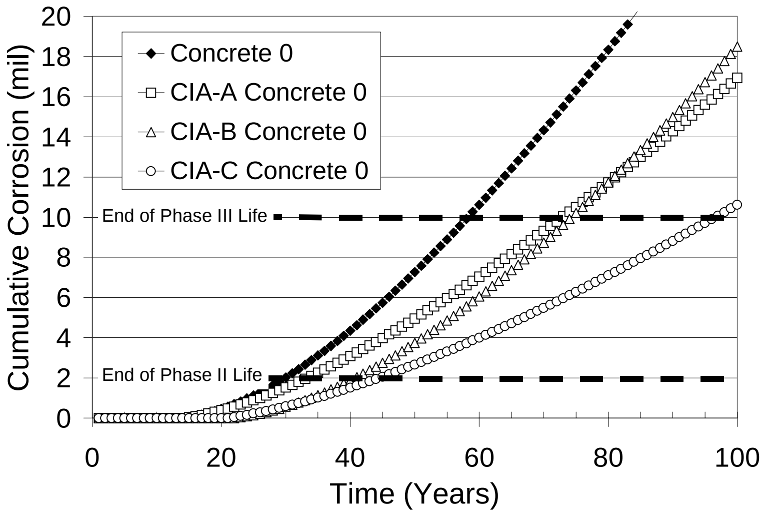


Figure 21. Cumulative Corrosion versus time for Concrete 0. [1 mm = 39 mil]

Table 10. Summary of life predictions for Concrete 0.

CIA	End of Phase I (years)	End of Phase II (years)	End of Phase III (years)
None	15	30	58
CIA-A	15	33	73
CIA-B	23	41	74
CIA-C	22	44	96

Table 11. Percent improvement in life extension by CIAs in Concrete 0.

CIA	End of Phase III (years)	Percent Improvement Over No CIA
None	58	
CIA-A	73	26%
CIA-B	74	28%
CIA-C	96	66%

recommended testing protocol is given in Appendix O. The performance criteria are based on predictive model and presented below.

For the Concrete 0, (high quality concrete with inert aggregate and a water-cement ratio of 0.40), discussed above, the performance predicted for no-CIA conditions was relatively good (58 years). For these conditions, performance criteria for acceptance of a CIA based on a 75-year life (original project goal) would correspond to a CIA that increased the expected life (based on the above model) by 29% over the 58-year life for no-CIA. A 75-year life goal as a performance criterion (or any other number of years) is not realistic, because variations in concrete batching and in laboratory procedures can have a significant impact on the life prediction results. Also, the selection (somewhat arbitrary) of the model parameters (e.g., definition of structure life) has a considerable effect on life predicted by the model. Therefore, a more appropriate criterion would be a percent life extension with respect to the control.

It is proposed that the performance criteria for qualifying a CIA designed to mitigate corrosion of reinforcing steel in concrete be as follows:

Criterion 1. The CIA should provide an improvement over the base (no-CIA) condition with respect to the predicted life by a minimum of 25 percent.

Criterion 2. Increase in life must be due to one or both of the following: (1) increased chloride threshold for initiation of corrosion or (2) a decrease in the slope of the regression fit of corrosion rate versus chloride concentration.

Criterion 3. The CIA should provide some improvement in corrosion performance for cracked concrete.

Criterion 4. The CIA should not adversely affect the concrete properties in such a manner that pertinent specifications are not met. At a minimum, these should include compressive strength, flexural strength, modulus of elasticity, slump, time-to-set, and air content. Other properties that were not specifically studied in this project but could have a significant effect on concrete performance are air distribution and shrinkage.

For the three CIAs tested in this program, Table 12 summarizes the above-performance criteria with the addition of the diffusion coefficient for comparison. CIA-C exceeds the first criterion of greater than 25% life extension as predicted by the model.

Table 12. Summary of performance criterion. [1 mm/yr = 39 mpy],[1 kg/m^3 = 1.67 lb/yd^3]

CIA	Life Extension Over No CIA (Percent)	Chloride Threshold (lb/yd^3)	Chloride-Corrosion Rate Slope (mpy/[lb/yd^3])	Diffusion Coefficient ($10^{-8}cm^2/s$)	Cracking Performance versus No CIA
None		4.0	0.032	1.86	
CIA-A	26%	7.0	0.018	2.92	Good*
CIA-B	28%	4.4	0.031	1.29	Good*
CIA-C	66%	4.9	0.013	1.51	Marginal**

*: Significant improvement in performance with CIA added.

**: Marginal improvement in performance with CIA added.

CIA-B exceeds the 25% goal by 3 percentage points and CIA-A exceeds it by 1 percentage point.

The second criterion requires an improvement in either the chloride threshold or the slope of the corrosion rate-chloride curve. CIA-A meets both parts of the second criterion. CIA-C met the corrosion rate-chloride slope improvement and showed a slight increase in the chloride threshold as compared to the control. CIA-B yielded a similar slope for the corrosion rate-chloride curve as the control and only a very slight increase in the chloride threshold. This indicates that most of the CIA-B benefit was due to a decrease in the chloride penetration rate.

The third criterion, to improve performance in the presence of cracks, was met by all three CIAs, with only marginal improvement for CIA-C.

Table 13 summarizes the four performance criteria for the CIAs tested. None of the CIAs tested passed all four criteria without at least one marginal performance rating. CIA-A passed all, except Criterion 1, where only marginal improvement was found (one percentage point over at 26%). CIA-B was marginal with respect to Criterion 1 (28%) and marginal for Criterion 2 (marginal for the chloride threshold part of Criterion 2 and did not pass the decrease in slope part of Criterion 2). CIA-C was marginal relative to Criterion 3.

It should be noted that all three CIAs provided an extended life based on the model calculations. However, by definition, “Corrosion Inhibiting Admixture” must extend life by mitigating the corrosion process. Also, it should be noted that the statistical reproducibility of the “Fixed Chloride Test,” which was used for the model predicting, was poor. Improved reproducibility is required for application of the criteria, especially in view of the marginal performance of the tested CIAs. Small changes in the regression equations used in the life predictions could have a significant impact on qualification of the CIAs based on the proposed criteria.

Table 13. Summary of Pass/Fail for performance criteria.

CIA	Criterion 1 25% Increase in Life	Pass Either/Or Criterion 2 Increase Chloride threshold	 Criterion 2 Decrease Slope of CR vs Cl*	Criterion 3 Improvement in Cracked Concrete	Criterion 4 Not Adversely Affect Properties
CIA-A	Marginal (+)	Pass	Pass	Pass	Pass
CIA-B	Marginal (+)	Marginal (+)	Fail	Pass	Pass
CIA-C	Pass	Marginal (+)	Pass	Marginal (+)	Pass

*: Corrosion rate vs chloride concentration.

COMPARISON OF LIFE PREDICTION MODEL WITH LONG-TERM CONCRETE SLAB TEST RESULTS

Overall Comparison

The model predictions utilized the following test methods:

- The short-term "Fixed Chloride Tests" for (1) corrosion rate versus chloride concentration relationship and (2) chloride threshold concentration for corrosion initiation.

- The AASHTO T 259 "Chloride Penetration Tests" for calculating the effective diffusion coefficient used to estimate chloride concentration versus time.

The fixed chloride test protocol involved a severe drying and ponding cycle to drive chloride into the test specimen, followed by a 98% relative humidity exposure with no further ponding. The AASHTO T 259 test involved an initial drying followed by continuous ponding with a 3% NaCl solution (no cyclic exposure). In comparison, the "Long-Term Concrete Slab Tests" consisted of a "7-day wet / 14-day dry" cycle exposure (ponding with a 15% NaCl solution).

Other differences in the tests procedures were: (1) the long-term slab had built-in macrocell corrosion couple (reinforcing steel exposed to chloride ponding coupled to steel ponded with no-chloride solution), while the steel in the fixed-chloride tests was exposed to uniform chloride concentration, and (2) the exposure time for the fixed-chloride tests was relatively short (2 to 3 months) compared to 14 to 15 months for the long-term slab tests.

Although caution has been mentioned in comparison of the CIAs performance from one method to another, it is inevitable within a given test program. For the long-tem concrete slab tests, CIA-B was the best performer. For the model prediction, CIA-C provided the greatest increase in life.

Final analysis indicates that all three CIAs enhanced performance based on both the life prediction model and the long-term concrete slab tests pointing to an overall agreement between the two test methodologies. Such agreement might be regarded as verification of the life prediction model. However, further analysis of the performance details is also of interest and includes determination of the root cause of the extended life as calculated by the model. The following factors are involved in the determining life extension:

- The threshold level of chloride concentration necessary to initiate corrosion.
- The rate of corrosion following initiation of corrosion.
- The effective diffusion coefficient for chloride penetration into the concrete.

Each of these factors will be examined individually through the comparison of the life-prediction model calculations to the results of the long-term concrete slab tests.

Chloride Threshold

Table 14 shows the comparison between the long-term concrete slab tests and the life prediction model results utilizing extrapolation of the fixed chloride tests. Note that the long-term concrete slab data pertains to 75% CIA concentrations and that the fixed chloride test-based estimates were for 100% CIA-A, 100% CIA-C, and 50% CIA-B dosages (see “Phase I Corrosion Initiation” for discussion of the use of 50% versus 100% CIA-B data for establishing chloride threshold concentration). From the long-term concrete slab data, a chloride threshold concentration can be inferred, which is either greater than (corrosion did not initiate) or less than (corrosion initiated) a chloride concentration measured following the exposure period.

From Table 14, the fixed chloride data indicate that only CIA-A provided a significant increase in chloride threshold over the control (no CIA). The long-term slab tests indicate a chloride threshold greater than 5.3, 5.0, and 3.9 kg/m^3 (8.8, 8.3, and 6.5 lb/yd^3) for CIA-A, CIA-B, and CIA-C, respectively. These values are large and would be expected to be a significant increase over the control (no-CIA) condition; however, the control can only be characterized with an upper limit (<8.2 kg/m^3 [<13.8 lb/yd^3]), which prohibits further quantification of the possible CIA benefits.

Rate of Corrosion Following Initiation

For the long-term concrete slab tests, the rate of corrosion following initiation can be examined by comparing the measured coupled currents. Table 15 provides these data for the conditions in which corrosion initiated (control, 10% CIA-A, and 10% CIA-C). Corrosion did not initiate for 10% CIA-B.

Table 14. Comparison of chloride threshold concentration data. [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Chloride Treshold From Long-Term Slab Test* (lb/yd^3)	Chloride Threshold From Fixed Chloride Tests** (lb/yd^3)
None	<13.8	4.0
A	>8.8	7.0
B	>8.3	4.4
C	>6.5	4.9

* Chloride measured at steel level at the conclusion of the test.

** Chloride estimated upon extrapolation to a corrosion rate of 0.05 mpy.

Table 15. Comparison of rate of corrosion data.

CIA	Rate of Corrosion Following Initiation Long-Term Slab Test* (μA)	Rate of Corrosion Following Initiation Fixed Chloride Tests**
None	31	--
A	9	Decreased
B	--	Similar
C	18	Decreased

* Coupled current following corrosion initiation.

** Based on comparison of corrosion rate versus chloride concentration curve to control (Table 6).

For the model predictions, the comparison of the corrosion rates calculated with the linear fit equations from Table 6 was used as an indication of the level of corrosion activity following initiation. Note that the CIA concentrations for the fixed chloride analysis were different than those used for the long-term concrete slab tests (see discussion above for “Chloride Threshold”).

Data for CIA-A and CIA-C indicate a decrease in corrosion rate (versus control) for both the long-term concrete slab tests and the fixed chloride tests used in the life prediction model. A complete data set was not available for CIA-B and, therefore, a comparison could not be made. Although, a quantitative comparison can not be made due to the differences in CIA concentrations, there is a similar trend for both test methods.

Chloride Penetration Rate

For the long-term slab tests, a comparison of the final chloride concentration at the steel surface serves as a relative measure of the chloride penetration rate (effective diffusion coefficient). In the model predictions, the AASHTO T 259 chloride penetration test method was used to measure effective diffusion coefficients. Table 16 summarizes these results. For both test methods, CIA-B and CIA-C indicate a lower penetration rate as compared to the control. The long-term slab tests indicate a lower penetration rate for CIA-A, while data from the AASHTO T 259 method (used in the life prediction model) indicate a higher penetration rate.

One possible reason for the discrepancies discussed above (chloride threshold and chloride penetration) between the long-term concrete slab tests and the life prediction model results is that the CIAs respond differently to the different exposure conditions (e.g., cyclic ponding, constant high-humidity exposure, continuous ponding). It is possible that numerous wet-dry cycles have an effect on the CIA action within the concrete matrix and pore structure.

For development of a test method, simulation of field conditions is a high priority. A cyclic exposure is present in actual applications, but a seven day continuous ponding would not be representative of a real situation. For substructures, continuous ponding does not exist, and the exposure is more of a high-to-low relative humidity exposure. A revised general testing protocol and associated performance criteria for qualifying CIAs are presented in Appendix O. This revised practice takes into account the exposure issues discussed above and standardizes on a cyclic exposure condition.

Table 16. Comparison of rate of chloride penetration data. [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Chloride Penetration Data from Long-Term Slab Test* (lb/yd^3)	Chloride Penetration Data from AASHTO T259** ($10^{-8}cm^2/s$)
None	13.8	1.86
A	8.8	2.92
B	8.3	1.29
C	6.5	1.51

* Chloride concentration at steel level.

** Effective diffusion coefficient.

CHAPTER **4**

CONCLUSIONS AND SUGGESTED RESEARCH

The following conclusions are based on the results of the testing program presented above.

1. The review of the literature identified three basic inhibitors, which are available as commercial inhibitors and which have shown beneficial performance in at least some of the studies performed prior to the present project.

   - Rheocrete 222 and Rheocrete 222+ (Master Builders). A water-based combination of amines and esters.

   - DCI and DCI-S (W.R. Grace). Calcium nitrite-based admixture.

   - Ferrogard 901, a modified version of Armatec 2000, (SIKA), and MCI 2000 (Cortec). A blend of surfactants and amine salts (specifically, dimethyl ethanolamine [DMEA], also referred to as alkanolamines or amino alcohols [AMA]) in a water carrier.

2. Concrete properties of compressive strength, flexural strength, modulus of elasticity, electrical resistivity, slump, time-to-set, and air content were not adversely affected by the addition of the three CIAs tested in this study. (That is, standard concrete admixtures could easily compensate the effects.)

3. In general, the effect of CIAs on the diffusion coefficient was consistent for both the standard and silica fume containing concrete.

4. The effect of CIA on corrosion of steel bar in concrete is dependent on the concrete mix, concentration of the CIA, chloride concentration, and exposure conditions.

5. A predictive model was developed that utilizes relatively short-term laboratory test results to predict structure life, thereby permitting quantitative measure of the benefits of a CIA. The model was designed to make a life prediction for the purpose of applying performance criteria, not to predict life of an actual structure. The life prediction model was applied to the control concrete (no CIA) and CIA-

containing concretes studied in this project. The model predicted significant life extension for all three CIAs examined in this study. Also, input into the model permits relative benefits to be attributed to one of three causes: (1) increase in the chloride threshold for corrosion initiation, (2) decrease in the slope of corrosion rate versus chloride concentration equation and, (3) decrease in the effective diffusion coefficient.

6. Performance criteria were developed that permit the qualification of a candidate formulation as a corrosion-inhibiting admixture (CIA). All four of the following performance criteria should be met for the formulation to qualify as a CIA:

   Criterion 1. The CIA should provide an improvement over the base (no-CIA) condition with respect to the predicted life by a minimum of 25 percent.

   Criterion 2. Increase in life must be due to one or both of the following: (1) increased chloride threshold for initiation of corrosion or (2) a decrease in the slope of the regression fit of corrosion rate versus chloride concentration.

   Criterion 3. The CIA should provide some improvement in corrosion performance for cracked concrete.

   Criterion 4. The CIA should not adversely affect the concrete properties in such a manner that pertinent specifications are not met. At a minimum, these should include compressive strength, flexural strength, modulus of elasticity, slump, time-to-set, and air content. Other properties that were not specifically studied in this project but could have a significant effect on concrete performance are air distribution and shrinkage.

7. Both the life prediction model (based on short-term tests) and the long-term slab tests indicated that all three CIAs provided improved performance compared to the control (no-CIA) condition. However, some discrepancies exist regarding the cause of the improved behavior when comparing the results of the two methodologies.

8. A recommended practice was developed for testing and qualifying a proposed admixture as a CIA. The testing protocol includes a modified procedure of the

fixed-chloride tests performed in this program and the performance criteria discussed above.

9. The discrepancies discussed in Conclusion 7 above, required changes in the testing protocol to account for the proposed causes of the discrepancies. Although a draft practice has been proposed in Appendix O, further testing and validation of the procedures would be required prior to its submittal as a standard practice.

10. Contributing to the problems of validation of the recommended practice is the poor statistical reproducibility for the “Fixed Chloride Tests.” This was attributed to difficulties in incorporating chlorides into the specimens (likely due to CIA interactions) and due to problems with chloride measurements.

Follow-on work is required to finalize the testing protocol proposed in this study. The following discusses the reasons why additional work is required.

1. The testing protocol was altered based on the results of the tests performed in this project. The primary change included cyclic exposures for all test methodologies. This is expected to produce a more consistent set of results among test methods, since it is believed that CIAs respond differently to wet-dry cyclic exposures than to continuous ponding/humidity exposures. Because the testing protocol was altered, the new testing protocol must be examined to establish its performance.

2. The statistical reproducibility of the fixed chloride tests was less than desired. It was proposed that the CIAs interfered with the chloride incorporation method used. This was identified early in the experimental program and adjustments were made to the method. Based on the final results, an improved methodology is required. The method used meets all of the necessary criteria for a method, i.e. (i) concrete should be cured prior to chloride incorporation to provide some time for the CIA to interact with the steel surface (film forming, etc.), (ii) the chloride concentration should be controllable, (iii) the chloride concentration should be constant over time, and (iv) the chloride concentration should be uniform over the entire steel surface. Relatively minor alterations in the chloride incorporation method should improve the reliability of the method to achieve the desired chloride concentration. These alterations could include (i) incorporating the chlorides at a higher temperature to enhance migration of the chlorides, (ii) permitting a longer time for migration of chlorides, and (iii)

utilizing multiple drying cycles instead of a single wet-dry cycle. An improved chloride incorporation method should greatly improve reproducibility of the data.

3. The method used for chloride sampling included trying to collect the smallest possible sample of chloride very near the steel surface. Although reasonable, in practice, taking a small sample of a heterogeneous material such as concrete resulted in a sample that could be high in cement phase and low in aggregate phase producing misleading chloride concentration results when reported in total weight of concrete. Further, the proportion of cement phase versus aggregate phase sampled was not consistent, leading to inconsistent chloride concentration results. Although, chloride sampling was repeated for several specimens, this could have contributed to the poor reproducibility of the test results.

4. Although comparisons between chloride measurement methodologies were performed, it is believed that more consistent results could be achieved by using the titration method (AASHTO T260-82) as opposed to the ion-probe method (Germann “Rapid Chloride Test”) primarily used in this study. It is proposed to use the titration method in any future work.

APPENDIX A

EXPERIMENTAL WORK PLAN

APPENDIX **A**

EXPERIMENTAL WORK PLAN

PREDICTIVE MODEL

One of the goals of the laboratory test plan was to propose performance criteria for assessing the service life of a structure up to 75 years. Therefore, the testing protocol was designed to produce the data required for meeting and establishing such criteria. The first step in defining criteria for 75-year service life is to establish a model for predicting life. The model was based on parameters that can be experimentally determined. The model selected was based on cumulative corrosion (cumulative metal loss of reinforcing steel), since corrosion rate is a measurable quantity in the laboratory and in the field. The following is a description of the model and a discussion of the information required to utilize the model.

The life prediction model was designed to permit the application of performance criteria for establishing the effectiveness of corrosion inhibitor admixtures (CIA). The criteria must be based on relatively short-term laboratory tests and not field measurements. The model was not designed to predict life of a bridge structure. Furthermore, the life prediction model was designed to predict inhibitor effectiveness for uncracked concrete only. Cracking was addressed in a separate analysis for inhibitor effectiveness. Other factors such as concrete quality or maintenance practices were not considered in the life prediction model.

The useful life of a concrete structure with respect to corrosion is governed by the rates of the three processes given below.

1. Rate of chloride ion penetration into the concrete.
2. Rate of corrosion (once initiated).
3. Rate of the development of cracking/spalling damage.

The life prediction model considered corrosion initiated by chloride permeation and not other mechanisms of initiation, such as carbonation. Cracking of the concrete, which was considered a special case, can provide an additional path for chlorides to the steel surface. Although the effectiveness of inhibitors to mitigate corrosion for the specific case of cracked concrete was part of the laboratory test plan, the predictive model did not address this condition.

The life prediction model considered three phases in the life of a structure.

Phase I – Corrosion Initiation. In Phase I, corrosion rate of the reinforcing steel is negligible and a critical chloride threshold for corrosion initiation has not yet been attained.

Phase II – Corrosion Propagation without Damage. In Phase II, the critical chloride threshold for corrosion initiation is exceeded. Corrosion is occurring at some rate, however, no damage of the concrete is apparent. The beginning of damage to the concrete signifies the end of Phase II.

Phase III – Damage to the Structure. In Phase III, corrosion along with damage continues until the damage becomes so great that significant maintenance of the structure is required.

Phase I – Corrosion Initiation

During Phase I, diffusion of chlorides has not yet resulted in concentrations above a critical threshold concentration for corrosion, i.e. the reinforcing steel remains passive and corrosion rates are minimal. To establish the time period for Phase I, the critical data required are (1) the penetration rate for chlorides and (2) the critical threshold concentration of chlorides to initiate corrosion. There are two approaches to establishing this time-to-corrosion initiation: (1) the use of relatively short-term testing to establish the above parameters or (2) the performance of a long-term test in which corrosion rate is monitored until the initiation of corrosion is detected.

Phase II – Corrosion Propagation without Damage

During Phase II, corrosion proceeds at some rate and chloride permeation continues to increase the chloride concentration over time. The continuous increase in chloride concentration will likely increase the corrosion rate as a function of time. This phase ends when damage begins. One approach for estimating the time period for Phase II is to utilize diffusion data to predict chloride concentration at the steel surface and to perform short-term laboratory tests to estimate corrosion rate as a function of chloride concentration. A simple equation would then predict cumulative corrosion versus time. Cumulative corrosion is determined by integrating corrosion rate over time. As before, a second approach is to perform long-term tests and measure corrosion rate versus time. With either method, a relationship between cumulative corrosion and damage is required.

Phase III – Damage to the Structure

In Phase III, damage to the structure proceeds until it results in cracking and spalling of the concrete and significant maintenance is required. As for Phase III, a damage versus cumulative corrosion dependence has to be known to successfully predict the end of useful life. Furthermore, the damage-corrosion relationship is expected to be a function of the mechanical properties of the concrete.

EXPERIMENTAL CONCRETES

Two concretes were selected for use in the laboratory program, which were identified as 9050-1 (Concrete 0) and 9050-2 (Concrete 1). The mixture proportions for the concretes are shown in Tables A1 and A2. Concrete No. 9050-1 was an air-entrained Portland cement concrete containing 423 kg/m^3 (705 lb/yd^3) of Type I cement. Concrete No. 9050-2 was an air-entrained concrete containing Type I Portland cement and 7% by weight silica fume in the cementitious component. In both concretes, the air content was maintained at 6 ± 1½% and the w/c (or w/cm) was maintained at 0.40. The cementitious material paste content was 30% by volume in both concretes. Both concretes contained the same fine aggregate (natural quartz sand) and the same coarse aggregate (9.5-mm

Table A1. Concrete mix design for Concrete 0 (no silica fume). [1 m^3 = 1.308 yd^3 = 35.3 ft^3], [1 kg/m^3 = 1.67 lb/yd^3]

Contract Constituent	lb. Constituent Per yd^3 of Concrete		Density of Constituent, lb/ft^3		ft^3 of Constituent per yd^3 of Concrete
Type I Portland Cement	705	÷	196.6	=	3.586
Sidley Quartz Sand (SSD)	1429	÷	165.4	=	8.640
Sidley No. 8 Quartz Aggregate (SSD)	1412	÷	163.5	=	8.638
Water	282	÷	62.4	=	4.510
Air Content (6 ± 11/2%)					1.620
	3828				27.003

Table A2. Concrete mix design for Concrete 1 containing silica fume. [1 m3 = 1.308 yd3 = 35.3 ft3], [1 kg/m3 = 1.67 lb/yd3]

Contract Constituent	lb. Constituent Per yd^3 of Concrete		Density of Constituent (lb/ft^3)		ft^3 of Constituent per yd^3 of Concrete
Type I Portland Cement	644	÷	196.6	=	3.276
Silica Fume	52	÷	143.5	=	0.362
Sidley Quartz Sand (SSD)	1429	÷	165.4	=	8.640
Sidley No. 8 quartz Aggregate (SSD)	1414	÷	163.5	=	8.648
Water	278	÷	62.4	=	4.455
Air Content (6 ±11/2%)					1.620
	3817				27.001

[3/8-in] maximum size silica aggregate from the same source as the fine aggregate phase).

Portland Cement

The Portland cement was a Type I (ASTM C150) manufactured by Southwestern Portland Cement Company, Fairborn, Ohio. The alkali content of the cement (expressed as Na_2O equivalent) was 0.75.

Silica Fume

The silica fume was manufactured by Elkem Materials, Pittsburgh, Pennsylvania, and was identified as Product EMS965. The silica fume was an undensified product with a loose bulk density under 12 kg/m^3 (20 lb/ft^3). The product was purchased through the Sika Corporation, Marion, Ohio, as their product identified as Sikacrete 950P. Silica fume was added at 7% by weight of cementitious material.

Sand

The silica sand was produced by R.W. Sidley, Inc., Painesville, Ohio, and was identified as their No. 400 Silica Sand. This sand conformed to the gradation requirements of ASTM C 33. It had a specific gravity of 2.65 and a fineness modulus of 2.84. The SiO_2 content of the sand was in excess of 99%.

No. 8 Coarse Aggregate

The coarse aggregate was pure quartz from the same source as the fine aggregate with a maximum size of 9.5 mm (3/8 in). The aggregate gradation conformed to the ASTM C 33 specification.

Admixtures

In addition to the corrosion-inhibiting admixtures that were used in the project, the concretes also contained three other chemical admixtures including: an air-entraining admixture, a conventional water reducing/set retarding admixture, and a high-range water reducer.

The air-entraining agent was Sika Chemical Corporation's Sika AEA-15, a concentrated solution of a sodium-salt-type soap. This product meets requirements of ASTM C 260 and AASHTO M-154.

The conventional water reducing/set-retarding admixture was Sika Chemical Corporation's Plastiment. Plastiment is a metallic salt of hydroxylated carboxylic acid. It meets the requirements of ASTM C 494 Type D, and AASHTO M-194 Type D.

The high-range water reducer was Mighty 150, which meets the requirements of ASTM C 494 Type F Chemical Admixture.

CONCRETE BATCHING, MIXING, AND CURING

Forty-five to 50 batches of concrete were required for the laboratory program. Batch sizes ranged from 0.034 to 0.065 m^3 (1.2 to 2.3 ft^3). All concrete mixes were prepared in a 0.085 m^3 (3.0 ft^3) capacity rotary drum mixer (Muller Manufacturing).

The addition of constituents to the mixer in the mixing period followed the guidelines outlined in ASTM C 192-90a, "The Standard Practice for Making and Curing Concrete Test Specimens In The Laboratory". Trial batches were made to determine the admixture levels required to obtain the desired slump (4 ± 1 in.) and air content (6 ± 1 ½%).

The air-entraining admixture was placed directly on the sand in the mixer. The high-range water reducer and conventional set retarding/water-reducing admixture was blended with the mix water.

Manufacturers' recommendations were followed with regard to the addition of the corrosion--inhibiting admixtures.

Slump, unit weight, and air content were measured on all of the concretes. Failure to achieve the desired slump level or air content was cause for rejection of that particular batch.

The batch water content was adjusted to reflect significant water contributions coming from the admixtures.

During the first 24-hour period after casting, specimens were maintained in a condition to assure that no moisture loss occurred. After removal from the molds, the specimens were stored in a moist room meeting the requirements of ASTM C 511-95, "The Standard Specification for Moist Cabinets, Moist Rooms, And Water Storage Tanks Used In The Testing Of Hydraulic Cements And Concretes".

CORROSION-INHIBITING ADMIXTURES

The CIAs examined were three of the commercially available CIAs. For purposes of this report, the CIAs tested were given designations CIA-A, CIA-B, and CIA-C.

The primary emphasis was to develop a testing protocol for evaluating CIA effectiveness. Therefore, it was necessary to have a range of inhibitors with different mechanisms of corrosion inhibitors. The three commercially available CIAs selected above represent such a range of mechanisms.

Each CIA was tested at four concentrations. The concentrations were established through discussions with the inhibitor manufacturer. It is desired to test a fairly broad range of CIA additions. The basic guideline for selecting the inhibitor concentration was as follows.

- Level 1: 10% of recommended maximum concentration.
- Level 2: 50% of recommended maximum concentration.

- Level 3: 75% of recommended maximum concentration.
- Level 4: 100% of recommended maximum concentration.

MECHANICAL AND PHYSICAL PROPERTIES

Concrete property data are important for two reasons: (1) certain critical concrete properties (in fresh and hardened state) should not be degraded by the addition of a CIA and (2) mechanical property data may be important in determining the damage relationships required to define the different phases of the life prediction model. The test methods to be performed are listed below:

- Compressive Strength (ASTM C 39).
- Flexural Strength (ASTM C 78).
- Modulus of Elasticity (ASTM C 469).
- Slump (ASTM C 143).
- Setting Time (ASTM C 403).
- Stability of the air void system (ASTM C457).
- Electrical Resistivity.

For the concrete property tests, two concretes and three CIAs were tested. Each CIA was tested at only two levels: an intermediate and maximum recommended concentration. The primary goal was to establish whether CIA additions detrimentally affect the concrete properties. Figure A1 gives the matrix of tests for the mechanical properties (compressive strength, flexural strength, and modulus of elasticity). Triplicate specimens were tested. Measurements were performed at 28 days and 360 days for the 100% of maximum concentration of the CIA and at 360 days for 50% of maximum concentration of the CIA. This gives a total of 66 specimens to be tested by each procedure.

Electrical Resistivity tests were performed for both concretes at all four levels of each of the three CIAs. The electrical resistivity tests were performed on duplicate specimens at 28 day and 360 day exposure periods. Figure A2 gives the test matrix for the electrical resistivity tests. Since the tests are non-destructive (same specimens tested at each of the two exposure periods), testing of duplicate specimens gives a total of 52 tests.

CHLORIDE DIFFUSION COEFFICIENT

Chloride diffusion data is important for estimating chloride concentrations at the reinforcing steel depth. Chloride penetration was measured using AASHTO Designation T-259-80 (1993). Both the standard period of 90 days and a one-year (365-days) test period were used. The longer time period was required to get measurable results for the low chloride diffusion coefficients expected for some of the concrete-inhibitor combinations examined in this study. A further modification to the standard procedure was that chloride concentrations were measured at approximately 3.2-mm (0.125-in) intervals down to the reinforcing steel depth. The test matrix shown in Figure A2 was

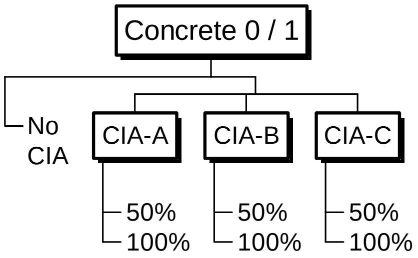


Figure A1. Test matrix for concrete property tests.

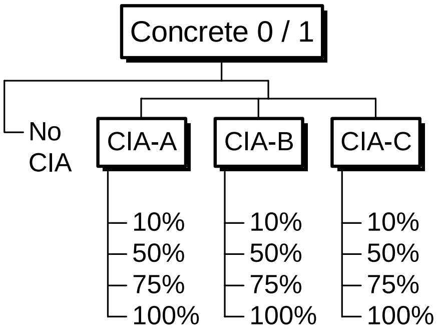


Figure A2. Test matrix for electrical resistivity and chloride diffusion tests.

used for the chloride permeability tests. Tests were performed for both concretes, the three CIAs, and all four CIA levels, in triplicate, for a total of 78 tests.

Effective diffusion coefficient was calculated based on the chloride measurements obtained from chloride diffusion tests. The calculation assumed a simplified model based on Fick's law for steady-state diffusion in a semi-infinite solid; it was further assumed that diffusion coefficient (D_{eff}) is independent of position x.

The boundary conditions for the equation are:

$C=C_0$ for t>0 at x=0

C=0 for t=0 at x>0

The solution to the diffusion equation takes the form:

$$c(x,t) = c_o\left[1-erf\left(\frac{x}{2\sqrt{D_{eff}t}}\right)\right] = c_o[1-erf(z)]$$

The chloride measurements at different depths (x) were entered into an Excel spreadsheet. Then, a built-in Excel analysis tool (SOLVER) was employed for a simultaneous fitting of the two variables (D_{eff} and C_0) to the experimental curve. However, SOLVER experienced considerable difficulties in fitting an experimental curve with only 3-4 datapoints. The algorithm requires the 'seed' values for both variables; once the values are entered, SOLVER attempted to fit the experimental curve in such a way that the sum of the squares of the actual/fitted pair variances be minimized (see Sum $(error)^2$ entries in Table I3). Given the small number of datapoints, the program ran through the iteration process, but 'froze' one the parameters and changed only the other one. The message "SOLVER could not find a feasible solution" typically appeared, and the resulting fitted curve was clearly far from the 'best fit' one.

In view of the above, it was decided to make further improvements to the fit manually, using the combination of SOLVER solutions and GOAL SEEK functions (works similarly to SOLVER, but changes only one specified variable at a time). The fitting was deemed finished when the sum of the squared variances was either at a certain value (typically 1 or less), or further changes to the fitting parameters did not lead to any marked improvements to the fit.

FIXED CHLORIDE TESTS

Knowledge of corrosion rate, as a function of chloride concentration, is required to establish the time of initiation of Phase II of the life prediction model. It is also necessary for estimating cumulative corrosion loss as a function of time. The following are important aspects of the experiments required to provide this information.

- The chlorides penetrate into the concrete by ponding following curing (not mixed in during batching).
- The chloride concentration at the steel surface is controlled at a desired level.

- A uniform chloride concentration is produced in the concrete that remains constant with time.
- Temperature and humidity are controlled variables.
- Specimen geometry allows chloride concentration to be measured within 2 to 3 mm (0.08" to 0.12") of the steel surface during post-test examination.

None of the published test methods conforms to these requirements. The majority of the test methods introduce chlorides into concrete by continuous or cyclic ponding. In these methods, chloride concentration increases with longer exposures. The test method used in this study was based on the premise that a majority of the free water is removed by vacuum drying at 38^{o}C (100^{o}F). The specimen is ponded with a solution containing a calculated amount of chlorides so that when the free water is replaced, the concrete will have the targeted chloride concentration. The specimens then are exposed to a constant humidity and constant temperature condition with no further chlorides added. The specimen design allows for chloride concentration at the steel surface to be measured during post-test examination. Although the test duration of three months may appear to be relatively short-term to provide the corrosion rate as a function of chloride and CIA concentration, this test method has been proven feasible for testing of high quality, low permeability concretes.

Test Specimen Design

Figure A3 shows the type of specimen configuration used in the fixed chloride tests. Figures A4 and A5 are photographs of the test specimens. The specimen design was such that the concrete environment was in contact with the cross-sectional face of the reinforcing steel bar rather than the circumferential area. The steel specimen was a #18 (57-mm [2.25-in] diameter) steel bar about 12.7-mm (0.5-in) long. Except for the cross-sectional area, which was in contact with the concrete, all other areas of the steel were sealed with coal tar epoxy. The total surface area of the steel in contact with the mortar was 2,548-mm^2 (3.98 in^2). The steel specimen was snugly fitted into a plastic mold (a PVC pipefitting) and 19 mm (0.75 in) cover of concrete was cast onto the cross-sectional steel surface. The specimen surface in contact with the mortar was given an 80-grit finish. Both the reference and counter electrode were fabricated from platinized niobium wire (niobium wire with 100-micron platinum coating).

All specimens were given a 28-day cure at room temperature by ponding with saturated calcium hydroxide solution.

Chloride Incorporation

The specimens were thoroughly dried after the 28-day curing cycle to facilitate chloride uptake. The following sequence was followed to prepare the samples before exposing them to the various environmental conditions:

1. Dry samples at 38^{o}C (100^{o}F) in a controlled temperature room for 7-days.
2. Apply epoxy concrete sealant (Sikagard) to joint between mortar and plastic mold.
3. Dry under vacuum at 38^{o}C (100^{o}F) for two additional-days.

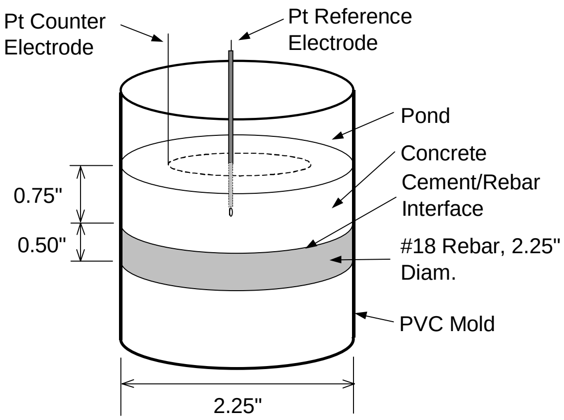


Figure A3. Specimen design for fixed-chloride corrosion tests. [1 mm = 0.039 in]

Figure A4. Photograph of individual fixed chloride test specimen.

Figure A5. Photograph of fixed chloride test specimens in humidity container.

4. Pond with 6 ml of the desired chloride solution for 14-days at 38^{o}C (100^{o}F). Ponding was carried out within an hour after completing step 3.
5. Place ponded specimens into autoclave and pressurize to 70 psi for 2-5 days.
6. Rinse off any excess chloride solution from the specimen surface pat dry with tissue and place them in the environmental chambers.

Weight checks after the drying cycle indicated that samples lost approximately 85 percent of the theoretical evaporable water.

Humidity Control In Environmental Test Chambers

Humidity control in the environmental test chambers was achieved by applying a layer of saturated salt solution at the bottom of the chamber. This method of humidity control is well established (ASTM E104 – "Maintaining Constant Relative Humidity By Means Of Aqueous Solutions"). Each of the test chambers (444 mm by 356 mm by 165 mm [17.5 in by 14 in by 6.5 in]) was filled with 1 L of the required salt solution, which gave an approximate 19-mm (0.75-in) layer of the solution at the bottom. The samples (24 in each chamber) were supported on a plastic grid above the surface of the solution. The actual humidity and temperature in each chamber was measured with a Thermo-hygrometer and were found to be within 2 to 3 percent (or degrees) of the desired values (98% relative humidity and 21^{o}C [70^{o}F]).

Measured Dependent Variables

The measured dependent variables in the fixed chloride tests were corrosion potential, corrosion rate, and chloride concentration at the steel surface. The potential of each specimen with respect to a copper/copper sulfate electrode (CSE) was made periodically during the exposure period.

Two measurement systems were used to more accurately determine the polarization resistance: PR Monitor 4500 by CC Technologies Systems, Inc. and Solartron Models 1255 and 1286 electrochemical impedance spectroscopy (EIS) measurement system. The PR Monitor 4500 performed a solution resistance measurement to correct the polarization resistance. The two measurement systems gave comparable results for corrosion rates.

Following breakdown of the test specimens, the concrete and the steel surface were separated to expose the concrete surface in contact with the steel. Concrete specimens for analysis were collected using a lathe to remove a few grams of concrete (approximately 1.6 mm [0.0625 in] thickness of the concrete near the steel surface). Chloride concentrations in the concrete test specimens were measured with a portable test kit manufactured by Germann Instruments.

Test Matrix for Fixed Chloride Tests

There are two important requirements for this test matrix.

1. The test matrix should provide a parameter that can be used to measure the reproducibility of the corrosion rate data.

2. The test matrix should maximize the predictive capability of the resulting regression model to estimate corrosion rate as a function of CIA level and chloride concentration.

After considering these two requirements and in view of the overall scope, the following test matrix was established. A full factorial matrix of tests (Figure A6) was performed utilizing duplicate specimens. For the control and 100% CIA conditions, there were four replicates for each of the 3 and 9 kg/m^3 (5 and 15 lb/yd^3) chloride concentrations. This gives 94 tests for each of the two concretes (188 total).

CHLORIDE THRESHOLD TESTS

Tests were performed in an attempt to establish the chloride threshold concentration for the initiation of corrosion for a given CIA level. This data was to be used in conjunction with the above described short-term corrosion tests to characterize the corrosion rate as a function of chloride and CIA concentration. The test specimens were identical to the "fixed chloride" tests. Following construction and 28-day curing of the test specimens, the specimens were ponded with a 15% NaCl solution. Periodic corrosion rate measurements were made until corrosion was initiated. Following initiation, the test specimen was destructively tested to measure the chloride concentration within 2 mm (0.0625 in) of the steel surface.

Figure A7 shows the test matrix for these tests. Both concretes were examined for each of the three CIAs at each of four levels. Utilization of duplicate specimens gave a total of 52 test specimens.

SIMULATED CRACK BEAM TESTS

These tests simulated the common case when corrosion of reinforcing steel in concrete is accelerated by the formation of surface cracks. These tests provided results with regard to the corrosion inhibiting capabilities of the CIAs in the presence of cracks down to the steel bar level. The design of a simulated crack beam specimen is shown in Figure A8. Figure A9 shows a photograph of the pre-cracked minibeams under test. The specimen design produces a 152-mm (6-in) long simulated longitudinal crack down to the top of the reinforcing steel. This design was selected because of the increased crack-reinforcing steel interface as compared to a transverse crack. The macrocell established by this relatively long crack-steel interface greatly enhanced the current measurement resolution over the very small (point) interface created by a transverse crack.

A crack was simulated by inserting a 0.25-mm (10-mil) thick shim aligned with the longitudinal dimension of the specimen and down to the surface of the top reinforcing steel specimen. The shim was inserted during casting and was pulled out after approximately 8 hours. In this manner, a uniform crack down to the reinforcing steel surface was simulated. The top surface of the slab was then ponded with 3% NaCl solution. The dependent variable measured was the coupled current between the top reinforcing steel specimen and the two steel bars in the bottom of the slab. This was accomplished using a zero-resistance ammeter. An increase of monitored macrocell

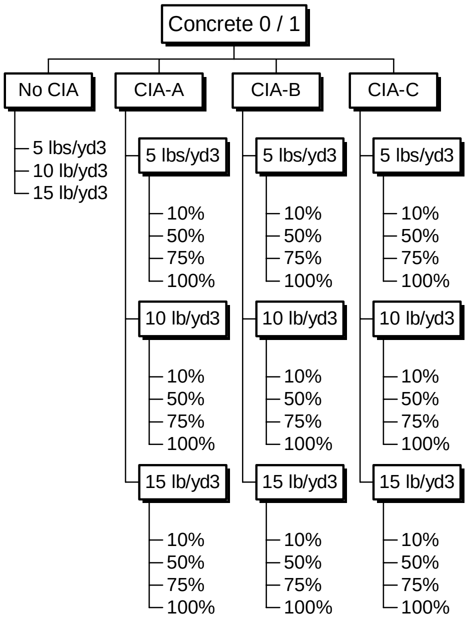


Figure A6. Test matrix for the fixed chloride corrosion tests. [1 kg/m^3 = 1.67 lb/yd^3]

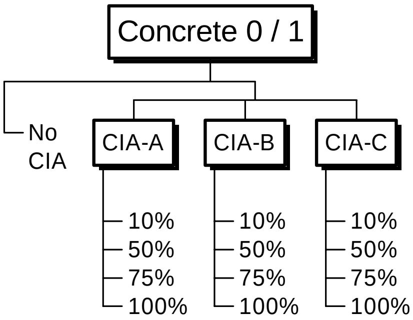
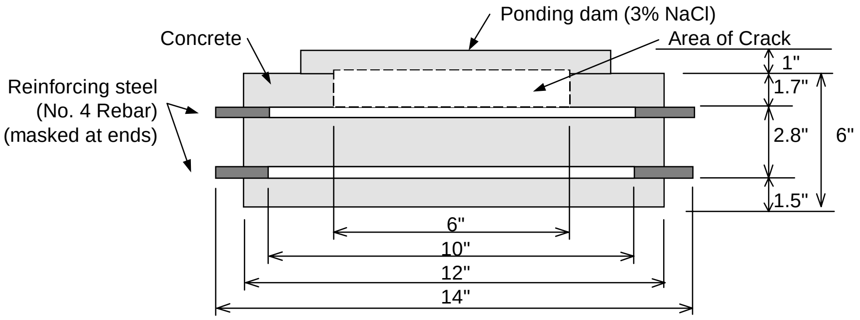
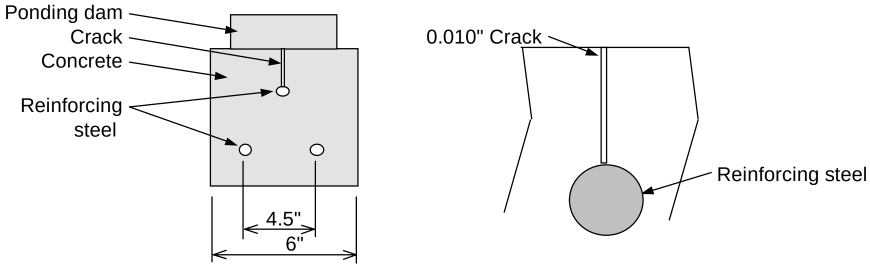


Figure A7. Test matrix for the chloride threshold tests.



a. Side view.



b. Front view.

c. Expanded front view of crack.

Figure A8. Schematic of the pre-cracked minibeam specimen. [1 mm = 0.039 in]

Figure A9. Photograph of pre-cracked concrete minibeam tests.

current between the top and bottom steel bars indicated the onset of active corrosion of the upper steel bar due to the aggressive action of chlorides.

The test matrix for the simulated crack beam specimens is shown in Figure A10. Only Concrete 0 (no silica fume) was tested. Two levels of CIA concentration (50 and 100%) were tested for each CIA. Testing of triplicate slabs gave a total of 21 pre-cracked test slabs to be tested.

LONG-TERM CONCRETE SLAB EXPOSURES

It was important to verify the predictions made by the short-term exposures for the fixed chloride tests through longer-term concrete slab exposures. In the slab exposures, only Concrete 0 (no silica fume) was used. It was assumed that meaningful data could not be collected within the allotted period of time if high performance concrete with silica fume was used in conjunction with CIA.

Current between the two reinforcing steel specimens coupled via a zero resistance ammeter (ZRA) was the primary measurement. Data was collected utilizing a computer acquisition system. LPR corrosion rate and potential measurements were made at the end of the exposure. Chloride concentration at the reinforcing steel depth was measured for each slab at the end of the test period.

Test Specimen Design

The standard specimen used in this task is shown in Figure A11. Figures A12 and A13 show photographs of the concrete slabs. The sides of the specimen were coated with an epoxy. The concrete surface above reinforcing steel bars ‘right’ and ‘center’ was ponded with a 15% NaCl solution. The concrete surface above reinforcing steel bar ‘left’ was ponded with deionized water. Chloride penetration into the concrete containing the right and center steel bars produced a corrosion couple between the center and left steel bars when electrically connected. The cover of the concrete to the top of the reinforcing steel bar was 19 mm (0.75 inch). This provided for a cover that was twice the maximum diameter of the large aggregate. By maintaining a minimum cover, chloride ingress down to the reinforcing steel level was maximized.

Exposure

The slab specimens were cured for a 28 days in 100% humidity room. They were exposed to the following cyclic ponding conditions at room temperature: ponded with 15% NaCl solution for 14 days and permitted to dry for 7 days.

Measurements

The following measurements were made to characterize the corrosion of the steel bars in the concrete slabs:

1. Coupled current measurements were made between the left and center steel bars for the concrete slab specimen (Figure A14). Coupled currents were measured periodically during the exposure period.

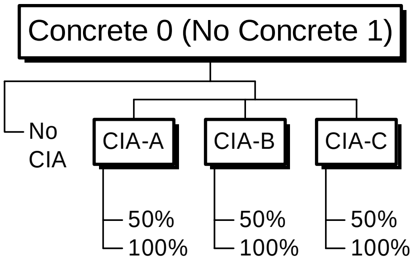


Figure A10. Test matrix for the pre-cracked minibeam tests.

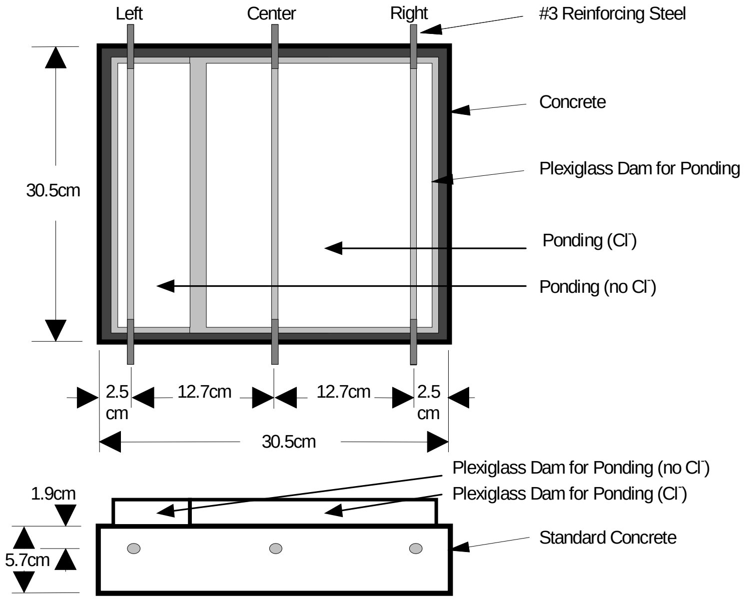


Figure A11. Schematic of the slab specimen. [1 mm = 0.1 cm = 0.039 in]

Figure A12. Photograph of concrete slab tests.

Figure A13. Photograph of individual concrete slab specimen.

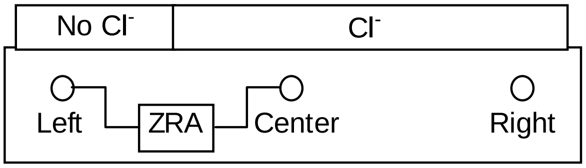


Figure A14. Coupled Current measurement using zero resistance ammeter (ZRA).

2. Linear polarization resistance (LPR) measurements were made for all three steel bars (uncoupled). The reference electrode is placed over the steel bar being measured and an adjacent steel bar is used as the counter electrode for the measurement (Figure A15).
3. Potential measurements were made with respect to a Cu/CuSO$_4$ (CSE) reference electrode performed in conjunction with the LPR measurements.

Acid-soluble chloride at the reinforcing steel depth was measured at the completion of the exposure period. Figure A16 shows the locations at which chloride analyses were performed.

Test Matrix

Figure A17 gives the test matrix for the slab tests. Four replicates of each condition were tested, giving a total of 28 slabs.

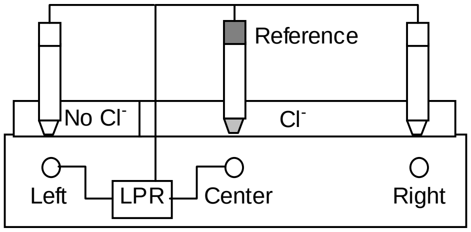


Figure A15. LPR corrosion rate measurement for the center steel bar.

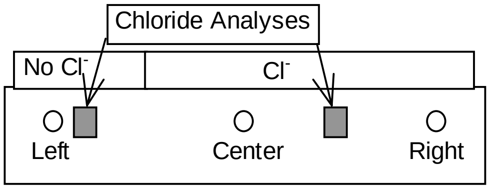


Figure A16. Schematic showing chloride analysis locations.

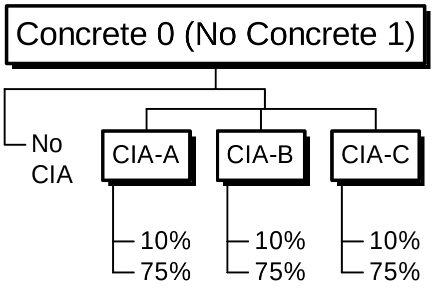


Figure A17. Test matrix for concrete slab exposures.

APPENDIX B

LITERATURE REVIEW

APPENDIX **B**

LITERATURE REVIEW

APPROACH

An extensive literature search of several in-house, industrial, and government databases was performed, including:

APILIT (1964-1996)
APIPAT (1964-1996)
CA SEARCH (1967-1996)
Ceramic Abstracts (1976-1995)
ChemEng and Biotec Abstracts (1970-1996)
CLAIMS (1950-1995)
COR-AB® (1980-1996)
Derwent WPI (1981-1995)
Ei Compendex Plus (1970-1996)
Energy SciTec (1974-1995)
European Patents (1978-1995)
FLUIDEX (1973-1995)
IAC Trade and Industry Database (1976-1996)
IHS International Standards and Specifications (1996)
INSPEC (1969-1996)
Kirk-Othmer Online (1995)
McGraw-Hill Publications (1985-1996)
Mechanical Engineering Abs (1973-1995)
METADEX® (1966-1996)
PAPERCHEM (1967-1996)
Pascal (1973-1996)
RAPRA Abstracts (1972-1996)
SciSearch® (1974-1996)
The Merck Index OnlineSM (1995)
TRIS (1970-1996)
World Transl. Index (1979-1996)
US Patent Database (1980-1996)

Vendors and manufacturers of inhibiting compounds (not necessarily for the concrete industry) were interviewed. Additionally, opinions from the panel members were solicited in the March 1996 Quarterly Report to NCHRP.

IDENTIFICATION OF INHIBITORS

This report primarily focuses on CIAs that are commercially available, although a brief section on "Other Inhibitors" is provided. This project is not attempting to develop new inhibitors, but is attempting to develop procedures for evaluating and qualifying corrosion-inhibiting admixtures. The vast majority of laboratory and field evaluations, beyond very preliminary screening in aqueous environments, have been performed on the commercially available CIAs. Also, this report is limited to corrosion inhibiting “admixtures” which, by definition, are the inhibitors added to the concrete during the batching process, and does not consider other methods of incorporating inhibitors into the concrete.

Commercially Available CIAs

Upon completion of the literature search and interviews, the following commercially available CIAs in four distinct classes were identified.

1. Rheocrete 222 and Rheocrete 222+, offered by Master Builders Inc., is a water-based combination of amines and esters. Rheocrete 222+ is a new and, supposedly, "improved" version.
2. DCI and DCI-S, offered by W.R. Grace and Company is a calcium nitrite-based admixture (about 30% concentration of the active ingredient). DCI-S contains a set-retarding admixture.
3. Armatec 2000, Ferrogard 901 (a modified version of Armatec) and MCI 2000 represent, a blend of surfactants and amine salts (specifically, dimethylethanolamine [DMEA], also referred to as alkanolamines or aminoalcohols [AMA]) in a water carrier. Cortec Corporation manufactures both Armatec and MCI; the Armatec version purportedly has proportions and concentrations of the ingredients slightly different from those for the MCI version. The newer CIA, Ferrogard 901, manufactured by Sika, has only recently been introduced to the market.
4. Catexol 1000 CI, offered by Axim Concrete Technologies, Inc., is a water-based solution of amine derivatives.

Generally, corrosion inhibitors are classified on the basis of their mechanism of protection, i.e., whether they affect anodic reaction, cathodic reaction, or, in some cases, both (mixed).

Rheocrete 222

Rheocrete 222, a water-based mixture of amines and esters, supposedly acts to form a protective organic layer on the steel bars (chelation process). [3, 4] This type of inhibitor is classified as "passive-active” system, meaning that besides mitigating iron dissolution it also provides an added benefit of "chloride screening". According to Nmai, "chloride screening", or act of reducing the ingress of chloride-ions into concrete, is facilitated by "lining the pores [in concrete] with chemical compounds that impart hydrophobic properties to the concrete". [2]

Inhibition of anodic and cathodic reactions is achieved by forming a layer of organic film, adsorbed to the surface of the reinforcing bar; the former supposedly acts as a physical barrier slowing all electrochemical reactions comprising the corrosion process. This type of inhibitor would inhibit both the anodic and cathodic reactions (mixed type).

DCI

The active ingredient of the DCI corrosion inhibitor, calcium nitrite, reportedly provides protection of reinforcing steel by facilitating the formation of oxide film on the steel surface via the following reaction and, therefore, falls into the category of anodic inhibitors. [1]

$$2Fe^{++} + 2OH^{-} + 2NO_2^{-} \rightarrow 2NO\uparrow + Fe_2O_3 + H_2O \qquad (1)$$

Nmai classifies this type of inhibitor as active as opposed to passive, where the term "passive" refers to systems that reduce the rate of chlorides ingression into concrete. [2]

Armatec/Ferrogard/MCI

The Armatec/Ferrogard/MCI brand inhibitors are organic-based and, according to the manufacturer(s), form a monomolecular film on both anodic and cathodic sites of the reinforcing steel. [5] Therefore, it can be classified as a mixed type inhibitor, with film forming capabilities much the same as described above for Rheocrete. Mäder and Bürge (both of SIKA Corp.) utilized X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS) to establish that amino alcohol (on which the Armatec/Ferrogard/MCI admixture is based) absorbs on the steel bar by forming a bond between "amino-functionality" hydroxide group on the steel surface.[6, 7] This can lead to the formation of insoluble iron oxide complexes which stabilize the oxide surface and inhibit further corrosion." It is also stated that XPS studies showed that amino alcohol "can displace chloride ions from the steel surface."

Catexol

Another organic inhibitor, Catexol 1000 CI, is said (by its manufacturer) to protect steel bars by forming a "protective barrier that stabilizes the passivating layer of iron oxide". At this time, no detailed information was made available to the research team regarding this product. However, it appears that the Catexol CIA combines ingredients of both an organic film forming inhibitor and a nitrite-based inhibitor.

Others

In addition to the above compounds, a wide range of chemicals is mentioned in the literature as having been studied (mostly in laboratory trials). A vast majority of these have been screening studies in aqueous simulated pore solutions, with only a few added as admixtures to concrete (or mortar mix). Among them are:

- Alkalis, tertramethyl and tetrabutyl quaternary ammonium salts. [8, 9]
- Barium metaborate, disodium -glycerophosphate, sodium metavanadate, sodium phosphate(s). [10, 11]
- Sodium tetroborate, sodium silicate, sodium carbonate, potassium nitrate, calcium borate, calcium sulfate, zinc borate. [11]
- Barium, strontium, lead, and potassium chromate, calcium and silicon fluorides, sodium benzoate and metasilicate, sodium nitrite, aluminum acetate, chrome oxide, chrome carbides, thiourea, mercaptobenzothiazole. [12, 11]
- Butyl ester emulsion and dimethylethanolamine (DMEA). [13]
- Formaldehyde. [14]
- Sodium molybdate. [11]
- Stannous and stannic chloride. [15-18]

- Water-soluble mono-and di-carboxylic acids. [19]
- Zinc oxide. [20]
- Sodium monofluorophosphate (MFP). [21, 11]

TEST METHODS

A variety of methods/techniques (short- and long-term) to assess the performance of corrosion inhibitor admixtures (CIA) have been used. These methods and special considerations for concrete and CIA testing are discussed below.

Concrete Quality Considerations

The issue of concrete quality, its permeability to chlorides in particular, is intrinsically controversial when it comes to short-term laboratory testing. On one hand, effective diffusion coefficient of chloride ions in high quality concretes is very low and, all conditions being equal, time-to-corrosion of reinforcing steel in such concrete are exceptionally long. On the other hand, laboratory tests (screening-type tests in particular) should create conditions in which time-to-corrosion is reasonable and practical. Therefore, compromises often are made with regard to concrete quality as far as w/c ratios and minimal concrete cover thickness are concerned.

Water-to-cement ratios (w/c) mentioned in research literature in connection with laboratory trials show considerable difference: from 0.28 to 0.5 to 0.65 to even 0.9. [22-25] Some authors suggest that a low w/c ratio (high quality concrete) be used for laboratory testing of CIAs; such an opinion is expressed by Berke. [13] It was also proposed that CIAs should be evaluated under conditions which would closely simulate the intended application, which for practical purposes means concrete with a maximum w/c of 0.4, which would be in compliance with ACI 318 code for concretes for corrosive environments. [26-27]

Concrete cover thickness for specimens used in evaluation trials of corrosion inhibiting admixtures is another factor which affects concrete quality with regard to time-to-corrosion. Cover thickness for the minibeam specimen range from 19 to 38 mm (0.75 to 1.5 in). [28-30, 4] Citing ACI building codes which call for a minimum cover thickness of 1.5 in for concretes exposed to aggressive environments, several authors recommend that the cover thickness of the test specimens be at least 25 to 38 mm (1 to 1.5 in). [27, 31, 26, 32] However, according to Berke et al, concrete with a higher (0.5 in) w/c ratio and a cover thickness less than 30 mm (1.2 in) could be used for screening-type tests. [16] Because concrete covers of 38 mm (1.5 in) would require prohibitively long testing times for most laboratory evaluations, the project team members of this NCHRP study have adopted the rule that concrete cover twice the maximum aggregate size is sufficient for laboratory studies.

Linear Polarization Resistance (LPR) Technique

This method is based on determination of polarization resistance of a specimen exposed to a corrosive environment. The polarization resistance is inversely proportional to corrosion rate.

The technique involves the application of a small (~10-20 mV) DC polarization at a specified rate and measurement of the resulting current. Typically, a lollipop specimen is employed which is submerged into the test solution. The following variations of testing parameters can be found.

Scan rates: 0.01 mV/s, 0.1 mV/s, and 10 mV/s. [30,9,29,31,33]

Lollipop dimensions:

- 11 mm (3/8 in) diameter reinforcing bar embedded in 76 mm (3 in) diameter by 152 mm (6 in) long concrete cylinder.[34]
- 12 mm (0.5 in) diameter reinforcing bar embedded in 76 mm (3 in) diameter by 152 mm (6 in) long concrete cylinder and in 101 mm (4 in) diameter by 202 (8 in) long concrete cylinder.[35]
- 16 mm (5/8 in) diameter reinforcing bar embedded in 100 mm (4 in) diameter cylinder.[36]
- 11 mm (7/16 in) diameter reinforcing bar embedded in 25 mm (1 in) diameter by 70 mm (2.75 in) long concrete cylinder.[37]

The polarization resistance (R_p) is defined as the slope at the free-corrosion potential (E_{corr}) of the resulting linear plot of voltage versus current density:

$$R_p = (\delta E/\delta I)_{Ecorr} \qquad (2)$$

The polarization resistance can be further related to the rate of corrosion via the Stern-Geary equation [38]:

$$i_{corr} = (1/Rp)\ \{\beta_a\beta_c / [2.303\ (\beta_a + \beta_c)]\} \qquad (3)$$

Where i_{corr} is the corrosion current density, and β_a and β_c are Tafel constants (slopes of the linear portions of anodic and cathodic polarization curves, respectively, on a E versus log i plot). The Tafel constants have to be known either from a separate experiment or from the published data. However, for simplicity sake, another equation is typically used:

$$i_{corr} = B/R_p \qquad (4)$$

Constant B is either calculated using equations 3 and 4, or is estimated as 0.026 V (when steel bars are actively corroding) or 0.052 V (when steel bars are passive). A value of B equal to 0.035 V is used by the authors of this report, based on the relatively large cathodic Tafel slope (measure) even during active corrosion of steel in concrete. The corrosion rate is calculated from i_{corr} via Faraday's law.

Various authors have used the following guidelines with regard to i_{corr} and R_p (to convert from polarization resistance to corrosion current, a constant B=0.035 V was used along with conversions of 1 μA/cm^2 = 11.7 μm/y = 0.46 mpy):

- Rp> 66,000 ohm-cm^2 (6.2 μm/y) - no corrosion
  Rp< 40,000 ohm-cm^2 (10.2 μm/y) - severe corrosion [4, 39]
- Rp> 50,000 ohm-cm^2 (8.2 μm/y) - no corrosion
  Rp< 20, 000 ohm-cm^2 (20.5 μm/y) - severe corrosion [40]

- $i_{corr} < 0.22$ μA/cm^2 (2.6 μm/y) - no corrosion
  0.22< i_{corr} <1.08 μA/cm^2 (2.6-12.6 μm/y) - damage possible in 10 to 15 years
  1.08< i_{corr} <10.8 μA/cm^2 (12.6-126 μm/y) - damage possible in 2 to 10 years [39]
- 0.1< i_{corr} < 0.2 μA/cm^2 (1.2 -2.3 μm/y) - no corrosion,
  i_{corr} > 0.22 μA/cm^2 (2.6 μm/y) - active corrosion
  i_{corr} ~ 1 μA/cm^2 (11.7 μm/y) - significant but not severe corrosion
  i_{corr} > 10 μA/cm^2 (117 μm/y) - very severe attack [33]

The value of polarization resistance (R_p') measured by DC electrochemical method is a composite number that also contains a solution, or concrete, resistance (R_s) that is not related to corrosion rate as is R_p ($R_p' = R_p + R_s$). The error produced by R_s is typically substantial for concrete measurements and requires compensation. There are various methods that have been developed to compensate for R_s in a DC electrochemical test. Alternatively, an AC technique can be used (electrochemical impedance spectroscopy, EIS) to determine the values of R_p and R_s. This technique is labor-intensive and requires training to interpret the results correctly and, therefore, is not typically recommended as a routine testing procedure. However, it can provide mechanistic information and has the ability to estimate a steady-state corrosion rate from non-steady-state data.

Cyclic Potentiodynamic Polarization (Based On ASTM G-5 And G-61 Test Methods)

In this method the potential of a reinforcing steel bar is scanned to a value that exceeds the pitting potential (also referred to as break-down potential) and then reversed and returned to some pre-determined value. The following values of the testing parameters can be found.

- 0.056 mV/s for forward scan and 0.42 mV/s for reverse, apex at 200 μA/cm^2. [40]
- 5 mV/s for both forward and reverse scans, apex at 250 μA/cm^2 (or at 100 μA/cm^2); specimen conditioned at -1V (SCE) for 15 min. [32,13]
- 20 mV/min (0.3 mV/s) for both forward and reverse scans. [37]
- 1 mV/s for both forward and reverse scans. [41]
- 100 mV/min (1.7 mV/s). [21]
- 40 mV/s. [6]

Cyclic potentiodynamic polarization (CPP) tests permit the corrosion behavior of the reinforcing steel in concrete (or a concrete pore solution) to be determined. The CPP test method is very effective in establishing the effect of inhibitors on the anodic and cathodic reactions.

Concrete Minibeam Test (ASTM G 109)

This test is based on measuring the coupled current of a macrocell formed by (1) reinforcing steel exposed to a chloride-rich top layer of a concrete slab (the anode of the macrocell couple) and (2) reinforcing steel at the bottom of the slab exposed to low-chloride concrete layer (the cathode of the macrocell couple). The following modifications to the method are described.

- One reinforcing bar on top, one at the bottom. [34]
- One reinforcing bar on top, two at the bottom. [32]
- Two on top, two at the bottom. [42]
- Two on top, four at the bottom. [23]
- Top and bottom reinforcing bars are shunted via 100 ohm, 10 ohm, 1 ohm, or 0.5-ohm resistor. [34,43,37,44]

The top beams are ponded with NaCl solution; a variety of concentrations have been used in the laboratory tests. Ponding protocols also vary considerably from study to study. The following regimens are mentioned.

- 15% solution, cycle: 4 days on at ambient temperature, 3 days drying at 38°C. [23]
- 15% solution, cycle: 5 days on, 2 days drying. [45]
- 15% solution, cycle: 4 days on, rinse, 3 days drying at ambient temperature. [30]
- 6% solution, cycle: continuous exposure, solution replenished every 2 weeks. [37]
- 6% solution, cycle: 4 days on, 3 days drying at 70°C. [4]
- 6% solution, cycle: 5 days on, 2 days drying at ambient temperature. [3]
- 3.5% solution, cycle: 2 weeks on, 2 weeks drying. [35]
- 3% solution, continuous exposure, solution replenished monthly. [46]
- 3% solution, cycle: 2 weeks on, 2 weeks drying. [34,7]
- 3% solution, sprayed 3 times per week, after 7 months - 3 times per month. [24]
- 1% solution, cycle: 1 week on, rinse, 1 week drying (used for “lollipop” specimen). [7]

The minibeam test is a simple, straightforward method, which has been used successfully by many laboratories. It has the advantage of establishing a macrocell couple similar to that expected in actual structures. However, under certain conditions, the method may not provide reliable quantitative data on corrosion rates of reinforcing bar. That is, the measured macrocell current may be smaller than the anodic dissolution current of the top reinforcing bar leading to underestimating the rate of corrosion. This effect is enhanced for a very local breakdown of the passive film on the top reinforcing bar (cracked specimen) and less for more general breakdown. Qualitative comparisons are typically very good.

Pre-Cracked Slab Test (Based on ACI 224R-89) [47]

An intentional crack is introduced to the slab with embedded reinforcing steel bar to simulate surface cracking of a real concrete structure. The following variations of the method are described.

- Maximum crack width of 0.18 mm (0.007 in). [47]

- Maximum crack width of 0.15 mm (0.006 in) for marine environments and 0.18 mm (0.007 in) if deicing compounds are present. [27]
- Crack width of 0.2-0.25 mm (0.008-010 in), with final widths of 0.13-0.33 mm (0.005 in - 0.013 in). [23]
- Crack width of 0.25 mm (0.01 in). [32, 6]
- Crack width of 0.3 mm (0.012 in). [24]

Large Scale Slab Test

Long-time exposure tests of large (e.g., 1524 x 700 x 152 mm [60 x 24 x 6 in]) concrete slabs to the outdoor conditions have been performed. Macrocell current between the top and bottom layers of reinforcing bars is generally monitored. The actual size and shape of large slabs can vary greatly. [44]

Harmlessness Testing

A steel specimen is embedded in cement with corrosion inhibitor admixture and exposed to chloride-containing solution. The specimen is polarized either potentiostatically or galvanostatically for a 24-hour period. The respective values of 260 mV (SCE) and 10 $\mu A/cm^2$ are mentioned. [16,48] Results of this test (potential or current measurements plotted versus test time) are compared with the control test (i.e. specimen embedded in cement with no corrosion-inhibiting admixture). [16]

Corrosion Potential Measurements

The method is described in detail in ASTM C 876 "Test Method for Half Cell Potentials of Reinforcing Steel in Concrete." The open circuit or corrosion potential (E_{corr}) of a steel bar is measured against a suitable reference electrode, such as copper/copper sulfate electrode (CSE). Both the magnitude and sign of the measured potential are important, since they reflect conditions at the metal/environment interface. The potential is typically measured via a high input impedance ($>10^{10}$ ohm) voltmeter.

Typically used criterion with regard to the onset of corrosion of reinforcing steel is -350 mV (CSE). [49] Referencing FHWA long-term exposure testing, one author states that according to the results of the project, steel bar corrosion is manifested at more positive potentials, namely -230 to -250 mV (CSE). [2, 23] This data has been disputed on the grounds that these findings are the result of the use of excessively dry concrete, which led to errors in potential measurements. [50]

The advantages of potential measurements are (1) the technique is simple and (2) it is a non-destructive technique. The method, however, does not provide any data with regard to corrosion rates, general or localized; only the probability that corrosion is occurring.

Immersion Tests

The steel specimen is exposed to an appropriate solution (e.g., simulated pore solution with or without corrosion inhibitor admixture and chlorides). Visual examination, electrochemical measurements, and weight loss of the specimen evaluate the degree of corrosion.

For weight-loss measurements the surface area of the coupon, type of the metal, anticipated corrosion rate, and the sensitivity of the balance used for weighing the coupons determine the time of exposure of the coupon. The coupons can be retrieved according to a set schedule and average corrosion rates can be calculated based on a weight loss.

The following equation is used to compute the corrosion rates:

$$CR\ \{mpy\} = WL\ \{g\} \times 394 / [D\ \{g/cm^3\} \times A\ \{cm^2\} \times t\ \{yr\}] \qquad (5)$$

CR is a corrosion rate, WL is a weight loss, D is the metal's density, A is the exposed area, and t is the exposure time.

The following simulated pore solutions have been mentioned.

- Saturated calcium hydroxide. [40]
- 0.2M potassium hydroxide + saturated calcium hydroxide. [6]
- 0.1M potassium hydroxide + 0.1M sodium hydroxide + saturated calcium hydroxide. [33]
- 0.6M potassium hydroxide + 0.2M sodium hydroxide + saturated calcium hydroxide. [37]
- 1.51% potassium hydroxide + 1.84% sodium hydroxide + saturated calcium hydroxide. [41]
- 0.6M potassium hydroxide + 0.3M sodium hydroxide + saturated calcium hydroxide. [28,11]

Although immersion tests utilizing weight-loss provide the most accurate information with regard to average corrosion rates, these tests are inadequate for determination of the performance characteristics of CIAs because they fail to accurately simulate the diffusion conditions/chemistry of the concrete.

LABORATORY PERFORMANCE OF CORROSION INHIBITING ADMIXTURES

One of the persistent difficulties in objectively evaluating inhibitor performance is that the manufacturers of the CIAs produce the vast majority of the data. Although it is believed that the results are accurate, it is also likely that the data presented by the manufacturer are only that which are favorable to their particular admixture. In addition, each of the primary manufacturers have developed "new and improved" versions of their CIA making comparisons with previous formulations difficult, if not impossible. The above observations are made only to qualify the data presented below and to indicate the difficulty in coming to conclusions.

DCI Corrosion Inhibitor

In an early study by Rosenberg and Gaidis (W.R. Grace), calcium nitrite was shown to increase the breakdown potential for carbon steel in chloride containing saturated calcium hydroxide solutions. [1] Potentiodynamic polarization curves were produced in a wide range of chloride and calcium nitrite concentrations. Berke presented similar results. [40]

Although the following study examines sodium nitrite and not calcium nitrite (DCI), it is included in this section. Andrade et al. noted that, over several years of testing concrete by several different methods, the addition of sodium nitrite is effective as an inhibitor against corrosion of steel in concrete. [51] It always reduced corrosion even if it did not completely eliminate it. The effectiveness of the inhibitor was dependent on the chloride-to-nitrite ratio and relative humidity. It was found that a nitrite concentration of 3% by weight of cement was generally beneficial. In addition, it was found that nitrite decreased corrosion in carbonated concrete. Alonso et al. (same group as Andrade) reported research using pore solutions and mortar samples in which it was shown that nitrites reduced corrosion of steel in carbonated concrete. [33, 52]

Berke (W.R. Grace) has performed several studies that have indicated the inhibiting capability of calcium nitrite (DCI). In a two year study using "lollipop" specimens immersed in 3% NaCl solution, several different concrete mixes were evaluated with respect to their corrosion performance with and without DCI added.[53] DCI was shown to improve the corrosion resistance of steel in concrete with w/c values of 0.5 or less. At w/c values greater than 0.5 the improvement was much less or none at all. In these studies, DCI addition rates (30% calcium nitrite solution) ranged from 15 to 30 L/m^3 (3 to 6 gal/yd^3). The protective effect of the DCI was related to the chloride-to-nitrite (original nitrite in the concrete) ratio. This study suggests a ratio of 1 or less is required to provide protection, although the data presented was insufficient to pinpoint the value accurately. Hartt and Rosenberg, using lollipop specimens immersed in filtered seawater, showed similar beneficial effects of calcium nitrite. [54] Potential measurements were periodically made to establish time to corrosion initiation. It was reported that the time corresponding to the onset of corrosion increased 50% with an addition of 2% calcium nitrite by weight of cement and 100% with an addition of 4% calcium nitrite.

An FHWA study examined the benefits of calcium nitrite inhibitor additions to the concrete utilizing large slabs (610 x 1524 x 153 mm [24 x 60 x 6 in]) exposed to the natural environment (northern Virginia) over a 7 year period. [44,55] The calcium nitrite addition was constant at 2.75% calcium nitrite by weight of cement. The chlorides were premixed into the concrete at concentrations of 0, 3, 6, 9, 12, 14.8, 20.7 kg/m^3 (0, 5, 10, 15, 20, 25, and 35 lb/yd^3). The concrete was designed to be of a relatively poor quality with a w/c ratio of 0.53. This study confirmed the beneficial effect of calcium nitrite in inhibiting corrosion of steel in concrete. Up to a chloride-to-nitrite ratio of 0.9, the calcium nitrite was effective in mitigating corrosion. For a chloride-to-nitrite ratio of 0.9 to 1.1, the macrocell currents were reduced by a factor of 10, and with a ratio between 1.3 to 1.8 a 2 to 7-fold reduction in the macrocell current was observed. At the higher ratios, cracking of the concrete with rusting coming through the cracks was found.

Berke et al. reported similar beneficial effects for calcium nitrite additions utilizing larger cracked (762 x 152 x 152 mm; 30 x 6 x 6 in) minibeam specimens ponded with 3% NaCl solution. [42,43] Electrochemical techniques (LPR and EIS) and visual inspection were utilized to characterize the degree of corrosion during the exposure.

Berke et al. showed that a combination of calcium nitrite inhibitor and silica fume could provide a significant improvement in the life of a concrete structure. [46, 34, 36, 56, 57] The silica fume significantly decreases chloride migration into the concrete and combined with the action of the calcium nitrite increases the chloride threshold for the initiation of corrosion.

Berke reported data on a 5-6-year study that utilized a relatively high quality concrete (w/c=0.45) and examined epoxy coated reinforcing steel. [58] This study used both lollipop and minibeam specimens and utilized LPR and EIS to characterize corrosion performance. DCI concentrations ranged from 13.5 to 27 L/m^3 with a majority of the data presented on 20 L/m^3. The epoxy coating was tested in the flawed and unflawed conditions along with no coating. The primary conclusion was that concretes containing DCI and black steel provided better performance than the flawed epoxy-coated steel specimens and similar to the unflawed epoxy-coated steel specimens. The combination of epoxy coated steel reinforcing with DCI added to the concrete performed the best.

An FHWA study on large concrete slabs provided similar results as those reported by Berke (above) regarding the benefits of calcium nitrite alone and in combination with epoxy-coated reinforcing steel. [59] For 2.75% calcium nitrite by weight of cement, a chloride-to-nitrate ratio of 1.25 or less afforded a factor of 10 or greater reduction in corrosion than the control slabs with no inhibitor.

In a study by Berke et al., calcium nitrite was found to reduce corrosion on galvanized steel and aluminum reinforcements. [29]

Dillard et al. reported work performed on a SHRP-funded project that involved a combination of mixing CIA into the repair material and subsequent ponding. [11, 60] The study utilized concrete slabs in which corrosion had been initiated, followed by removing the concrete directly above the reinforcing steel and replacing it with repair concrete containing a CIA. Although, the application is different from the primary focus of this project, the results are of interest. Several inhibitors were examined, including DCI and Cortec 1609 (similar to MCI 2000). DCI provided the most consistent data and the greatest decrease in the measured corrosion rate (corrosion current by LPR). Although Cortec 1609 performed well, the magnitude of decrease in corrosion was not as great as that of DCI.

Rheocrete 222 Corrosion Inhibitor

Nmai (Master Builders) and Krauss have reported beneficial effects of a water-based organic inhibitor (Rheocrete 222) in a relatively low quality concrete (w/c ratio of 0.5). [23] The tests were performed using concrete slabs with cyclic ponding with a 15% NaCl solution. The coupled current measurements were used as the basis for evaluating the corrosion performance. Nmai et al. present additional data indicating the beneficial effects of Rheocrete 222. [4, 61]

Bobrowski and Youn (Master Builders) showed that the Rheocrete 222 increased time to corrosion for cracked beam tests. [3] Nmai and Krauss have reported similar results using doses of Rheocrete 222 at 5 L/m^3 (1 gal/yd^3). [23]

In tests preformed by Master Builders, a comparison of Rheocrete 222 and Rheocrete 222+ was presented. [62] Linear polarization resistance and electrochemical impedance spectroscopy measurements were made on steel specimens immersed in saturated calcium hydroxide containing 0.2 molal NaCl. Manufacturer's recommended dosages (5 L/m^3 [1 gal/yd^3]) of the Rheocrete 222 and Rheocrete 222+ were used. Both

Rheocrete formulations inhibited corrosion as compared to the solution with no inhibitor. Some crevice corrosion was observed at the mounting gaskets for the Rheocrete 222 specimens as indicated by a lower polarization resistance (higher corrosion rate) and a more active corrosion potential. Specimens immersed in Rheocrete 222+ containing solution showed no signs of corrosion. Based on the LPR measurements, the latter conditions exhibited a significantly lower corrosion activity than the solution with Rheocrete 222. It should be noted that tests in aqueous environments should be used as a screening tool and their results should not be extrapolated to the performance of a CIA in actual concrete.

Armatec/Ferrogard/MCI Corrosion Inhibitor

Armatec, Ferrogard 901(Ferrogard), and MCI are all amino alcohol-based inhibitors. Ferrogard is the newest formulation by Sika. The majority of information on the Ferrogard pertains to the tests in aqueous media. Concrete tests are reported to be underway, but the results have not yet been published. Maeder (Sika) presents data for the pitting potential of steel in calcium hydroxide plus 0.09 molal sodium chloride solutions adjusted to pH 11.5 and 10 with and without aminoalcohol (AMA) inhibitor and Ferrogard. [6] The pitting potentials were determined by a potentiostatic method. The AMA inhibited solution (2% amino alcohol) increased (in a positive direction) the pitting potential by values between 70 and 100 mV. The Ferrogard increased the pitting potential by 200 mV. In potentiodynamic tests at a scan rate of 40 mV/s, which is considered to be extremely high, a pitting potential increase of 400 to 450 mV is reported. Ferrogard (3%) was also shown to inhibit corrosion in a 9.25 pH solution with excess CO_2, indicating the possibility of beneficial effect of the formulation on corrosion caused by carbonation.

Buerge (Sika) reports findings, but presents little data, on the Armatec and Ferrogard- type inhibitors (reported as alkanolamine mixed inhibitor) tested as lollipop mortar specimen immersed half way in 1% NaCl solution. [7] The corrosion performance assessment was based on visual examination. Beneficial effects were observed when 3 to 4% (by cement weight) of the inhibitor was used.

Data was published by Maeder for cracked specimens exposed to a ponding (with a 3% NaCl) and drying cycle (2 weeks wet, 2 weeks dry). [6, 63] The concrete had a w/c ratio of approximately 0.42. The current following the initiation of corrosion was decreased by a factor of 10 for the Armatec inhibitor (3%) and a factor of 6 to 4 for the Ferrogard formulation (addition of 2 to 4%, respectively) after 10 ponding cycles, when compared to control beams. [63] The article showed similar data for a calcium nitrite admixture (4% addition). However, after 15 ponding cycles, the current magnitude for Ferrogard containing specimens increased to 44 and 36% of that of control specimens. [6]

Maeder also presented data from which it can be inferred that the AMA-containing inhibitors can diffuse through concrete in a matter of a few days after a topical application. In fact, the article states that an average transport rate inhibitor was 10 mm/day. [6] The resultant profile of AMA in the concrete (w/c ratio of 0.51) after a 28-day exposure looks unusual and suggests that the mechanism of transport is other than diffusion. A very similar paper published by Buerge references the same study as

Maeder and states that "AMA penetrate through...concrete and seek out the steel". [7A] This conclusion was not substantiated by the data presented.

Studies by State Departments of Transportation

In an early study by the Idaho Department of Transportation, concrete minibeams were exposed for one year to cyclic ponding with 5% NaCl solution, which also contained various other compounds. [64] The compounds investigated were calcium hydroxide, sodium carbonate, calcium silicate, and calcium nitrite. In addition to the above salts being added to the ponding solution, calcium nitrite was added to the concrete mix for some of the minibeams. A certain beneficial effect was observed for the calcium silicate, but the most corrosion reduction was observed for the calcium nitrite mixed into the concrete. The dosage used was 2.5% calcium nitrite by weight of cement. Further reduction of corrosion rate was observed when calcium nitrite was added to the ponding solution in addition to being mixed into the concrete.

Louisiana DOT performed a study that examined calcium nitrite based corrosion inhibitor. [65] Concrete minibeam specimens were exposed to two environments: (1) continuous ponding with a 3% NaCl solution and (2) a salt fog. The "continuous" ponding included a 24-hour drying cycle prior to monthly measurement of potentials. The calcium nitrite dosage was 2% by weight of cement. Based on potential measurements and visual examination, the calcium nitrite inhibitor provided some protection against corrosion and warranted further study.

In a California DOT study, lollipop specimens with admixed calcium nitrate were partially immersed in a saturated NaCl solution to examine the effectiveness of calcium nitrite in inhibiting corrosion of steel in concrete. [66] Corrosion potential measurements and visual examination were used to characterize the corrosion performance. It was found that a 4% calcium nitrite by cement weight dosage significantly delayed the onset of corrosion and delayed damage to the concrete.

In another California DOT study, the possibility of applying calcium nitrite to the surface of mature concrete for the purpose of the calcium nitrite diffusing into the concrete down to the steel surface was examined. [67] It was concluded that the penetration into the concrete was not sufficient to permit a topical application of calcium nitrite to be effective.

Very few comparison studies are available that were not performed by the manufacturers. One such study was performed by the Indiana DOT, which evaluated MCI products 2020 and 2000, Rheocrete 222, and DCI. [68] In this study, minibeam concrete specimens were used in conjunction with coupled current measurements to evaluate corrosion performance. The concrete had a w/c ratio of 0.5 and the minibeams were cyclically ponded with 3% NaCl solution. Visual examination following testing confirmed the validity of the current measurements. Cracked beam tests were also performed.

DCI and Rheocrete 222 provided significant corrosion inhibition as compared to the control specimens for the tests with no cracks. The concretes with the MCI products performed only marginally better than the control concrete. None of the CIAs performed very well in the cracked beam tests.

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APPENDIX C

TRANSPORTATION AGENCY SURVEYS

APPENDIX **C**

TRANSPORTATION AGENCY SURVEYS

CURRENT PRACTICES

A detailed questionnaire (see Table C1) was used to collect information from state DOTs inquiring about their experiences with corrosion inhibiting admixtures. Several of the DOTs were interviewed by phone and others were mailed the questionnaire. Their responses are summarized in Tables C2 through C5.

As seen from this data, the most commonly employed inhibitor is DCI (regular and set-retardant containing version) with the other admixtures used on an experimental basis by only a handful of DOTs.

Six DOTs submitted specifications used for CIA; all six were for DCI. The specifications ranged in applications typically divided into marine and non-marine applications, dosages, and verification test requirements. Also, at least one DOT specifically addressed prestressed concrete application. The dosages ranged from 20 to 27 L/m^3 (4 to 5.4 gal/yd^3) of the DCI. One DOT specified 20 L/m^3 (4 gal/yd^3) for non-marine applications and 27 L/m^3 (5.4 gal/yd^3) for marine applications. Other DOTs specified either 20 or 27 L/m^3 (4 or 5.4 gal/yd^3) for general use, but not both. In each case, the DOTs required contractors to perform manufacturers-recommended testing to verify the presence of calcium nitrite in the concrete and, in some cases, a test to determine the concentration of calcium nitrite. It is not possible to indicate current practice for the other inhibitors because of the lack of experience. At present, use of CIAs other than DCI is mostly experimental. The DOTs using the other CIAs are basing their practice primarily on manufacturers-recommended procedures.

According to responses to the questionnaire, CIAs have been used at one time or another for most types of structures (i.e., prestressed piles, piers and caps, prestressed beams, abutments, decks, ramps, and parapets). Because of the lack of follow-up examinations and the fact that most applications are relatively new, there is no available information on the CIAs performance as a function of type of structure.

Only two DOTs reported the use of an inhibitor applied to the surface of an existing structure (MCI 2020 by Cortec and Postrite by W.R. Grace). No details are available on follow-up examination. Like many others, CC Technologies has experience in experimental trial exposures, but as of the date of this report, no reportable findings. CC Technologies has used two CIAs as topically applied inhibitors as remedial measures for rehabilitation of a concrete structure (parking garage deck). Ferrogard 901 (Sika) was applied to the surface for a 24 hour period. DCI-S (W.R. Grace) was applied to the concrete surface after removal of the concrete down to within one inch of reinforcing steel. DCI-S was applied for 24 hours and DCI was mixed at a concentration of 20 L/m^3 (4 gal/yd^3) into the concrete that was used to bring the deck back to its original level. The above is provided as an example of topical applications, but no information is yet available as to their performance.

Table C1. Industry Survey

QUESTIONNAIRE: USE and ACCEPTANCE of CORROSION-INHIBITING ADMIXTURES

1. Transportation Department: ____
2. Individual Completing Questionnaire: __________________________________
3. Position of Individual: ____
4. Telephone Number: ____
5. Has your agency used corrosion-inhibiting admixtures (CIA) in concrete? Yes___No__
6. Has your agency applied corrosion inhibitors in concrete surfaces? Yes___No__
7. Which CIA have your agency used (please check all that apply)?
   Darex Corrosion Inhibitor___, Armatec 2000___, Rheacrete 222___, Postrite___, Armatec 3020___, Others (please name)_______________________________
8. In what types of concrete structures have you used CIA (please check all that apply)?
   Precast prestressed piles___, Piers and caps___, Precast prestressed beams___, Abutments___, Decks___, Parapets___, Others (please name)________________
9. What material property tests do you perform to approve CIA and concretes that contain them (please check all that apply and indicate test method and the acceptance criteria)?

Test	Method	Acceptance Criteria
Slump_______________	_________	______________________________
Air Content____________	_________	______________________________
Compressive strength____	_________	______________________________
Flexural strength________	_________	______________________________
Bond strength__________	_________	______________________________
Length change__________	_________	______________________________
Freeze thaw____________	_________	______________________________
Permeability____________	_________	______________________________
Ponding_______________	_________	______________________________
Other_________________	_________	______________________________

10. What CIA performance tests do you conduct (please check all that apply)?

Test	Method	Acceptance Criteria
Visual inspection________	_________	______________________________
Half-cell Potentials_______	_________	______________________________
Rate of corrosion________	_________	______________________________
Bond strength__________	_________	______________________________
Laboratory performance__	_________	______________________________
Other_________________	_________	______________________________

1. What has been the performance of the concretes with CIA with respect to batching, mixing and placing the concrete (please indicate good, fair, poor, uncertain)?_______
2. What has been the corrosion performance of the structures with CIA (please indicate good, fair, uncertain)?_____________________________________
3. Do you have specifications for CIA or concretes that contain them? Yes___No___
4. Do you have reports on CIA or concretes that contain them? Yes___No___

Additional comments, reports, and specifications would be appreciated and can be sent to:

CC Technologies
6141 Avery Road, Dublin, Ohio 43016-8761
Tel: (614) 761-1214 Fax: (614) 761-1633

Table C2. Corrosion inhibiting admixtures used by state dots.

Armatec 2000	DCI/DCI-S	MCI	Rheocrete 222	None
	AK[a]			MS
	CA			ND
	CP			NM
	CT			OR
	FL			UT
	GA			WA
	IA			NE[c]
	ID			
	IL/IL		**IL**	
	IN	**IN**[b]	**IN**	
	KY		KY	
	LA			
ME	**ME/ME**		**ME**	
	MI			
	MN		**MN**	
	MO			
NJ	NJ		NJ	
	NC			
	NY/NY		NY/NY	
OH	OH			
PA	**PA**			
	RI			
	SC			
	TX			
VA	VA		VA	

[a] Boldface font denotes use of admixtures on experimental basis.

[b] Also used MCI 2020, topical application formulation.

[c] Used Postrite (W. R. Grace and Co., topical application).

Table C3. DOTs performing tests to assess the influence of corrosion inhibiting admixtures on concrete properties.

Slump	Air Content	Compressive Strength	Other
AK	AK	AK	AK
CT	CT	CT	
FL	FL	FL	FL
GA	GA	GA	
IA	IA	IA	
IL	IL	IL	
ME	ME	ME	
MN	MN	MN	
NC	NC	NC	**NC**[a]
NJ	NJ	NJ	
NY	NY	NY	NY
PA	PA	PA	PA
SC	SC	SC	
TX	TX	TX	TX
VA	VA	VA	**VA**

[a] Boldface font denotes use of admixtures on experimental basis.

Table C4. DOTs performing tests to assess the corrosion inhibiting properties of admixtures.

Visual Inspection	Half-Cell Potential	Corrosion Rate	Laboratory Tests	Bond Strength
			FL[a]	
			NC	
ME	ME			
ID	**ID**	**ID**	**ID**	
MN	**MN**	**MN**		
		MO		
PA		PA	PA	PA
VA	**VA**	**VA**	**VA**	**VA**

[a] Boldface font denotes use of admixtures on experimental basis.

Table C5. DOTs' comments on CIA-related experience.

DOT	Effect on Concrete Properties	Effect On Corrosion
CA	Fair	Uncertain
CT	Uncertain	Uncertain
FL	Good	Good
GA	Fair	Uncertain
IA	Uncertain	Uncertain
IL	Good	Uncertain
KY	Good	Good
ME	Good[a]	Good[b]
MN	Good	Uncertain
NC	Uncertain	Uncertain
NJ	Uncertain	Uncertain
NY	Good	Uncertain
OH	Fair	Better Than without[b]
PA	Good	Uncertain
RI	Good	Uncertain
SC	Good	Uncertain
TX	Meets specifications	Uncertain
VA	Good	Uncertain

[a] With respect to DCI-S.

[b] Only with respect to DCI, the only one used for a prolonged period of time.

Other than the previously mentioned tests to ascertain the presence and concentration of calcium nitrite, routine tests involving corrosion inhibiting admixtures pertain exclusively to concrete properties, such as slump, air content, and compressive strength. Except for the well-established effect of DCI as a set accelerator, no detrimental effects have been reported with respect to the properties of fresh or hardened concrete. One DOT indicated that since DCI-S replaced DCI, no further problems were experienced with accelerated setting.

In a SHRP study, methods of application were provided for DCI and MCI 2000 CIAs. [1] Although, the emphasis was on the repair of existing structures, for those applications where the concrete is removed down to the top layer of reinforcing steel and the CIA is added to the repair material, the application is similar to new placement of concrete. The rate of addition of DCI was 30 L/m^3 (6 gal/yd^3). The rate of addition for MCI 2000 was 1.2 kg/m^3 (2 lb/yd^3).

Tests with regard to corrosion performance are confined primarily to experimental facilities (California, Florida, Idaho, Indiana, Louisiana, Minnesota, Missouri, and Virginia DOTs). The results of the laboratory tests programs were presented as part of Appendix B of the present report.

FIELD PERFORMANCE

Field performance data of any significance is nearly non-existent. The primary reason for this is the extremely long time to corrosion periods in high quality concretes in use today, especially when a corrosion inhibitor has been added. A few specific studies performed for the purpose of evaluating the inhibitor performance are presented below. The general statements collected by the questionnaire are presented in the following paragraph.

Nineteen DOTs reported using CIAs in the past. Of the 19, the vast majority (14) reported uncertain performance with respect to corrosion, primarily due to insufficient exposure time. In addition, several of the DOTs have not performed any follow-up studies. Three indicated good performance and one indicated better performance with the inhibitor than without. As previously stated, the vast majority of the DOTs experience is with DCI. (The DOT that mentioned superior performance was referring to DCI.)

SHRP/FHWA Experience

In a SHRP study by Prowell et al., several concrete decks and substructures were treated with repair concretes containing DCI and MCI CIAs. [1] At the time of the original report, assessment of the CIA performance was not possible. It was established that CIAs could be applied with field repair and rehabilitation techniques.

Subsequent analysis of the original SHRP project has been performed under a FHWA study by Weyers et al. [2] In these post evaluations, potential and LPR measurements were used to compare performance of a control area with the one containing CIA. [9] Recall that these test sites were repair areas with the CIA mixed into the repair concrete and not new construction. As the following descriptions indicate, the results after 2.5 to 4 years of exposure are inconclusive. Because the evaluation of these sites will likely continue as part of an on-going FHWA project, it was felt it was important to review them briefly below.

U.S. Route 460 Over Virginia Route 723, Christiansburg, Virginia

Both a bridge deck and a column were repaired with DCI in July 1990. [2] The data discussed by Weyers were collected in September 1994. It was concluded that "over the 4 year treatment period, it appears that the calcium nitrite corrosion inhibitor impregnation, plugging, and patching treatments stabilized the corrosion on the reinforcing steel and thus prevented the continuation of corrosion damage." The authors of this review agree that the corrosion does not appear to be worse after 4 years, but the performance does appear to be significantly different from the control.

Elmwood Avenue Over New York Route 198, Buffalo, New York

Cast-in-place repairs were performed on a substructure following removal of delaminated concrete using MCI 2000 (1.2 kg/m^3 [2 lb/yd^3]) and DCI-S (30 L/m^3 [6 gal/yd^3]) additives to the repair concrete. [3] This evaluation was approximately two years following repair of the substructure. Somewhat conflicting results are observed upon analyzing the potential and LPR data. The potential data indicate a marked improvement in the potentials of the CIA treated patches. However, the corrosion rates measured by LPR technique indicate the corrosion rate of the CIA treated repairs are greater than the control sections. It was concluded that "the effectiveness of using corrosion inhibitor admixtures in patching concrete to backfill cavities containing unclean reinforcing steel cannot be assessed at this time." These results were partly attributed to significant shrinkage cracking, which had occurred in the CIA repair patched concrete.

Trunk Highway 3 Over Southview Boulevard, South St. Paul, Minnesota

Repair of a bridge deck consisted of removing unsound areas and milling off 1.5 inch of original chloride-contaminated concrete. [4] The deck was overlaid with 2 inches of low-slump-dense concrete. There was one control area (no CIA), one area with 1.2 kg/m^3 (2 lb/yd^3) MCI 2000 added, and one area with 20 L/m^3 (4 gal/yd^3) DCI added. After approximately 3 years, all three areas are indicating similar behavior. This makes it impossible at this time to differentiate between the CIA-treated areas and the control area.

Pennsylvania State Road 2042 Over I-81, Wilkes-Barre, Pennsylvania

Bridge piers were selected to evaluate shotcrete post-treatment corrosion inhibitor repair treatments. [5] Treatments consisted of shotcrete containing 30 L/m^3 (6 gal/yd^3) of DCI, 1.2 kg/m^3 (2 lb/yd^3) of MCI 2000, and a control (no CIA). The original concrete was removed to 0.75 inch below the reinforcing steel level and the steel was sandblasted to white metal prior to treatment. After two years of exposure, there are insufficient differences among the two CIA-treated areas and the control area to assess performance of the CIAs.

Washington Route 104 Across the Hood Canal, Port Gamble, Washington

The site was a floating bridge across a brackish tidal water canal. [6] Repair of the deck consisting of exposing the reinforcing steel and sandblasting it to white metal prior to replacement. The treatments included additions to the concrete of 30 L/m3 (6 gal/yd3) of DCI and 1.2 kg/m3 (2 lb/yd3) MCI 2000, and a control (no CIA). For the 2.5 year evaluation period, the control area exhibited better performance based on potential measurements and LPR corrosion rate measurements, which could be attributed to the original condition being slightly less aggressive at that location.

DCI Corrosion Inhibitor

In a study by Berke et al., (W.R. Grace), twelve structures with service lives of up to 14 years were evaluated using potential and LPR measurement methods. [7] Each structure had utilized DCI as a corrosion inhibitor. It was determined that all structures were performing well. In two cases, adjacent structures were showing signs of corrosion. (This illustrates one of the problems with field studies: they require a very long time to prove the benefit of the CIA.) It was also shown that the calcium nitrite has remained in the concrete and is stable.

In yet another study by Berke et al., a method of impregnating a concrete structure with DCI was reviewed in which the concrete was dried out by heating, which was followed by ponding with an aqueous solution of DCI. [39] Field trials on a deck and substructure were performed. Although the study encompassed only a relatively short time (one year), it was reported that a decrease in corrosion rate (measured by LPR method) in the treated area was observed compared to the control area. Much longer time periods are required to establish a long-term benefit. This study presented one example of impregnating an existing concrete structure with inhibitor.

In a California DOT study, calcium nitrite inhibitor was used to protect bridge deck reinforcing steel from corrosion. [8] The latest inspection was following six years of performance. The calcium nitrite at a dosage of 2% by weight of cement was performing well, and is concluded that "even the six-year evaluation is far too little to base DCI performance at this site."

Armatec/Ferrogard/MCI Corrosion Inhibitor

No systematic field studies performed for the purpose of establishing performance were identified.

Rheocrete 222 Corrosion Inhibitor

Krauss (Wiss, Janney, Elstner) and Nmai (Master Builders) presented a preliminary inspection of prestressed concrete piles exposed to a marine environment that were constructed using 5L/m3 (1 gal/yd^3) of a "water-based organic corrosion inhibitor consisting of amines and fatty acid esters" (Rheocrete 222). [9] The corrosion evaluation was performed primarily by corrosion potential measurements on the pile surfaces and a single core that was extracted. As indicated by Krauss, conventional interpretation of potential data is often times not applicable to immersed structures and, therefore, minimum information on the corrosion condition of the steel can be inferred. The single concrete core taken through the steel reinforcement indicated corrosion had not initiated. As is the problem with the majority of the field data, sufficient time has not elapsed to make an evaluation of the benefit of the CIA.

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APPENDIX D

FIXED CHLORIDE CORROSION VS TARGETED CHLORIDE MAPS FOR CONCRETE 0

APPENDIX **D**

FIXED CHLORIDE CORROSION VS TARGETED CHLORIDE MAPS FOR CONCRETE 0

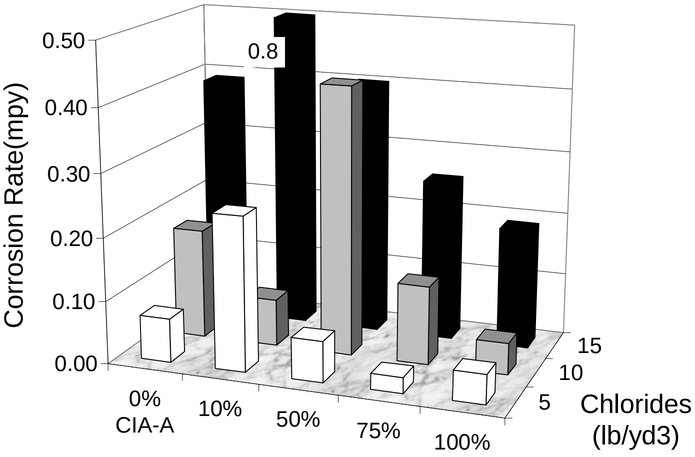


Figure D1. Corrosion rate map for CIA-A and Concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

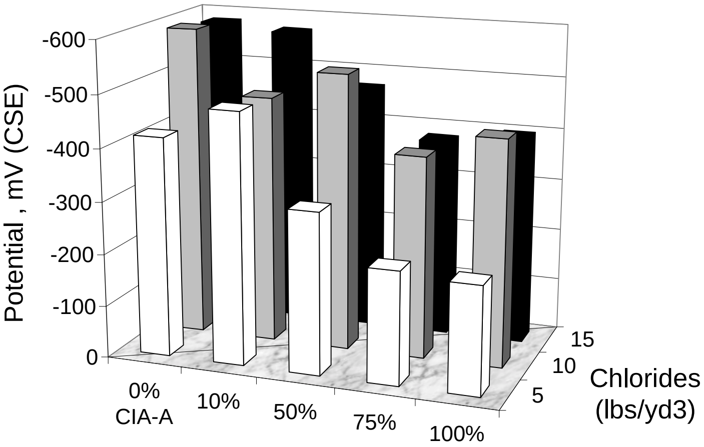


Figure D2. Corrosion potential map for CIA-A and Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3]

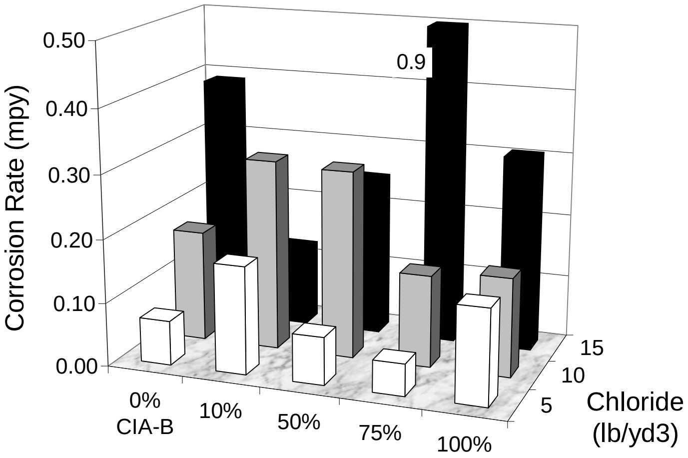


Figure D3. Corrosion rate map for CIA-B and Concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

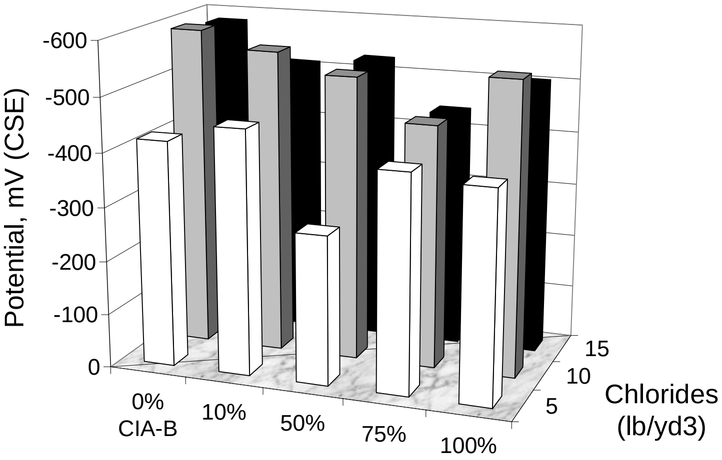


Figure D4. Corrosion potential map for CIA-B and Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3]

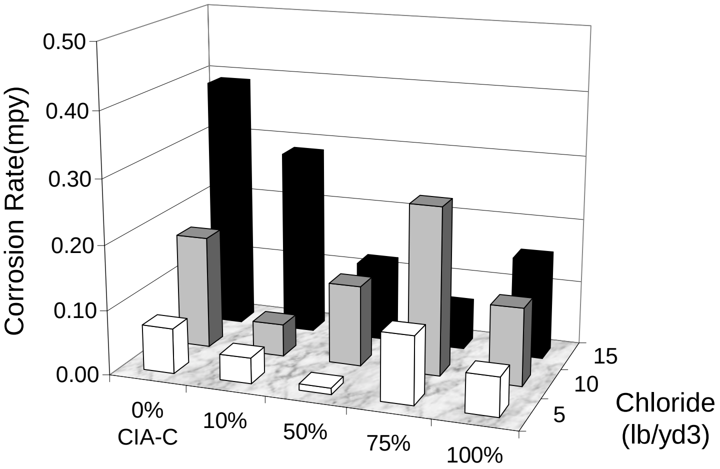


Figure D5. Corrosion rate map for CIA-C and Concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

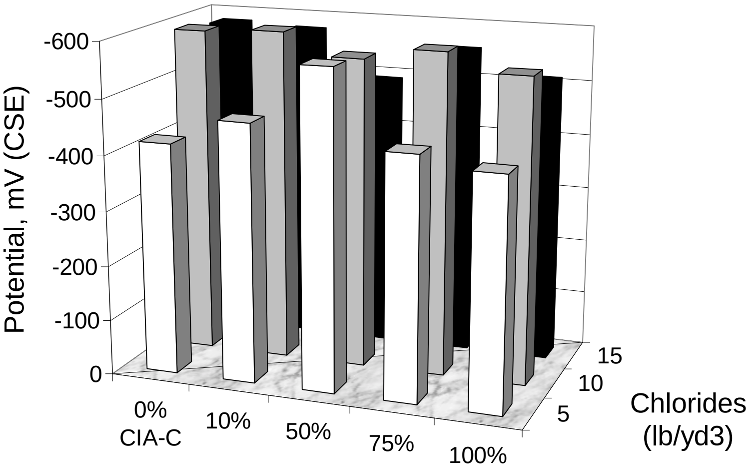


Figure D6. Corrosion potential map for CIA-C and Concrete 0 [1 kg/m^3 = 1.67 lb/yd^3]

APPENDIX E

FIXED CHLORIDE DATA TABLES

APPENDIX **E**

FIXED CHLORIDE DATA TABLES

Table E1. Fixed chloride data for the control Concrete 0 (no CIA). [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
0 Control	a	114	15	11.8	0.20	-578
0 Control	b	114	15	12.3	0.95	-556
0 Control	c	114	15	16.0	0.15	-608
0 Control	d	114	15	13.3	0.27	-586
0 Control	e	114	10	8.7	0.15	-620
0 Control	f	114	10	7.0	0.20	-567
0 Control	g	113	5	7.1	0.15	-442
0 Control	h	113	5	5.3	0.06	-472
0 Control	I	113	5	6.2	0.02	-322
0 Control	j	113	5	5.6	0.05	-442

Table E2. Fixed chloride data for the Concrete 0 with CIA A. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
A	0	10	a	160	15	16.5	0.66	-599
A	0	10	b	160	15	12.7	1.08	-543
A	0	10	c	161	10	15.2	0.06	-490
A	0	10	d	161	10	14.7	0.09	-456
A	0	10	e	161	5	9.7	0.20	-481
A	0	10	f	161	5	11.4	0.29	-477
A	0	50	a	160	15	12.3	0.58	-442
A	0	50	b	160	15	15.9	0.22	-498
A	0	50	c	160	10	16.9	0.48	-525
A	0	50	d	160	10	16.4	0.37	-531
A	0	50	e	160	5	10.9	0.07	-328
A	0	50	f	160	5	10.1	0.06	-289
A	0	75	a	160	15	12.0	0.19	-446
A	0	75	b	160	15	14.6	0.32	-315
A	0	75	c	161	10	10.4	0.12	-224
A	0	75	d	161	10	12.8	0.13	-549
A	0	75	e	161	5	8.0	0.02	-193
A	0	75	f	161	5	6.7	0.03	-239
A	0	100	a	160	15	12.7	0.03	-303
A	0	100	b	160	15	12.4	0.37	-449
A	0	100	c	160	15	13.6	0.17	-445
A	0	100	e	161	10	10.1	0.01	-471
A	0	100	f	161	10	11.6	0.09	-393
A	0	100	h	161	5	8.5	0.05	-251
A	0	100	I	161	5	6.0	0.02	-129
A	0	100	j	162	5	5.5	0.07	-240

Table E3. Fixed chloride data for the Concrete 0 with CIA B. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
B	0	10	a	159	15	12.9	0.25	-522
B	0	10	b	159	15	12.3	0.00	-499
B	0	10	c	160	10	10.5	0.37	-511
B	0	10	d	160	10	12.5	0.23	-614
B	0	10	e	160	5	8.5	0.27	-599
B	0	10	f	160	5	7.7	0.07	-307
B	0	50	a	160	15	11.3	0.30	-527
B	0	50	b	159	15	12.0	0.19	-547
B	0	50	c	160	10	10.9	0.17	-532
B	0	50	d	160	10	10.9	0.42	-521
B	0	50	e	160	5	7.0	0.07	-231
B	0	50	f	160	5	4.7	0.08	-321
B	0	75	a	159	15	10.9	0.19	-395
B	0	75	b	159	15	9.4	1.68	-485
B	0	75	c	160	10	8.0	0.17	-421
B	0	75	d	160	10	9.8	0.12	-480
B	0	75	e	160	5	4.3	0.08	-416
B	0	75	f	160	5	4.5	0.02	-389
B	0	100	a	159	15	9.8	0.51	-536
B	0	100	b	160	15	9.1	0.09	-450
B	0	100	c	159	15	9.8	0.32	-526
B	0	100	e	160	10	10.5	0.14	-510
B	0	100	f	160	10	11.6	0.17	-575
B	0	100	h	160	5	4.4	0.06	-322
B	0	100	I	160	5	4.2	0.15	-403
B	0	100	j	160	5	7.1	0.24	-468

Table E4. Fixed chloride data for the Concrete 0 with CIA C. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
C	0	10	a	114	15	na	0.28	-582
C	0	10	b	114	15	13.4	0.29	-570
C	0	10	c	114	10	14.3	0.01	-657
C	0	10	d	114	10	13.3	0.09	-545
C	0	10	e	114	5	6.3	0.07	-436
C	0	10	f	114	5	6.4	0.01	-500
C	0	50	a	114	15	14.2	0.13	-535
C	0	50	b	114	15	12.4	0.11	-447
C	0	50	c	113	10	9.7	0.13	-567
C	0	50	d	113	10	9.8	0.12	-556
C	0	50	e	113	5	6.3	0.02	-570
C	0	50	f	113	5	7.4	0.00	-578
C	0	75	a	114	15	13.4	0.05	-492
C	0	75	b	114	15	14.2	0.08	-620
C	0	75	c	114	10	10.2	0.35	-588
C	0	75	d	114	10	9.8	0.17	-578
C	0	75	e	113	5	8.2	0.08	-389
C	0	75	f	113	5	6.0	0.13	-488
C	0	100	a	114	15	15.4	0.13	-559
C	0	100	b	114	15	11.9	0.15	-402
C	0	100	c	114	15	9.8	0.10	-547
C	0	100	d	114	15	11.1	0.24	-536
C	0	100	e	113	10	9.2	0.19	-523
C	0	100	f	113	10	9.8	0.05	-579
C	0	100	g	113	5	7.5	0.01	-339
C	0	100	h	113	5	6.8	0.04	-461
C	0	100	I	113	5	7.2	0.07	-342
C	0	100	j	113	5	7.2	0.12	-530

Table E5. Fixed chloride data for the control Concrete 1 (no CIA). [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
1 Control	a	141	15	0.3	0.03	-76
1 Control	b	141	15	6.7	0.25	-528
1 Control	c	141	15	0.4	0.05	-163
1 Control	d	141	15	7.7	0.34	-539
1 Control	e	141	10	1.0	0.05	-182
1 Control	f	141	10	0.9	0.01	-181
1 Control	h	141	5	0.2	0.01	-210
1 Control	I	141	5	0.4	0.03	-141

Table E6. Fixed chloride data for the Concrete 1 with CIA A. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
A	1	10	a	141	15	6.1	0.72	-391
A	1	10	b	141	15	0.3	0.00	-59
A	1	10	c	141	10	5.4	0.21	-466
A	1	10	d	141	10	0.2	0.00	-111
A	1	10	e	141	5	0.4	0.00	-176
A	1	10	f	141	5	4.6	0.22	-456
A	1	50	a	141	15	2.5	0.01	-54
A	1	50	b	141	15	9.0	0.41	-593
A	1	50	c	141	10	8.9	0.32	-525
A	1	50	d	141	10	8.2	0.10	-419
A	1	50	e	141	5	5.3	0.05	-376
A	1	50	f	141	5	5.9	0.14	-470
A	1	75	a	131	15	0.2	0.00	-108
A	1	75	b	131	15	9.3	0.01	-184
A	1	75	c	131	10	0.3	0.04	-597
A	1	75	d	131	10	0.2	0.06	-311
A	1	75	e	131	5	0.3	0.01	-76
A	1	75	f	131	5	0.3	0.00	-106
A	1	100	a	139	15	0.2	0.00	-44
A	1	100	b	139	15	0.8	0.00	-297
A	1	100	c	139	15	0.1	0.00	-100
A	1	100	d	139	15	0.1	0.00	-50
A	1	100	e	138	10	0.1	0.00	-124
A	1	100	f	138	10	0.2	0.00	-155
A	1	100	g	137	5	0.3	0.00	-24
A	1	100	h	137	5	0.2	0.01	-44
A	1	100	I	137	5	0.3	0.00	-51
A	1	100	j	137	5	0.2	0.00	-12

Table E7. Fixed chloride data for the Concrete 1 with CIA B. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
1	B	10	a	139	15	0.6	0.06	-449
1	B	10	b	139	15	0.1	0.01	-85
1	B	10	c	139	10	8.4	0.19	-437
1	B	10	d	139	10	0.1	0.01	-203
1	B	10	e	134	5	0.2	0.00	-70
1	B	10	f	134	5	0.3	0.00	-76
1	B	50	a	139	15	1.7	0.06	-374
1	B	50	b	139	15	0.2	0.00	-125
1	B	50	c	137	10	5.0	0.07	-213
1	B	50	d	137	10	0.2	0.00	-42
1	B	50	e	134	5	0.9	0.01	-135
1	B	50	f	134	5	0.2	0.00	-22
1	B	75	a	139	15	0.2	0.00	-151
1	B	75	b	139	15	0.1	0.00	-125
1	B	75	c	137	10	0.2	0.02	-95
1	B	75	d	138	10	0.2	0.00	-67
1	B	75	e	134	5	0.2	0.00	-67
1	B	75	f	134	5	0.2	0.00	-84
1	B	100	a	139	15	0.1	0.01	-248
1	B	100	b	139	15	6.6	0.42	-357
1	B	100	c	139	15	0.2	0.01	-206
1	B	100	d	139	15	0.2	0.00	-137
1	B	100	e	138	10	0.2	0.00	-53
1	B	100	f	138	10	0.2	0.00	-72
1	B	100	g	134	5	0.3	0.00	-27
1	B	100	h	134	5	6.0	0.10	-616
1	B	100	I	134	5	0.2	0.00	-77
1	B	100	j	137	5	3.6	0.06	-381

Table E8. Fixed chloride data for the Concrete 1 with CIA C. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

CIA	Concrete	CIA Concentration (%)	Cell	Exposure (days)	Target Chloride (lbs/yd^3)	Measured Chloride (lbs/yd^3)	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)
C	1	10	a	141	15	7.0	1.30	-430
C	1	10	b	141	15	0.3	0.00	-87
C	1	10	c	141	10	4.0	0.01	-369
C	1	10	d	141	10	4.8	0.03	-348
C	1	10	e	141	5	0.8	0.03	-307
C	1	10	f	141	5	6.1	0.08	-336
C	1	50	a	144	15	9.2	0.32	-531
C	1	50	b	144	15	0.2	0.00	-69
C	1	50	c	144	10	4.6	1.18	-441
C	1	50	d	144	10	0.6	0.00	-59
C	1	50	e	144	5	0.8	0.00	-18
C	1	50	f	144	5	1.0	0.00	-84
C	1	75	a	149	15	0.3	0.01	-122
C	1	75	b	149	15	0.1	0.01	-189
C	1	75	c	149	10	5.0	0.08	-647
C	1	75	d	149	10	5.1	0.09	-380
C	1	75	e	149	5	0.1	0.01	-108
C	1	75	f	149	5	0.5	0.00	-130
C	1	100	a	141	15	0.2	0.00	-138
C	1	100	b	141	15	7.7	0.04	-408
C	1	100	c	141	15	0.5	0.01	-141
C	1	100	d	141	15	0.7	0.00	-151
C	1	100	e	141	10	0.2	0.00	-124
C	1	100	f	141	10	5.8	0.06	-349
C	1	100	g	144	5	2.6	0.04	-196
C	1	100	h	144	5	4.0	0.06	-272
C	1	100	I	144	5	0.6	0.01	-208

APPENDIX F

CHLORIDE THRESHOLD DATA

APPENDIX **F**

CHLORIDE THRESHOLD DATA

Table F1. Chloride threshold data for Concrete 0. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA Concentration (%)	Cell	Exposure (days)	Corrosion Initiated	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)	Chlorides Previous lbs /yd^3	Chlorides** 5g Sample lbs /yd^3
0	None	None	k	98	yes	0.09	-362	7.4	7.5
0	None	None	l	49	yes	0.07	-262	6.5	4.3
0	A	10	g	115	yes	0.11	-342	9.0	7.0
0	A	10	h	217	yes	0.03	-412	na	14.2
0	A	50	g	209	no	NM	-160	na	14.3
0	A	50	h	209	no	NM	-120	17.6	13.8
0	A	75	g	217	yes	0.04	-217	na	na
0	A	75	h	185	yes	0.09	-384	12.6	12.3
0	A	100	k	66	yes	0.15	-420	9.8	8.0
0	A	100	l	98	yes	0.11	-272	11.9	8.5
0	B	10	g	209	no	0.03	-528	9.2	17.2
0	B	10	h	209	no	NM	-171	7.4	na
0	B	50	g	209	no	NM	-116	4.9	13.7
0	B	50	h	209	no	NM	-127	8.8	16.0
0	B	75	g	209	no	NM	-123	4.7	14.3
0	B	75	h	209	no	NM	-83	7.6	14.4
0	B	100	k	209	no	NM	-26	4.0	11.6
0	B	100	l	209	no	0	-225	5.4	13.3
0	C	10	g	10	yes	0.12	-512	5.1	0.6
0	C	10	h	30	yes	0.13	-568	5.4	0.9
0	C	50	g	38	yes	0.05	-578	3.9	0.8
0	C	50	h	38	yes	0.13	-278	3.3	0.7
0	C	75	g	77	yes	0.18	-467	3.0	3.4
0	C	75	h	29	yes	0.24	-576	1.6	1.1
0	C	100	k	197	yes	0.19	-454	7.9	5.7
0	C	100	l	217	yes	0.21	-65	3.0	10.9

NM: Not Measured
na: not available
*: Corrosion did not initiate and test was terminated.
**: Final chloride analysis performed by collecting larger than previous concrete sample (5-6 g)

Table F2. Chloride threshold data for Concrete 1. [1 mm/yr = 39 mpy], [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA Concentration (%)	Cell	Exposure (days)	Corrosion Initiated	Corrosion Rate (mpy)	Corrosion Potential (mV vs CSE)	Chlorides Previous lbs /yd^3	Chlorides** 5g Sample lbs /yd^3
1	None	None	k	209	no	NM	-110	2.4	0.9
1	None	None	l	209	no	NM	-104	1.4	1.5
1	A	10	g	185	no	NM	-121	3.9	0.8
1	A	10	h	185	no	NM	-199	2.1	0.7
1	A	50	g	185	no	0	-244	1.2	0.8
1	A	50	h	185	no	NM	-92	1.5	1.4
1	A	75	g	185	no	NM	-173	1.8	1.5
1	A	75	h	185	no	NM	-123	1.3	1.0
1	A	100	k	185	no	NM	-97	6.9	4.1
1	A	100	l	185	no	NM	-112	1.7	1.2
1	B	10	g	185	no	NM	-187	3.2	0.5
1	B	10	h	185	no	NM	-117	0.5	0.3
1	B	50	g	185	no	NM	-141	0.3	0.3
1	B	50	h	185	no	0	-456	1.4	0.5
1	B	75	g	185	no	0	-182	1.7	1.0
1	B	75	h	185	no	0.02	-322	2.1	0.3
1	B	100	k	185	no	0.02	-293	0.6	0.4
1	B	100	l	185	no	NM	-188	2.5	0.8
1	C	10	g	185	no	NM	-167	1.1	0.5
1	C	10	h	185	no	NM	-132	0.8	0.6
1	C	50	g	185	no	NM	-98	0.8	0.3
1	C	50	h	185	no	NM	-113	1.0	0.8
1	C	75	g	185	no	NM	-101	0.7	0.3
1	C	75	h	185	no	NM	-120	0.7	0.2
1	C	100	k	185	no	NM	-74	0.6	0.4
1	C	100	l	185	no	NM	-177	1.2	0.4

NM: Not Measured
na: not available
*: Corrosion did not initiate and test was terminated.
**: Final chloride analysis performed by collecting larger than previous concrete sample (5-6 g)

APPENDIX G

SIMULATED CRACK MINIBEAM DATA

APPENDIX **G**

SIMULATED CRACK MINIBEAM DATA

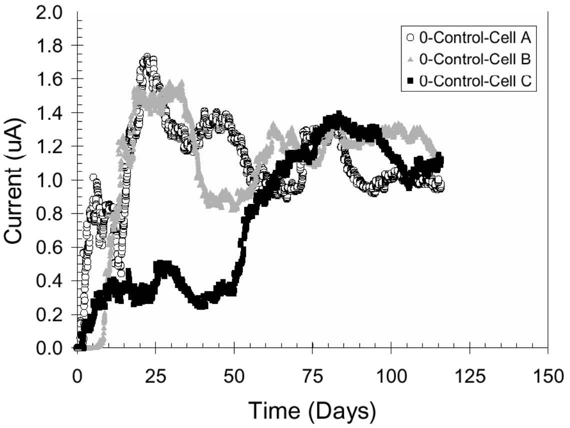


Figure G1. Performance of Control (no CIA) in presence of a crack.

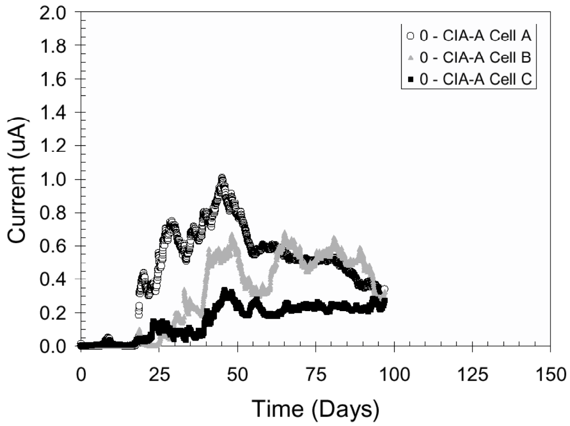


Figure G2. Performance of 50% CIA-A in presence of a crack.

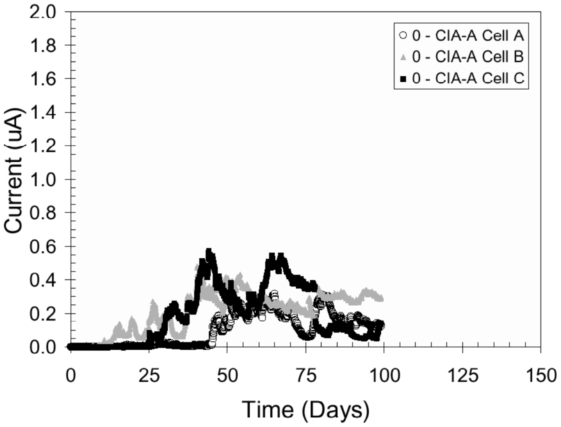


Figure G3. Performance of 100% CIA-A in presence of a crack.

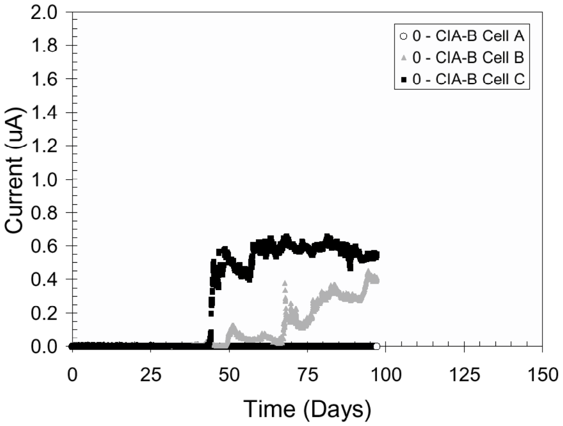


Figure G4. Performance of 50% CIA-B in presence of a crack.

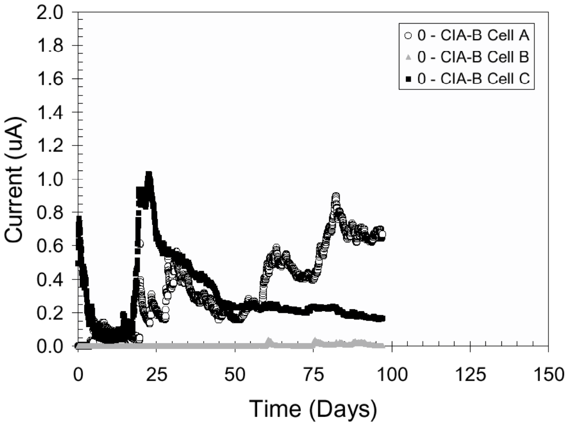


Figure G5. Performance of 100% CIA-B in presence of a crack.

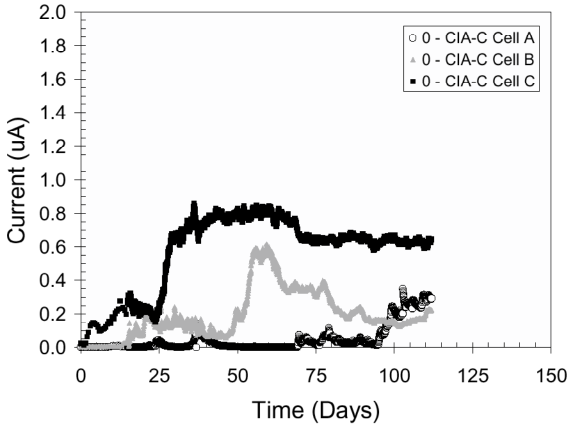


Figure G6. Performance of 50% CIA-C in presence of a crack.

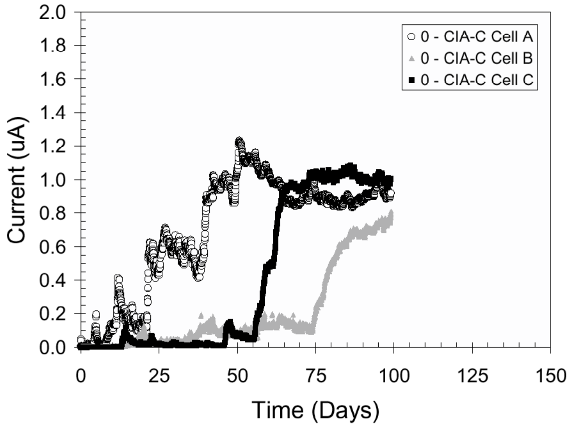


Figure G7. Performance of 100% CIA-C in presence of a crack.

APPENDIX H

LONG-TERM CONCRETE SLAB DATA

APPENDIX **H**

LONG-TERM CONCRETE SLAB DATA

Table H1. Data for individual slab specimens. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA (%)	Slab	Time to Initiation (days)	Current (µA)	Corrosion Coverage[(b)] (%)	Chlorides (lb/yd^3)
0	None	None	a	100	40	25	9
0	None	None	b	325	30	25	15
0	None	None	c	310	25	25	20
0	None	None	d	325	30	25	12
0	C	10	a	440	3	10	11
0	C	10	b	100	40	10	13
0	C	10	c	375	7	10	10
0	C	10	d	310	20	10	14
0	C	75	a	(a)	1	<10	3
0	C	75	b	(a)	<1	<10	8
0	C	75	c	(a)	<1	<10	6
0	C	75	d	(a)	1	<10	9
0	A	10	a	360	5	10	14
0	A	10	b	390	10	10	14
0	A	10	c	330	12	10	10
0	A	10	d	330	10	10	13
0	A	75	a	(a)	1	<10	4
0	A	75	b	(a)	2	<10	9
0	A	75	c	(a)	<1	<10	9
0	A	75	d	(a)	<1	<10	13
0	B	10	a	(a)	<1	<10	12
0	B	10	b	(a)	1	<10	10
0	B	10	c	(a)	1	<10	13
0	B	10	d	(a)	1	10	8
0	B	75	a	(a)	<1	0	7
0	B	75	b	(a)	<1	0	6
0	B	75	c	(a)	<1	<10	12
0	B	75	d	(a)	<1	0	8

(a): Did not initiate corrosion after approximately 450 day exposure period.

(b): Post test examination.

0%: No corrosion.
<10%: Some slight surface corrosion possible but negligible.
10%: Definite corrosion, but limited to approximately 10% of the steel surface.
25%: Approximately 25% surface coverage of corrosion.

APPENDIX I

CHLORIDE DIFFUSION CALCULATIONS AND DATA

APPENDIX **I**

CHLORIDE DIFFUSION CALCULATIONS AND DATA

EFFECTIVE DIFFUSION COEFFICIENT DETERMINATION

Effective diffusion coefficient was calculated based on the chloride measurements obtained from chloride diffusion tests. The calculation assumed a simplified model based on Fick's law for steady-state diffusion in a semi-infinite solid; it was further assumed that diffusion coefficient (D_{eff}) is independent of position x.

The boundary conditions for the equation are:

$C=C_0$ for t>0 at x=0

C=0 for t=0 at x>0

The solution to the diffusion equation takes the form:

$$c(x,t) = c_o\left[1 - erf\left(\frac{x}{2\sqrt{D_{eff}t}}\right)\right] = c_o\left[1 - erf(z)\right]$$

The chloride measurements at different depths (x) were entered into an Excel spreadsheet. Then, a built-in Excel analysis tool (SOLVER) was employed for a simultaneous fitting of the two variables (D_{eff} and C_0) to the experimental curve. However, SOLVER experienced considerable difficulties in fitting an experimental curve with only 3-4 datapoints. The algorithm requires the 'seed' values for both variables; once the values are entered, SOLVER attempted to fit the experimental curve in such a way that the sum of the squares of the actual/fitted pair variances be minimized (see Sum $(error)^2$ entries in Table I3). Given the small number of datapoints, the program ran through the iteration process, but 'froze' one the parameters and changed only the other one. The message "SOLVER could not find a feasible solution" typically appeared, and the resulting fitted curve was clearly far from the 'best fit' one.

In view of the above, it was decided to make further improvements to the fit manually, using the combination of SOLVER solutions and GOAL SEEK functions (works similarly to SOLVER, but changes only one specified variable at a time). The fitting was deemed finished when the sum of the squared variances was either at a certain value (typically 1 or less), or further changes to the fitting parameters did not lead to any marked improvements to the fit.

DIFFUSION DATA

Table I1. Chloride versus depth in concrete for Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3], [1 mm = 0.039 in]

Concrete	CIA	CIA %	Slab	Chloride Concentration (lb/yd^3) Slice (0.125 inch)							
				1	2	3	4	5	6	7	8
0	None	None	a		4.2	2.5	0.6		0.3		
0	None	None	b		7.6	5.7	3.8	1.9	0.7	0.2	0.1
0	None	None	c	7.4	5.9	3.4	0.3				
0	None	None	d		5.0	4.8	3.4		0.7		
0	A	10	a		5.8	4.2	2.2		0.2		
0	A	10	b		3.4	2.5	1.3		0.1		
0	A	10	c		3.3	2.7	1.3		0.2		
0	A	50	a		3.6	2.2	1.1		0.1		
0	A	50	b		3.8	3.2	1.6		0.2		
0	A	50	c		5.0	3.0	1.2		0.1		
0	A	75	a		7.4	7.2	2.4		0.1		
0	A	75	b		8.8	8.3	5.4		0.7		
0	A	75	c		6.7	5.7	3.1		0.1		
0	A	100	a		8.5	6.2	5.5		0.4		
0	A	100	b		7.2	6.4	4.1		0.2		
0	A	100	c		4.1	4.0	2.9		0.3		
0	B	10	b		3.0	2.4	1.1		0.1		
0	B	10	c		3.4	3.2	2.4		0.2		
0	B	50	a		2.7	2.1	1.5		0.1		
0	B	50	b		3.8	2.9	1.9		0.3		
0	B	50	c		3.9	2.6	1.6		0.9		
0	B	75	a		6.2	3.1	1.9		0.1		
0	B	75	b		2.4	1.5	0.7		0.2		
0	B	75	c		1.9	0.9	0.3		0.1		
0	B	100	a		8.2	5.1	2.7		0.2		
0	B	100	b		3.6	2.1	1.0		0.2		
0	B	100	c		3.3	1.8	0.7		0.2		
0	C	10	a		4.3	3.7	2.2		0.2		
0	C	10	b		6.1	5.7	4.2		1.8		
0	C	10	c		5.3	4.6	4.2		1.5		
0	C	50	a		7.8	5.3	3.7		1.0		
0	C	50	b		5.9	5.0	3.7		1.6		
0	C	50	c		3.1	3.9	1.4		0.1		
0	C	75	a		4.8	3.4	1.7		0.2		
0	C	75	b		6.7	6.5	3.6		0.2		
0	C	75	c		6.5	5.0	1.7		0.1		
0	C	100	a		5.4	2.9	1.6		0.2		
0	C	100	b		3.5	2.6	0.6		0.1		
0	C	100	c		5.2	3.4	1.4		0.1		

Table I2. Chloride versus depth in concrete for Concrete 1. [1 kg/m3 = 1.67 lb/yd3], [1 mm = 0.039 in]

Concrete	CIA	CIA %	Slab	Chloride Concentration (lb/yd^3) Slice (0.125 inch)							
				1	2	3	4	5	6	7	8
1	None	None	a		9.2	2.6	0.4				
1	None	None	b		6.7	2.2	0.3				
1	None	None	c		4.8	1.0	0.2				
1	None	None	d		4.9	1.7	0.5				
1	A	10	a		3.2	1.7	0.2				
1	A	10	b		1.8	0.7	0.1				
1	A	10	c		2.6	0.7	0.2				
1	A	50	a		3.5	0.8	0.3				
1	A	50	b		3.3	0.9	0.2				
1	A	50	c		2.6	0.7	0.2				
1	A	75	a		4.5	2.1	0.2				
1	A	75	b		3.4	1.0	0.3				
1	A	75	c		7.2	3.4	0.4				
1	A	100	a		6.5	2.2	0.4				
1	A	100	b		4.4	2.3	0.4				
1	A	100	c		3.6	1.5	0.2				
1	A	100	d		6.4	3.4	0.4				
1	B	10	b		4.7	2.0	0.3				
1	B	10	c		6.2	1.5	0.3				
1	B	50	a		5.0	1.5	0.2				
1	B	50	b		3.7	0.7	0.2				
1	B	50	c		5.3	1.2	0.1				
1	B	75	a		3.5	0.5	0.1				
1	B	75	b		1.9	0.3	0.2				
1	B	75	c		1.6	0.3	0.1				
1	B	100	a		4.0	0.5	0.1				
1	B	100	b		2.1	0.1	0.1				
1	B	100	c		2.4	0.3	0.1				
1	C	10	a		7.0	3.8	0.4				
1	C	10	b		3.4	1.2	0.3				
1	C	10	c		4.7	1.9	0.3				
1	C	10	d		3.4	0.7	0.3				
1	C	50	a		4.3	0.8	0.2				
1	C	50	b		2.8	0.7	0.2				
1	C	50	c		3.4	0.7	0.3				
1	C	75	a		3.8	0.6	0.1				
1	C	75	b		4.8	1.0	0.2				
1	C	75	c		2.7	0.5	0.2				
1	C	100	a		3.6	0.6	0.2				
1	C	100	b		7.2	1.4	0.1				

Table I3. Diffusion coefficients for Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA (%)	Slab	Effective Diffusion Coefficient (cm^2/s)	Sum of (Error)^2	Average Diffusion Coefficient (cm^2/s)	D$_{eff}$ Standard Deviation (cm^2/s)	Surface Concentration C$_o$ (lb/yd^3)	Average C$_o$ (lb/yd^3)
0	None	None	a	2.68E-08	0.49			11.2	
0	None	None	b	1.60E-08	0.48			13.6	
0	None	None	c	9.77E-09	2.78			14.0	
0	None	None	d	2.20E-08	0.90	1.86E-08	7.38E-09	9.3	12.0
0	A	10	a	2.02E-08	0.66			13.0	
0	A	10	b	1.59E-08	0.10			5.4	
0	A	10	c	1.99E-08	0.13	1.87E-08	2.40E-09	7.9	8.8
0	A	50	a	1.83E-08	0.02			8.5	
0	A	50	b	2.67E-08	0.70			8.5	
0	A	50	c	1.85E-08	0.47	2.12E-08	4.79E-09	11.7	9.5
0	A	75	a	2.20E-08	6.00			18.0	
0	A	75	b	2.53E-08	4.27			15.6	
0	A	75	c	2.03E-08	2.10	2.25E-08	2.54E-09	11.5	15.0
0	A	100	a	3.18E-08	3.87			16.7	
0	A	100	b	2.98E-08	2.81			14.3	
0	A	100	c	2.59E-08	1.17	2.92E-08	3.00E-09	8.6	13.2
0	B	10	b	1.39E-08	0.17			6.6	
0	B	10	c	2.82E-08	1.03	2.11E-08	1.01E-08	6.2	6.4
0	B	50	a	2.40E-08	0.18			5.1	
0	B	50	b	2.20E-08	0.19			6.1	
0	B	50	c	2.51E-08	0.11	2.37E-08	1.57E-09	6.4	5.8
0	B	75	a	1.40E-08	0.18			14.3	
0	B	75	b	1.46E-08	0.03			5.0	
0	B	75	c	1.14E-08	0.02	1.33E-08	1.70E-09	7.2	8.8
0	B	100	a	1.58E-08	0.20			21.2	
0	B	100	b	1.18E-08	0.28			10.2	
0	B	100	c	1.10E-08	0.05	1.29E-08	2.57E-09	9.0	13.5
0	C	10	a	1.48E-08	0.50			9.8	
0	C	10	b	3.20E-08	0.82			9.1	
0	C	10	c	2.65E-08	0.49	2.44E-08	8.78E-09	9.2	9.3
0	C	50	a	1.55E-08	0.26			13.6	
0	C	50	b	1.75E-08	0.37			9.1	
0	C	50	c	1.44E-08	1.97	1.58E-08	1.57E-09	6.7	9.8
0	C	75	a	1.24E-08	0.80			11.3	
0	C	75	b	9.64E-09	2.07			16.7	
0	C	75	c	1.68E-08	1.72	1.29E-08	3.61E-09	16.7	14.9
0	C	100	a	1.34E-08	0.09			15.3	
0	C	100	b	1.46E-08	0.45			9.3	
0	C	100	c	1.73E-08	0.29	1.51E-08	2.00E-09	10.5	11.7

Table I4. Diffusion coefficients for Concrete 1. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete	CIA	CIA (%)	Slab	Effective Diffusion Coefficient (cm^2/s)	Sum of $(Error)^2$	Average Diffusion Coefficient (cm^2/s)	D_{eff} Standard Deviation (cm^2/s)	Surface Concentration Co (lb/yd^3)	Average Co (lb/yd^3)
1	None	None	a	5.18E-09	0.12			50.2	
1	None	None	b	5.30E-09	0.06			33.4	
1	None	None	c	4.63E-09	0.04			40.2	
1	None	None	d	5.74E-09	0.04	5.21E-09	4.57E-10	22.8	36.6
1	A	10	a	7.56E-09	0.24			9.7	
1	A	10	b	6.45E-09	0.02			7.0	
1	A	10	c	7.13E-09	0.17	7.05E-09	5.60E-10	8.9	8.5
1	A	50	a	8.88E-09	0.36			13.3	
1	A	50	b	5.56E-09	0.14			11.3	
1	A	50	c	5.08E-09	0.04	6.51E-09	2.07E-09	9.8	11.4
1	A	75	a	6.31E-09	0.23			14.5	
1	A	75	b	5.05E-09	0.06			12.8	
1	A	75	c	5.89E-09	0.53	5.75E-09	6.42E-10	24.5	17.3
1	A	100	a	5.04E-09	0.07			24.8	
1	A	100	b	6.49E-09	0.21			14.0	
1	A	100	c	5.24E-09	0.06			13.5	
1	A	100	d	6.90E-09	0.70	5.92E-09	9.17E-10	19.3	17.9
1	B	10	b	6.43E-09	0.05			17.6	
1	B	10	c	5.57E-09	0.17	6.00E-09	6.08E-10	29.2	23.4
1	B	50	a	4.44E-09	0.04			21.7	
1	B	50	b	3.57E-09	0.04			20.2	
1	B	50	c	3.87E-09	0.08	3.96E-09	4.42E-10	26.1	22.7
1	B	75	a	3.01E-09	0.06			24.0	
1	B	75	b	2.27E-09	0.02			20.7	
1	B	75	c	2.22E-09	0.02	2.50E-09	4.42E-10	17.8	20.8
1	B	100	a	3.04E-09	0.08			26.6	
1	B	100	b	2.26E-09	0.02			22.0	
1	B	100	c	2.33E-09	0.01	2.54E-09	4.32E-10	24.7	24.5
1	C	10	a	6.71E-09	0.90			24.5	
1	C	10	b	6.55E-09	0.02			12.2	
1	C	10	c	6.31E-09	0.12			21.3	
1	C	10	d	4.65E-09	0.06	6.06E-09	9.51E-10	16.8	18.7
1	C	50	a	3.41E-09	0.04			25.1	
1	C	50	b	3.59E-09	0.01			15.7	
1	C	50	c	3.73E-09	0.06	3.58E-09	1.60E-10	17.6	19.4
1	C	75	a	3.24E-09	0.04			23.2	
1	C	75	b	3.80E-09	0.05			24.2	
1	C	75	c	2.69E-09	0.02	3.24E-09	5.55E-10	22.2	23.2
1	C	100	a	3.13E-09	0.06			23.3	
1	C	100	b	3.36E-09	0.08	3.25E-09	1.63E-10	42.3	32.8

APPENDIX J

MECHANICAL PROPERTY DATA

APPENDIX **J**

MECHANICAL PROPERTY DATA

Table J1. Mechanical property data for Concrete 0. [1 kPa = 0.145 psi]

	Compressive Stength (psi)		Modulus of Elasticity (psi x 10^6)		Flexural Strength (psi)	
Condition	28-Day	365-Day	28-Day	365-Day	28-Day	365-Day
Control	6,540	8,190	5.18	5.52	820	828
CIA-A 50%	7,030	NM	5.58	NM	830	NM
CIA-A 100%	7,280	8,670	4.94	5.35	680	750
CIA-B 50%	6,250	NM	4.98	NM	750	NM
CIA-B 100%	6,450	8,080	5.47	5.64	820	754
CIA-C 50%	6,840	NM	5.63	NM	830	NM
CIA-C 100%	7,030	7,890	5.79	5.65	820	726

Table J2. Mechanical property data for Concrete 1. [1 kPa = 0.145 psi]

Condition	Compressive Stength (psi)		Modulus of Elasticity (psi x 10^6)		Flexural Strength (psi)	
	28-Day	365-Day	28-Day	365-Day	28-Day	365-Day
Control	8,090	8,520	5.79	5.94	1,040	1,020
CIA-A 50%	9,760		6.19		990	
CIA-A 100%	10,000	10,040	5.72	6.53	950	1,080
CIA-B 50%	8,370		5.81		1,020	
CIA-B 100%	8,500	9,630	5.90	6.11	1,040	950
CIA-C 50%	8,990		6.18		960	
CIA-C 100%	8,920	9,710	5.97	6.33	1,015	1,170

APPENDIX K

RESISTIVITY DATA

APPENDIX **K**

RESISTIVITY DATA

Table K1. Resistivity data for Concrete 0.

Concrete	CIA	CIA (%)	Average Electrical Resistivity (ohm-cm)					
			Day 1	Day 7	Day 28	Day 90	Day 180	Day 365
0	None	None	645	4,801	5,900	7,956	8,368	9,465
0	A	10	591	5,419	7,476	8,573	9,259	10,770
0	A	50	528	3,875	5,418	6,996	7,681	9,260
0	A	75	549	3,160	4,801	6,584	7,407	8,916
0	A	100	460	686	4,252	5,898	7,339	8,367
0	B	10	576	4,732	6,859	7,339	9,328	10,015
0	B	50	576	4,527	6,516	7,407	8,985	10,015
0	B	75	658	5,075	6,173	8,093	10,220	11,590
0	B	100	555	5,007	7,133	7,544	9,534	10,770
0	C	10	675	4,595	6,175	7,407	8,848	9,877
0	C	50	679	3,841	5,900	7,819	8,916	9,945
0	C	75	672	4,665	7,476	9,671	12,002	13,717
0	C	100	645	4,115	6,653	8,505	11,111	12,414

Table K2. Resistivity data for Concrete 1.

Concrete	CIA	CIA (%)	Average Electrical Resistivity (ohm-cm)					
			Day 1	Day 7	Day 28	Day 90	Day 180	Day 365
0	None	None	610	8,230	No data	52,126	49,383	48,695
0	A	10	631	5,761	41,152	50,754	53,498	48,011
0	A	50	521	3,772	28,806	41,152	41,838	40,466
0	A	75	576	4,390	25,514	39,780	39,780	37,037
0	A	100	521	3,841	21,537	32,922	34,979	32,236
0	B	10	583	9,053	45,953	63,100	63,100	57,613
0	B	50	590	7,682	39,095	54,870	54,870	50,069
0	B	75	562	8,710	45,953	58,985	58,985	53,498
0	B	100	576	8,642	45,953	61,728	61,728	57,613
0	C	10	644	7,270	41,152	49,383	46,639	46,639
0	C	50	693	5,898	37,037	46,639	45,953	44,581
0	C	75	617	6,174	36,351	48,011	46,639	46,639
0	C	100	665	6,310	41,152	55,556	55,556	53,498

APPENDIX L

FRESH CONCRETE DATA

APPENDIX **L**

FRESH CONCRETE DATA

Table L1. Fresh concrete property data for Concrete 0.

Concrete	CIA	CIA (%)	Slump (inch)	Air (%)	Unit Weight (lb/ft^3)	Time of Set (Hr:Min) Initial	Time of Set (Hr:Min) Final
0	None	None	4.00	7.5	140.3	5:21	6:55
0	A	10	4.75	7.0	141.2	6:20	8:07
0	A	50	8.00	6.4	142.6	6:06	7:27
0	A	75	6.50	7.2	141.3	5:26	6:46
0	A	100	6.50	7.1	141.0	5:45	6:47
0	B	10	7.00	5.6	143.9	7:55	9:11
0	B	50	4.75	6.0	143.1	7:39	9:20
0	B	75	5.00	5.7	143.4	7:46	9:22
0	B	100	5.25	6.0	142.7	8:23	10:01
0	C	10	5.75	6.5	142.6	5:43	7:05
0	C	50	8.00	7.2	140.5	7:05	8:34
0	C	75	5.00	5.2	144.8	6:50	8:23
0	C	100	6.75	5.4	144.3	8:03	9:28

Table L2. Fresh concrete property data for Concrete 1.

Concrete	CIA	CIA (%)	Slump (inch)	Air (%)	Unit Weight (lb/ft^3)	Time of Set (Hr:Min) Initial	Time of Set (Hr:Min) Final
1	None	None	5.75	6.2	141.7	7:02	8:20
1	A	10	4.50	5.2	143.6	7:16	8:28
1	A	50	3.50	4.6	144.1	8:25	9:43
1	A	75	5.00	6.1	142.3	8:22	9:55
1	A	100	3.50	4.8	144.0	8:22	9:33
1	B	10	4.25	6.7	141.8	7:50	9:05
1	B	50	7.00	6.3	141.9	8:05	9:17
1	B	75	5.75	5.2	143.6	8:42	10:03
1	B	100	7.00	6.4	141.8	8:52	10:20
1	C	10	4.75	5.8	142.7	6:30	7:57
1	C	50	5.75	4.8	143.6	8:15	9:37
1	C	75	5.75	5.8	142.7	9:20	10:39
1	C	100	5.75	5.7	142.8	9:02	10:17

APPENDIX M

TABULATED CHLORIDE VERSUS TIME PREDICTIONS

APPENDIX **M**

TABULATED CHLORIDE VERSUS TIME PREDICTIONS

Table M1. Predictions for Concrete 0. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete 0 Time (yr)	No CIA (lb/yd^3)	CIA-A (lb/yd^3)	CIA-B (lb/yd^3)	CIA-C (lb/yd^3)	Time (yr)	No CIA (lb/yd^3)	CIA-A (lb/yd^3)	CIA-B (lb/yd^3)	CIA-C (lb/yd^3)
1	0.0	0.0	0.0	0.0	51	12.4	15.4	9.7	10.9
2	0.0	0.0	0.0	0.0	52	12.5	15.5	9.8	11.0
3	0.0	0.2	0.0	0.0	53	12.6	15.6	10.0	11.1
4	0.1	0.6	0.0	0.0	54	12.8	15.7	10.1	11.3
5	0.3	1.1	0.0	0.1	55	12.9	15.8	10.3	11.4
6	0.5	1.7	0.1	0.2	56	13.0	15.9	10.4	11.5
7	0.8	2.3	0.2	0.4	57	13.1	16.1	10.5	11.7
8	1.2	2.9	0.4	0.6	58	13.3	16.2	10.6	11.8
9	1.5	3.6	0.6	0.9	59	13.4	16.3	10.8	11.9
10	1.9	4.2	0.8	1.2	60	13.5	16.4	10.9	12.0
11	2.3	4.7	1.0	1.5	61	13.6	16.5	11.0	12.1
12	2.7	5.3	1.3	1.8	62	13.7	16.6	11.1	12.3
13	3.1	5.8	1.5	2.1	63	13.8	16.7	11.2	12.4
14	3.5	6.3	1.8	2.5	64	13.9	16.8	11.3	12.5
15	3.9	6.8	2.1	2.8	65	14.0	16.8	11.5	12.6
16	4.3	7.3	2.3	3.1	66	14.1	16.9	11.6	12.7
17	4.7	7.7	2.6	3.4	67	14.2	17.0	11.7	12.8
18	5.0	8.1	2.9	3.8	68	14.3	17.1	11.8	12.9
19	5.4	8.5	3.2	4.1	69	14.4	17.2	11.9	13.0
20	5.7	8.9	3.4	4.4	70	14.5	17.3	12.0	13.1
21	6.0	9.2	3.7	4.7	71	14.6	17.4	12.1	13.2
22	6.4	9.5	4.0	5.0	72	14.7	17.4	12.2	13.3
23	6.7	9.9	4.2	5.2	73	14.8	17.5	12.3	13.4
24	7.0	10.2	4.5	5.5	74	14.9	17.6	12.4	13.5
25	7.2	10.5	4.8	5.8	75	15.0	17.7	12.5	13.6
26	7.5	10.8	5.0	6.1	76	15.1	17.7	12.6	13.7
27	7.8	11.0	5.2	6.3	77	15.1	17.8	12.7	13.8
28	8.1	11.3	5.5	6.6	78	15.2	17.9	12.7	13.8
29	8.3	11.5	5.7	6.8	79	15.3	18.0	12.8	13.9
30	8.5	11.8	5.9	7.0	80	15.4	18.0	12.9	14.0
31	8.8	12.0	6.2	7.3	81	15.5	18.1	13.0	14.1
32	9.0	12.2	6.4	7.5	82	15.5	18.2	13.1	14.2
33	9.2	12.5	6.6	7.7	83	15.6	18.2	13.2	14.3
34	9.5	12.7	6.8	7.9	84	15.7	18.3	13.3	14.3
35	9.7	12.9	7.0	8.1	85	15.8	18.3	13.3	14.4
36	9.9	13.1	7.2	8.3	86	15.8	18.4	13.4	14.5
37	10.1	13.2	7.4	8.5	87	15.9	18.5	13.5	14.6
38	10.3	13.4	7.6	8.7	88	16.0	18.5	13.6	14.6
39	10.5	13.6	7.8	8.9	89	16.0	18.6	13.6	14.7
40	10.6	13.8	8.0	9.1	90	16.1	18.6	13.7	14.8
41	10.8	13.9	8.1	9.3	91	16.2	18.7	13.8	14.9
42	11.0	14.1	8.3	9.5	92	16.2	18.8	13.9	14.9
43	11.2	14.3	8.5	9.6	93	16.3	18.8	13.9	15.0
44	11.3	14.4	8.6	9.8	94	16.4	18.9	14.0	15.1
45	11.5	14.6	8.8	10.0	95	16.4	18.9	14.1	15.1
46	11.6	14.7	9.0	10.1	96	16.5	19.0	14.2	15.2
47	11.8	14.8	9.1	10.3	97	16.6	19.0	14.2	15.3
48	11.9	15.0	9.3	10.4	98	16.6	19.1	14.3	15.3
49	12.1	15.1	9.4	10.6	99	16.7	19.1	14.4	15.4
50	12.2	15.2	9.6	10.7	100	16.7	19.2	14.4	15.5

Table M2. Predictions for Concrete 1. [1 kg/m^3 = 1.67 lb/yd^3]

Concrete 1 Time (yr)	No CIA (lb/yd^3)	CIA-A (lb/yd^3)	CIA-B (lb/yd^3)	CIA-C (lb/yd^3)	Time (yr)	No CIA (lb/yd^3)	CIA-A (lb/yd^3)	CIA-B (lb/yd^3)	CIA-C (lb/yd^3)
1	0.0	0.0	0.0	0.0	51	3.6	4.4	0.8	1.5
2	0.0	0.0	0.0	0.0	52	3.7	4.5	0.8	1.5
3	0.0	0.0	0.0	0.0	53	3.8	4.6	0.9	1.6
4	0.0	0.0	0.0	0.0	54	4.0	4.7	0.9	1.7
5	0.0	0.0	0.0	0.0	55	4.1	4.8	1.0	1.8
6	0.0	0.0	0.0	0.0	56	4.2	4.9	1.0	1.8
7	0.0	0.0	0.0	0.0	57	4.3	5.1	1.1	1.9
8	0.0	0.0	0.0	0.0	58	4.4	5.2	1.1	2.0
9	0.0	0.0	0.0	0.0	59	4.5	5.3	1.2	2.0
10	0.0	0.0	0.0	0.0	60	4.6	5.4	1.2	2.1
11	0.0	0.1	0.0	0.0	61	4.7	5.5	1.3	2.2
12	0.0	0.1	0.0	0.0	62	4.8	5.6	1.3	2.2
13	0.1	0.1	0.0	0.0	63	4.9	5.7	1.4	2.3
14	0.1	0.2	0.0	0.0	64	5.0	5.8	1.4	2.4
15	0.1	0.2	0.0	0.0	65	5.1	5.9	1.5	2.5
16	0.2	0.3	0.0	0.0	66	5.2	6.0	1.5	2.5
17	0.2	0.4	0.0	0.0	67	5.3	6.1	1.6	2.6
18	0.3	0.4	0.0	0.0	68	5.4	6.2	1.6	2.7
19	0.3	0.5	0.0	0.0	69	5.5	6.3	1.7	2.7
20	0.4	0.6	0.0	0.1	70	5.6	6.4	1.7	2.8
21	0.5	0.7	0.0	0.1	71	5.7	6.5	1.8	2.9
22	0.5	0.8	0.0	0.1	72	5.8	6.6	1.9	2.9
23	0.6	0.9	0.0	0.1	73	5.8	6.7	1.9	3.0
24	0.7	1.0	0.0	0.1	74	5.9	6.8	2.0	3.1
25	0.8	1.1	0.0	0.1	75	6.0	6.9	2.0	3.2
26	0.9	1.2	0.1	0.2	76	6.1	7.0	2.1	3.2
27	1.0	1.4	0.1	0.2	77	6.2	7.1	2.1	3.3
28	1.1	1.5	0.1	0.2	78	6.3	7.2	2.2	3.4
29	1.2	1.6	0.1	0.3	79	6.4	7.3	2.2	3.4
30	1.3	1.7	0.1	0.3	80	6.5	7.4	2.3	3.5
31	1.4	1.9	0.1	0.4	81	6.6	7.4	2.3	3.6
32	1.5	2.0	0.2	0.4	82	6.6	7.5	2.4	3.6
33	1.6	2.1	0.2	0.4	83	6.7	7.6	2.5	3.7
34	1.7	2.2	0.2	0.5	84	6.8	7.7	2.5	3.8
35	1.8	2.4	0.2	0.5	85	6.9	7.8	2.6	3.8
36	1.9	2.5	0.2	0.6	86	7.0	7.9	2.6	3.9
37	2.1	2.6	0.3	0.6	87	7.1	8.0	2.7	4.0
38	2.2	2.8	0.3	0.7	88	7.1	8.0	2.7	4.0
39	2.3	2.9	0.3	0.7	89	7.2	8.1	2.8	4.1
40	2.4	3.0	0.4	0.8	90	7.3	8.2	2.8	4.2
41	2.5	3.1	0.4	0.9	91	7.4	8.3	2.9	4.2
42	2.6	3.3	0.4	0.9	92	7.4	8.4	3.0	4.3
43	2.7	3.4	0.5	1.0	93	7.5	8.4	3.0	4.4
44	2.9	3.5	0.5	1.0	94	7.6	8.5	3.1	4.4
45	3.0	3.6	0.5	1.1	95	7.7	8.6	3.1	4.5
46	3.1	3.8	0.6	1.2	96	7.8	8.7	3.2	4.6
47	3.2	3.9	0.6	1.2	97	7.8	8.7	3.2	4.6
48	3.3	4.0	0.7	1.3	98	7.9	8.8	3.3	4.7
49	3.4	4.1	0.7	1.3	99	8.0	8.9	3.3	4.8
50	3.5	4.2	0.7	1.4	100	8.0	9.0	3.4	4.8

APPENDIX N

TABULATED CORROSION RATE AND CUMULATIVE CORROSION VERSUS TIME PREDICTIONS

APPENDIX **N**

TABULATED CORROSION RATE AND CUMULATIVE CORROSION VERSUS TIME PREDICTIONS

Table N1.a. Data for Concrete 0. [1 mm/yr = 39 mpy], [1 mm = 39 mil]

	Concrete 0							
Time	No CIA		CIA-A		CIA-B		CIA-C	
(yr)	(mpy)	(mil)	(mpy)	(mil)	(mpy)	(mil)	(mpy)	(mil)
1	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
2	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
3	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
4	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
5	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
6	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
7	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
8	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
9	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
10	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
11	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
12	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
13	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
14	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
15	0.05	0.0	0.05	0.0	0.00	0.0	0.00	0.0
16	0.06	0.1	0.05	0.1	0.00	0.0	0.00	0.0
17	0.07	0.2	0.06	0.2	0.00	0.0	0.00	0.0
18	0.08	0.3	0.07	0.2	0.00	0.0	0.00	0.0
19	0.09	0.4	0.08	0.3	0.00	0.0	0.00	0.0
20	0.10	0.5	0.08	0.4	0.00	0.0	0.00	0.0
21	0.12	0.6	0.09	0.5	0.00	0.0	0.00	0.0
22	0.13	0.7	0.10	0.6	0.00	0.0	0.05	0.1
23	0.13	0.8	0.10	0.7	0.04	0.0	0.05	0.1
24	0.14	1.0	0.11	0.8	0.05	0.1	0.06	0.2
25	0.15	1.1	0.11	0.9	0.06	0.2	0.06	0.2
26	0.16	1.3	0.12	1.0	0.07	0.2	0.07	0.3
27	0.17	1.5	0.12	1.1	0.07	0.3	0.07	0.4
28	0.18	1.6	0.13	1.3	0.08	0.4	0.07	0.4
29	0.19	1.8	0.13	1.4	0.09	0.5	0.07	0.5
30	0.20	2.0	0.14	1.5	0.10	0.6	0.08	0.6
31	0.20	2.2	0.14	1.7	0.10	0.7	0.08	0.7
32	0.21	2.4	0.14	1.8	0.11	0.8	0.08	0.7
33	0.22	2.7	0.15	2.0	0.12	0.9	0.09	0.8
34	0.22	2.9	0.15	2.1	0.12	1.0	0.09	0.9
35	0.23	3.1	0.16	2.3	0.13	1.1	0.09	1.0
36	0.24	3.3	0.16	2.4	0.14	1.3	0.09	1.1
37	0.24	3.6	0.16	2.6	0.14	1.4	0.10	1.2
38	0.25	3.8	0.17	2.8	0.15	1.6	0.10	1.3
39	0.26	4.1	0.17	2.9	0.15	1.7	0.10	1.4
40	0.26	4.4	0.17	3.1	0.16	1.9	0.10	1.5
41	0.27	4.6	0.17	3.3	0.16	2.0	0.11	1.6
42	0.27	4.9	0.18	3.4	0.17	2.2	0.11	1.7
43	0.28	5.2	0.18	3.6	0.17	2.4	0.11	1.8
44	0.28	5.5	0.18	3.8	0.18	2.6	0.11	1.9
45	0.29	5.8	0.19	4.0	0.18	2.8	0.11	2.1
46	0.29	6.0	0.19	4.2	0.19	2.9	0.12	2.2
47	0.30	6.3	0.19	4.4	0.19	3.1	0.12	2.3
48	0.30	6.6	0.19	4.6	0.20	3.3	0.12	2.4
49	0.31	7.0	0.20	4.8	0.20	3.5	0.12	2.5

Table N1.b. Data for Concrete 0. [1 mm/yr = 39 mpy], [1 mm = 39 mil]

	Concrete 0							
Time	No CIA		CIA-A		CIA-B		CIA-C	
(yr)	(mpy)	(mil)	(mpy)	(mil)	(mpy)	(mil)	(mpy)	(mil)
50	0.31	7.3	0.20	5.0	0.21	3.7	0.12	2.7
51	0.32	7.6	0.20	5.2	0.21	4.0	0.13	2.8
52	0.32	7.9	0.20	5.4	0.22	4.2	0.13	2.9
53	0.33	8.2	0.20	5.6	0.22	4.4	0.13	3.0
54	0.33	8.6	0.21	5.8	0.23	4.6	0.13	3.2
55	0.33	8.9	0.21	6.0	0.23	4.9	0.13	3.3
56	0.34	9.2	0.21	6.2	0.23	5.1	0.13	3.4
57	0.34	9.6	0.21	6.4	0.24	5.3	0.14	3.6
58	0.35	9.9	0.21	6.6	0.24	5.6	0.14	3.7
59	0.35	10.3	0.22	6.8	0.25	5.8	0.14	3.9
60	0.35	10.6	0.22	7.0	0.25	6.1	0.14	4.0
61	0.36	11.0	0.22	7.3	0.25	6.3	0.14	4.1
62	0.36	11.3	0.22	7.5	0.26	6.6	0.14	4.3
63	0.36	11.7	0.22	7.7	0.26	6.8	0.15	4.4
64	0.37	12.1	0.23	7.9	0.26	7.1	0.15	4.6
65	0.37	12.4	0.23	8.2	0.27	7.4	0.15	4.7
66	0.37	12.8	0.23	8.4	0.27	7.6	0.15	4.9
67	0.38	13.2	0.23	8.6	0.27	7.9	0.15	5.0
68	0.38	13.6	0.23	8.9	0.28	8.2	0.15	5.2
69	0.38	13.9	0.23	9.1	0.28	8.5	0.15	5.3
70	0.39	14.3	0.23	9.3	0.28	8.7	0.15	5.5
71	0.39	14.7	0.24	9.6	0.29	9.0	0.16	5.6
72	0.39	15.1	0.24	9.8	0.29	9.3	0.16	5.8
73	0.39	15.5	0.24	10.0	0.29	9.6	0.16	6.0
74	0.40	15.9	0.24	10.3	0.30	9.9	0.16	6.1
75	0.40	16.3	0.24	10.5	0.30	10.2	0.16	6.3
76	0.40	16.7	0.24	10.8	0.30	10.5	0.16	6.4
77	0.41	17.1	0.24	11.0	0.30	10.8	0.16	6.6
78	0.41	17.5	0.25	11.2	0.31	11.1	0.16	6.8
79	0.41	17.9	0.25	11.5	0.31	11.4	0.17	6.9
80	0.41	18.3	0.25	11.7	0.31	11.7	0.17	7.1
81	0.42	18.8	0.25	12.0	0.31	12.1	0.17	7.3
82	0.42	19.2	0.25	12.2	0.32	12.4	0.17	7.4
83	0.42	19.6	0.25	12.5	0.32	12.7	0.17	7.6
84	0.42	20.0	0.25	12.7	0.32	13.0	0.17	7.8
85	0.43	20.4	0.25	13.0	0.32	13.3	0.17	7.9
86	0.43	20.9	0.25	13.3	0.33	13.7	0.17	8.1
87	0.43	21.3	0.26	13.5	0.33	14.0	0.17	8.3
88	0.43	21.7	0.26	13.8	0.33	14.3	0.17	8.5
89	0.43	22.2	0.26	14.0	0.33	14.7	0.18	8.6
90	0.44	22.6	0.26	14.3	0.34	15.0	0.18	8.8
91	0.44	23.0	0.26	14.5	0.34	15.3	0.18	9.0
92	0.44	23.5	0.26	14.8	0.34	15.7	0.18	9.2
93	0.44	23.9	0.26	15.1	0.34	16.0	0.18	9.4
94	0.44	24.4	0.26	15.3	0.35	16.4	0.18	9.5
95	0.45	24.8	0.26	15.6	0.35	16.7	0.18	9.7
96	0.45	25.3	0.27	15.9	0.35	17.1	0.18	9.9
97	0.45	25.7	0.27	16.1	0.35	17.4	0.18	10.1
98	0.45	26.2	0.27	16.4	0.35	17.8	0.18	10.3
99	0.45	26.6	0.27	16.7	0.36	18.1	0.18	10.4
100	0.46	27.1	0.27	16.9	0.36	18.5	0.18	10.6

APPENDIX O

PROPOSED STANDARD METHOD FOR QUALIFYING CORROSION INHIBITING ADMIXTURES THAT MITIGATE CORROSION OF REINFORCING STEEL IN CONCRETE

APPENDIX **O**

PROPOSED STANDARD METHOD FOR QUALIFYING CORROSION INHIBITING ADMIXTURES THAT MITIGATE CORROSION OF REINFORCING STEEL IN CONCRETE

1. SCOPE

1.1 This practice is focussed on relatively short-term laboratory test procedures for qualifying corrosion inhibiting admixtures. The results are utilized in a life prediction model to establish the benefits of the admixture.

1.2 The performance criteria for a corrosion inhibiting admixture include corrosion resistance of non-cracked concrete, corrosion resistance of cracked concrete, and concrete properties.

2. PURPOSE

2.1 The purpose is to provide testing protocols and performance criteria for qualifying admixtures as a “corrosion inhibiting admixture” as defined the American Concrete Institute Manual of Concrete Practice: “chemical admixtures to be added to the portland cement concrete mixtures, usually in very small concentrations, for the primary purpose of corrosion protection.”

3. REFERENCED DOCUMENTS

3.1 AASHTO Standards:

- AASHTO T 259-80 (1993): Resistance of Concrete to Chloride Ion Penetration.
- AASHTO T 260-82: Sampling and Testing for Total Chloride Ion in Concrete and Concrete Raw Materials.

3.2 ASTM Standards:

- ASTM C 33-93: Specification for Concrete Aggregates.
- ASTM C 39-94: Test Method for Compressive Strength of cylindrical Concrete Specimens.
- ASTM C 78-94: Test Method for Flexural Strength of Concrete.
- ASTM C 143-90a: Test Method for Slump of Hydraulic Cement Concrete.
- ASTM C 150-95: Specification for Portland Cement.
- ASTM C 192-90a: Practice for Making and Curing Concrete Test Specimens in the Laboratory.
- ASTM C 403-95: Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance.
- ASTM C 457-90: Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete.
- ASTM C 469-94: Test Method for Static Modulus of Elasticity and Poisson’s Ratio of Concrete in Compression.
- ASTM C 511-95: Specifications for Moist Cabinets, Moist Rooms and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes.
- ASTM E 104-85(1991): Practice for Maintaining Constant Relative Humidity By Means Of Aqueous Solutions.

4. SIGNIFICANCE

4.1 Corrosion inhibiting admixtures (CIA) are becoming more used in concrete structures for the purpose of mitigating corrosion of the reinforcing steel. In concrete, corrosion is a complex mechanism that involves oxidation (corrosion) and reduction reactions, and requires (1) a critical concentration of chloride ions to initiate corrosion (breakdown the natural passivity of steel in a high pH environment) and (2) water and oxygen (necessary for the reduction reaction). Since corrosion requires the simultaneous production (oxidation) and consumption (reduction) of electrons to occur, corrosion mitigation can be accomplished by mitigating either the oxidation reaction or the reduction reaction, or both.

4.2 There are many different possible inhibitors that are either on the market or that may be introduced. A major problem for the bridge engineer is to be able to evaluate these products by comparable methods. In addition, the claims made by the inhibitor manufacturers are often vague and the mechanisms by which inhibitors mitigate corrosion are often not well understood.

4.3 The need of a standard testing protocol that utilizes relatively short-term laboratory tests is addressed by this standard. In addition, this standard also provides performance criteria for the acceptance of a CIA. To be qualified as a “corrosion inhibitor”, the CIA should affect the corrosion mechanism and not just impede chloride migration through the concrete. This is not to say that an admixture that impedes chloride migration is not worthwhile, but that it is not a “corrosion inhibiting” admixture.

4.4 No single methodology based on relatively short-term laboratory tests will be able to predict or replicate the performance of a CIA in the diverse environments and field conditions present around the world. The performance will be dependent on numerous variables impossible to reproduce in the laboratory including concrete mix design, structural design (loading characteristics, etc.), climatic conditions, salting procedures, maintenance procedures, etc. This standard is not meant to provide a ranking of different CIAs because such a ranking is likely dependent on the variables discussed above. This standard will qualify individual CIAs as to their benefit in mitigating corrosion.

5. USE

5.1 This standard is designed for use by bridge engineers whose responsibilities include designing, specifying materials, etc. This standard will permit the engineer, through standard testing methodologies, to qualify a CIA for use. Once qualified, the engineer can be reasonably confident that the CIA will provide some significant benefit to extending the life of a structure.

6. EQUIPMENT

6.1 The equipment necessary includes:

- LPR corrosion rate instrument with correction for solution resistance.
- Humidity chamber for 98% humidity exposures.
- Oven (or hot room) capable of 100^{o}F (38^{o}C) exposures.
- Vacuum chamber.
- Chloride measurement equipment for AASHTO T 260-82 titration method.
- Core drill for 51-mm (2-inch) cores.
- Saw to slice core in 3.2-mm (0.125-in) slices.

- Crusher/grinder to crush concrete slices.
- Standard concrete batching equipment.
- Mechanical test equipment for concrete properties (compressive strength, flexural strength, and modulus of elasticity).
- Fresh-state concrete property measurement equipment (slump, time to set, and air void content).

7. EXPERIMENTAL CONCRETE

7.1 The experimental concrete is intended to be high quality concrete that would meet specifications for a typical concrete bridge structure. The recommended concrete is an air-entrained Portland cement concrete containing 423 kg/m^3 (705 lb/yd^3) of Type I cement (ASTM C150). The air content should be maintained at 6 $\underline{+}$ 1.5%, w/c at 0.40, and the cementitious material paste content at 30% by volume.

7.2 The fine and coarse aggregates should conform to gradation requirements of ASTM C 33. The fine sand should be silica sand (SiO_2 content in excess of 99%) with a specific gravity of approximately 2.65 and a fineness modulus of approximately 2.84. the coarse aggregate should be a pure quartz with a maximum size of 9.5 mm (3/8-inch).

7.3 Additional admixtures may be required to maintain the concrete properties including an air-entraining admixture and a conventional water reducing/set-retarding admixture.

7.4 Batching of the concrete should be performed in large enough batches to ensure uniform concrete properties throughout the specimens prepared. Minimum batches of

0.028 to 0.071 m^3 (1 to 2.5 ft^3) prepared in a 0.085-m^3 (3.0-ft^3) capacity rotary drum mixer are recommended.

7.5 The addition of the components to the mixer follows the guidelines outlined in ASTM C 192. Trial batches should be made to determine the admixture levels required to obtain the desired slump and air content. The air-entraining admixture was placed directly on the sand in the mixer. The set retarding/water-reducing admixture was blended with the mix water. Manufacturer’s recommendations should be followed with regard to the addition of the CIA. The batch water should be adjusted to reflect any water contributions coming from the admixtures.

7.6 During the first 24-hr period after casting, the specimens should be maintained in a condition to assure that no moisture loss occurs. After removal from the molds, the specimens should be stored in a moist room meeting the requirements of ASTM C 511. The minimum curing period for all specimens was 28 days.

8. CONCRETE PROPERTY MEASUREMENTS

8.1 It is important to establish that the addition of CIA does not degrade mechanical or physical properties of concrete. The following tests should be performed:

ASTM C 39 - Compressive Strength.

ASTM C 78 - Flexural Strength.

ASTM C 469 - Modulus of Elasticity.

ASTM C 143 - Slump.

ASTM C 403 - Setting Time.

ASTM C 457 - Air Void System.

8.2 Triplicate samples should be tested for the mechanical property tests: compressive and flexural strength and modulus of elasticity. These tests should be performed with no CIA added and at two levels of CIA concentrations (100 and 50% of the maximum recommended dosage).

8.3 Slump, setting time, and stability of the air void system are performed at the time of batching.

8.4 The analysis is to determine whether the CIA caused any detrimental effect on the concrete properties that would cause the concrete to not meet specifications for its intended use.

9. CHLORIDE DIFFUSION MEASUREMENTS

9.1 Chloride diffusion coefficients are required to estimate the chloride concentration as a function of time for the prediction model. Chloride penetration should be measured using AASHTO Designation T-259-80 (1993). The standard time of 90-days may be insufficient to get results for low chloride diffusion coefficients of a good quality concrete. 365 days has been shown sufficient for even high performance silica fume concrete. In addition to the proposed longer exposure time, it is proposed to alter the exposure conditions of the standard test to include cyclic ponding. The recommended cyclic exposure is two-weeks ponded and two-weeks dry.

9.2 The following modification for collecting the concrete samples for analysis is recommended. Chloride concentration is required at 3.2-mm (1/8-in) intervals down from the concrete surface. One method capable of accomplishing this is coring the concrete slab with a 51-mm (2-in) bit. The core is sliced in 3.2-mm thick slices using a cut-off saw (the thickness of the saw should be accounted for in determining depth of the slice). Each slice can be ground or crushed for chloride analysis. It is recommended that the chloride analysis follow AASHTO T 260-82.

9.3 Effective diffusion coefficient is calculated based on the chloride measurements obtained from chloride diffusion tests. The calculation assumes a simplified model based on Fick's law for steady-state diffusion in a semi-infinite solid; it is further assumed that diffusion coefficient (D_{eff}) is independent of position x.

The boundary conditions for the equation are:

$C=C_0$ for t>0 at x=0

C=0 for t=0 at x>0

The solution to the diffusion equation takes the form:

$$c(x,t) = c_o\left[1 - erf\left(\frac{x}{2\sqrt{D_{eff}t}}\right)\right] = c_o[1 - erf(z)]$$

The actual test data is then fitted with a trendline, utilizing any available algorithm software, which is capable of optimizing the fit by varying D_{eff} and C_o parameters simultaneously.

The goal of such optimization is to achieve the minimum possible sum of the squared variances for each pair of the test/fitted datapoints.

10. CORROSION RATE MEASUREMENTS (FIXED-CHLORIDE TESTS)

10.1 Corrosion rate measurements as a function of chloride concentrations are required to estimate total cumulative corrosion for the life prediction model. Also required is the chloride threshold for corrosion initiation. Both of these data are provided by the following test procedures.

Specimen Design

10.2 Figures 1 and 2 show the specimen configuration. The specimen design is such that the concrete environment is in contact with the cross-sectional face of the reinforcing steel bar rather than the circumferential area. The steel specimen is a #18 (57-mm [2.25-in] diameter) steel bar about 12.7-mm (0.5-in) long. Except for the cross-sectional area, which is in contact with the concrete, all other areas of the steel are sealed with coal tar epoxy. The steel surface in contact with the concrete is given an 80-grit finish. The total surface area of the steel in contact with the concrete is 2,548-mm^2 (3.98 in^2). The steel specimen is snugly fitted into a plastic mold (a PVC pipefitting) and all edges are sealed. A 19-mm [0.75-in] cover of concrete is cast onto the cross-sectional steel surface. Both the reference and counter electrode were fabricated from platinized niobium wire (niobium wire with 100-micron platinum coating).

10.3 All specimens were given a 28-day cure at room temperature by ponding with saturated calcium hydroxide solution.

Chloride Incorporation

10.4 The specimens are thoroughly dried after the 28-day curing cycle to facilitate chloride uptake. The following sequence is followed to prepare the samples before exposing them to the 98% humidity:

1. Dry samples at 38 ^{o}C (100 ^{o}F) in a controlled temperature room for 7-days.
2. Apply epoxy concrete sealant (Sikagard) to joint between mortar and plastic mold.
3. Dry under vacuum at 38 ^{o}C (100 ^{o}F) for two additional-days.

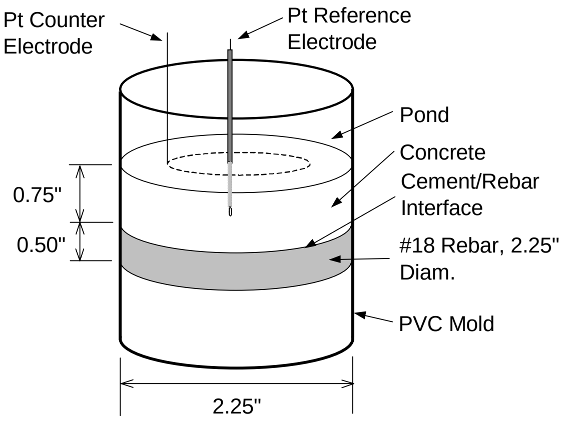


Figure 1. Specimen design for fixed-chloride corrosion tests. [1 mm = 0.039 in]

Figure 2. Photograph of individual fixed-chloride test specimen.

4. Pond with 6 ml of the desired chloride solution for 14-days at 38^{o}C (100^{o}F). Ponding is carried out within an hour after completing step 3.
5. Rinse off any excess chloride solution from the specimen surface pat dry with tissue and place them in the environmental chambers.

10.5 Weight-loss measurements are performed after the drying cycle to determine the amount of water loss by the specimens. Weight-loss must account for the epoxy applied in Step 2 above. The weight-loss will provide a measure of water loss and is required to calculate the chloride concentration in the ponding solution (Paragraph 10.6).

10.6 The chloride ponding in step 4 is designed to replace the pore water loss with sufficient chloride to achieve the desired chloride concentration. The following is the calculation for the chloride concentration in the ponding solution.

1. This example calculation is for a targeted 6 kg/m^3 (10 lb/yd^3) chloride concentration.
2. The volume of concrete that is added to the mold to get 17.5-mm (0.75-in) cover is 0.000049 m^3 (0.000064 yd^3).
3. The weight of chloride to be added to the concrete specimen is equal to the volume of concrete times the weight of chloride per cubic yard of concrete [0.000049 m^3 x 6 kg/m^3 = .00029 kg = 0.29 g of chloride].
4. The total water lost due to drying can be either calculated from theoretical free-water content or measured directly during drying. For theoretical free-water calculations, it is assumed that 100% of free-water is removed during drying and that free-water makes up 4.3% of the concrete weight. A volume of 0.000049 m^3 times a density of

2291 kg/m^3 (3820 lb/yd^3), density of concrete used in this study, gives a concrete weight of 112 g (0.247-lb). The free-water in the concrete specimen is equal to the concrete weight times the percent of free-water [111 g x 4.3% = 4.8 g of water].

5. The amount of chloride calculated in (3) when dissolved in the amount of free-water computed in (4) [0.29 g of chloride / (0.29 g + 4.8 g of water)], gives a 5.7% chloride solution or 9.4% NaCl.

6. Drying the majority of free-water and ponding the specimen with 9.4% NaCl solution should result in a concrete with 6 kg/m^3 (10 lb/yd^3) chloride, once all of the free-water is replaced with the ponding solution. With the pore water and the ponding solution having the same chloride concentration, equilibrium exists with respect to chloride concentration and no further chloride diffusion is expected. This assumes that there is no drying of the concrete or binding of the chlorides in the paste (outside of the pore water).

Humidity Control

10.7 Following incorporation of the chloride into the concrete, the following is the recommended cyclic exposure: (1) one week 98% relative humidity at 21^oC [70^oF]), (2), one week ponded with saturated CaOH solution (no chloride) (3) one week dry, and repeat. No further chloride ponding is performed. It is assumed that the severe drying and ponding provides a relatively uniform and constant chloride concentration at the steel surface.

10.8 Humidity control in the environmental test chambers is achieved by applying a layer of saturated salt solution at the bottom of the chamber. This method of humidity control is well established (ASTM E104). Each of the test chambers (444 mm by

356 mm by 165 mm [17.5 in by 14 in by 6.5 in]) was filled with 1 L of the required salt solution, which gave an approximate 19-mm (0.75-in) layer of the solution at the bottom. The samples (24 in each chamber) were supported on a plastic grid above the surface of the solution (see Figure 3). The actual humidity and temperature in each chamber was measured with a Thermo-hygrometer and were found to be within 2 to 3 percent (or degrees) of the desired values (98% relative humidity and 21°C [70°F]).

Measured LPR Corrosion Rate

10.9 The measured dependent variables in the fixed chloride tests are corrosion potential, corrosion rate, and chloride concentration at the steel surface. The potential of each specimen with respect to a copper/copper sulfate electrode (CSE) is made periodically during the exposure period. Final measurements are made during each of the three exposure conditions (high humidity, wet, and dry).

10.10 Corrosion rate measurements are determined using the linear polarization resistance (LPR) technique while correcting for the solution resistance component in the measurement. This correction can be accomplished by conventional LPR measurements while compensating for voltage (IR-drop) created by the solution resistance, or by measuring the solution resistance directly with a high frequency technique. Electrochemical impedance spectroscopy can also be used. It is imperative that some proven technique be used since the error in a conventional DC electrochemical method of LPR measurement can be significant. [The concept that LPR applies a small potential perturbation to the steel and therefore IR-drop is small and insignificant is wrong.]

Figure 3. Photograph of fixed chloride test specimens in humidity container.

10.11 The exposure period for the fixed-chloride corrosion rate measurements is a minimum of three months (90 days). During this exposure, it is recommended that corrosion rates be measured periodically. However, the most important data is collected at the end of the exposure. The corrosion rate should be measured three separate times during the final week of exposure and averaged to give the corrosion rate for that particular condition.

Measured Chloride Concentration

10.12 Following breakdown of each test specimen, the concrete and the steel surface are easily and cleanly separated (following cutting opposite sides of the mold and removing the mold) to expose the concrete surface in contact with the steel. Concrete sample for chloride analysis is collected using a lathe or cut-off saw. The initial concrete layer in contact with the steel is removed to clean any corrosion products from the concrete surface (by grinding or a lathe). The sample for chloride analysis should provide a minimum of 7 to 10 grams of concrete.

10.13 It is recommended that chloride analyses be performed using AASHTO T 260-82.

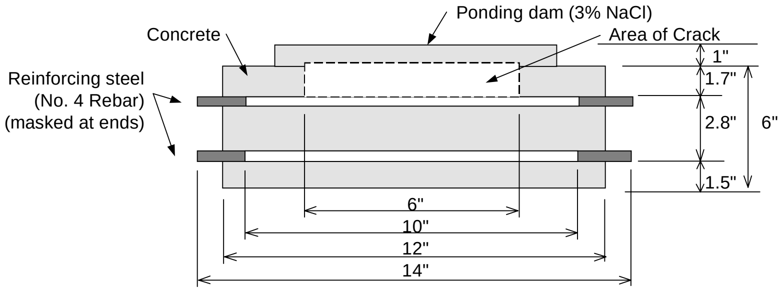
Test Matrix

10.14 Fixed-chloride tests are performed at three chloride concentrations (3, 6, and 9 kg/m^3 [5, 10, and 15 lb/yd^3]). It is recommended that tests be performed at two to three CIA concentrations (10, 50, and 100% of maximum recommended dosage). A control with no CIA is also included in the test matrix. Four replicates for each condition are to be tested. This provides a matrix of 36 tests specimens (3 chloride concentrations – two inhibitor concentrations and a control – four replicates). A matrix of 48 tests are required if three CIA concentrations are tested.

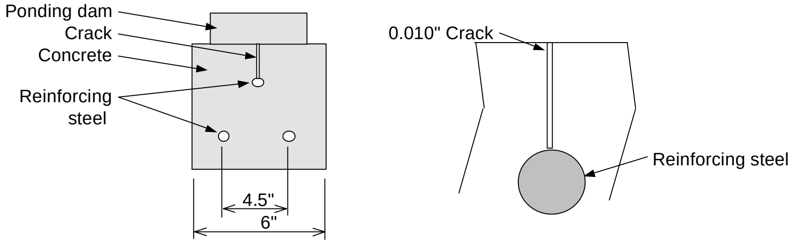
11. SIMULATED CRACK BEAM TEST

11.1 This test simulates the common case when corrosion of reinforcing steel in concrete is accelerated by the formation of surface cracks. This test provides results with regard to the corrosion inhibiting capabilities of the CIA in the presence of cracks down to the steel bar level. The design of a simulated crack beam specimen is shown in Figure 4. Figure 5 shows a photograph of the pre-cracked minibeams under test. The specimen design produces a 152-mm (6-in) long simulated longitudinal crack down to the top of the reinforcing steel. This design was selected because of the increased crack-reinforcing steel interface as compared to a transverse crack. The macrocell established by this relative long crack-steel interface greatly enhances the measurement current resolution over the very small (point) interface created by a transverse crack.

11.2 A crack is simulated by inserting a 0.25-mm (10-mil) thick shim along the length and down to the surface of the top reinforcing steel specimen. The shim is inserted during casting and is pulled out after approximately 8 hours. In this manner, a uniform crack down to the reinforcing steel surface is simulated. The top surface of the slab is ponded with 3% NaCl solution. The dependent variable measured is the coupled current between the top reinforcing steel specimen and the two steel bars in the bottom of the slab. This is accomplished using a zero-resistance ammeter. An increase in the macrocell current between the top and bottom steel bars indicate the onset of active corrosion of the upper steel bar due to the aggressive action of chlorides. Figure 6 shows the results for the experimental concrete with no CIA added.



a. Side view.



b. Front view.

c. Expanded front view of crack.

Figure 4. Schematic of the pre-cracked minibeam specimen. [1 mm = 0.039 in]

Figure 5. Photograph of pre-cracked concrete minibeam tests.

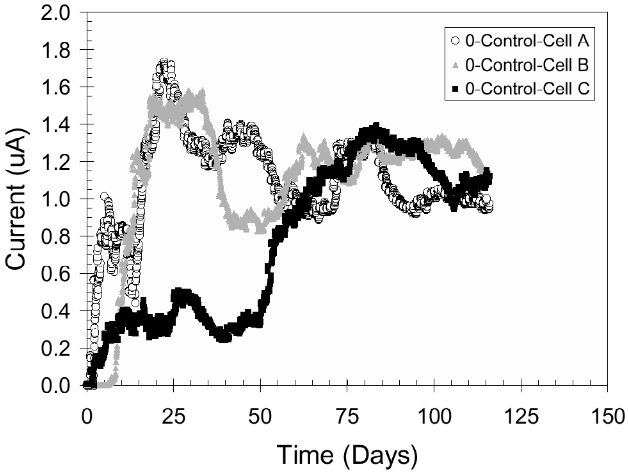


Figure 6. Coupled current versus time for pre-cracked minibeam tests.

11.3 The simulated crack beam tests are performed in triplicate using the maximum recommended dosage of the CIA or other dosages if recommended by the manufacturer. In addition, a control (no CIA) is also performed in triplicate. The control should be performed with each set of tests because of the sensitivity of this test to specimen preparation.

12. PREDICTIVE MODEL (CUMULATIVE CORROSION VERSUS TIME)

Model Description

12.1 One performance criterion for qualifying a CIA is based on a quantitative value related to life extension of a concrete structure. The first step in this process is to develop a life prediction model. The sole purpose of this model is to facilitate the application of the performance criterion for a "typical" concrete structure and is not designed to include all the necessary variables that might be required to predict life of a particular structure in a specific location. In addition, the specific case of cracked structures is not addressed in this prediction model. The following presents the proposed life prediction model for a non-cracked concrete structure.

12.2 The concrete structure parameters used in this model include a concrete cover of 64 mm (2.5 in) above the reinforcing steel. It is assumed that salt applications occur at the beginning of the structure's life. Following the first application and for all times thereafter, the concentration of salt at the structure's surface is equivalent to 18 kg/m^3 (30 lb/yd^3). This significantly simplifies the calculation of chloride concentration as a function of time. Also, once cracking occurs, the corrosion rate is not affected by the presence of cracks.

12.3 The life of a structure is divided into the three phases: Phase I - Corrosion Initiation, Phase II - Corrosion Propagation without Damage, and Phase III - Damage to Structure. The following information is required for the above model and can be determined by the testing protocol presented above.

Phase I - Corrosion Initiation

12.4 Phase I is defined as the time prior to corrosion initiation. The calculation Phase I life requires (1) diffusion coefficients for chloride and (2) critical chloride concentration required to initiate corrosion. The diffusion coefficient is determined in Section 9 above and averaged for the replicate specimens. A typical value for the experimental concrete is 1.9×10^{-8} cm^2/s. Using this value and the diffusion equation in Paragraph 9.3 above, the chloride concentration as a function of time can be calculated (see Figure 7).

12.5 The chloride threshold concentration to initiate corrosion can be either (1) determined from the extrapolation of the corrosion rate versus chloride concentration to negligible corrosion rates or (2) assumed based on other direct experiments used to calculate chloride threshold. It should be noted that chloride threshold may be dependent on the specific test conditions and concrete mix variables.

12.6 To calculate chloride threshold based on the above experimental procedures, it is assumed that, once corrosion initiates, the corrosion rate versus chloride is a linear function. For the experimental concrete, a value of the linear corrosion rate versus chloride calculated using the procedures in Section 10 is:

$$CR = 0.032Cl - 0.078.$$

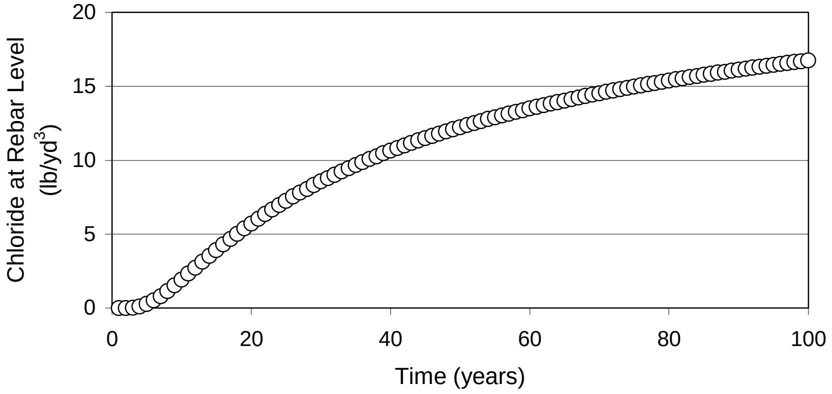


Figure 7. Chloride as a function of time. [1 kg/m^3 = 1.67 lb/yd^3]

Where CR in the corrosion rate in mpy and Cl is the chloride concentration in lb/yd^3. At a corrosion rate of 0.0013 mm/yr (0.05 mpy) (negligible corrosion), the estimated chloride threshold for corrosion initiation is 2.4 kg/m^3 (4.0 lb/yd^3). For a corrosion threshold of 2.4 kg/m^3 (4 lb/yd^3), Figure 7 gives a Phase I life of 15 years.

Phase II - Corrosion Propagation without Damage

12.7 Phase II life extends until damage occurs. The three parameters necessary to calculate Phase II life are (1) corrosion rate as a function of chloride concentration, (2) chloride concentration as a function of time, and (3) cumulative corrosion necessary to initiate damage. Items 1 and 2 are previously discussed above. For the final item, 0.05 mm (2 mil) of cumulative corrosion is assumed to initiate cracking damage.

12.8 The corrosion rate and cumulative corrosion as a function of time can be calculated by combining chloride concentration as a function of time and the corrosion rate as a function of chloride concentration. For the experimental concrete being used in the example calculation, Figure 8 gives the cumulative corrosion versus time. The end of Phase II life is defined when the predicted cumulative corrosion is 0.05 mm (2 mil). From Figure 8 this gives an end of Phase II life at 30 years.

Phase III - Damage to Structure

12.9 Phase III is defined as the life of the structure from when damage starts to when damage becomes significant. The two parameters necessary to calculate Phase III life are (1) cumulative corrosion versus time (Figure 8) and (2) cumulative corrosion to end life. The cumulative corrosion to end Phase III life is a difficult parameter to establish and depends on many variables including maintenance procedures during the life of a structure, concrete variables, structure loading, etc. Also, what constitutes “end of life”

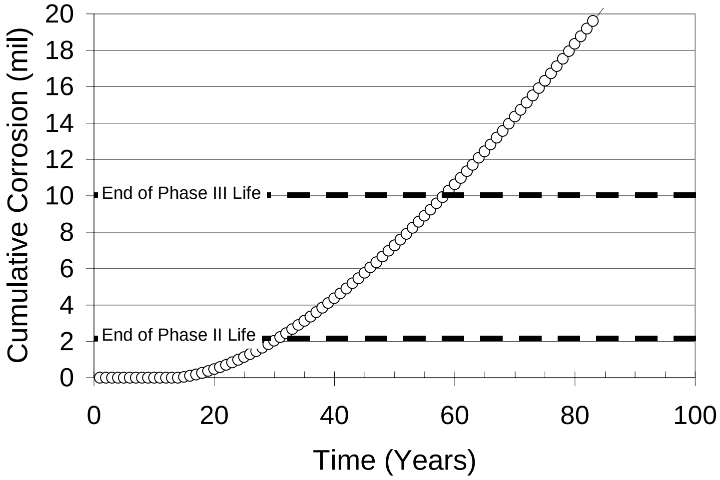


Figure 8. Cumulative corrosion versus time. [1 mm = 39 mil]

is important. In this scenario, it is assumed that no maintenance is performed and that Phase III life is the point in time when sufficient damage has occurred to require significant repairs. The value of 0.25 mm (10 mil) of cumulative corrosion over the entire structure is assumed to define the end of Phase III life.

12.10 From Figure 8, the end of Phase III life is estimated as 58 years for the example calculation.

13. PERFORMANCE CRITERIA

13.1 The performance criteria for qualifying a CIA designed to mitigate corrosion of reinforcing steel in concrete are as follows:

> Criterion 1. The CIA should provide an improvement over the base (no CIA) condition with respect to the predicted life by a minimum of 25 percent.
>
> Criterion 2. Increase in life must be due to one or both of the following: (1) increased chloride threshold for initiation of corrosion or (2) a decrease in the slope of the regression fit of corrosion rate versus chloride concentration.
>
> Criterion 3. The CIA should provide some improvement in corrosion performance for cracked concrete.
>
> Criterion 4. The CIA should not adversely affect the concrete properties in such a manner that pertinent specifications are not met. At a minimum, these should include compressive strength, flexural strength, modulus of elasticity, slump, time-to-set, and air content. Other properties that were not specifically studied in this project but could have a significant effect on concrete performance are air distribution and shrinkage.

13.2 Performance Criterion 1 is a quantitative comparison of the Phase III life prediction of a control (no CIA) concrete to a CIA concrete. The calculation is a simple percent increase (100 x [CIA – Control]/control) in predictive life for the inhibitor to be qualified by this standard.

13.3 Performance Criterion 2 is a qualitative criterion that requires a portion of the benefit of the CIA be attributed to a decrease in the corrosion properties. It is possible that a CIA only impedes chloride permeability. Although this type of CIA can extend the predicted life, it is not considered a “corrosion inhibitor.”

13.4 Performance Criterion 3 is a qualitative criterion that requires the CIA to provide some beneficial effect when measured in the presence of a preformed crack in the concrete. This benefit can be (1) an increase in time to initiation of corrosion or (2) a decrease in the measured coupled current following initiation, or both.

13.5 Performance Criterion 4 is a qualitative criterion that requires the CIA additive not significantly alter concrete properties in a detrimental manner. The concrete properties are those discussed in Section 8 above: compressive strength, flexural strength, modulus of elasticity, slump, setting time, and stability of the air void system.