**PROTECTION OF REINFORCEMENT
WITH CORROSION INHIBITORS, PHASE II**

Final Report
December 2000

JHR 00-279                    Project 96-2 Phase II
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16. Abstract

Costs due to corrosion of reinforcement in concrete caused by deicing salts have been estimated at up to $1 billion per year in the U.S. alone. For most situations, corrosion-inhibiting admixtures offer significant advantages over other protection methods to delay initiation of corrosion for the service life of the structure.

Phase II of this project continued the evaluation of two new corrosion inhibiting chemicals, described as alkali metal and ammonium slats of an alkenyl-substituted succinic acid. Tests compared them with two commercial corrosion-inhibiting admixtures as well as with a typical air-entrained control concrete. Corrosion testing consisted of weekly wetting (with salt solution) and drying cycles applied to slab type specimens and to 2-inch and 3-inch diameter lollipop specimens. Lollipops with a longitudinal saw-cut or with a preformed longitudinal "crack" were also tested. Linear polarization techniques measured corrosion rates. Reinforcing bars were removed and visually examined at completion of testing. Other test results included compression strength, absorption, setting time, and chloride penetration.

The new chemicals produced significant improvements in corrosion protection compared with the control concrete and with the two commercial inhibitors. After about 24 months or corrosion monitoring, specimens with the two prototype chemicals showed no sign of corrosion. For specimens with a saw-cut or preformed crack, the chemicals produced greatly reduced amounts of reinforcement corrosion.

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# SI* (MODERN METRIC) CONVERSION FACTORS

## APPROXIMATE CONVERSIONS TO SI UNITS

| Symbol | When You Know | Multiply By | To Find | Symbol |
|---|---|---|---|---|
| | | **LENGTH** | | |
| in | inches | 25.4 | millimetres | mm |
| ft | feet | 0.305 | metres | m |
| yd | yards | 0.914 | metres | m |
| mi | miles | 1.61 | kilometres | km |
| | | **AREA** | | |
| in² | square inches | 645.2 | millimetres squared | mm² |
| ft² | square feet | 0.093 | metres squared | m² |
| yd² | square yards | 0.836 | metres squared | m² |
| ac | acres | 0.405 | hectares | ha |
| mi² | square miles | 2.59 | kilometres squared | km² |
| | | **VOLUME** | | |
| fl oz | fluid ounces | 29.57 | millilitres | mL |
| gal | gallons | 3.785 | Litres | L |
| ft³ | cubic feet | 0.028 | metres cubed | m³ |
| yd³ | cubic yards | 0.765 | metres cubed | m³ |

NOTE: Volumes greater than 1000 L shall be shown in $m^3$

| Symbol | When You Know | Multiply By | To Find | Symbol |
|---|---|---|---|---|
| | | **MASS** | | |
| oz | ounces | 28.35 | grams | g |
| lb | pounds | 0.454 | kilograms | kg |
| T | short tons (2000 lb) | 0.907 | megagrams | Mg |
| | | **TEMPERATURE (exact)** | | |
| °F | Fahrenheit temperature | 5(F-32)/9 | Celcius temperature | °C |

## APPROXIMATE CONVERSIONS TO SI UNITS

| Symbol | When You Know | Multiply By | To Find | Symbol |
|---|---|---|---|---|
| | | **LENGTH** | | |
| mm | millimetres | 0.039 | inches | in |
| m | metres | 3.28 | feet | ft |
| m | metres | 1.09 | yards | yd |
| km | kilometres | 0.621 | miles | mi |
| | | **AREA** | | |
| mm² | millimetres squared | 0.0016 | square inches | in² |
| m² | metres squared | 10.764 | square feet | ft² |
| ha | hectares | 2.47 | acres | ac |
| km² | kilometres squared | 0.386 | square miles | mi² |
| | | **VOLUME** | | |
| mL | millilitres | 0.034 | fluid ounces | fl oz |
| L | litres | 0.264 | gallons | gal |
| m³ | metres cubed | 35.315 | cubic feet | ft³ |
| m³ | metres cubed | 1.308 | cubic yards | yd³ |
| | | **MASS** | | |
| g | grams | 0.035 | ounces | oz |
| kg | kilograms | 2.205 | pounds | lb |
| Mg | megagrams | 1.102 | short tons (2000 lb) | T |
| | | **TEMPERATURE (exact)** | | |
| °C | Celcius temperature | 1.8C+32 | Fahrenheit temperature | °F |

°F  -40    0    32    40    80   98.6  120   160   180   200   212
°C  -40  -20       0       20   37   40        60        80   100
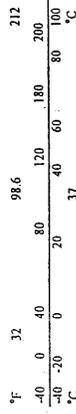
* SI is the symbol for the International System of Measurement

# PREFACE

This is the final report on the JHRAC Project 96-2 Phase II entitled "Protection of Reinforcement with Corrosion Inhibitors."  This work was sponsored by the Joint Highway Research Advisory Council of the University of Connecticut and the Connecticut Department of Transportation.  This work was originally presented in the first author's thesis to the Graduate Faculty of the University of Connecticut in partial fulfillment of the requirements for the degree of Master of Science. The support of the Connecticut Department of Transportation is gratefully acknowledged.

# TABLE OF CONTENTS

# LIST OF TABLES

# LIST OF FIGURES

**Solution Resistance and Corrosion Rate Data**

**Solution resistance charts:**

**2-inch diameter lollipops**:

**3-inch diameter lollipops:**

**Slabs**:

**Pre-cracked lollipop specimens:**

**Corrosion rate charts:**

**2-inch diameter lollipops:**

**3-inch diameter lollipops:**

**Slabs**:

**Pre-cracked lollipop specimens:**

# CHAPTER 1.0
# INTRODUCTION

## 1.1 PROBLEM STATEMENT

Steel corrodes actively in oxygen rich environments due to its large content of iron. Chloride ions found in deicing salt and seawater accelerate the corrosion of steel and therefore are a concern for reinforced concrete structures. Structures located in areas of harsh winters or near marine environments are very susceptible to corrosion due to the high presence of chloride ions.

The Bureau of Public Records (now the Federal Highway Administration) mandated the current policy for acceptable winter road conditions in 1955. This wet road policy required that in the winter, vehicle tires should be in direct contact with pavement on federal funded highways [1]. This policy calls for bare roadway surfaces and in an effort to achieve this, deicing salts are used.

The corrosion of concrete reinforcement is a very expensive problem, amounting in billions of dollars of damage worldwide. Corrosion repair costs due to the use of deicing salts have been estimated at $325 million to $1 billion per year in the United States [2].

When steel is embedded in concrete a passive film develops and covers the steel surface. Cement paste is alkaline (pH between 12 and 14) and enables the formation of this thin passive film coating on steel that protects the steel from corroding. After passing through the hardened concrete the chloride ions present in deicing salts destroy this protective layer.

Several solutions to this problem have been proposed and tested, though to date no ideal solution has been found. Some of these methods involve increasing the concrete cover over the rebar, reducing water/cement ratios, using denser concrete, using latex or polymer modified concrete overlays, adding waterproofing membrane with asphalt overlay, coating the rebar with epoxy or zinc, protecting the rebar cathodically, and using corrosion inhibiting admixtures. Initially, the use of epoxy coatings was thought to be the "ideal" solution for the prevention of rebar corrosion, but the long-term effectiveness of this method is being questioned [3].

Ideally, a concrete corrosion prevention system would protect the reinforcing steel from the initiation of corrosion for the duration of the structure's service life. The characteristics of corrosion inhibiting admixtures appear to make them the current leading candidates for achieving this goal. The high costs or lack of effectiveness for some of the other methods reveal several advantages for the using corrosion inhibiting admixtures. These advantages are:

1. The admixture is uniformly distributed throughout the concrete, therefore all of the steel in the structure is equally protected.
2. The use of the admixtures is not skill dependent, it only consists of adding the correct amount of admixture.
3. Construction quality control generally is not a concern with admixtures as it is with other methods (i.e., damaging of epoxy coatings, difficulty of working

with low water/cement ratio or low slump concrete mixes).
4. There is no maintenance required with an inhibiting admixture system.

Some of the many compounds and chemicals that have been tried as corrosion inhibiting admixtures [4, 5] produced favorable results as an inhibitor. However, some of these chemicals had adverse effects on some of the concrete properties, and some were also too expensive. Presently there are few corrosion inhibiting admixtures in use that do not have detrimental effects on other aspects of the concrete properties. Furthermore, these existing inhibitors only delay the onset of corrosion in laboratory tests [6].

## 1.2 INTENT OF RESEARCH

The objective of this research was to evaluate the performances of two prototype corrosion inhibitors in a concrete environment relative to a standard air-entrained control concrete mix and to two existing commercial concrete corrosion inhibitors. These prototype chemicals were disodium tetrapropenyl succinate and diammonium tetrapropenyl succinate, hereafter referred to as DSS and DAS, respectively. These chemicals were described as alkali metal and ammonium salts of an alkenyl-substituted succinic acid. They were supplied as aqueous solutions at about 20 % active ingredient concentration. Appendix B contains Material Safety Data Sheets for the DSS and DAS.

A prior phase of the research used lollipop and slab type specimens [48]. This second phase of the research (this portion of the research) continued to monitor and evaluate the Phase I specimens. In addition, a new type of specimen was developed to study the behaviors of these chemicals in cracked concrete. Also different concentrations of the prototype inhibitors were used in the Phase II mixes, based on results from the Phase I research.

The effectiveness of the inhibitors was evaluated using linear polarization measurements and visual inspections. Other effects of the inhibitors were also evaluated, including concrete strength, absorption, setting time, and chloride ingress.

## 1.3 ORGANIZATION OF REPORT

Chapter 2.0 provides a brief review of corrosion principles and electrochemistry that pertain to this particular study. Chapter 3.0 is a literature review of past research on corrosion of steel reinforcement embedded in concrete. Chapter 4.0 discusses the experimental program, including materials, corrosion tests (specimen design, test environment, specimen fabrication, cycling procedure, and corrosion testing method), and other tests (compression strength, absorption, setting time, and chloride penetration). The results are presented in Chapter 5.0. Finally, Chapter 6.0 provides the conclusions and recommendations based on the results of this research. The corrosion test data for all specimens is presented fully in Appendix A.

This report is essentially the thesis of Mr. Paul D. Goodwin, former Graduate Research Assistant, submitted as part of the requirements for the Master of Science degree [52].

# CHAPTER 2.0
# CORROSION PRINCIPLES AND MONITORING TECHNIQUE

## 2.1 PRINCIPLES OF CORROSION

There are many variables in corrosion but this study is specific. This study investigated the corrosion of steel through wetting and drying cycles. For this purpose, only pertinent corrosion principles will be discussed.

Corrosion is defined as the deterioration of a material through a chemical or electrochemical reaction with its environment. For reinforcing steel in concrete, the steel will react with the chlorides and oxygen in its surroundings.

The corrosion of metal is a process that returns metal to its natural form. Steel is primarily made of iron. The natural state of iron is iron oxide, as it exists in iron ore. Iron oxide is the natural state of iron because it is more thermodynamically stable in this form.

When an electrolyte or aqueous solution is in the presence of the material, wet corrosion occurs. This pertains directly to the corrosion of steel reinforcement in concrete that becomes saturated with water. This study attempted to mirror this wet corrosion state because it is responsible for the majority of deterioration of metal.

### 2.1.1 Types of Corrosion

There are several types of corrosion that can occur. All are identical in the chemical process of corrosion. They differ in how and where they attack the metal. This study tried to maintain a state of general corrosion, where the corrosives attack the steel uniformly. An evenly spread general corrosion reduces the possibility of a more concentrated attack on a small area of the metal. However, other forms of corrosion can occur. Crevice corrosion is one possibility. When an electrolyte is present in a small gap such as one between a metal and its protective coating, an intensive localized corrosion occurs [7]. The electrolyte may have been trapped upon application of the coating or penetrated the coating through a hole or gap. These conditions can cause an aggressive attack in the area of the electrolyte. Another form is galvanic corrosion. This occurs when two different metals are connected electrically in the presence of an electrolyte. There exists a natural potential difference between any two metals. The greater this potential difference, the greater the rate of corrosion in the more negative metal. When two different metals are in contact, the more positive metal will force the more negative metal to corrode at an accelerated rate. The area of exposure also has an effect on the corrosion rate. If a large area of the more positive metal is exposed while only a small area of the more negative metal is, then the corrosion rate of the more negative metal is increased.

### 2.1.2 Passivity of Steel

Under certain conditions steel is passive, where the corrosion rate for the metal is relatively low. Iron is considered an active-passive metal and therefore steel behaves

similarly.  Passivity, defined simply, refers to a loss of chemical reactivity under certain conditions [7].  Steel achieves this by having a passive film form along its surface.

### 2.1.3 Corrosion Factors

The cement paste in concrete is alkaline with a pH typically between 12 and 14.  This paste surrounds reinforcing steel in concrete.  Many researchers believe this alkaline environment facilitates the protective passive film around the steel [4, 6, 8, 9, 13, 14].  The passive film is not invulnerable, though.  It can be damaged both chemically and mechanically.

Some examples of chemical damage are carbonation and chloride ingress.  Carbonation is the result of the reaction of atmospheric carbon dioxide and hydroxides in the cement paste.  Through this reaction carbonates and water are formed.  The carbonates that form from this reaction consume the hydroxides present and therefore can lower the pH of the concrete below the value of 8.0.  This action causes the steel to depassivate, leaving it susceptible to attack from corrosives.  The likelihood of this occurrence is relative to the impermeability characteristics of the concrete.  Adequate depths of the concrete cover for the bars and the use of good quality concrete mixes have greatly reduced the concern for carbonation and its effect on corrosion.

Ingress of chlorides, on the other hand, is far more destructive to the steel.  These damaging chlorides are common in concrete environments.  They are mainly present in marine environments and in deicing salts, however they can also be due to admixtures containing chlorides and chloride contaminated cements, aggregates, and batch water.  The majority of researchers believe that chlorides that migrate through the concrete, as opposed to those already present in it, are the most destructive [14].

Most researches also believe that the presence of chlorides causes the depassivation of reinforcing steel [6, 8, 9, 16, 17].  The exact cause is not yet fully understood.  Even at pH levels where the concrete should be passivated, chlorides allow corrosion.

Concrete is a permeable material and thus will absorb moisture.  Different mixtures of concrete have different rates of moisture infusion.  Along with moisture, oxygen and chlorides also diffuse through the uncracked concrete to the surface of the steel.  A cathodic reaction is induced by the presence of these elements.  The moisture and chlorides act as an electrolyte which facilitate the flow of ionic current.  The chlorides initiate corrosion and oxygen fuels the reaction.  It is reasonable to expect that the lower the absorption and permeability of the concrete, the longer corrosion requires to initiate.

### 2.1.4 Corrosion Inhibitors

The rate of corrosion can also be reduced by using corrosion inhibitors.  These inhibitors are chemical compounds that reduce the corrosion rate of metals existing in actively corrosive environments.  Using corrosion inhibitors on embedded steel is difficult.  There are corrosion inhibitors known to protect bare steel, but some of these compounds have ill effects on the properties of hardened concrete.  Setting time, strength, and durability are key elements that can be affected by the corrosion inhibitors.  Few commercial

corrosion inhibitors are available for concrete due to the stringent requirements that must be met for concrete.

To increase the service life of steel imbedded in concrete, some form of corrosion protection must be provided. This is accomplished by using corrosion inhibitors or reducing the rate of chloride ingress to the steel reinforcement. The ideal protection would accomplish both of these.

## 2.2 CORROSION MONITORING TECHNIQUE

There are several methods of monitoring the corrosion of steel reinforcement in concrete for laboratory tests. The more common of these methods are the half cell, macro-cell, linear polarization and AC impedance. The half cell method only predicts the probability of corrosion activity and the macro-cell method can not measure the actual corrosion rate. Linear polarization and AC impedance are capable of measuring the corrosion rate that occurs in a system. The experiments in this research used a linear polarization technique, therefore only this technique will be discussed.

## 2.2.1 Linear Polarization

The linear polarization method is capable of measuring the corrosion rate of a system. It is achieved by shifting the corrosion potential typically 10 to 20mV towards more noble or more active. Both the potential and the current required to achieve this potential are recorded. The potential is stepped in increments (called over-potentials, typically 1mV) from one extreme to the other and each of these steps is recorded. The plotting of these data yields a polarization curve. When this is done, it is observed that the applied current density is a linear function of the electrode potential. The polarization resistance, $R_p$, is equal to the slope of this curve ($\Delta E/\Delta i_{app}$). The corrosion rate can then be determined through the relations of the Stern-Geary equation:

$$R_p = \frac{\Delta E}{\Delta i_{app}} \qquad B = \frac{\beta_a \beta_c}{2.3\,(\beta_a + \beta_c)} \qquad I_{corr} = \frac{B}{R_p}$$

where $\beta_a$ and $\beta_c$ are the anodic and cathodic Tafel slopes, respectively, B is the Tafel constant, and $I_{corr}$ is the corrosion current. By dividing $I_{corr}$ (Amps) by the surface area (cm$^2$) of the polarized steel, we obtain the corrosion current density, $i_{corr}$. The corrosion rate in mils per year (mpy) is multiplied by a constant, C (457,164 for steel), and is directly proportional to the corrosion current density when

The Tafel slopes are difficult to obtain without damaging the system. For this reason, many researchers assume values for the Tafel slopes. Their basis for assumption comes from related published work. The assumed Tafel slopes (both anodic and cathodic) for corrosion of reinforcement in concrete are typically 0.120V. This yields a Tafel constant of 26mV.

There are inherent errors possible with the linear polarization method. To avoid these errors the measurements should be performed by measuring a significant number of data points within 5mV of the corrosion potential to ensure maximum accuracy [7]. The electrolyte resistance should also be considered for tests in low-conductivity electrolytes.

The solution resistance, $R_s$, (a component of the electrolyte resistance) for the steel reinforcement in the concrete environment is the resistance of the concrete that is subjected to polarization currents (used to produce the over-potential). When combined with the polarization currents, the $R_s$ for concrete (typically a large value) produces an IR drop. When the $R_s$ and/or currents are large values, the IR drop is meaningful and should be accounted for. If we do not account for this reistance, then we will overestimate the polarization resistance and consequently underestimate the corrosion rate.

In order to correct the errors that propagate from the solution resistance we must determine its value. There are various methods for correcting the IR drop. One of these is positive feedback. This is a method that multiplies the applied current by a portion of the uncompensated resistance, $R_u$. That product is then added to the electrode potential through a current-voltage converter. This value is then input back into the potentiostat [25]. This continues until the compensated resistance, $R_c$, is equal to $R_u$. This method is performed under the assumption that $R_u$ is constant. If this value varies, the correction that is applied to the polarization resistance is not correct. One disadvantage of using this method is that the potentiostat can become unstable at 100% compensation, therefore the total compensation is typically limited to 75% to 90% [26]. An advantageous aspect is that the correction is continuous and can be used during fast scan rates (100mV/s or greater). If the Rs (~Ru when neglecting the resistances of the electrolytic and connection leads) can be assumed as constant during each polarization, then the IR drop can be corrected mathematically with the positive feedback method.

# CHAPTER 3.0
# LITERATURE REVIEW

## 3.1 GENERAL

The problems of corrosion of steel in concrete still exist despite the extensive research conducted over the last 20 years.  The numerous factors involved in this type of corrosion instigate a number of studies to help understand the phenomenon and an equal number to bring about its prevention.

## 3.2 STUDIES OF CONCRETE & REBAR PROPERTIES

With 124-5 ft. by 4 ft. by 6 in. reinforced concrete slabs, Clear et. al. [19] used half cell potentials, visual inspection, and chloride analyses to monitor corrosion.  In the testing, the mixes, construction methods, and treatments were all varied.  The slabs had reinforcement in a grid pattern using No.4 steel reinforcement.  These slabs were cycled daily, ponding with a 3% sodium chloride solution over a span of approximately six years.  The results indicated that the water/cement ratio, depth of cover, and degree of consolidation had a considerable effect on the time to corrosion.

Lorentz et. al. [9] used prismatic concrete specimens as well as slabs to evaluate the impact of water/cement ratio, silica fume content, entrained air content, type of reinforcing steel and coating, and cracked versus uncracked concrete on corrosion of steel reinforcement embedded in concrete.  This test program used 96 specimens with dimensions of 12.5 in. by 12.5 in. by 7 in. for the prisms and 12 in. by 48 in. by 7 in. for the slabs.  To create an aggressive corrosion environment, some of the specimens had chloride mixed into the top layer of the concrete.  Over a test period of 35 to 40 months, the specimens were cycled through wet and dry periods, using a 15% NaCl solution and air-drying.  There were two layers of reinforcement in both types of specimens with a cover of 1 inch.  The two layers of steel were electrically connected so that a galvanic couple was formed.  Measurements were taken during the test period using macro-cell current measurements, instant off potential measurements, concrete resistance measurements, and half cell potential measurements.  A traditional macro-cell formed in 85% of the specimens, with top layer corrosion, but in the other 15% of specimens corrosion occurred on the lower mat of reinforcement, as shown upon visual examination.  Addition of condensed silica fume reduced the corrosion macro-cell current and significantly increased the concrete resistance.  Results also indicated there was no correlation between the air entrainment and the corrosion resistance of the concrete.  Other researchers [14, 18] also evaluated the effects of varying concrete properties using similar tests.

## 3.3 EVALUATION OF CORROSION INHIBITORS

Research on corrosion inhibitors [4, 5, 6] has revealed some inhibitors that showed promise, while others caused problems with some of the concrete properties.  Two methods were generally used to evaluate the inhibitors.  One method used simulated pore

solution testing [29, 30], and the other used actual concrete or mortar specimens [6, 17, 23, 30, 31, 32, 33, 34]. The two methods were sometimes used in conjunction.

Simulated pore solution testing is a screening process that evaluates the performance of chemicals for inhibiting steel reinforcement corrosion in a high alkaline environment, usually simulated by saturated calcium hydroxide solution. For these tests linear polarization or AC impedance methods are typically used.

The common specimen types used to investigate potential corrosion inhibitors in concrete are slab and lollipop specimens. These are typically monitored using either macro-cell, linear polarization, or AC impedance methods. Berke et. al. [31] used corrosion specimens similar to ASTM G-109 slabs to compare the effect of macro-cell and linear polarization on the evaluation of the inhibiting effects of calcium nitrite on galvanized steel and aluminum in concrete. He exposed these specimens continuously to wet/dry cycling that consisted of two-week periods of ponding in a 3% NaCl solution and air drying at room temperature. The linear polarization technique detected both localized and macro-cell corrosion, and therefore he concluded that if a true corrosion rate is desired, linear polarization should be used.

Trepanier [6] used 2-inch diameter lollipop mortar specimens and also ASTM G-109 concrete slab specimens for evaluating four commercial concrete inhibitors. The lollipops were constantly submerged in 3.5% NaCl solution to approximately mid-height of the specimens, and the slabs were treated per ASTM G-109 procedures. Half cell potentials, linear polarization, and AC impedance methods were used to monitor the lollipops whereas the slabs were monitored using macro-cell current measurements. All four of the corrosion inhibitors delayed the initiation of corrosion to some degree. However, none of them completely prevented the corrosion from occurring. The performance of the inhibitors was indirectly proportional to the water/cement ratio. The effectiveness of the inhibitors increased as the w/c ratio decreased. The results from both the AC impedance and linear polarization measurements were comparable. After nearly one year of cyclic ponding only the control specimens of the ASTM G-109 slabs were actively corroding, which had initiated after 271 days. From this it was evident that a considerable amount of time was needed in order to obtain any results from this type of test.

Though this form of testing is not quick to produce results, it is felt that the evaluation of corrosion inhibitors should be conducted using concrete specimens representative of real world conditions [35].

Allyn et. al. [48] performed tests using slab (6 in. tall by 4.5 in. wide by 11 in. deep) and lollipop (both 2 in. and 3 in. diameter by 6 in. tall) specimens. The testing was performed with the same four corrosion inhibitors used in the present study: two commercially available inhibitors and two experimental ones. That program included tests for compression strength, absorption, freeze-thaw durability, and corrosion rates. The slabs, similar to the ASTM G-109 specimens, had two layers of No. 4 reinforcement, the lower mat having twice as much steel as the upper mat. Most of the slab specimens had the two layers electrically connected to form a galvanic couple, but some were left unconnected. The lollipops also used No. 4 reinforcing steel and concrete, not mortar. The concrete resistance was measured using positive feedback, and the corrosion rates were determined using linear polarization. That research concluded the lollipop specimens produced results faster than the slab specimens and also showed the two new

chemicals may be more effective than existing commercial admixtures, pending further research.

10

# CHAPTER 4.0
# EXPERIMENTAL PROGRAM

## 4.1 GENERAL

These experiments evaluated and compared commercial and prototype corrosion inhibitors and studied how the geometry of different types of specimens affected the corrosion results.  This chapter presents information on the corrosion tests as well as on the absorption, compression strength, setting time, and chloride penetration tests.  Test results are discussed in Chapter 5.0.

The experimental program of this Phase II research was divided into two parts. The first part was a continuation of the previous Phase I begun by Allyn [48], extending the data collection of 2-inch and 3-inch diameter lollipop specimens and slab specimens. The second part of Phase II consisted of additional corrosion, absorption, and compression strength testing and included new data on setting time and chloride penetration.  Phase II developed new corrosion specimens, which were modified 3-inch diameter by 6-inch tall lollipop specimens, having the same dimensions as the Phase I 3-inch lollipops but with a preformed crack cast along the side of each specimen.  No new slab specimens were cast in Phase II.

## 4.1.1 Prototype Inhibitors

The new chemicals were disodium tetrapropenyl succinate and diammonium tetrapropenyl succinate, hereafter referred to as DSS and DAS, respectively.  They were described as alkali metal and ammonium salts of an alkenyl-substituted succinic acid. Appendix B contains Material Safety Data Sheets.

The specimens continued from Phase I evaluated concentrations of 2%, 1%, and ½% for the DAS, and of 2% and 1% for the DSS.  The second phase included concentrations of ½%, ¼%, and 1/8% for both DAS and DSS.  The concentrations of the DAS and DSS chemicals were based on the amount of active ingredients compared to the weight of the cement.

## 4.1.2 Commercial Inhibitors

Two commercially available corrosion inhibitors were studied.  These inhibitors, referred to here as Inhibitor A and Inhibitor B, are frequently used in reinforced concrete. Inhibitor A was a calcium nitrite based chemical.  The manufacturer recommended dosages of 2.0, 4.0, or 6.0 gallons per cubic yard, depending on the desired amount of protection. Phase I and Phase II used a dosage of 4.0 gallons per cubic yard.  Inhibitor B was an organic based chemical consisting of esters and amines.  The manufacturer described it as acting as a dual protection inhibitor by also reducing the ingress of moisture and chlorides.  The recommended dosage for this inhibitor was 1.0 gallon per cubic yard for all corrosive environments.

### 4.1.3 Cement

We used the same brand Type I/II cement for Phase II as was used in Phase I. The chemical analysis of the cement in Table 4.1 is for the Phase I cement and was provided by the supplier.

**Table 4.1 Chemical Analysis of Type I/II Cement.**

| Chemical | Content |
|---|---|
| $SiO_2$ | 21.0% |
| $Al_2O_3$ | 4.7% |
| $Fe_2O_3$ | 3.3% |
| CaO | 63.0% |
| MgO | 3.2% |
| $SO_3$ | 3.1% |
| $Na_2O_{Equiv.}$ | 0.58% |
| Ignition Loss | 0.90% |
| Insoluble Residue | 0.30% |
| **Potential Compounds** | |
| $C_3S$ | 52% |
| $C_3A$ | 7% |

### 4.1.4 Steel Reinforcement

Reinforcement was No. 4, Grade 60 rebar. Bars were purchased from the same manufacturer as for the Phase I research. The bars for Phase II were all from the same production lot to maintain similar material properties. The manufacturer provided mechanical and chemical analysis for the reinforcement for the Phase I rebar but not for those of Phase II. Table 4.2 has data for the Phase I rebar. It was assumed that the steel from Phases I and II did not have significant differences.

**Table 4.2 Mechanical and chemical analysis of No. 4, Grade 60 reinforcement.**

| Mechanical Properties | |
|---|---|
| Yield Strength (psi) | 63,000 |
| Tensile Strength (psi) | 98,500 |
| %Elongation | 10.0 |
| **Chemical Analysis** | |
| **Chemical** | **Content** |
| C | 0.45% |
| Mn | 0.84% |
| P | 0.072% |
| S | 0.033% |

### 4.1.5 Deicing Salt

ConnDot provided the deicing salt for this project. Its chemical content was primarily sodium chloride. This salt was mixed into a 15% sodium chloride solution (approximate solution found on bridge decks). To aid dissolving of the salt in solution, the salt was crushed into smaller particles and mixed with hot water. The solution was then stirred vigorously for approximately 30 seconds. This solution stood for at least one day to allow settlement of impurities and undissolved salt and then filtered through a No. 200 sieve. The filtered solution was stored in five-gallon plastic containers.

### 4.2 CONCRETE MIXES

A good quality, air-entrained concrete was used in both phases of the study as a control to compare the corrosion inhibiting characteristics of the commercial and prototype inhibitors.

### 4.2.1   Mix Design

The control concrete mix was identical for both phases. It was based on Connecticut Department of Transportation (ConnDOT) Form 814 [39], the guidelines in the Portland Cement Association's (PCA) "Design and Control of Concrete Mixtures" [40] and the ConnDOT Bridge Design Manual [41]. A previous research project [42] developed this mix and it conformed to ConnDOT's Class F concrete for bridges:

> Minimum 28 day compressive strength ($f_c$') = 4,000 psi
> Maximum water/cement ratio (w/c) = 0.44
> Minimum cement content = 658 pounds per cubic yard

The coarse aggregate for the experiments was limited to 3/8 inch crushed basaltic trap rock rather than the ¾ and ½ inch aggregate blend typically used for Class F concrete due to the small size of the test specimens. The stone was prepared by washing over a large No. 30 pan sieve and then drying in an oven at 200°F for one day. This assured better consistency and reproducibility of results.

A bank sand was used for the fine aggregate. It was obtained from a local gravel plant and arrived as a bulk load. It was dried the same as the coarse aggregate and conformed to the ConnDOT requirements for gradation.

These materials were received from the same sources as two recently completed University of Connecticut research projects. The absorption and specific gravity values of the aggregates for this project were assumed identical to the values from the two previous studies (Table 4.3) [42, 48].

**Table 4.3 Aggregate Properties [42, 48]**

| Properties | Coarse Aggregate | Fine Aggregate |
|---|---|---|
| Bulk Specific Gravity (dry) | 2.88 | 2.73 |
| Bulk Specific Gravity (saturated-surface-dry) | 2.92 | 2.75 |
| Absorption | 1.29% | 0.85% |
| Fineness Modulus | N/A | 2.73 |

To provide the water/cement ratio specified by ConnDOT (assuming it is based upon saturated-surface-dry aggregates), additional batch water was added to account for the absorption of the oven-dried coarse and fine aggregates. The batch water was also adjusted to account for the water contained in the corrosion inhibiting solutions, excluding the Inhibitor B mixes, which did not need adjusting per instructions from the manufacturer.

The mix proportions were the same as in Phase I. To remain consistent with the Phase I program, the maximum water/cement, w/c, ratio was set at 0.44 with a slump of 4 inches. The slump obviously depended greatly on the amount of water present in the mixture. However, some admixtures also affected the consistency of the mixture. An advantage of some admixtures is that they allow the desired consistency to be reached while maintaining a lower w/c ratio. Additionally, the cement content can have an affect on the performance of some inhibitors. These factors led to the conclusion that the amount of cement in the mixture should be held constant, not the w/c ratio. Therefore, to achieve the proper consistency the w/c ratios for the new study varied from 0.38 to 0.45. The average was 0.427 while the median value was 0.44. Only one mix had the value of 0.45, exceeding the limit, but the mix was still used due to the minimal divergence from the maximum.

The intended maximum slump for the mixtures was 4.0 inches. In order to maintain individual mix characteristics, the slumps were allowed to vary. All values were below the maximum and ranged from 1.0 to 3.5 inches.

When the consistency is affected by the admixtures it is possible that the set time of the mixture could also be altered. For the new study, the set times of the mixes were measured. The results of this testing are in Section 5.2.3.

The air content of concrete mixtures must also be controlled to achieve a desired product. The DAS and DSS mixes did not use any air entraining admixture. The commercial inhibitors and the control mixes used air entraining admixtures per recommendations of the manufacturers. The air content is recommended to be 7.5% with a range of –1% to +2% for concrete with a maximum aggregate size of 3/8 inch and exposure to "severe" conditions [40]. All but two mixes were within this range. The deviant mixes were both below the lower limit.

Batch sizes were 1.0, 1.1, and 1.25 c.f., depending on the number of specimens required for testing. Table 4.4 shows the mix proportions for the corrosion specimens.

**Table 4.4 Corrosion Mix Properties**

| Mix Type[1] | w/c Ratio | Air-Entrain Admixture | Inhibitor Concentration[2] | Air Content (%) | Slump (in) |
|---|---|---|---|---|---|
| Control-5 | 0.44 | Yes | N/A | 8.0 | 3.5 |
| Inhibitor A | 0.38 | Yes | 4.0 gal/c.y. | 5.5 | 1.25 |
| Inhibitor B | 0.41 | Yes | 1.0 gal/c.y. | 6.5 | 2.75 |
| ½% DAS | 0.45 | No | ½% | 7.5 | 1.75 |
| ¼% DAS | 0.43 | No | ¼% | 7.5 | 1.5 |
| 1/8% DAS | 0.43 | No | 1/8% | 7.0 | 1.5 |
| ½% DSS | 0.44 | No | ½% | 8.5 | 2.5 |
| ¼% DSS | 0.42 | No | ¼% | 7.5 | 1.5 |
| 1/8% DSS | 0.44 | No | 1/8% | 6.0 | 1.0 |

[1] All mixes contained Type I/II cement @ 27.85 lb., coarse agg. (oven-dried) @ 48.59 lb., and fine agg. (oven-dried) @ 54.26 lb. – Based on 1.0 c.f. mix.
[2] Concentrations for Inhibitors A and B were as suggested by the supplier. Concentrations for the DAS and DSS were based on weight of cement.

### 4.2.2 Mixing Procedure

A uniform mixing procedure was used. A drum mixer with a maximum capacity of 2.0 c.f. was used with batches of 1.0, 1.1, and 1.25 c.f. as needed. All mixes were done as a single batch.

The general mixing procedure followed the procedure performed in the Phase I program and was as follows [48]:

1. Wet the inside of the drum. Drain all excess water from drum.
2. Add all coarse and fine aggregate and mix for 1.5 minutes.
3. With mixer running, add approximately 85% of batch water to mixer and mix for 2.0 minutes.
4. Add air entraining admixture, DAS, or DSS (as required) and mix for 2.0 minutes.
5. Add all cement over a 1.0 minute span and then mix for an additional minute.
6. Add Inhibitor A or B (as required) and mix for 1.0 minute.
7. Stop mixer and scrape drum (over an approximate 1.0 minute span). Restart mixer.
8. Add remaining batch water as needed. Mix for 2.0 minutes.
9. Stop mixer and let stand for 2.0 minutes.
10. Restart mixer and mix for 2.0 minutes.
11. Pour mixture into pre-wetted floor pan.
12. Perform a slump test and air content test.
13. Cast specimens.

The slump was determined by ASTM C143-90a [44] and air content by the pressure method, ASTM C231-91b [45]. However an aggregate correction factor was not applied.

**4.3 SPECIMEN DESIGN AND FABRICATION FOR PHASE II**

The testing of the remaining 2-inch and 3-inch diameter lollipop specimens and slab specimens of Phase I continued. Phase II also explored the use of "cracked" concrete specimens. Using the basic geometry of the 3-inch diameter cylinders of the Phase I project, a preformed crack was added to the cylinder (Fig. 4.1) extending from the outer concrete surface to a depth approximately 1/8 inch from the surface of the reinforcing steel.
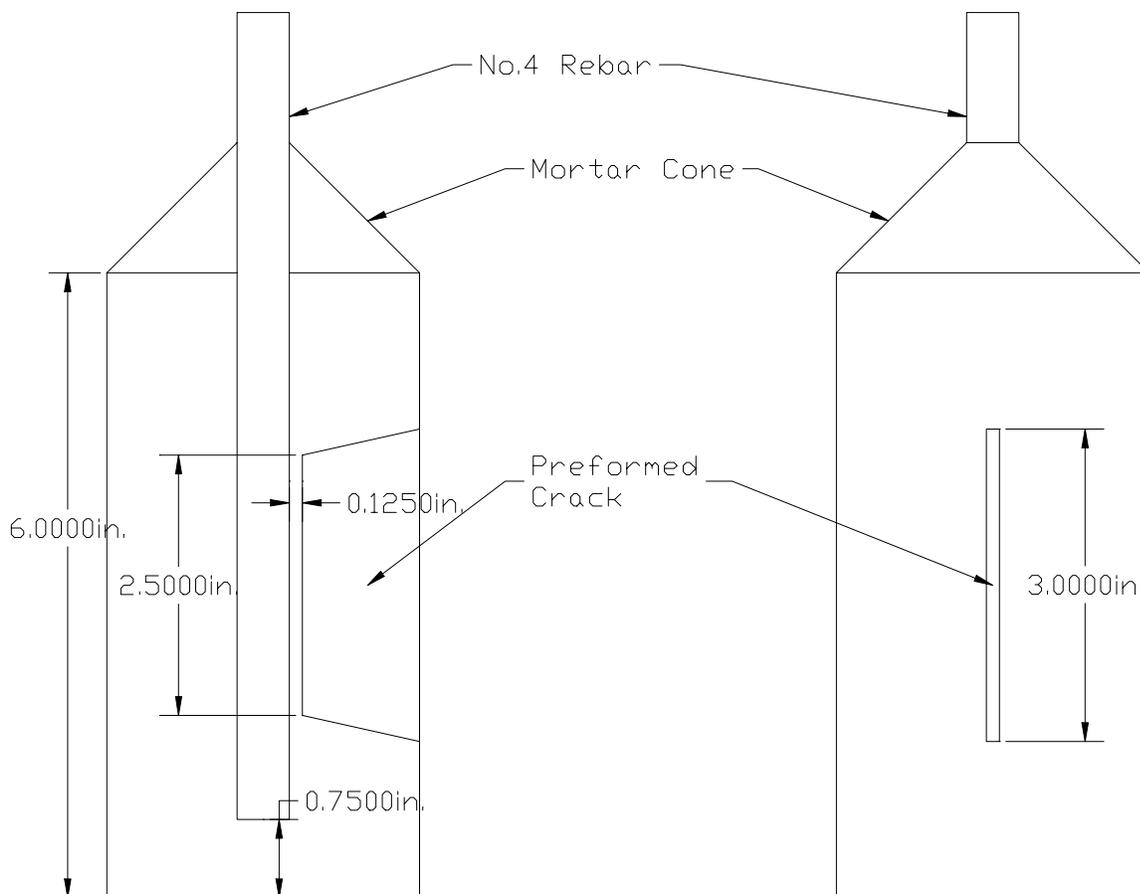
**Figure 4.1  Pre-cracked cylinder.**

The molds were modified slightly by putting a long, thin cut along the side of the mold (Fig. 4.2) that was 1/8 inch wide by 3 inches long and centered along the height of the cylinder at 1½ inches from both the top and bottom of the cylinder. This cut provided a slot for a teflon wedge that created the preformed crack in the specimens. The wedge (Fig. 4.3) was tapered in both directions to ease in its removal after the concrete had hardened. The wedge created a 1/16 inch wide crack near the reinforcing steel that

16

extended nearly the entire length of the exposed steel (see Fig. 4.4).  The crack at the surface of the concrete was 1/8 inch wide due to the tapering of the wedge.
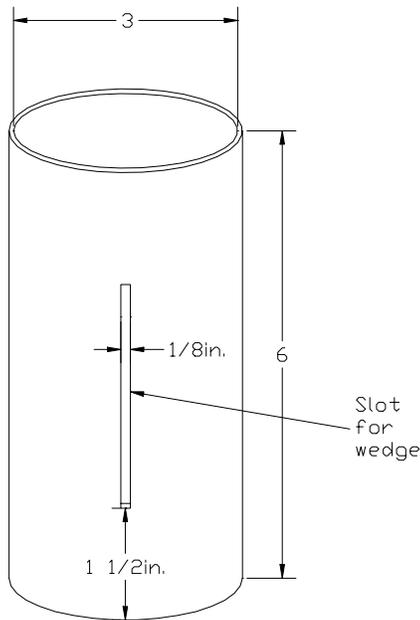


**Figure 4.2  Slotted cylinder for casting pre-cracked specimens.**
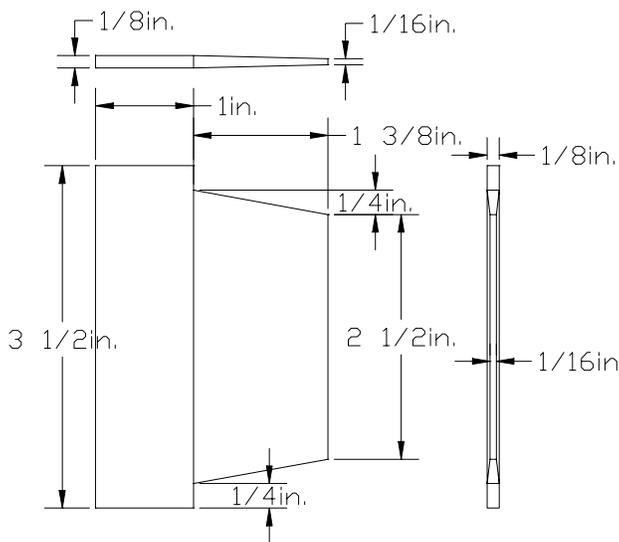


**Figure 4.3  Wedge used to cast pre-formed crack.**

### 4.3.1    Casting and Preparation

For Phase I mixes, there were three replicate specimens for each mix except the lollipop control specimens (six) and the slab Control 1 specimens (two).  For Phase II, specimens were as follows:

- 18 of the Control 5 specimens.
- Eight each of the ½%DAS and DSS (half of which were air dried).
- Six each of Inhibitors A and B (two of each were air dried).
- Four each of the ¼% and 1/8% DAS and DSS with the exception of ¼%DAS, which only had three (one was lost in the casting process).

The reinforcing bars arrived in lengths of 20 feet, which were cut into 14.75 inch lengths. The bars were cleaned with a wire wheel brush and were stored in an oven at 100°F to inhibit the corrosive effects of moist air. The bars were then pickled in a 2.5M sulfuric acid solution for ten minutes, rinsed with tap water, and dried. Allyn concluded in the Phase I research that electroplaters tape (as recommended by ASTM G109-92) was not effective in preventing corrosion in the taped area. Therefore the bars received an epoxy paint coating in three layers leaving a desired area of exposure (Fig. 4.4). The epoxy layers were applied one per day and the bars returned to the oven. On the day before casting, the painted bars were pickled again in the same acid solution and returned to the oven. Prior tests showed the epoxy coating was unaffected by the brief submergence in 2.5M sulfuric acid, thus the second round of pickling did not affect the protective coating. During and after cleaning, the bars were handled using vinyl gloves to prevent contamination. The purpose for all of this preparation was so that the starting point could be assumed at no corrosion.
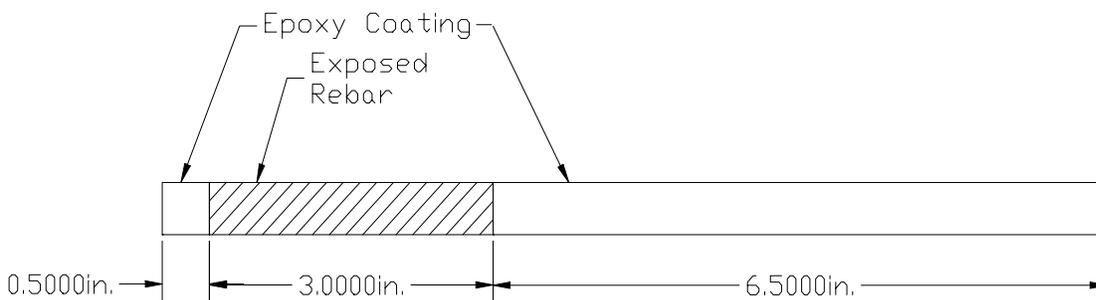


**Figure 4.4  No. 4 rebar with 3" long area of exposure.**

The insides of the cylinder molds and the wedges were coated with a thin film of mineral oil. As previously, a special casting fixture was used (Fig. 4.5) that consisted of upper and lower plywood decks measuring 24 inches square. The molds fit snugly into holes cut in these decks and as many as eight 3-inch specimens could be cast at one time.

PLAN VIEW
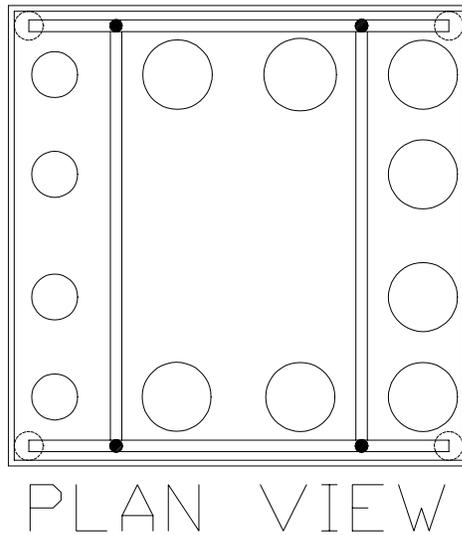
**Figure 4.5 Modified (from Phase I) casting form with eight 3-inch diameter openings instead of six, adapted from Reference 48.**

The rebar was carefully aligned in each cylinder using the same nylon mounting templates as Allyn. The rebar was positioned with the preformed crack along the transverse ribs of the rebar and not the longitudinal spine. After the molds and rebar were in place, the wedges were inserted and secured with duct tape to keep them from shifting during casting and vibrating (Fig. 4.6).

Three casting fixtures were made so that 3 different mixes could be cast or a large number of cylinders for one mix could be cast. The fixtures were clamped to the vibration tables one at a time. The cylinders were cast in 3 layers with each layer being rodded approximately 10 times with a 3/16-inch rod, vibrated for 10 seconds, and tapped on the sides with a 3/16-inch diameter rod during each vibration. This eliminated the problems of surface honeycombing. A mortar cone (Fig. 4.1) was molded to the top of the cylinders. After demolding, any gaps between the cone and rebar were sealed with a silicone caulk to ensure no water would enter from this point.
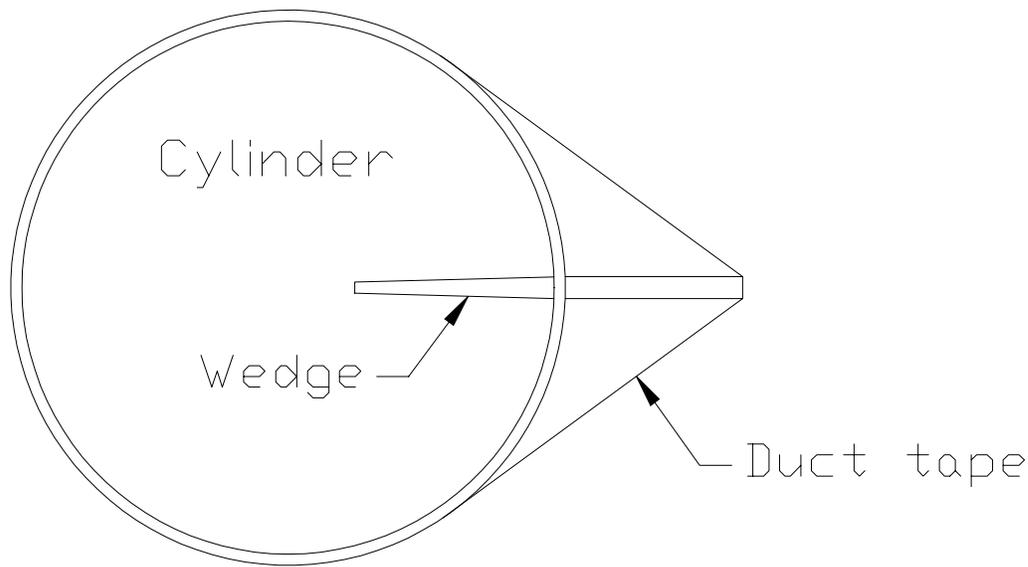
**Figure 4.6 Plan view of wedge held in position using duct tape bracing.**

For strength data, 12 cylinders for the ¼ and 1/8 DAS and DSS mixes and 8 each for the other mixes were made. These cylinders were cast in two layers in a similar fashion to the pre-cracked specimens. The pre-cracked cylinders had three layers to provide uniform concrete consistency near the rebar. There was no rebar present in the strength cylinders and therefore only two layers were needed.

After casting, the specimens were covered with 4 mil plastic sheeting. The following morning, specimens were demolded and placed into a moist curing room for 13 days at a temperature of 70°F. The curing room had open shelves for storing the specimens. Multiple misting jets maintained the humidity of the room at 95% (±5%). After moist curing, specimens were moved into a store room where they remained until testing. Some of the corrosion specimens stayed in the store room for a slightly longer period of time than others, but no more than three additional days, so that when they were introduced to the wet/dry cycling test schedule they would always start at the beginning of a cycle.

During the storage period, the rebars were then cut to a final length of 3 inches above the top of the mortar cone. The epoxy coating was removed from the exposed end of the rebar. One side along the circumference of each rebar was flattened and then two grooves, one on the flattened surface and one on the end of the rebar, were cut into the rebar into which the working and sense leads would lay. The bare steel was then sandblasted to remove as many impurities and stains as possible to facilitate bonding with the solder. The Phase I specimens had the lead wires brazed on with silver solder. It was found that this made the wires brittle and they frequently snapped off due to repeated handling during tests. For many of the Phase II specimens the leads were soldered onto the rebar using a soldering gun and tin solder. The rebar acted as a heat sink, though, and as a result the tin did not bond well with the rebar causing the wires to frequently detach. After concluding that neither soldering nor brazing were ideal, brazing was settled upon as the better choice and thus the lead wire connections returned to this method for the

Inhibitors A and B specimens.  After the leads had been attached to the rebar, the exposed areas were then coated with three layers of the same epoxy paint as before.

The strength cylinders were tested at 14, 28, and 56 days of age.  Some cylinders were used for measuring absorption data.  Absorption tests began at about 28 days of age.

Most of the Phase I slab specimens had the upper and lower layers of reinforcing steel electrically connected.  Some of the control specimens did not have this connection.  These specimens are referred to as "conn" or "unconn," respectively in the Chapter 5.0 discussion.  The slab specimen is shown in Figure 4.7.
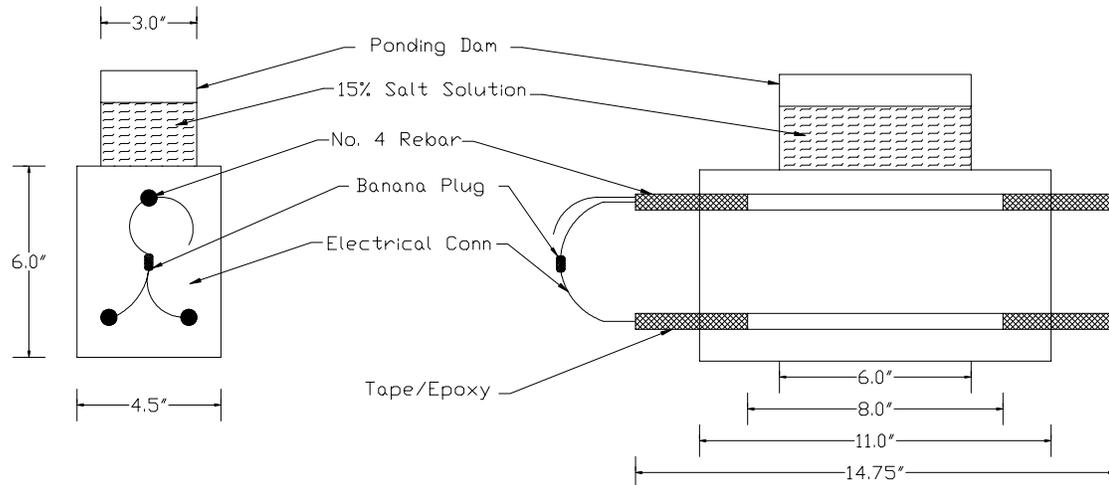


**Figure 4.7  Slab Specimen, from Reference 48.**


## 4.4 WETTING AND DRYING PROCEDURE

The lollipops were ponded to a depth of 5 inches for four consecutive days in a 15% salt solution.  Some previous research used 3.0% [6, 14, 19, 31] or 15% [9, 18, 32, 34] salt solutions.  Salt concentrations on actual bridge decks have been shown to vary between 7% and 15%.  The high end salt concentration expedites the overall test time.

All specimens had a three day drying period following the ponding period for a total cycle length of seven days.

Slab specimens were dried at room temperature in both phases.  For Phase I, all lollipop specimens were oven dried at 100°F.  The absorption testing results (Section 5.2.2) indicated that absorption is reduced for specimens dried at lower temperatures.  Phase II varied the drying conditions of the new specimens.  To observe the effects of air drying versus oven drying for the lollipop specimens,  four of the eight lollipop specimens of ½% DAS and DSS along with two of the six specimens of Inhibitors A and B were air dried at room temperature for the three days.  All of the other lollipop specimens were dried in an oven at 100°F (±3°F).  The drying oven was 50 inches high, 24 inches wide, and 24 inches deep with multiple shelves.  It was vented to exhaust the evaporating moisture.  Each specimen was placed in the oven so that there was at least an inch of space between it and the specimens next to it.

The oven drying in conjunction with the ponded cycle simulated a "southern

exposure" environment that is described as an aggressive corrosion environment with an abundance of chlorides and oxygen. This should accelerate the overall test time.

In Phase II, the sizes of the lollipop ponding containers were changed. These containers were made from standard plastic cylinder molds. Phase I used 4-inch diameter by 6-inch tall containers for the 2-inch diameter specimens and 6-inch diameter by 6-inch tall containers for the 3-inch diameter specimens. Phase II used 4-inch diameter by 6-inch tall containers for the 3-inch specimens and 3-inch diameter by 6-inch tall containers for the 2-inch specimens. With the smaller containers, evaporation and absorption still did not have a significant effect on the level of solution. These smaller containers were used for the remainder of the testing to lessen the amount of solution consumption.

The procedure was as follows [48]:

1. Place the specimens in their individual ponding containers.
2. Fill the ponding containers with fresh 15% salt solution to a depth of 5 inches.
3. Four days later, remove the specimens from their ponding containers and rinse the specimens with warm water to remove any exterior salt buildup.
4. Place specimens on the racks in the drying oven.
5. Empty the ponding containers and rinse with warm water.
6. After three days of drying, remove the specimens from the oven and proceed to step one. Repeat steps one through six.

The ponding and drying procedure for the slabs was maintained from The Phase I research through the second phase of this project and was as follows [48]:

1. Fill each ponding dam (Fig. 4.7) to a depth of 2.0 inches with fresh 15% salt solution.
2. Place a plastic cover over each dam during the ponding cycle to prevent evaporation of the salt solution.
3. Four days later, vacuum out the salt solution (once per month rinse out the ponding dams and exposed concrete with fresh water to remove any salt buildup).
4. Let the slabs air dry for three days at room temperature.
5. Proceed to step one. Repeat steps one through five.

## 4.5 CORROSION TESTING METHODS

Linear polarization is an accurate method with a moderate cost. It also has the ability to measure both macro-cell and localized corrosion. For these reasons this method was chosen for this testing program.

### 4.5.1 Test Setup

An EG&G PARC Potentiostat/Galvanostat, Model 273A was used to make the polarization measurements. This equipment was used in conjunction with a three electrode probe setup (Figs. 4.8 & 4.9) that consisted of:

1. One saturated calomel reference electrode, RE.
2. One working electrode, WE (No. 4 reinforcing bar cast into specimens).
3. One or two ½-inch diameter graphite counter electrodes for the slab and lollipop specimens, respectively, CE.



Plan View                    Elevation View
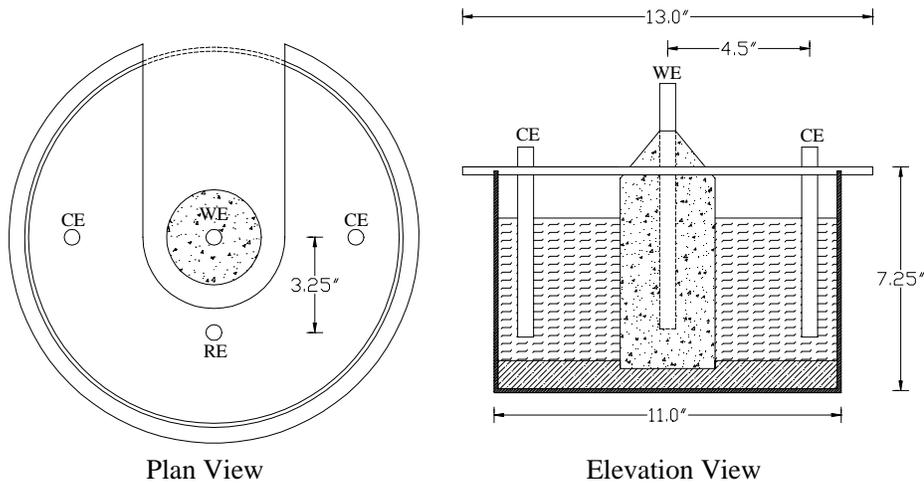
**Figure 4.8  Lollipop Test Cell Setup, from Reference 48.**



Plan View                    Elevation View
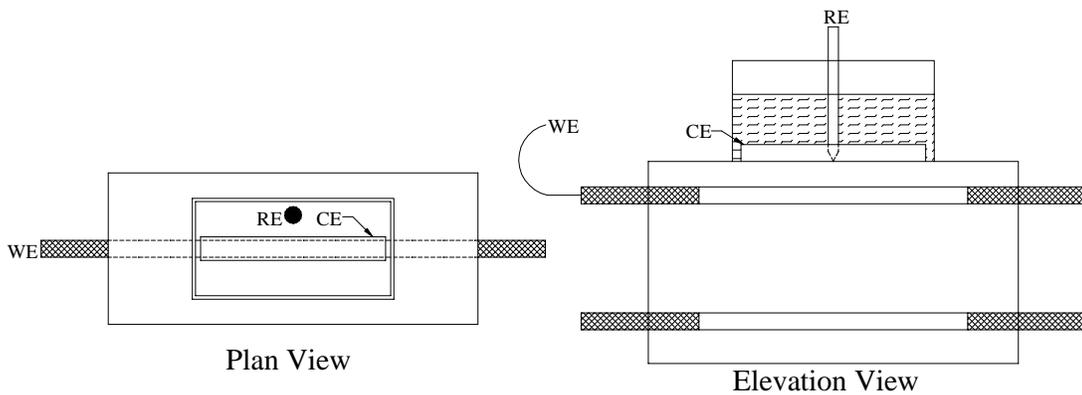
**Figure 4.9  Slab Specimen Test Cell Setup, from Reference 48.**
        For the lollipop specimens (Fig. 4.8), a plexiglass lid placed on the lower

container held the reference and counter electrodes in place. Multiple counter electrodes provided a more uniform polarization for the specimens. A separate sense wire was attached to the working electrode to help stabilize the linear polarization measurements in the event of high currents. A high current would cause a considerable voltage drop in the working electrode lead. The solution in the container was the same 15% salt solution used for the wet/dry cycling.

The slab specimens (Fig. 4.9) had a built-in container on each specimen. The reference electrode was inserted into a hole in a wooden support slat that lay across the top of the container. This setup held the reference electrode (RE) in a steady, vertical position. The counter electrode (CE) was laid parallel to the rebar centered across the bottom of the container aligned directly over the rebar, centered in the ponding dam. Similarly to the lollipop specimens, the working and sense leads were attached to the leads on the working electrode. These leads were disconnected from the lower mat of reinforcing for the duration of the testing so that they could be linked to the test cell, and then rejoined following the test.

### 4.5.2   Software and Programs

CorrWare™ software, by Scribner Associates, Inc., was used to control the polarization resistance measurements. This software was chosen after difficulties were encountered with EG&G PARC M352 SoftCorr™ III software in Phase I of this project. The software was run on a Gateway P133 Pentium Processor Computer through a National Instruments® GPIB-PCII A interface.

The same Tafel constant, B, of 0.026V was used for both active and passive systems. The same working electrode material (iron) was used for all of the specimens, therefore the specific Tafel slopes were not obtained.

In most instances, corrosion is a localized effect. However for calculating corrosion rates, researchers typically assume that the corrosion is occurring over the entire exposed surface of the bar. This same assumption was used in this research. Under this assumption an average corrosion rate is calculated and the calculated corrosion rates are at best nominal.

The cell was polarized by ±10mV across the open circuit potential at a scan rate of 0.1mV/sec. The IR drop, rather than accounted for during the test, was corrected for in the specially written "in-house" data reduction program for this project. This program is described in greater detail in Section 5.5.

This data reduction program calculated the actual $R_p$ value for each test. The slope of the corrected current versus the potential curve between the potential where the current equals zero, $E_{I=0}$ (the true open circuit potential), and the potential +5mV from $E_{I=0}$ is the $R_p$ value. By limiting the $R_p$ calculation to +5mV from the true open circuit potential, accuracy was maintained. Figure 4.10 shows a polarization curve of an actively corroding lollipop specimen from this experiment. It is similar in shape to a typical polarization curve and has been corrected for the IR drop. A "best fit" linear function was applied to the segment that spans from the true potential at open circuit to +5mV above that value.
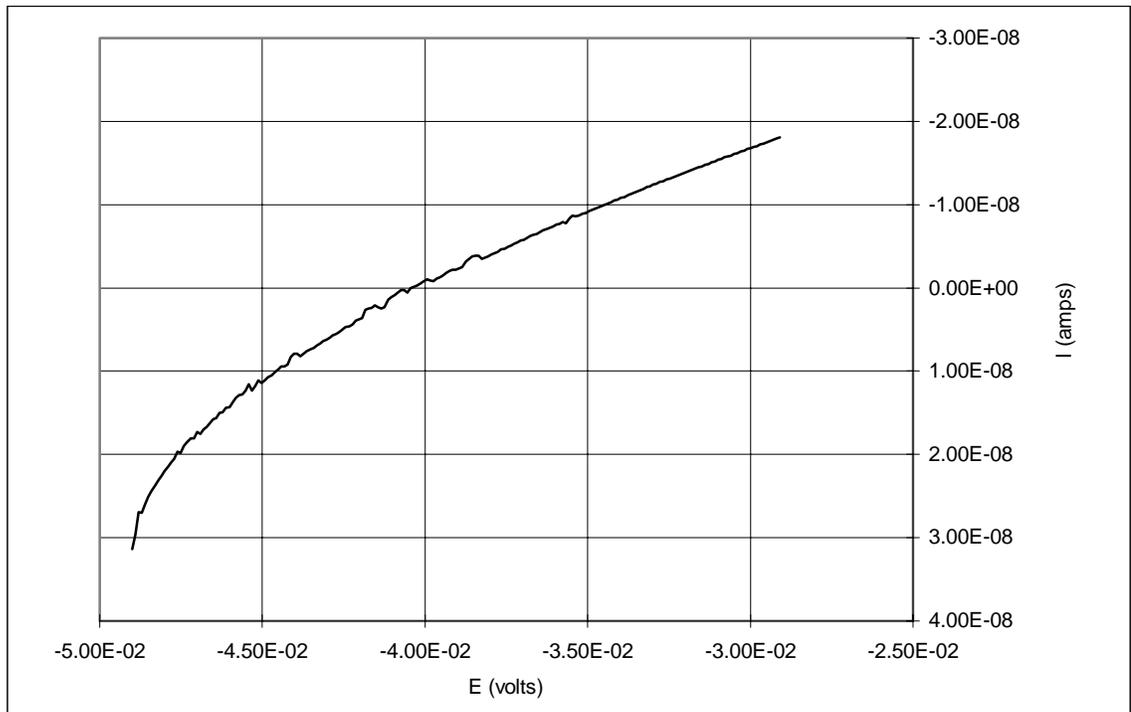
**Figure 4.10  Polarization curve for an actively corroding lollipop specimen.**

### 4.5.3   Testing Schedule

The lollipop specimens cast for Phase II of this project were tested biweekly. There was very little corrosion activity in any of the DAS and DSS lollipop specimens that remained from Phase I and these specimens were therefore placed on a monthly test schedule to save time, although the wet/dry cycles continued on a weekly cycle.  The control and commercial inhibitor specimens were still monitored biweekly due to the higher level of corrosion activity observed in the polarization measurements.  The testing schedule for the slab specimens from Phase I was also altered similarly.  The lack of activity in the DAS and DSS specimens allowed them to be placed on a bimonthly testing schedule while the control and commercial inhibitor remained on the once per month schedule. Based on the experiences from Phase I, only one measurement of each specimen was typically done per test day.  This allowed a large number of specimens to be tested for Phase II because each test was so time consuming.  Each set of results was scrutinized after each test.  If any discrepancies in the results seemed apparent, the tests were rerun multiple times to determine where errors were occurring and which result was the correct result.

To account for the IR drop, the solution resistance was measured by the positive feedback method with EG&G PARC M270 software.  The identical three electrode test cell described previously was used.  Settings for this test included the compensation held at 85% and the pulse height of 10mV.  The current ranges were varied frequently because the resistances differed greatly from specimen to specimen.  The values varied from 10mA to 100μA for the Phase II specimens and 1ma to 100μA for the Phase I specimens. The resistance of the concrete was assumed to be constant during each test period, a

reasonable assumption considering the short test period.  The IR drop was then accounted for mathematically as described in Section 2.2.1.

The concrete resistance was assumed to be the solution resistance.  The resistance of the electrolyte solution and of the testing leads can increase the IR drop in certain cases, however the 15% salt solution was very conductive and the leads were limited to a moderate length.  The resistance of the solution and of the leads would be relatively insignificant when compared to the concrete resistance and were therefore discounted.

## 4.6 COMPRESSION STRENGTH TEST PROCEDURE

The evaluation of the compressive strength for the corrosion mixes was conducted as outlined in ASTM C39-94 "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens."

Compression strength cylinders were cast at the same time as the corrosion specimens.  Six 3-inch diameter by 6-inch tall cylinders were cast for the control, ½% DAS and DSS, Inhibitor A, and Inhibitor B mixes.  Ten cylinders were cast for the ¼% and 1/8% DAS and DSS mixes.  All cylinders were wet cured for 14 days on shelves in a moist cure storage room and then stored in a dry storage room until tested.

The cylinders were loaded at a rate of 15 kips per minute using elastometric end caps.  Three cylinders per mix were used for each test age and the results were averaged.  The control, ½% DAS and DSS, Inhibitor A, and Inhibitor B were tested at 28 and 56 days after casting.  The ¼% and 1/8% DAS and DSS specimens were also tested at 14 days.

## 4.7 ABSORPTION TEST PROCEDURE

Two (excluding Inhibitor A and Inhibitor B) absorption specimens, 3 inches in diameter by 6 inches tall, were cast concurrently with the corrosion specimens.  Absorption specimens were cast in the same manner as the corrosion specimens.  After casting, the specimens were wet cured for fourteen days on shelves in the moist cure room.  The specimens were then placed in the storage room for an additional 14 days.  After the 28 days, specimens were entered into the absorption testing.

ASTM C642-90 "Standard Test Method for Specific Gravity, Absorption, and Voids in Hardened Concrete" was generally followed to evaluate the absorption characteristics of each concrete mix.  To maintain consistency with the Phase I absorption testing, the specimens were not boiled and were dried at 40$^{o}$C, which varied from the ASTM standard.

The procedure for absorption testing was as follows [48]:

1. Remove the cylinders from the store room, record initial weight, and dry on an individual basis in an oven at 40°C until the weight loss between successive weight measurements is equal to or less than 0.1%.
2. Once the final dry weight is achieved, submerge the cylinders in a water bath at room temperature until, on an individual basis, the weight gain between successive weight measurements is equal to or less than 0.1%

3. Towel dry the excess surface moisture prior to weighing.

At 40$^o$C it generally took four to five weeks to dry the specimens and about four to five weeks to saturate them. Final absorption values were based on the average of the two specimens. The absorption was calculated using:

$$Absorption = [(B - A) / A] \times 100\%, where:$$

A = Final weight, in air, of oven-dried sample.
B = Final weight, in air, of surface-dry sample after immersion.

## 4.8 SETTING TIME TEST PROCEDURE

As part of the Phase II testing, setting times were measured. This test followed the procedure outlined in ASTM C403. The setting time is the amount of time that a mortar sample of the mix takes to stiffen to a penetration resistance of 4000psi.

The testing procedure required approximately 0.25 cubic feet of mortar from the same mix as the corrosion test specimens. To obtain the mortar, the concrete mixes were sieved to remove the coarse aggregate. The resultant mortar was placed into cylindrical molds six inches in diameter to a depth of six inches. The mix was rodded 25 times in three layers. Two cylindrical molds were used for each mix.

The compression strength of the mortar mix was determined by inserting a needle with a known surface area into the mix. The needle was inserted to a depth of 1 inch. The force required to insert the needle was read from a dial gage affixed to the needle insertion apparatus. Knowing the surface area of the needle and the force required to insert it, the pressure (penetration resistance) required to insert the needle was determined. As many as eight needles of various known surface areas were used for the test to compensate for the limited range of the dial gage.

The first measurement was taken three hours after the mix had been cast. The following measurement was taken one hour after the first measurement. From this point on, the measurements were taken every half-hour until the mix obtained a resistance greater than that of 4000psi.

The penetration resistance and time of measurement were recorded and plotted. The point where the plot crosses the 4000psi limit is considered the setting time.

## 4.9 CHLORIDE PENETRATION TEST PROCEDURE

The remaining Phase I specimens were split open at the end of the Phase II research. This allowed concrete samples to be removed from the interiors of the Phase I specimens. Using these samples, the amount of chloride penetrating to the rebar could be determined.

Surface concrete samples were removed using a ½-inch diameter masonry drill to a depth of approximately ¼-inch. After splitting open the corrosion specimens, additional interior samples were removed from the rebar imprint at the center of each half. The samples were removed from "clean" surfaces of concrete, avoiding portions stained with rust, if possible. A minimum sample of 13 grams was removed from each specimen used.

The powdered samples were analyzed for chloride content by ConnDot, Bureau of Engineering and Highway Operations, Division of Material Testing. The testing was based on Report Number FHWA-RD-77-85 and the method used was Gran endpoint determination.  The corrosion threshold for chloride content is about 1.0 to 1.6 lbs. of acid soluable chloride per cubic yard of concrete [18].  This amount of chloride ion concentration is what is assumed to be required for corrosion to initiate in normal concrete.

# CHAPTER 5.0
# PRESENTATION AND DISCUSSION OF RESULTS

## 5.1  GENERAL

The compression strength, absorption, and setting time results for the Phase II specimens are found in Sections 5.2.1 through 5.2.3, respectively.  The chloride penetration results are in Section 5.3.  For results of the Phase I specimens for compression strength, absorption, and freeze-thaw durability, please refer to reference [48].  Sections 5.4 and 5.5 contain the solution resistance results and the polarization resistance data for both Phases.  The visual inspections of all specimens are discussed in Section 5.6.  Some test results and discussion, previously presented by Allyn [48] in Phase I, is repeated here for completeness and for ease of the reader.

## 5.2  CONCRETE CHARACTERISTICS

### 5.2.1  Compression Strength

As seen in Table 5.1 and Figure 5.1, Inhibitor A had the highest strength of all mixes in Phase II at approximately 150% relative to the control mix, supporting the Phase I results (120%).  The Inhibitor A mix also had the lowest w/c ratio.  The lowest strength was for ½%DSS, at about 70% of the control strength (90% in Phase I).  Inhibitor B had a compression strength consistently around 95% of the control for Phase II (84% in Phase I).

As expected, increasing the concentration of DAS or DSS typically produced lower strengths.  The exception was the 1/8%DAS that was consistently lower than the ¼%DAS and higher than the ½%DAS.  There was very little difference between the DAS and DSS.  All of the mixes, with the exception of the ½%DSS mix (3702 psi), had 28 day compression strengths greater than 4000 psi which satisfied the requirements of ConnDot's Class "F" concrete for bridges, the design basis for the "control" mix.

**Table 5.1  Corrosion Mixes:  14, 28, and 56 Day Compression Strengths**

| Mix Type[1] | w/c | Air Content | 14 Day (psi) | 28 Day (psi) | 56 Day (psi) | Relative 28 Day (psi)[2] |
|---|---|---|---|---|---|---|
| Control 5-C | 0.44 | 8.0% | 4376 | 5382 | 5284 | 1.00 |
| ½%DAS-C | 0.45 | 7.5% | 3319 | 4150 | 4267 | 0.77 |
| ¼%DAS-C | 0.43 | 7.5% | 3595 | 4501 | 4894 | 0.84 |
| 1/8%DAS-C | 0.43 | 7.0% | 3084 | 4215 | 4577 | 0.78 |
| ½%DSS-C | 0.44 | 8.5% | 2890 | 3702 | 4023 | 0.69 |
| ¼%DSS-C | 0.42 | 7.5% | 3573 | 4480 | 4839 | 0.83 |
| 1/8%DSS-C | 0.44 | 6.0% | 3652 | 4730 | 5200 | 0.88 |
| Inhib. A-C | 0.38 | 5.5% | 6675 | 8305 | 8268 | 1.54 |
| Inhib. B-C | 0.41 | 6.5% | 4064 | 4984 | 5305 | 0.93 |

[1]All mixes contained Type I/II cement @ 27.85 lb., coarse agg. (oven-dried) @ 48.59 lb., and fine agg. (oven-dried) @ 54.26 lb. – Based on a 1.0 cu. ft. mix.
[2]Relative strength:  Control 5-C mix equals 100%.
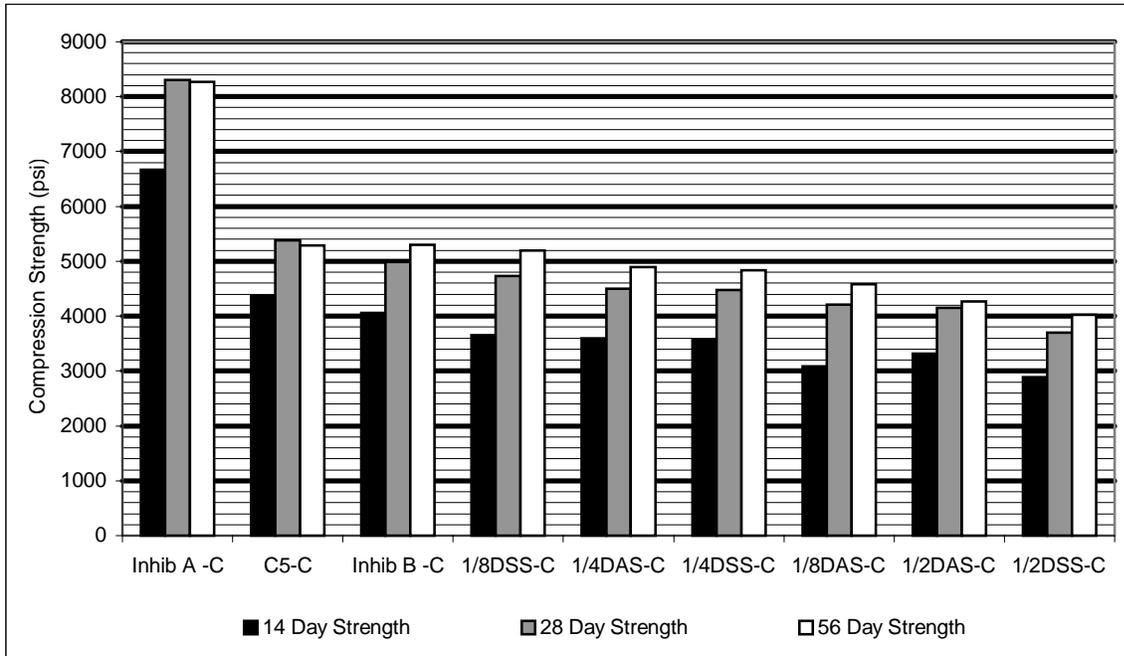
**Figure 5.1 Compression Strengths in descending order for 28 day strength.**
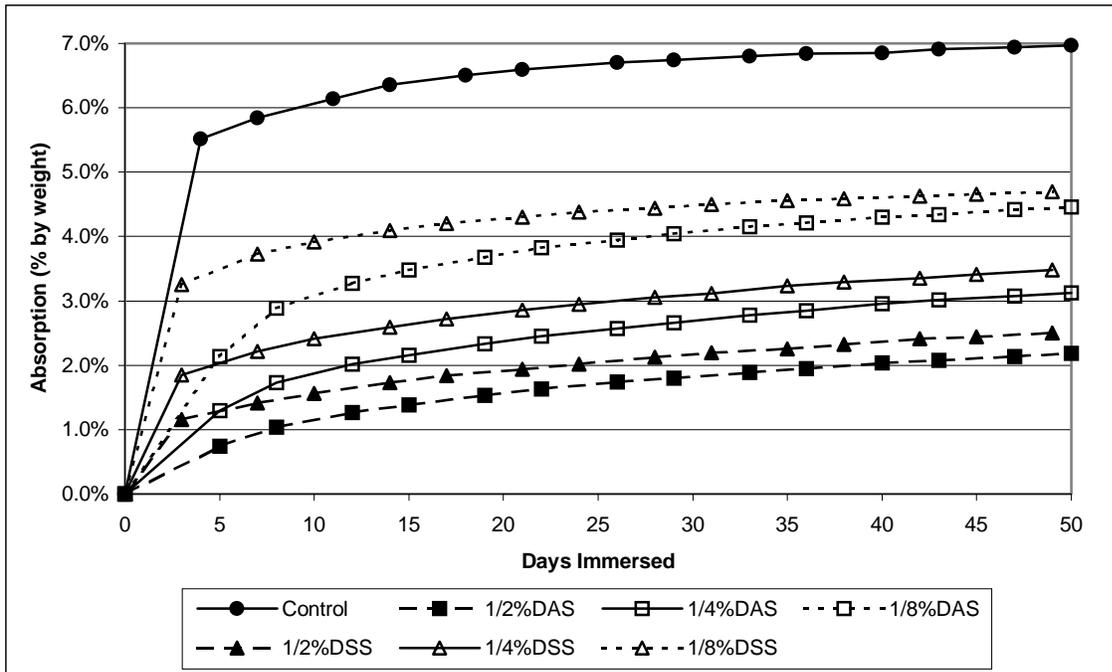


**Figure 5.2 Absorption versus time: Control, DAS and DSS specimens, dried at 40$^o$C, Phase II specimens.**

### 5.2.2 Absorption

Figure 5.2 shows the absorption versus time for the mixes tested in Phase II. Table 5.2 is a listing for specimens in both phases and Figure 5.3 is a ranked chart for this data.

**Table 5.2  Final absorption:  all specimens  (Phases I and II)**

| Specimens | Phase | Average Absorption (%) Dried @ 40°C |
|---|---|---|
| Control 2 | I | 5.10 |
| Control 5 | II | 7.08 |
| Inhibitor A | I | 5.06 |
| Inhibitor B | I | 3.88 |
| 2%DAS | I | 1.28 |
| 1%DAS | I | 1.45 |
| ½%DAS | I | 2.81 |
| ¼%DAS | II | 3.70 |
| 1/8%DAS | II | 4.83 |
| 2%DSS | I | 1.51 |
| 1%DSS | I | 1.82 |
| ½%DSS | I | 3.25 |
| ¼%DSS | II | 3.94 |
| 1/8%DSS | II | 4.92 |
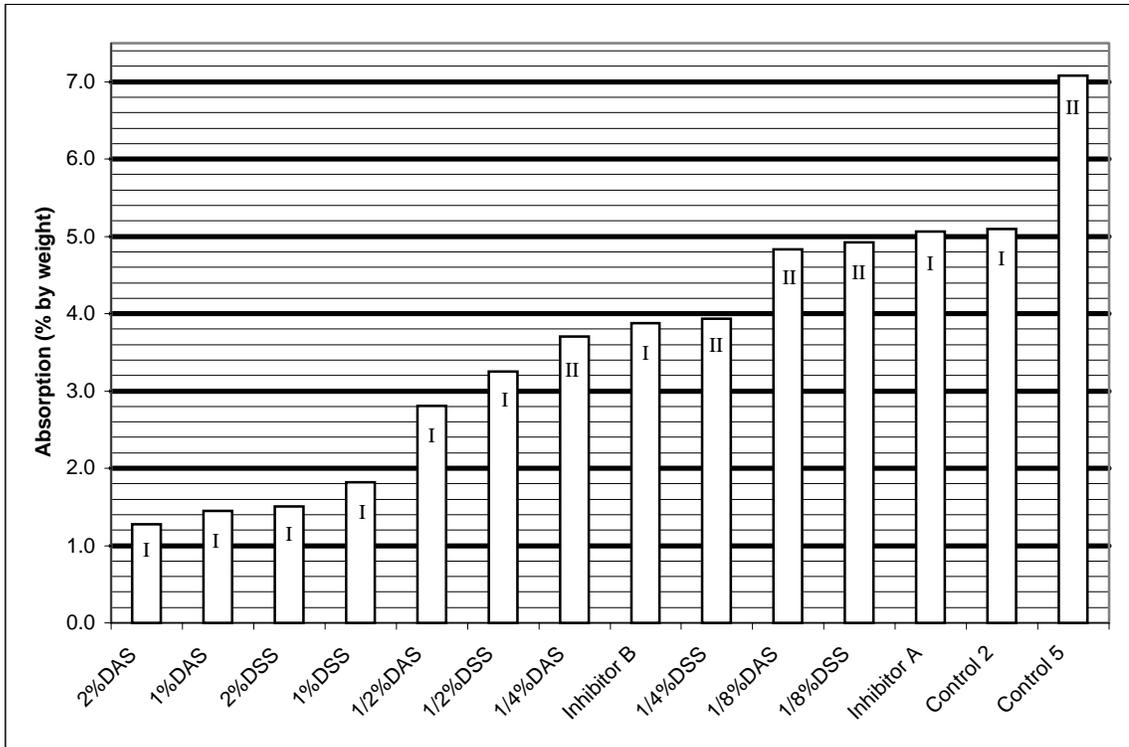


**Figure 5.3  Final absorption:  all specimens (Phases I and II), dried at 40°C.**

The controls from Phases I and II had the highest absorption (5-7%). No absorption data was taken for Inhibitors A and B in the Phase II testing. In Phase I, Inhibitor A was similar to the Control 2.

For both the DAS and DSS chemicals, the absorption decreased as the concentration of inhibitor increased. The absorption in the DAS specimens was consistently slightly lower than in the DSS specimens. At the ¼% concentration they were similar to the absorption of Inhibitor B (about 4%). The 1/8% concentration was relatively ineffective in reducing absorption, with absorption values of about 5% and close to that of the control for the Phase I testing. A concentration of ½% for both chemicals produced an absorption of about 3%.

### 5.2.3    Setting Time

The setting time curves are displayed in Figure 5.4. This test was not performed for Inhibitor A or Inhibitor B. It is apparent that both the DAS and DSS chemicals acted as accelerators for setting time.
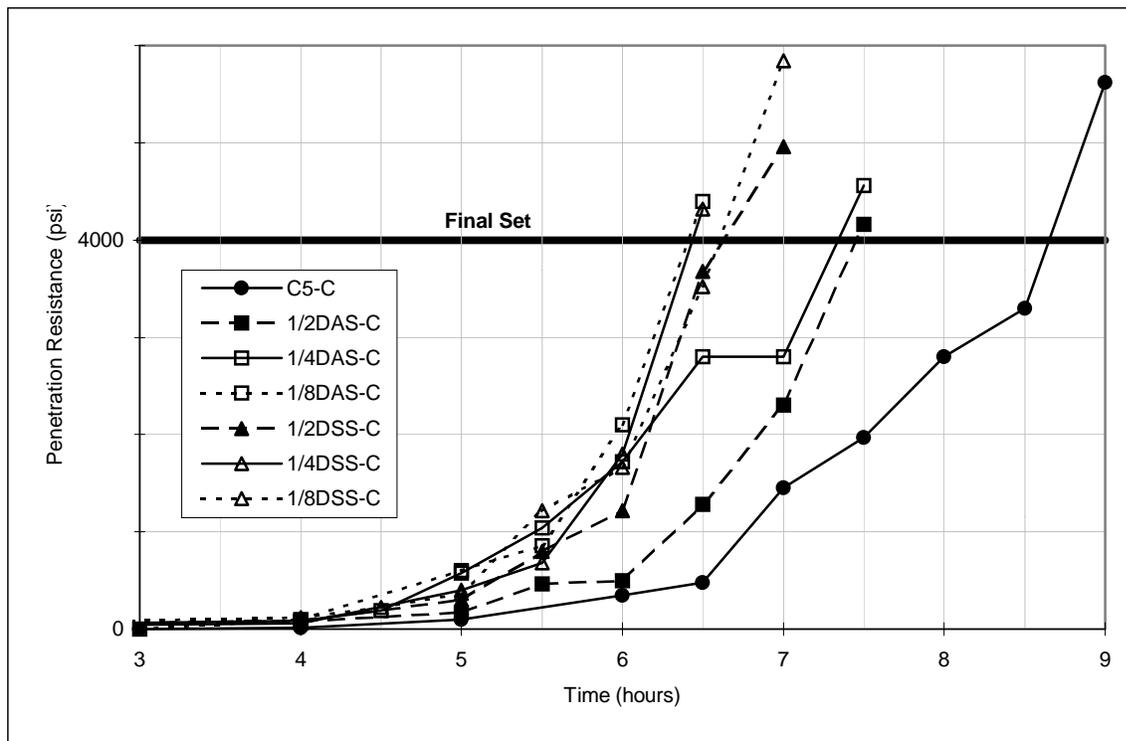


**Figure 5.4  Setting times for Phase II specimens.**

Varying the amount of the DAS chemical produced unexpected results. As the concentration of chemical decreased, so did the setting time. The ½%DAS achieved a penetration resistance of 4000psi at just under 7.5 hours, about 15% faster than the setting time for the Control (8.6 hours). The ¼%DAS set at about 7.3 hours (18% faster) and the 1/8%DAS at approximately 6.4 hours (34% faster). Since the DAS chemical acted as an

accelerator, it seemed strange for the setting time to be reduced with lesser amounts of the chemical.

The DSS chemical produced different results from the DAS. The variance in concentrations for the DSS had little effect on the setting time for those mixes. The setting times of all DSS mixes was around 6.5 hours.

## 5.3  CHLORIDE PENETRATION

Figures 5.5 and 5.6 present the chloride results obtained from the ConnDot laboratory. These results clearly demonstrate one reason the DAS and DSS chemicals are effective at preventing corrosion of the rebar.

As expected from the absorption test results, the amount of chlorides present in the concrete decreased as the concentration of solution increased for both chemicals. Inhibitor A and Inhibitor B performed better than the Control, though Inhibitor A was not significantly better. The chloride content at the exterior surface was inversely proportional to the concentration of solution for the DAS chemical, whereas both DSS mixes had about the same low concentration of chloride. On average, the DSS performed slightly better than the DAS. The amount of chloride present at the surface of the rebar was extremely low. A value this low indicates that this is probably the "background" amount of chloride present in concrete mixing materials.
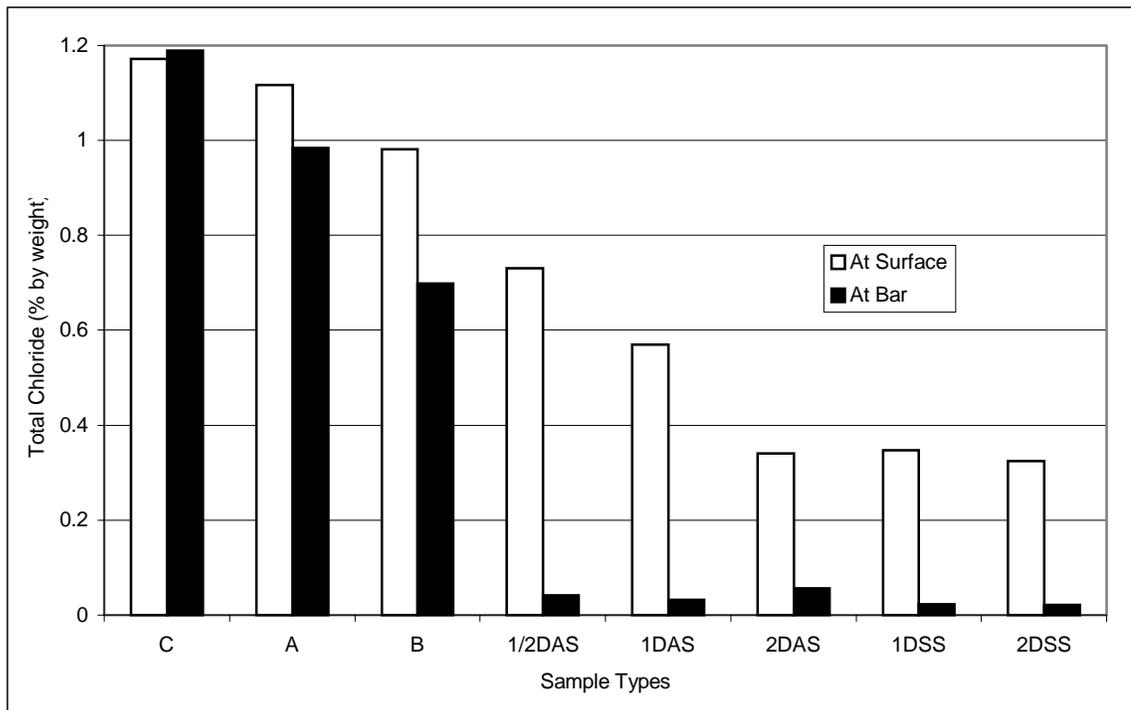


**Figure 5.5  Total chlorides (% by weight) present in the concrete at both the surface and rebar.**
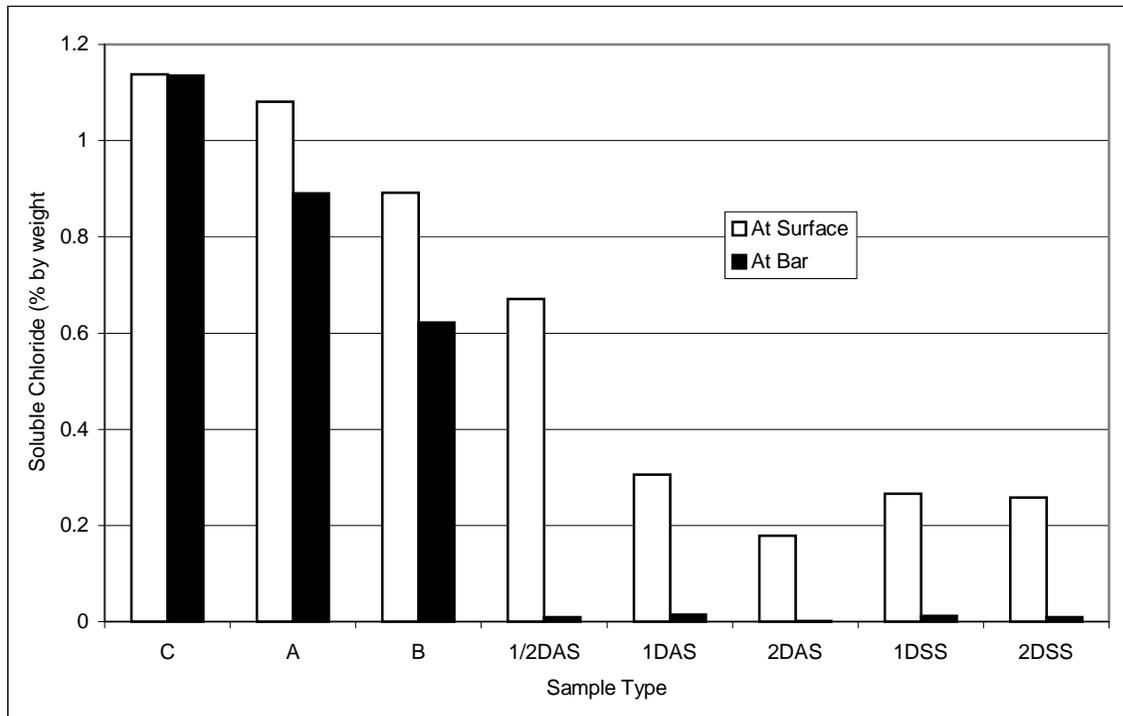
**Figure 5.6  Soluble chlorides (% by weight) present in the concrete at both the surface and rebar.**

## 5.4  SOLUTION RESISTANCE

The solution resistance, $R_s$, was plotted versus time. The solution resistances shown are based on averages of replicate specimens.  Individual plots for each specimen of each group and type are in Appendix A.  Generally the replicates produced very similar results.

The Phase I research was initially reported after approximately 48 weeks of cycling for the lollipops and about 45 weeks for the slabs.  Phase II continued these specimens through about 100 weeks for the lollipops and 97 weeks for the slabs.  Phase II specimens had approximately 37 weeks of cycling.

Phase I data were continued into Phase II and are plotted in Figures 5.7 through 5.12.  Figures 5.13 and 5.14 are for Phase II specimens.  Note the significant difference in the vertical scales ($R_s$) in the Phase I specimens for the DAS and DSS specimens versus the other specimens.  For the Phase II pre-cracked specimens the scales were similar, as expected, because of the low amount of concrete cover in the pre-cracked area and therefore very little solution resistance.  As some of the Phase I specimens cracked from corrosion, their $R_s$ values dropped as less resistive paths for solution existed through these cracks.

### 5.4.1  Control Specimens

The Control 1 and Control 2 specimens of Phase I had similar w/c ratios, air contents, and curing environments.  The compression strength of the Control 1 mix was about 20%

stronger than the Control 2 mix.

The solution resistance for the 2-inch diameter lollipop specimens remained constant at around 50 ohms.  It never rose higher than 70 ohms or fell lower than 40 ohms.  The 3-inch diameter lollipop specimens had similar results averaging initially about 80 ohms.  At approximately 48 cycles (weeks) the solution resistance began dropping off and averaged about 50 ohms.  The Control 2 slab specimens (both "conn" and "unconn") averaged about 80 to 100 ohms for the duration of the cycling.  The Control 1 slab specimens had a very steady rise from about 150 ohms to 400 ohms during the test period.  The pre-cracked 3-inch diameter lollipops had a nearly constant 40 ohms throughout the 37 week period with one deviant point of about 5 ohms at 28 weeks.

The solution resistance for the 3-inch diameter specimens was nearly double that of the 2-inch specimens until approximately 48 weeks of cycling had passed.  At this point the 3-inch diameter results dropped to similar values as the 2-inch and remained there.  From this it appeared as if after approximately 1 year of cycling, the 3-inch specimen resistance is comparable to the 2-inch specimen resistance.  It is interesting to note that the solution resistance for the pre-cracked specimens was only slightly less than the 2-inch specimens.  The 2-inch specimens had ¾ inches of cover whereas the pre-cracked cylinders had relatively no cover (approximately 1/8 inch).  The "whole cylinder" specimens of Phase I had a w/c ratio of 0.38 compared to 0.44 for the pre-cracked Phase II specimens.

## 5.4.2   Inhibitor A and Inhibitor B Specimens

Inhibitor A lollipop specimens displayed results similar to the control specimens.  The slab specimens showed a steady increase in solution resistance similar to, but greater than the Control 1 specimens.  The slab specimens were dried at room temperature and the lollipop specimens were oven dried at $100^o$F.  The absorption testing results (Section 5.2.2) indicated that absorption is reduced for specimens dried at lower temperatures.  This could have been the reason why the solution resistance for the slabs increased over time.  This same trend occurred in the pre-cracked specimen testing, where the solution resistance increased from approximately 40 ohms at the beginning of the testing to over 100 ohms at 30 cycles (the end of the testing).

Inhibitor B showed significantly higher solution resistance values than the Control and Inhibitor A specimens throughout the study for all geometries except the pre-cracked specimens.  There was a sharp rise from the beginning for both the 2-inch and 3-inch specimens from 170 ohms to 360 ohms in 10 cycles, and from 260 to 850 ohms in 35 cycles, respectively.  Both specimen types then had significant decrease with the 3-inch specimens declining to the starting value of 250 ohms after 95 cycles and the 2-inch specimens approaching the Control and Inhibitor A specimens at about 55 cycles.  The Inhibitor B slab specimens showed a larger increase than the Control 1 and Inhibitor A specimens, with the solution resistance increasing to 1900 ohms by the end of testing at 100 cycles.  The pre-cracked Inhibitor B cylinders had comparable results to the oven-dried Control and Inhibitor A specimens.  The air-dried cylinders of Inhibitor B also displayed an increasing resistance with time, but it was surprisingly high when compared to the other specimens.  It was about the same as the ½%DSS (air-dried) specimens but

lower than the ½%DAS (air-dried), until those specimens had a significant decrease at around 22 cycles.

### 5.4.3   DAS and DSS Specimens

Generally, the solution resistance for the DAS and DSS specimens increased over time. Over a span of nearly 100 cycles, the 2-inch cylinders increased by approximately 50 to 60 times the starting value.  The highest increase was in the 2%DAS specimens, starting at 837 ohms and finishing at 49,150 ohms, an increase of 59 times.  The only exception was in the ½%DAS, which increased from 527 to 4350 ohms (an increase of only 8 times).  The 3-inch cylinders also showed dramatic increases of 20 to 45 times in the DAS and about 170 times in the DSS.  The slab specimens also had an increase, though not of the same magnitude.  Again the DSS showed a greater increase of about 4 times compared to 1.5 to 2.5 times for the DAS specimens.  The drying periods probably allowed continuing drying of the concrete but the DAS and DSS chemicals did not permit re-saturation during the soaking periods.  The pre-cracked specimens had an increase on a scale similar to the slabs.  The 1/8%DSS had a basically constant $R_s$ value throughout the testing program.

It is important to note the scale of the DAS and DSS plots when comparing these results with those of the Control and Inhibitor  A and B specimens.  The $R_s$ values for the DAS and DSS were much higher than those of the Control and Inhibitors A and B, with the exception of the pre-cracked cylinders.  Table 5.3 shows how much greater the values for the DAS and DSS were in most cases.

For all specimen types, the solution resistance for both Phase I and Phase II data generally increased as the concentration of either DAS or DSS increased from 1/8% to 2%.

**Table 5.3  Highest and lowest solution resistance values for DAS and DSS relative to Control, Inhibitor A, and Inhibitor B specimens.**

| Specimen Type | | $R_s$ for DAS/DSS Specimens | | | |
|---|---|---|---|---|---|
| | | Vs. Control 1 | Vs. Control 2 | Vs. Inhibitor A | Vs. Inhibitor B |
| 2" cylinders | High | -- | 325x | 325x | 185x |
| | Low | -- | 70x | 70x | 40x |
| 3" cylinders | High | -- | 1200x | 1200x | 190x |
| | Low | -- | 155x | 155x | 25x |
| Slabs | High | 15x | 100x | 60x | 3x |
| | Low | 4x | 25x | 15x | 0.75x |
| Pre-cracked 3" cylinders | High | -- | 3.5x[2] | 2.5x | 2x |
| | Low[1] | -- | 1.2x[2] | 0.9x | 0.75x |

[1]The low end value was from the 1/8%DSS-C specimens.  This low of a concentration was ineffective and produced results comparable to those of the control specimens.
[2]These values are comparisons to Control 5.  The proportions of this mix were nearly identical to those of Control 2.
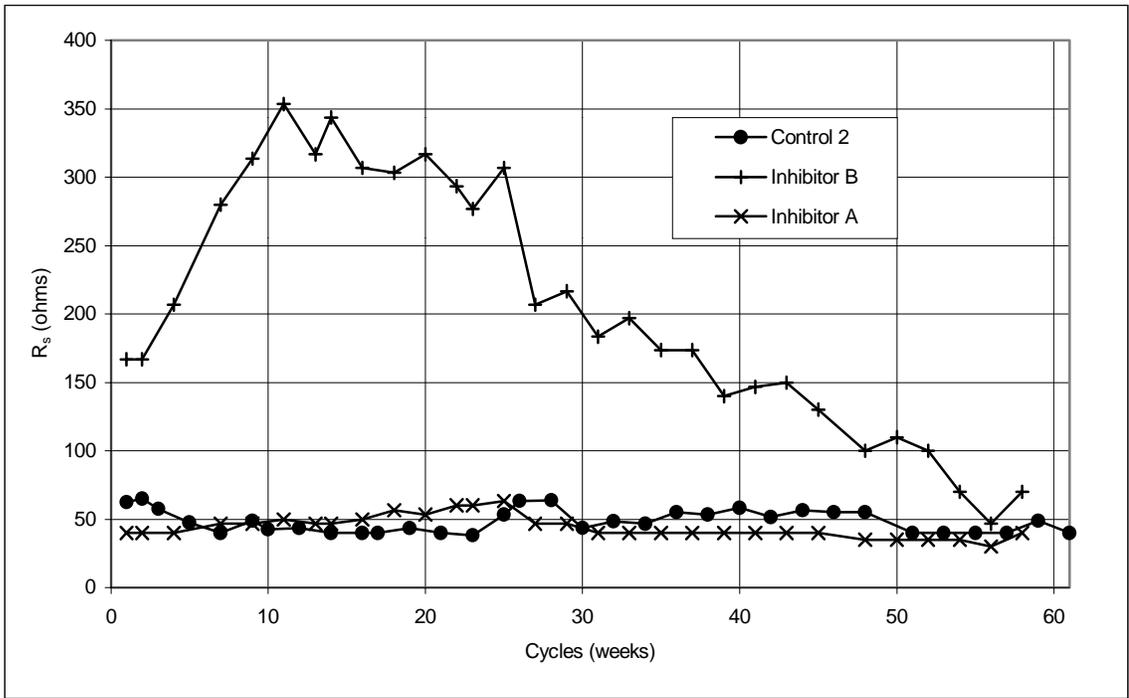
36

**Figure 5.7  Solution resistance, 2-inch cylinders:  Control, Inhibitors A and B.**
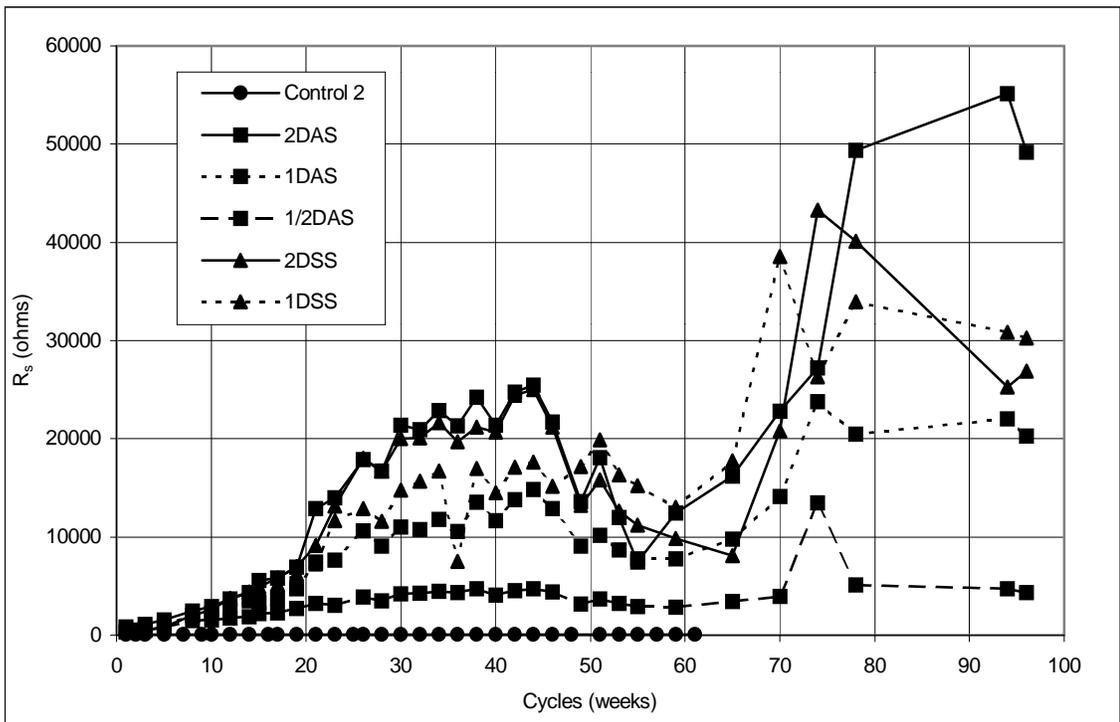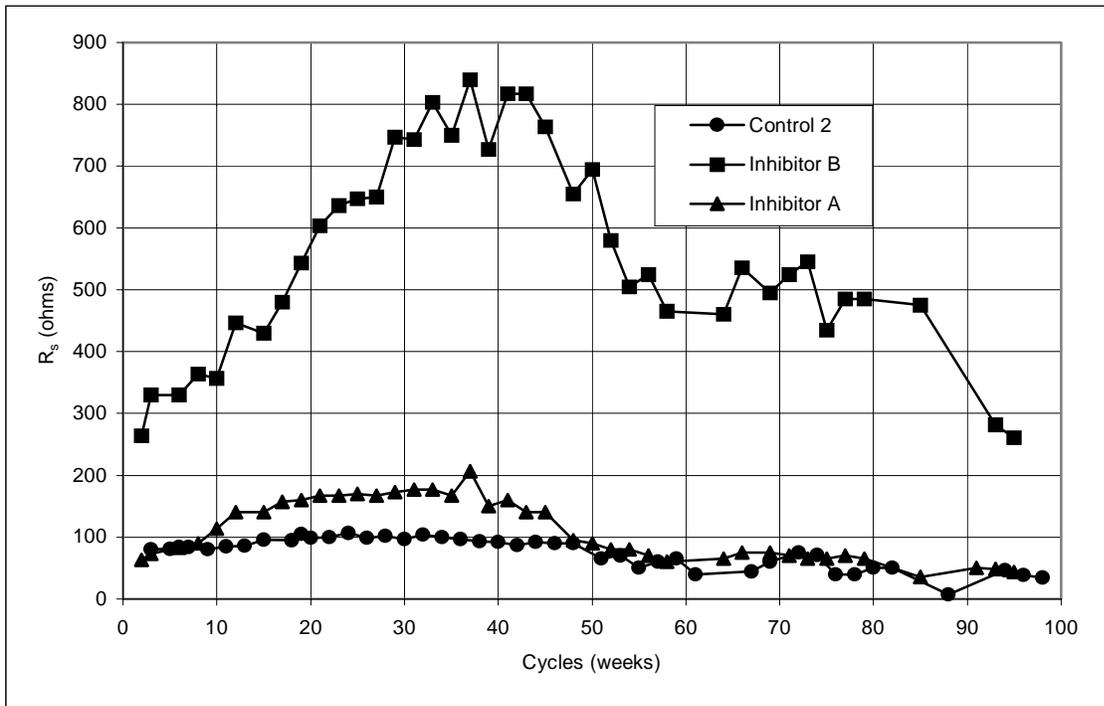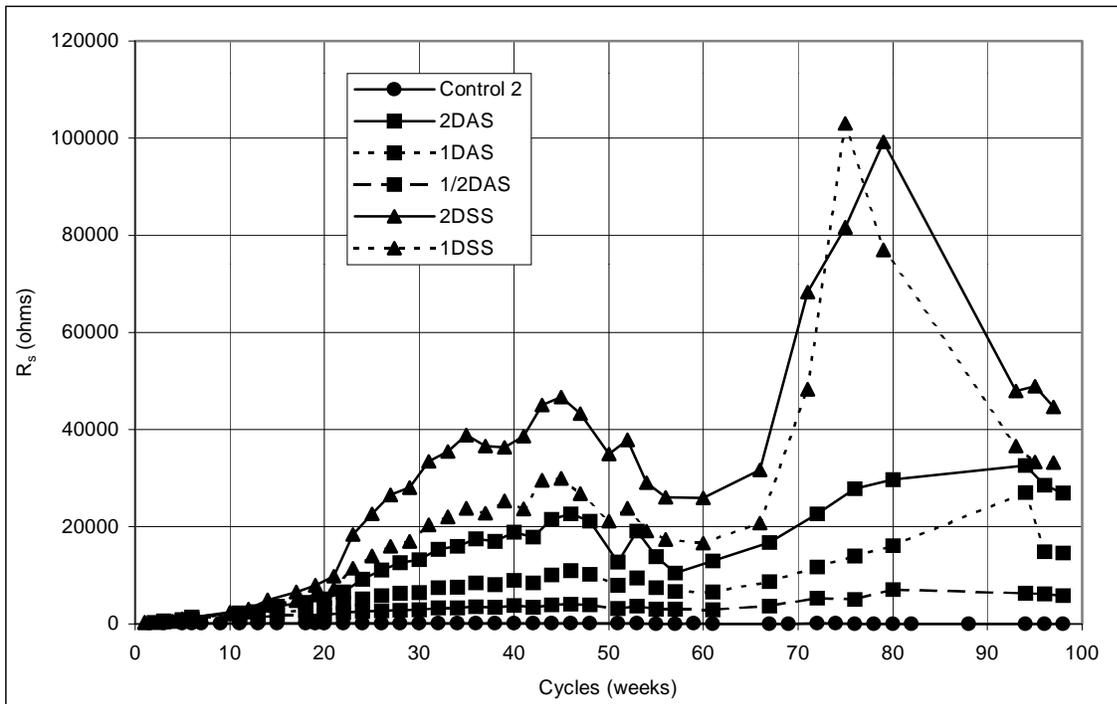


**Figure 5.8  Solution Resistance, 2-inch cylinders:  Control, DAS, and DSS**

**Figure 5.9  Solution resistance, 3-inch cylinders:  Control, Inhibitors A and B.**



**Figure 5.10  Solution resistance, 3-inch cylinders:  Control, DAS, and DSS.**
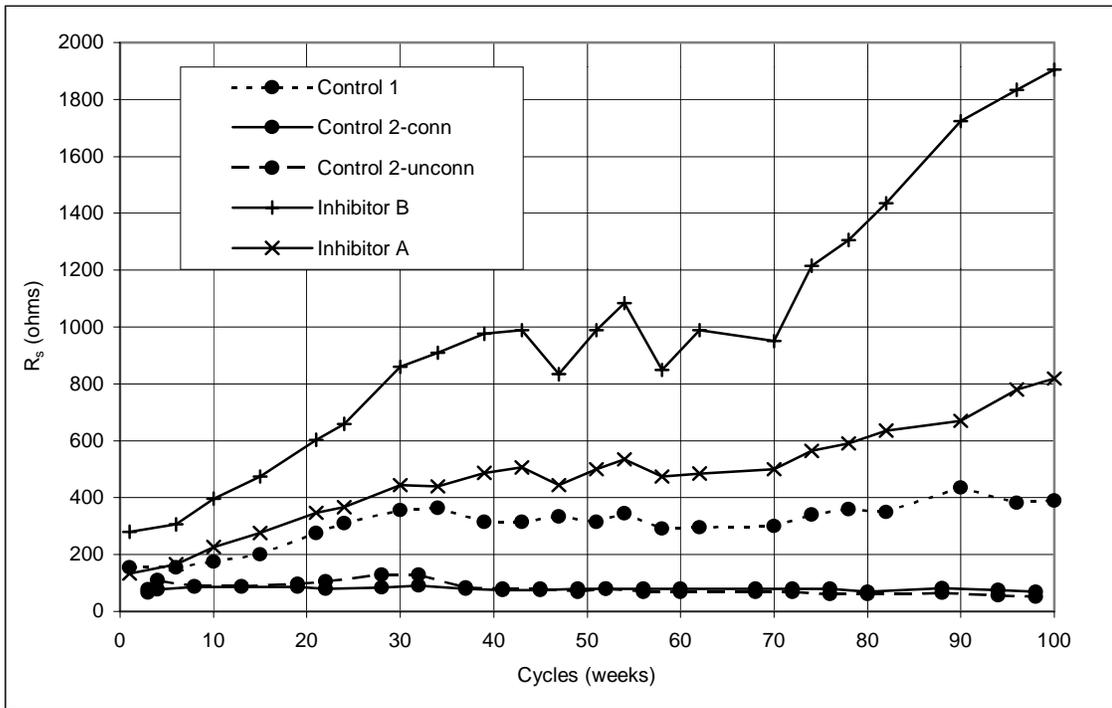
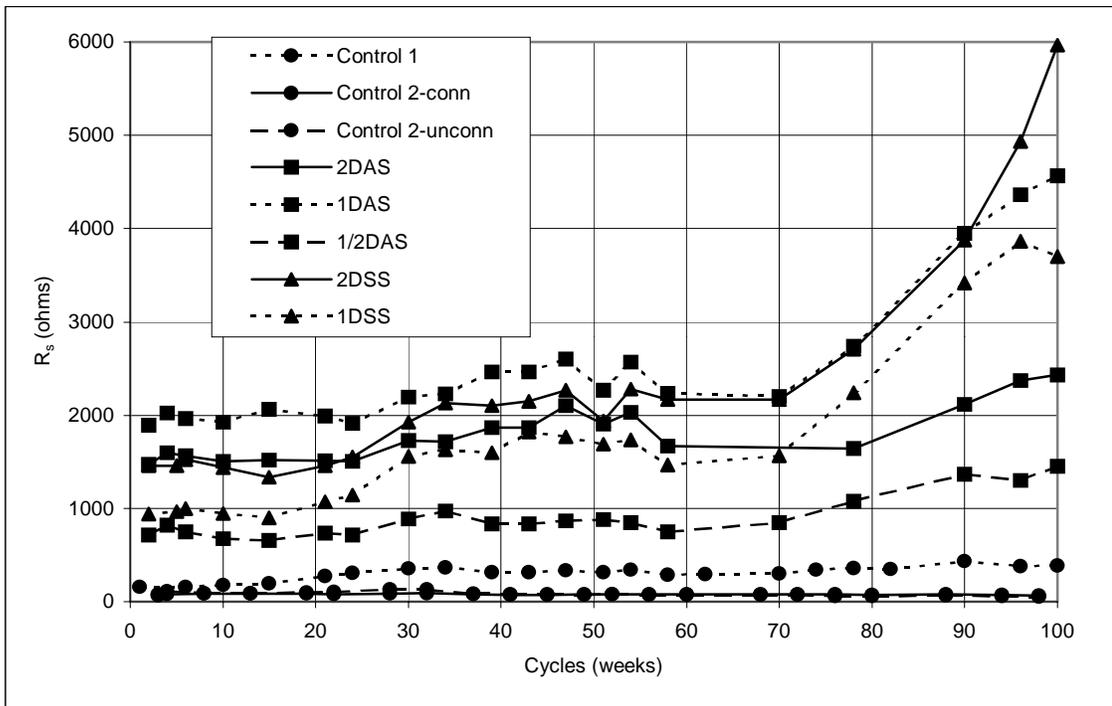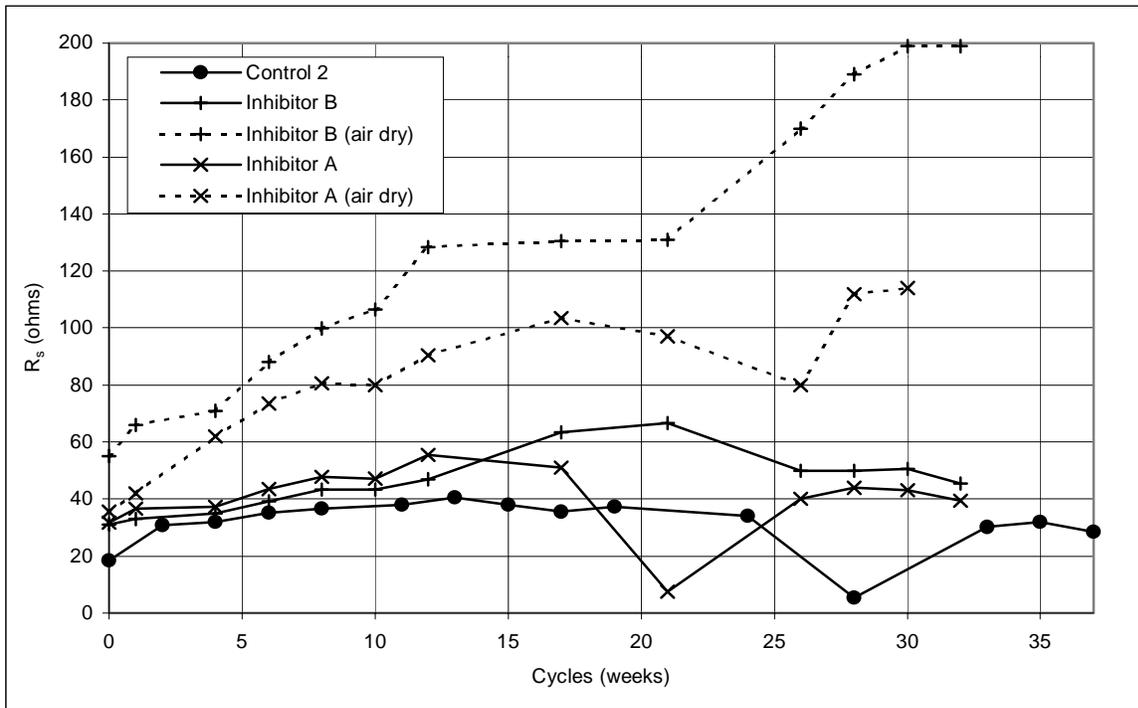**Figure 5.11  Solution resistance, slabs:  Controls 1 and 2, Inhibitors A and B.**



**Figure 5.12  Solution resistance, slabs:  Controls 1 and 2, DAS, and DSS.**

**Figure 5.13  Solution resistance, pre-cracked cylinders: Control, Inhibitors A and B.**
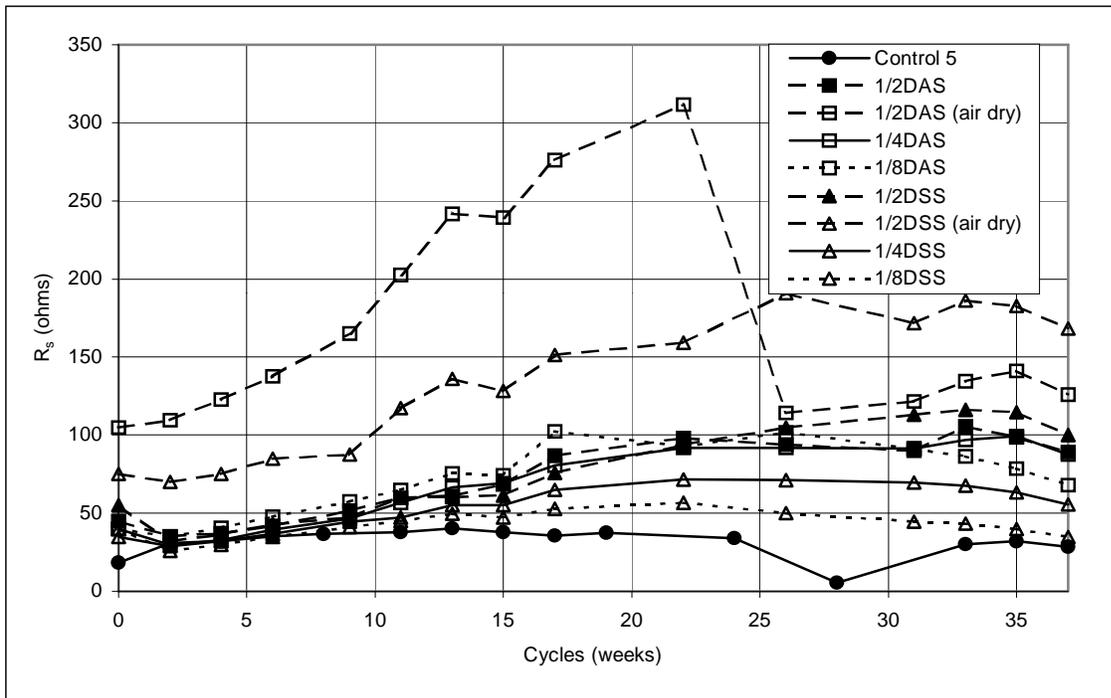


**Figure 5.14  Solution resistance, pre-cracked cylinders:  Control, DAS, and DSS.**

40

## 5.5  CORROSION RATES

Multiplying the current density ($i_{corr}$) by a constant yields corrosion rates (CR), see Section 2.2.1.  This is shown in the following:

$$I_{corr} = \frac{B \text{ (Volts)}}{R_p \text{ (ohms)}} \qquad i_{corr} = \frac{I_{corr}}{\text{Electrode Area (cm}^2)} \qquad CR = i_{corr} \times \text{Constant}$$

From these equations, a direct relationship for calculating the corrosion rates from the polarization is derived and expressed as:

$$CR = \frac{B}{(R_p) \times \text{(electrode area)}}$$

Including the electrode area allows comparisons of specimens with different exposed areas of rebar (electrode area).  In the research reported here, this area is implied as inclusive when the formula is expressed as $1/R_p$.  Therefore, the charts for $1/R_p$ include the area of exposed rebar.  The value of $1/R_p$ is directly proportional to corrosion rates, therefore higher values of $1/R_p$ are synonymous with higher corrosion rates.  The units of $1/R_p$ are the specific area of conductance, which is $\mu$mhos/cm$^2$, where $\mu$mhos equal ($\mu$ohms)$^{-1}$.

For the Phase I specimens, the calculated exposed reinforcing bar area (electrode area) was 36.10 cm$^2$ and 81.07 cm$^2$ for the lollipop (2- and 3-inch) and slab specimens, respectively.  The exposed area on the Phase II specimens was 30.97 cm$^2$, slightly smaller than in Phase I.  In Phase II, the bottom ½ inch of the rebar was coated with epoxy paint to prevent corrosion on the highly vulnerable end.

The corrosion rates, expressed in terms of 1/Rp, for the continued Phase I specimens are shown in Figures 5.15 through 5.20 and for the Phase II specimens in Figures 5.21 and 5.24.  Similarly to the solution resistance values in Section 5.4, the rates were averaged for each set of replicates.  Refer to Appendix A for individual data for all specimens.  Generally, all replicates had similar results.  It must be noted that where there is a sudden change in the rates for some specimens, one of the specimens was behaving differently (specimens in the 1%DSS, 2-inch and Inhibitor B, 3-inch categories in particular).  The deviant specimens were removed from the testing shortly afterward and visually inspected for causes to this behavior.  The plots show where this removal occurs, as the trend line returns to the original path before the deviations had begun.

When a noticeable relative increase in the corrosion rate occurred (and the rate increase continued), this point was assumed to be the initiation of corrosion.  Table 5.4 lists the average time to corrosion for each mix and specimen type.  The final corrosion rates for all specimens and geometries are listed in Table 5.5.

### 5.5.1  Control Specimens

The 2-inch control specimens, with a concrete cover of ¾ inches, had corrosion initiation after 5 cycles.  The 3-inch specimens, with 1¼ inches of cover, had corrosion initiation

after 9 cycles.  These limited results demonstrated that an increase in cover of ½ inch delayed the initiation of corrosion by a factor of approximately two.

Both the conn and unconn Control 2 slab specimens had corrosion initiation at about the same time.  The conn specimens had initiation of corrosion after 3 cycles while the unconn slabs had initiation after 4 cycles with a significant increase in corrosion after 8 cycles.  The delay of the significant increase in the unconn specimens was expected.  The connected slabs acted as both micro- (localized corrosion) and macro-cell (galvanic corrosion), therefore causing an earlier initiation of corrosion than the unconn specimens.  The Control 1 specimens had no indications of corrosion until after 6 cycles.  The average corrosion rate for these specimens increased from a $1/R_p$ value of about three to about 10 from weeks 6 to 21 and remained approximately constant for the duration of the testing.  On the contrary, the Control 2 specimens (both conn and unconn) continued their sharp rises through the end of the testing.

The pre-cracked specimens had corrosion initiating immediately.  This was expected because of the loss of cover protection due to the preformed crack.  Although the corrosion of the pre-cracked specimens initiated earlier than the Phase I counterparts, the trend lines for corrosion rate increased comparable to the Phase I specimens.

When the 2-inch specimens were discontinued after 61 cycles, the final corrosion rate was 168 $\mu$mhos/cm$^2$.  The 3-inch control specimens had a rate of 191 $\mu$mhos/cm$^2$ after 98 cycles.  For the slab specimens, the Control 1 specimens had a rate of only 12 $\mu$mhos/cm$^2$ after 100 cycles, whereas the Control 2-conn and Control 2-unconn had rates of 65 and 81 $\mu$mhos/cm$^2$ after 98 cycles, respectively.  The pre-cracked lollipop specimens, after only 37 cycles, had a rate of 187 $\mu$mhos/cm$^2$.

### 5.5.2   Inhibitor A and Inhibitor B Specimens

Corrosion initiated in the 2-inch specimens of Inhibitor A after 9 cycles, while its 3-inch counterparts started after 25 cycles.  Inhibitor A appeared to delay the onset of corrosion by a factor of nearly 3 when the cover increased by ½ inch, whereas the factor for the controls was only 2.  Inhibitor A had final corrosion rates of 245 $\mu$mhos/cm$^2$ after 58 cycles and 161 $\mu$mhos/cm$^2$ after 95 cycles for the 2- and 3-inch specimens, respectively.  After 30 cycles, the corrosion rate of Inhibitor A in the 2-inch lollipops was higher than the controls.  The difference continued to widen throughout the remainder of the testing with Inhibitor A finishing nearly 80 $\mu$mhos/cm$^2$ higher than the controls, an indication that probably there was an insufficient amount of Inhibitor A remaining after 30 cycles.  The slab specimens showed no indication of corrosion throughout the test period.  In fact, the corrosion rate decreased steadily with time and finished with a rate of 1.89 $\mu$mhos/cm$^2$ after 100 cycles.  For Inhibitor A with the pre-cracked specimens, the oven-dried lollipops had an immediate onset of corrosion, as did the control specimens.  However, the rate then lowered to a minimum of 55 $\mu$mhos/cm$^2$ after 12 cycles.  From this point on, though, the rate increased to 86 $\mu$mhos/cm$^2$ after 32 cycles.  The air-dried specimens displayed similar results to the slabs showing a steadily decreasing rate over time.  These specimens finished with an average of 23 $\mu$mhos/cm$^2$ after 30 cycles.

In general, the Inhibitor B specimens performed better than both the controls and the Inhibitor A specimens.  Corrosion did not initiate in the 2-inch cylinders until after 11

cycles and showed only a slight increase in rate compared to the controls and Inhibitor A. Inhibitor B did momentarily reach the controls' rate in week 56. At 58 weeks, the Inhibitor B specimens finished with a corrosion rate of 153 $\mu$mhos/cm$^2$ for the 2-inch cylinders. In the 3-inch cylinders, corrosion did not initiate until after the 48$^{th}$ cycle. After that time the rate rose steadily to approximately 20 $\mu$mhos/cm$^2$. The rates towards the end of the testing were erratic, but the final rate after 95 cycles was 44 $\mu$mhos/cm$^2$. The Inhibitor B slab specimens were similar to Inhibitor A. It too had a steadily decreasing rate that finished at 1.06 $\mu$mhos/cm$^2$ after 100 weeks of cycling. The results for the pre-cracked specimens were nearly identical to those of Inhibitor A. For the oven-dried specimens, corrosion initiated immediately before dropping to a low of 71 $\mu$mhos/cm$^2$ after 17 cycles and then increasing to a final value of 123 $\mu$mhos/cm$^2$ after 32 weeks. This was the only case where Inhibitor A finished with a lower corrosion rate than Inhibitor B.

### 5.5.3   DAS and DSS Specimens

With the exception of the dropped 2-inch 1%DSS specimen (discussed later), none of the specimens showed any corrosion initiation for the 2- and 3-inch cylinders and slabs for the duration of this project. Only one specimen may have had a small amount of corrosion occurring. The rate for one of the 2-inch ½%DAS specimens increased from about 0.50 $\mu$mhos/cm$^2$ to around 1.20 $\mu$mhos/cm$^2$ (Fig. A.42). This is still a very low corrosion rate and it was difficult to determine from these results whether or not corrosion did in fact initiate. It should also be noted that a 2-inch 1%DSS specimen was accidentally dropped at 23 cycles, creating a large crack, and the onset of corrosion was apparent (Fig. A.44). It is also important to see that even after being cracked, the corrosion rate was not nearly as high as for the control, Inhibitor A, and Inhibitor B specimens. The average corrosion rates, after 96 weeks of cycling, were as follows:

- 2%DAS—0.07 $\mu$mhos/cm$^2$
- 1%DAS—0.03 $\mu$mhos/cm$^2$
- ½%DAS—0.76 $\mu$mhos/cm$^2$
- 2%DSS—0.16 $\mu$mhos/cm$^2$
- 1%DSS—0.10 $\mu$mhos/cm$^2$


The fact that each has a value for corrosion rate does not mean that there is really any corrosion occurring on the specimens. The calculated corrosion rate will always have a value unless the polarization resistance has a value of infinity, since the corrosion rate is inversely proportional to the value of polarization resistance (see Section 2.2.1).
As with the 2-inch cylinders, the 3-inch specimens showed a decreasing corrosion rate. The final rates after 97 cycles were all extremely low, with none greater than 0.30 $\mu$mhos/cm$^2$, and were as follows:

- 2%DAS—0.10 $\mu$mhos/cm$^2$
- 1%DAS—0.06 $\mu$mhos/cm$^2$

- ½%DAS—0.25 μmhos/cm$^2$
- 2%DSS—0.07 μmhos/cm$^2$
- 1%DSS—0.13 μmhos/cm$^2$

The slabs showed a similar trend with the rate decreasing towards zero. When comparing the slab results to those of the cylinders, the air-drying apparently slows the decrease in rate as it approaches zero. This conclusion is based on the fact that the lowest rate was still greater than 0.4 μmhos/cm$^2$ after 100 weeks of cycling. The final corrosion rates after 100 weeks of cycling for all of these specimens were as follows:

- 2%DAS—0.75 μmhos/cm$^2$
- 1%DAS—0.45 μmhos/cm$^2$
- ½%DAS—0.65 μmhos/cm$^2$
- 2%DSS—0.54 μmhos/cm$^2$
- 1%DSS—0.62 μmhos/cm$^2$

The results for the pre-cracked specimens did not quite follow the trends of the other geometries. Due to the minimal amount of concrete cover, corrosion initiated immediately in all of the oven-dried specimens. After the initial increase in corrosion rate, all of the DAS and DSS specimens had lowering rates. In the 1/8%DSS specimens, though, the rate soon began to rise again. The ¼%DSS also had a rising trend after 30 cycles, though not to the same degree as the 1/8%DSS. The 1/8%DAS began rising after 25 weeks of cycling. All oven-dried specimens showed a slight increase in corrosion rate towards the end of the cycling, though it could not be determined for sure if this trend would continue. The air-dried specimens exhibited the same behavior as the 2- and 3-inch cylinders. The corrosion rate decreased immediately and proceeded towards lower values with time. The ½%DAS specimens showed a rise after 22 weeks of cycling but the trend leveled immediately. The final ratings for the DAS and DSS specimens after 37 cycles were seen as follows:

- ½%DAS (oven)—61 μmhos/cm$^2$
- ½%DAS (air)—43 μmhos/cm$^2$
- ¼%DAS—62 μmhos/cm$^2$
- 1/8%DAS—88 μmhos/cm$^2$
- ½%DSS (oven)—59 μmhos/cm$^2$
- ½%DSS (air)—33 μmhos/cm$^2$
- ¼%DSS—90 μmhos/cm$^2$
- 1/8%DSS—155 μmhos/cm$^2$

### 5.5.4  Comparison Among All Inhibitors

Table 5.5 summarizes the corrosion rates of all specimens. The final corrosion rates relative to the inhibitor concentration of DAS and DSS were inconsistent. For uncracked specimens, the 1%DAS always had the best (lowest) corrosion rate. The ½%DAS always had the highest rate (excluding the pre-cracked specimens), except for the slabs, though it

was near the highest. In terms of decreasing performance in corrosion resistance in uncracked specimens, the order was DAS, DSS, Inhibitor B, Inhibitor A, and then the controls.

Figures 5.23 and 5.24 compare the results for all materials for both oven-dried and air-dried pre-cracked specimens, respectively. In the pre-cracked specimens where lower concentrations of DAS and DSS were used the values were much closer. For oven-dried cylinders, the ½%DSS specimens had the lowest corrosion rates. The highest (excluding the controls) were the 1/8%DSS specimens. The overall order from best (lowest rate) to worst (highest) is as follows: ½%DSS, ½%DAS, ¼%DAS, Inhibitor A, 1/8%DAS, ¼%DSS, Inhibitor B, 1/8%DSS, Controls. Surprisingly, the air-dried cylinders had an order that was nearly reversed from the oven-dried specimens. For these cylinders, Inhibitor B was best, followed by Inhibitor A, ½%DSS, and then ½%DAS.

Generally, the ½%DAS and ½%DSS pre-cracked specimens performed comparably to or better than Inhibitors A and B. Also notice that for the oven-dried pre-cracked specimens, the Inhibitors A and B specimens had corrosion rates that were tending to increase at the end of testing. Conversely, the ½%DSS and ½%DAS specimens had rates that were still low and still "bottoming out" (Fig. 5.23).
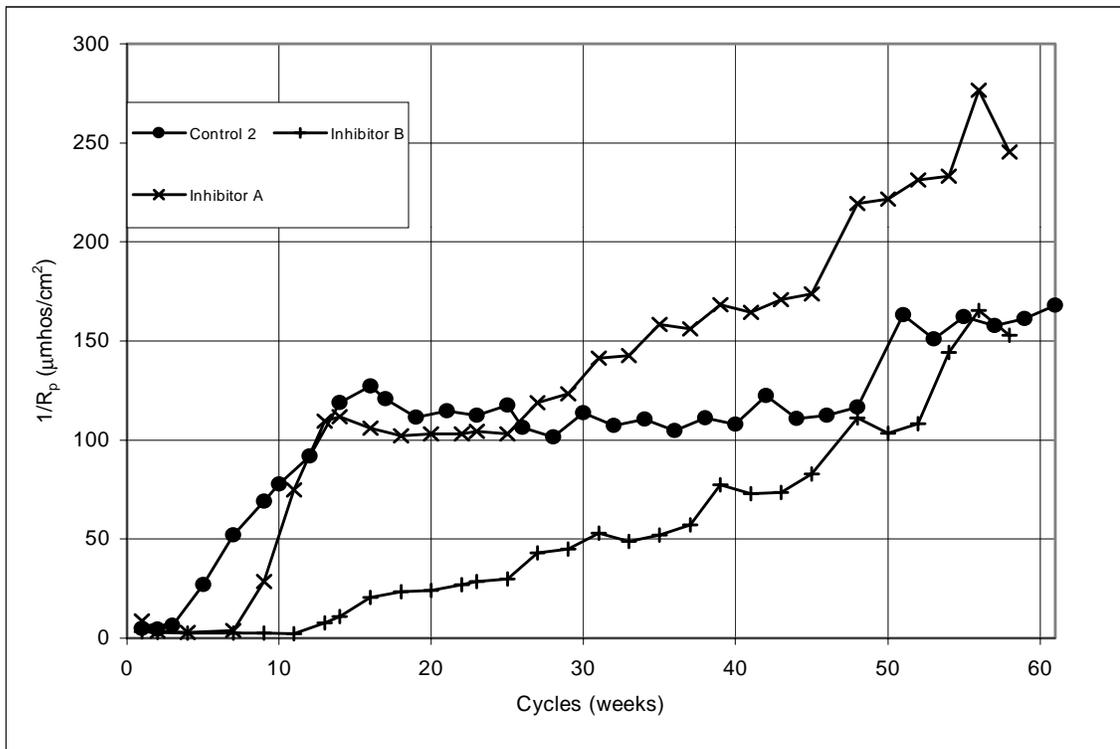


**Figure 5.15  Corrosion rates, 2-inch cylinders:  Control, Inhibitors A and B.**
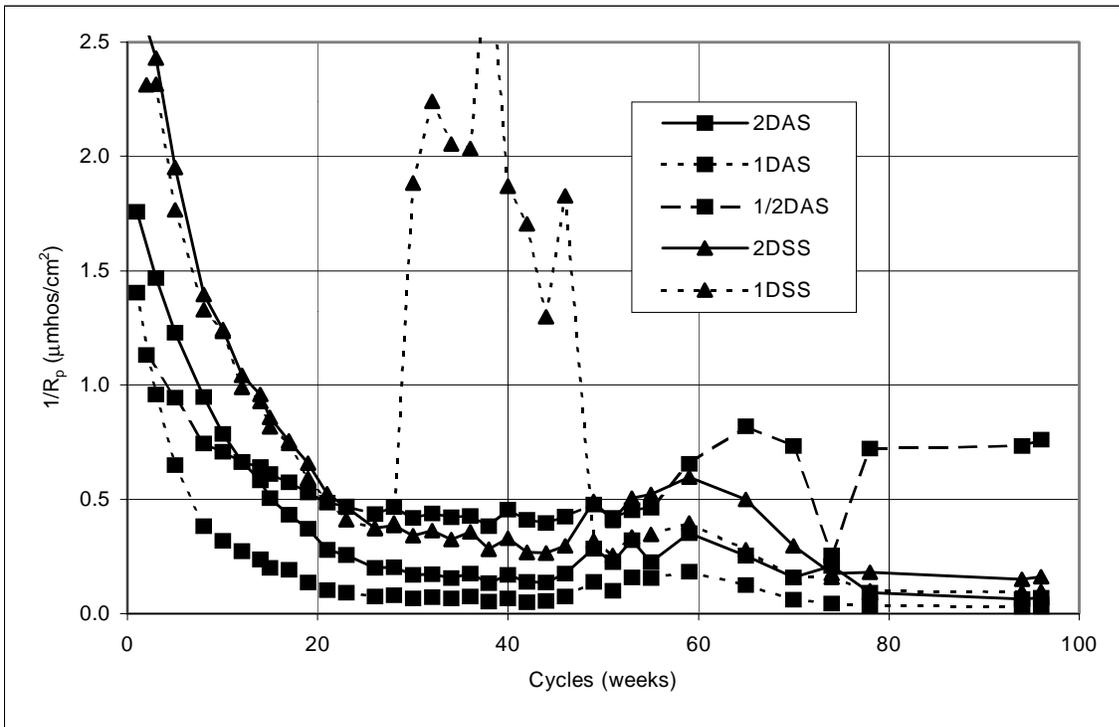
**Figure 5.16  Corrosion rates, 2-inch cylinders:  DAS and DSS.**
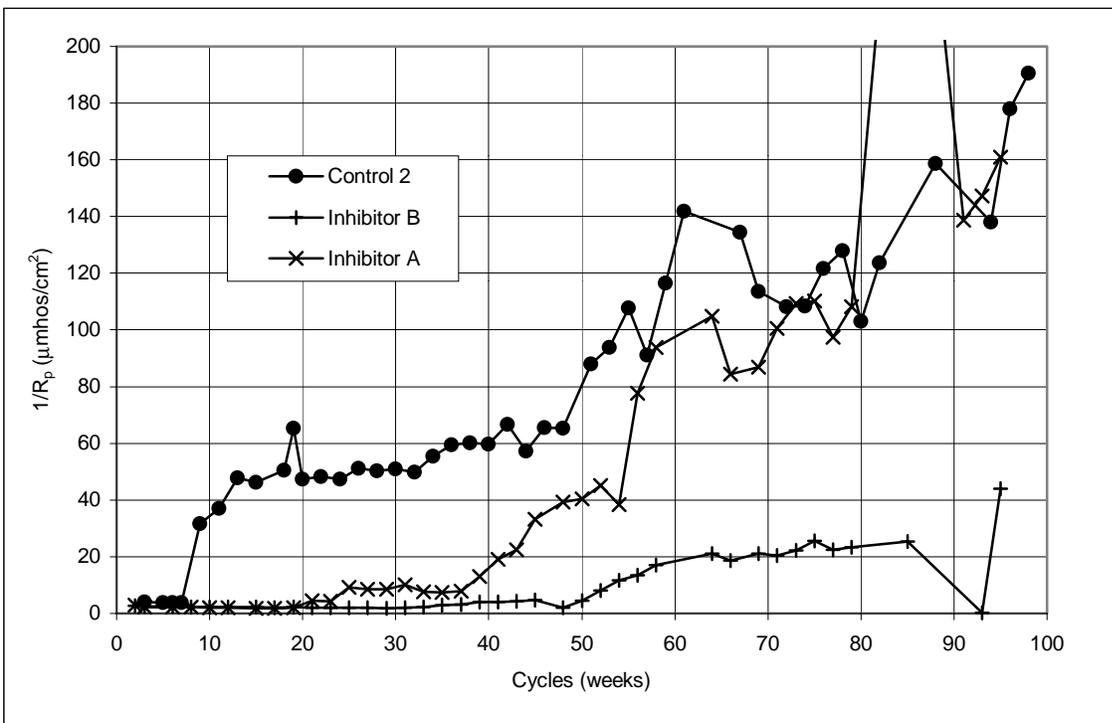


**Figure 5.17  Corrosion rates, 3-inch cylinders:  Control, Inhibitors A and B.**
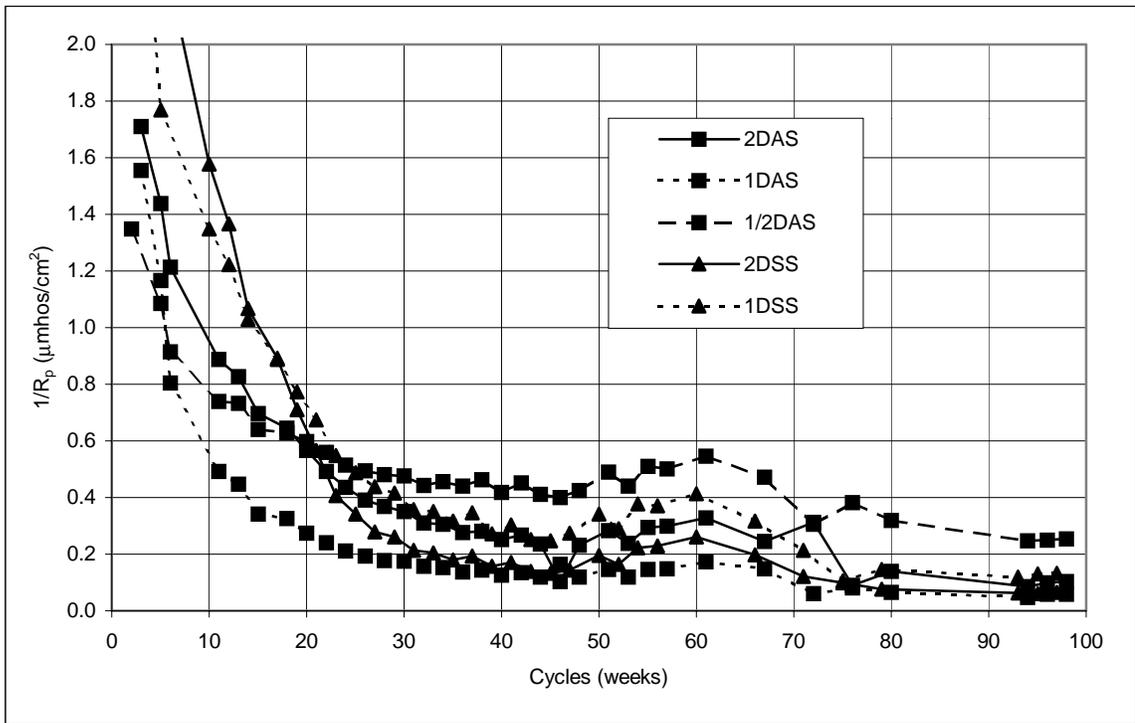
**Figure 5.18  Corrosion rates, 3-inch cylinders:  DAS and DSS.**
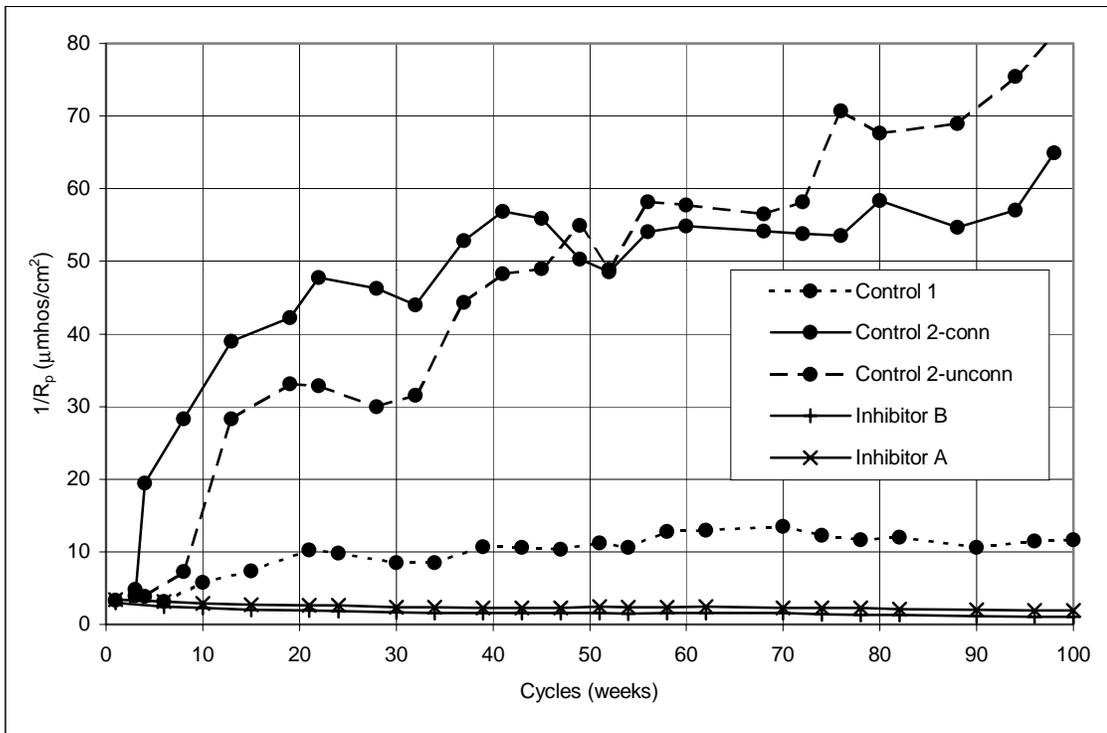


**Figure 5.19  Corrosion rates, slabs:  Control, Inhibitors A and B.**
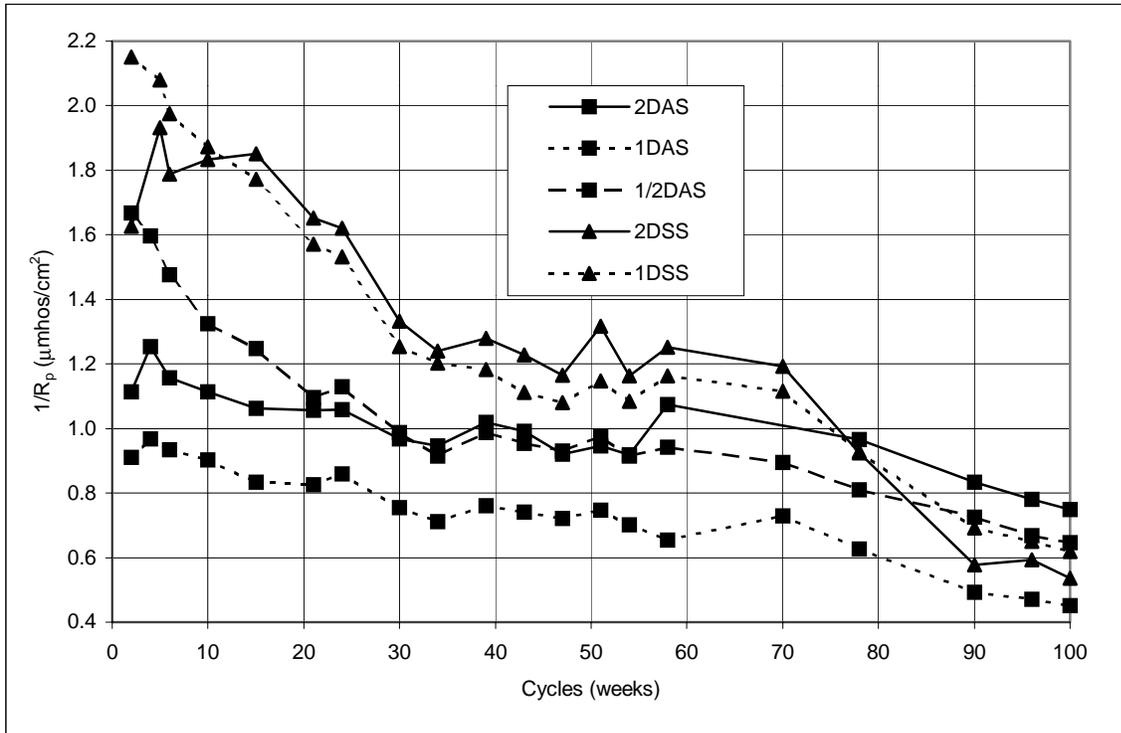
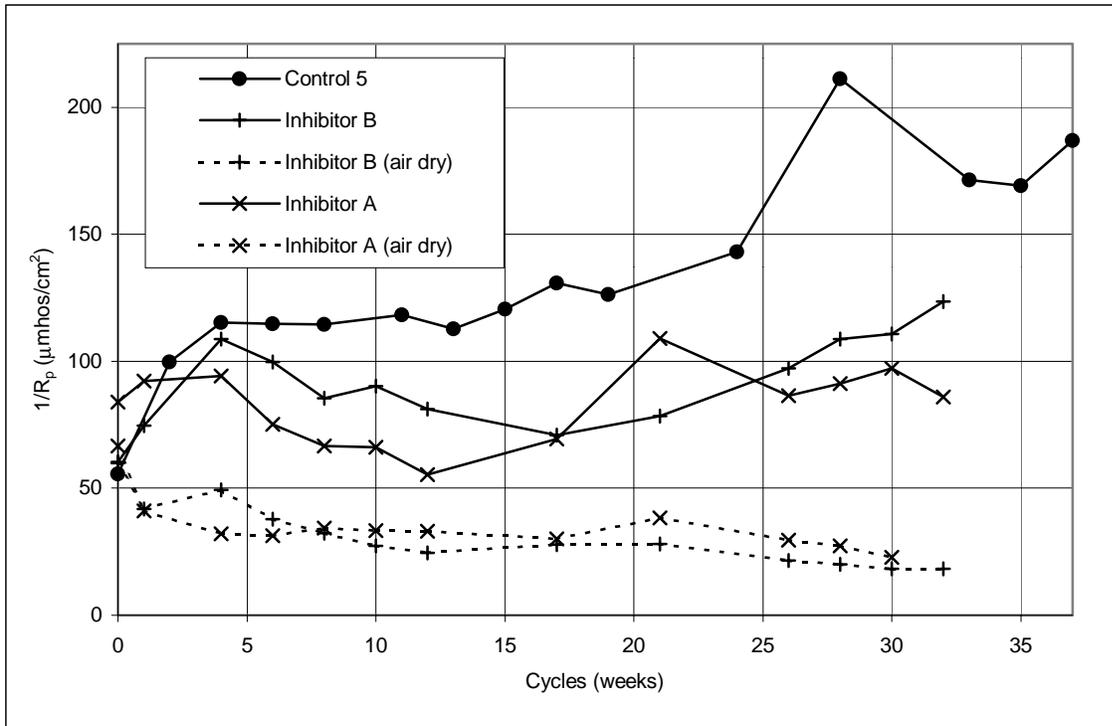**Figure 5.20  Corrosion rates, slabs:  DAS and DSS.**



**Figure 5.21  Corrosion rates, pre-cracked cylinders:  Control, Inhibitors A and B.**
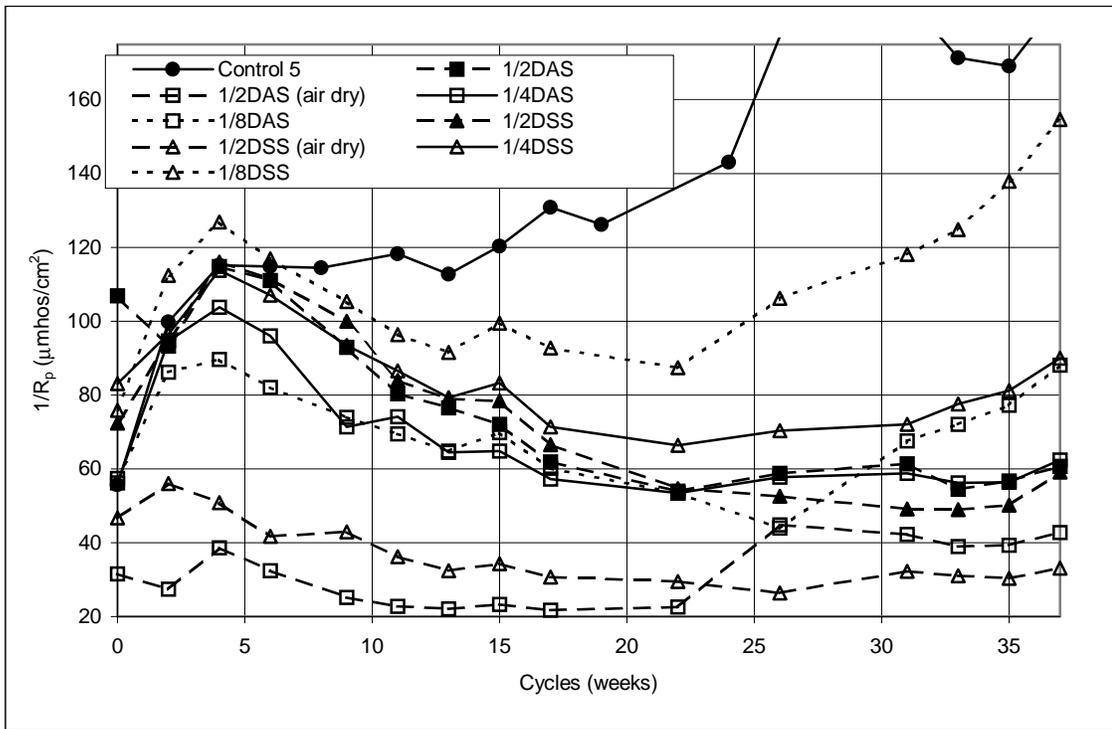
**Figure 5.22  Corrosion rates, pre-cracked cylinders:  Control, DAS, and DSS.**
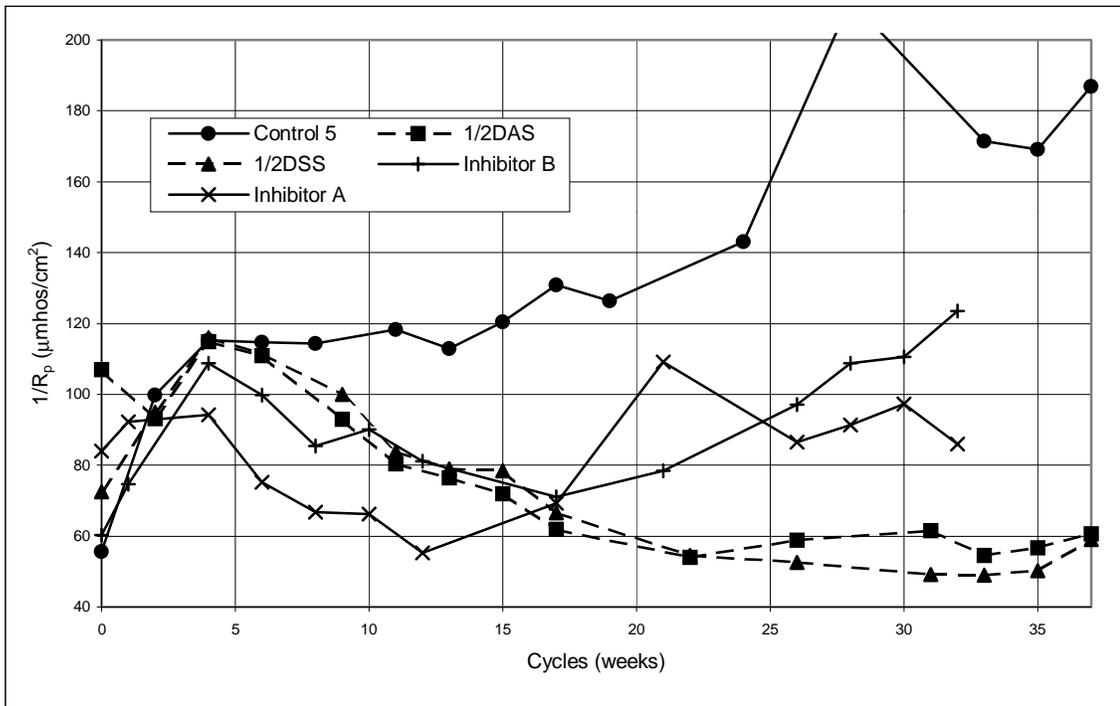


**Figure 5.23 Corrosion rates, pre-cracked cylinders:  Control, ½%DAS and DSS, and Inhibitors A and B.**
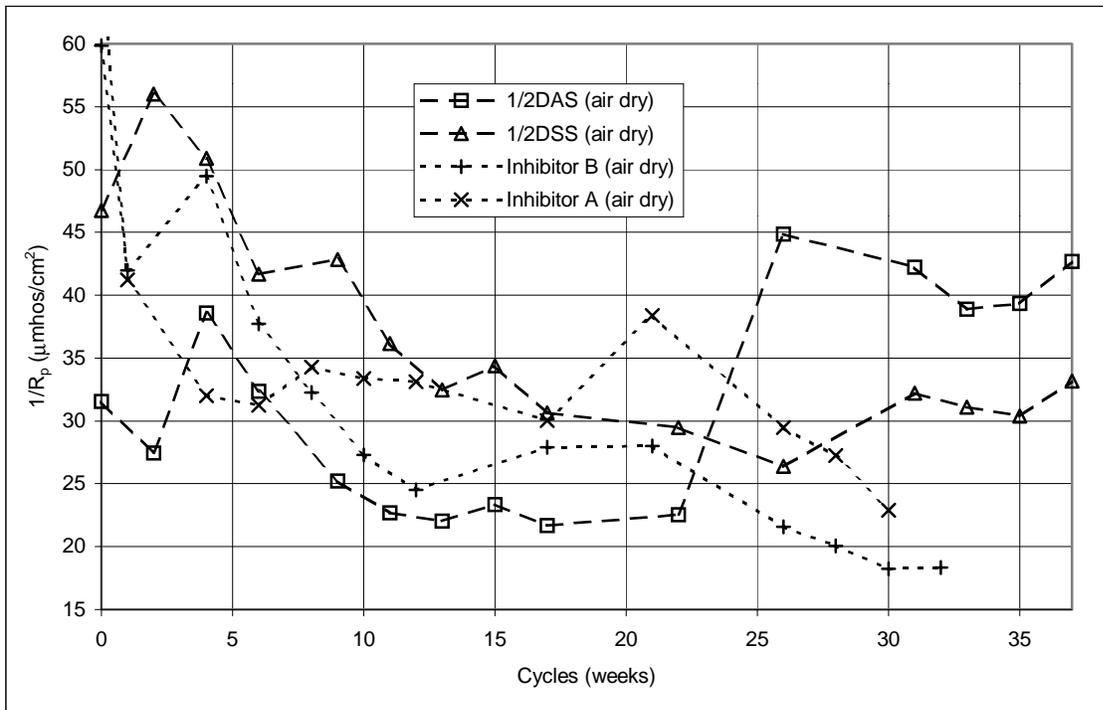
**Figure 5.24 Corrosion rates, pre-cracked cylinders (air-dried): ½%DAS and DSS, Inhibitors A and B.**

**Table 5.4 Average time to corrosion: Corrosion mixes.**

| Mix Type | 2-inch Lollipop (weeks) | 3-inch Lollipop (weeks) | Slab (weeks) |
|---|---|---|---|
| Control 1 | --- | --- | 6 |
| Control 2 | 5 | 9 | --- |
| Control 2-conn | --- | --- | 3 |
| Control 2-unconn | --- | --- | 8 |
| Inhibitor A | 9 | 25 | * |
| Inhibitor B | 11 | 48 | * |
| 2%DAS | * | * | * |
| 1%DAS | * | * | * |
| ½%DAS | * | * | * |
| 2%DSS | * | * | * |
| 1%DSS | 30 [1] | * | * |

--- means "No specimen of this type."

* means "No corrosion initiated by the end of testing."

[1]One of the 1%DSS specimens was accidentally dropped and a crack formed allowing for the initiation of corrosion.

50

**Table 5.5  Corrosion rates for all specimens.**

| Mix Type | 2-inch Lollipop ($\mu$mhos/cm$^2$) ~100 weeks | 3-inch Lollipop ($\mu$mhos/cm$^2$) ~100 weeks | Slab ($\mu$mhos/cm$^2$) ~100 weeks | Pre-cracked Lollipop ($\mu$mhos/cm$^2$) ~35 weeks |
|---|---|---|---|---|
| Control 1 | --- | --- | 12 | --- |
| Control 2 | 168 [1] | 191 | --- | --- |
| Control 2-conn | --- | --- | 65 | --- |
| Control 2-unconn | --- | --- | 81 | --- |
| Control 5 | --- | --- | --- | 187 |
| Inhibitor A | 245 [1] | 161 | 1.89 | 86 |
| Inhibitor A (air dry) | --- | --- | --- | 23 [2] |
| Inhibitor B | 153 [1] | 44 | 1.06 | 123 |
| Inhibitor B (air dry) | --- | --- | --- | 18 |
| 2%DAS | 0.07 | 0.10 | 0.75 | --- |
| 1%DAS | 0.03 | 0.06 | 0.45 | --- |
| ½%DAS | 0.76 | 0.25 | 0.65 | 61 |
| ½%DAS (air dry) | --- | --- | --- | 43 |
| ¼%DAS | --- | --- | --- | 62 |
| 1/8%DAS | --- | --- | --- | 88 |
| 2%DSS | 0.16 | 0.07 | 0.54 | --- |
| 1%DSS | 0.10 | 0.13 | 0.62 | --- |
| ½%DSS | --- | --- | --- | 59 |
| ½%DSS (air dry) | --- | --- | --- | 33 |
| ¼%DSS | --- | --- | --- | 90 |
| 1/8%DSS | --- | --- | --- | 155 |

--- means "No specimen of this type."

[1]The rates for these specimens are for approximately 60 weeks of cycling.

[2]This rate is for 30 weeks of cycling.

## 5.6  VISUAL EXAMINATION OF SPECIMENS

At the completion of all wet-dry cycling, all lollipop specimens (2-inch, 3-inch, and pre-cracked) were saw cut longitudinally on two sides, split open, and visually inspected. Slabs were opened in a similar fashion by saw cutting along two sides of the rebar of interest. Some Phase I specimens were opened at the end of that phase. The remaining Phase I specimens were opened following the Phase II research.

After splitting open the specimens, they were photographed (Figures 5.25 through 5.34). The "front" and "back" of each concrete specimen and the rebar are shown so that the entire circumference can be seen. The "backs" of the pre-cracked concrete cylinders are not displayed because the corrosion was induced on the "front" sides where the pre-formed cracks were located. Cracking is clearly visible on the concrete surfaces of the 3-inch diameter Control and Inhibitors A and B specimens. The Control specimens were heavily cracked during the Phase I tests [48]. Tables 5.6 through 5.9 are a tabulation of quantitative values for the corroded area of each specimen.

The corrosion of the reinforcing bars was generally localized. The surfaces of some bars were almost completely covered in corrosion products, therefore it could not be determined for sure where the corrosion had begun initially. Immediately after each rebar was extracted from its concrete casing, any corroding bars exhibited a dark green-

black crust of corrosion. This indicated that the visible corrosion products were in the form of ferrous hydroxide that is green in color. The color of these corrosion products changed from green to the commonly seen red rust color after being exposed to air. This indicated the change from the ferrous hydroxide to ferric hydroxide due to the abundance of oxygen found in air.

The use of the epoxy paint directly on the rebar either prevented or greatly reduced the problems of crevice corrosion found beneath the electroplater's tape from the Phase I specimens. The epoxy formed a much tighter seal around the rebar preventing the infusion of moisture. However, some corrosion was discovered beneath the epoxy coating. In all instances, this corrosion was found under the epoxy coating on the bottom half-inch of the rebar.

### 5.6.1   Corrosion Monitored Specimens

There was good correlation between the linear polarization results and what was observed visually. Every specimen for which the linear polarization results had indicated corrosion, clearly had some corrosion forming on the bar surfaces after they were split open. Those specimens that had no visible corrosion also had this indicated by the linear polarization results. There was also a strong correlation between the amount of visible corrosion and the corrosion rate indicated by the linear polarization data.

### 5.6.2   Phase I Specimens

All DAS and DSS specimens appeared to be undamaged by the extreme conditions of the cycling. The specimens shown in Figures 5.25 and 5.26 are representative examples of each mix. After approximately 100 weeks of cycling the concrete surfaces looked as if they had just been cast. The topmost inch of concrete surface on the cylinders was not submerged in the sodium chloride solution and darkened over time. The submerged portion of each cylinder was relatively unmarked from its original appearance. The rebars had clean surfaces after removal with no visible corrosion (Figures 5.27 and 5.28). The gray material visible on the surface of some of the rebars is concrete that was not removed. The rebars from the slab specimens revealed similar results with clean, unstained surfaces (Figure 5.29).

The Control, Inhibitor A, and Inhibitor B specimens did not perform as well in the 2-inch and 3-inch cylinders. This was most noticeable in the 2-inch cylinders that had less concrete cover and were discontinued after approximately 60 cycles due to the high corrosion rates (Figure 5.15). The 3-inch cylinders were terminated after about 100 cycles. There was a very distinguishable difference in the appearance on the surface of the concrete. There were noticeable surface cracks on the 3-inch Control, Inhibitor A, and Inhibitor B specimens, appearing as white lines across the surfaces of the concrete Figure 5.26. There was also a discoloration on the surface. All three specimens slowly changed hue to a dull red-orange. The relative color differences between the DAS and DSS specimens and the control, Inhibitor A, and Inhibitor B specimens is clearly seen in Figure 5.26. Corrosion was visible on the rebar surfaces for these three series (Figure 5.28). As seen in the polarization data, only the control specimens (both Control 1 and

| 1%DSS | 2%DAS | 1%DAS | ½%DAS | 2%DSS |

**Figure 5.25  Final exterior appearance of 2-inch DAS and DSS specimens.**

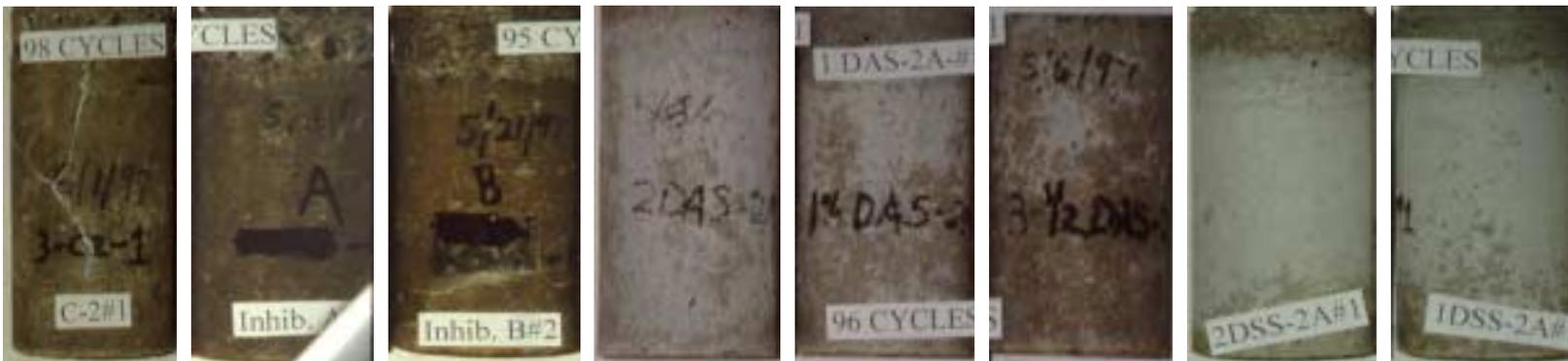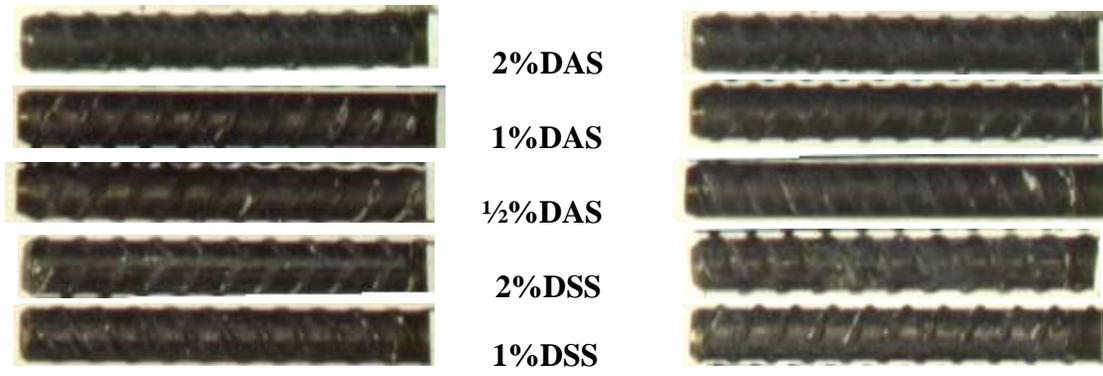| Control 2 | Inhibitor A | Inhibitor B | 2%DAS | 1%DAS | ½%DAS | 2%DSS | 1%DSS |

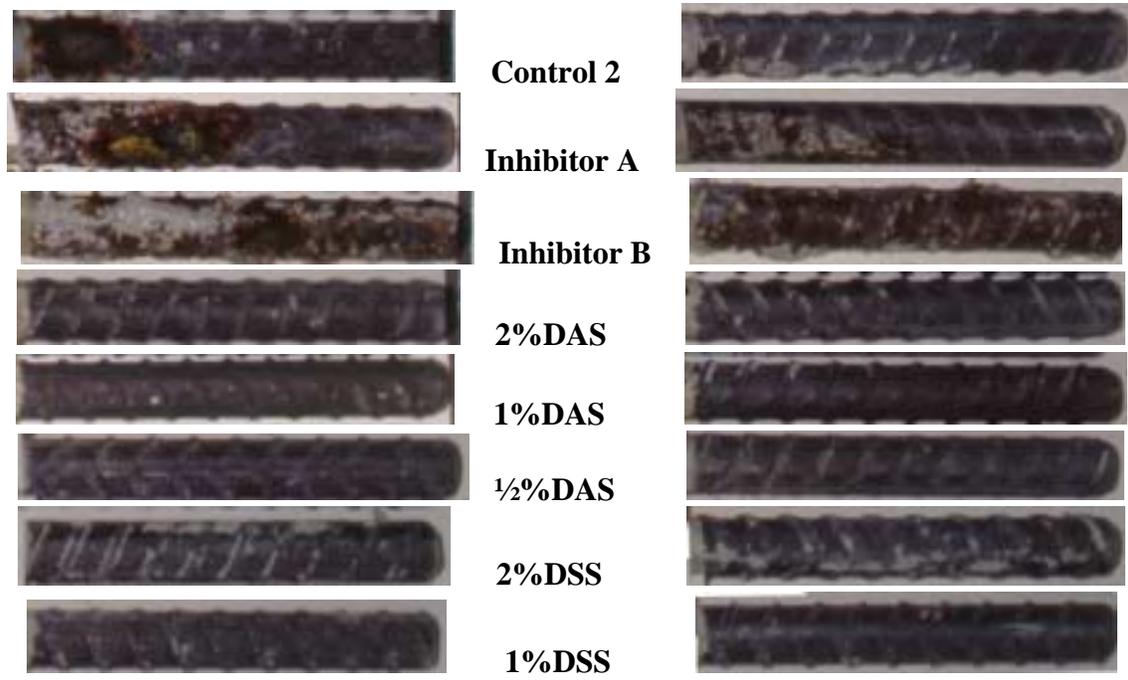**Figure 5.26 Final exterior appearance of the 3-inch specimens.**

**2%DAS**

**1%DAS**

**½%DAS**

**2%DSS**

**1%DSS**

"Front"                                                      "Back"

**Figure 5.27  No. 4 rebar from the 2-inch DAS and DSS specimens.**



**Control 2**

**Inhibitor A**

**Inhibitor B**

**2%DAS**

**1%DAS**

**½%DAS**

**2%DSS**

**1%DSS**

"Front"                                                      "Back"

**Figure 5.28  No. 4 rebar from the 3-inch specimens.**

**Control 1**

**Ctrl 2-conn**

**Ctrl 2-unconn**

**Inhibitor A**

**Inhibitor B**

**2%DAS**

**1%DAS**

**½%DAS**

**2%DSS**

**1%DSS**

**"Front"**                    **"Back"**
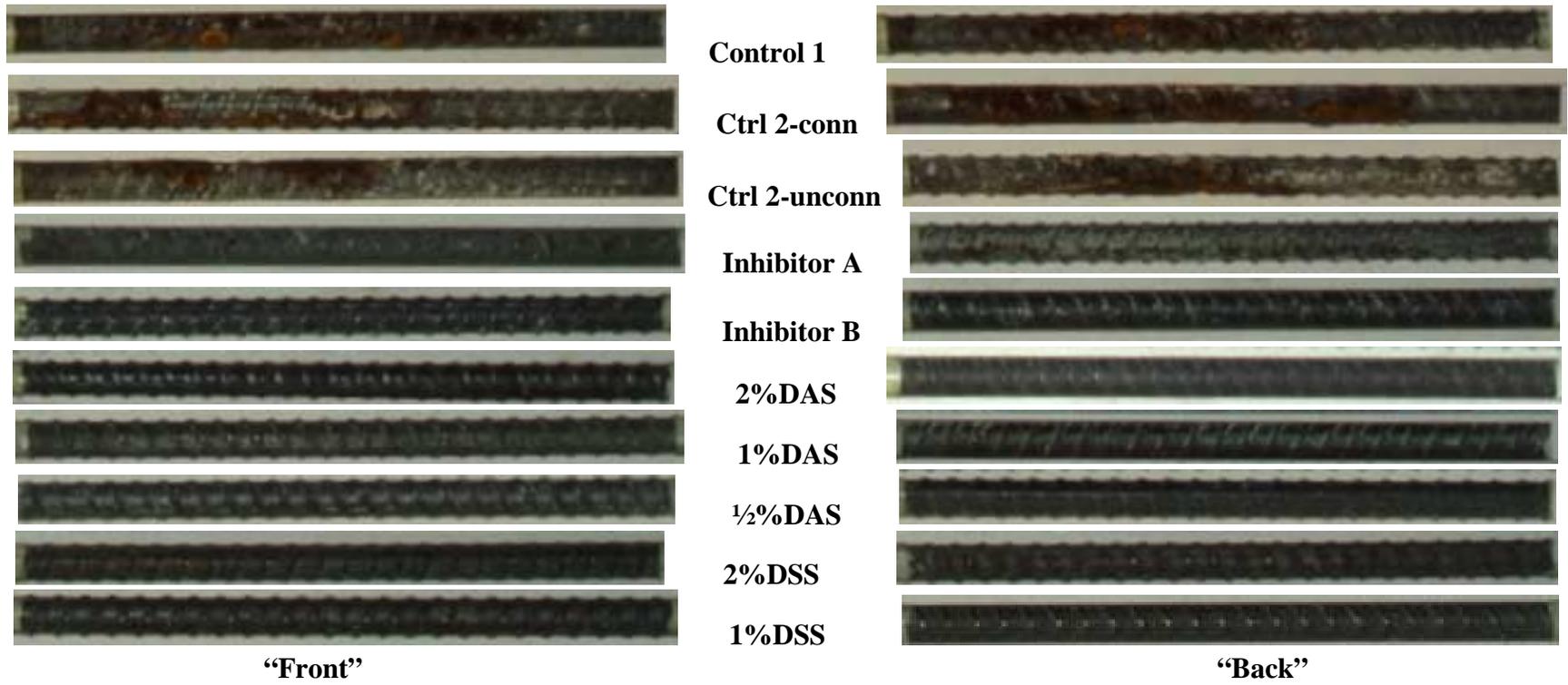
**Figure 5.29 No. 4 rebar from the slab specimens.**

**2%DAS**

**1%DAS**

**2%DSS**

**1%DSS**

**"Front"**　　　　　　　　　　　　　　　　　**"Back"**
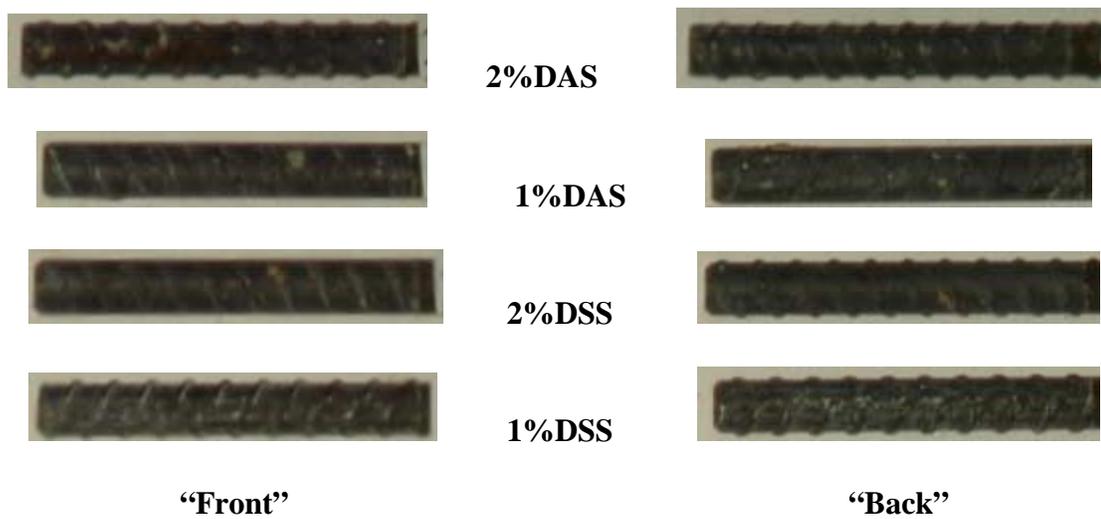
**Figure 5.30  No. 4 rebar from the "Saw-Cut" 3-inch DAS and DSS specimens.**

**Control**

**Inhibitor A**

**Inhibitor B**

**½%DAS**

**¼%DAS**

**1/8%DAS**

**½%DSS**

**¼%DSS**

**1/8%DSS**

**"Front"**          **"Back"**
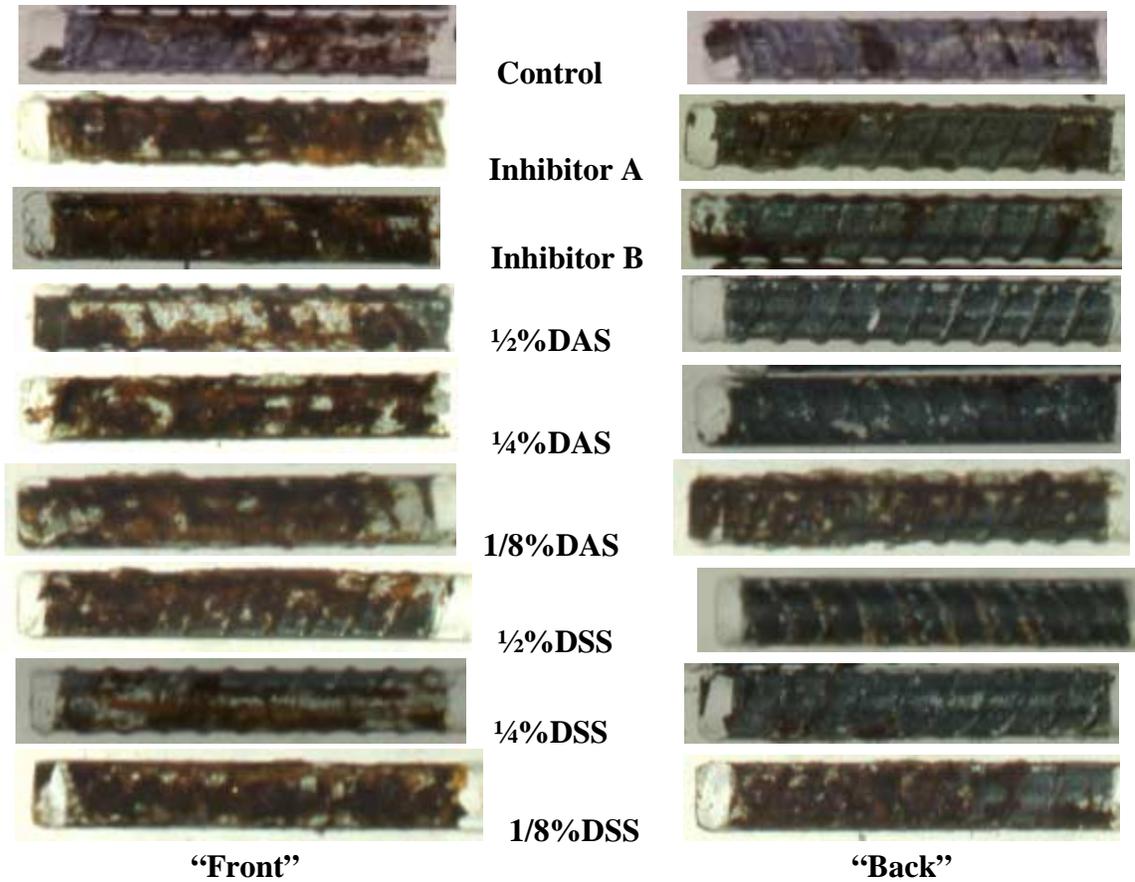
**Figure 5.31  No. 4 rebar from the oven-dried pre-cracked specimens.**



**Inhibitor A**

**Inhibitor B**

**½%DAS**

**½%DSS**

**"Front"**          **"Back"**
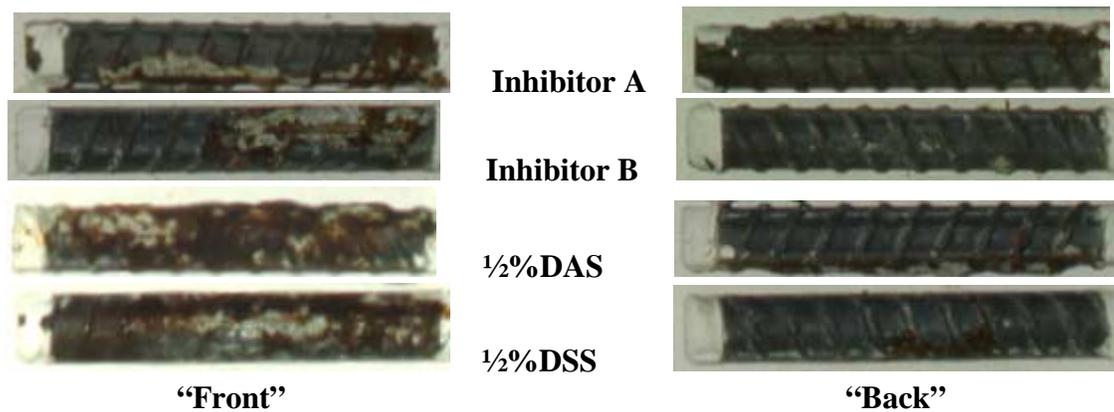
**Figure 5.32  No. 4 rebar from the air-dried pre-cracked specimens.**

| Control | Inhibitor A | Inhibitor B | ½%DAS | ¼%DAS | 1/8%DAS | ½%DSS | ¼%DSS | 1/8%DSS |

**Figure 5.33 Final exterior appearance of the oven-dried pre-cracked specimens. Only the "front" side is shown because this is where the corrosion was induced.**

| Inhibitor A | Inhibitor B | ½%DAS | ½%DSS |

**Figure 5.34 Final exterior appearance of the air-dried pre-cracked specimens. Only the "front" side is shown because this is where the corrosion was induced (the camera tripod leg hides the lower-right corner of the Inhibitors A and B specimens).**

Control 2) for the slabs were actively corroding.  This was witnessed visually on the
exposed rebar seen in Figure 5.29.

**Table 5.6  Visual inspection results for the lollipop specimens (not "saw-cut" nor
pre-cracked).**

| Specimen | Total Corrosion Area (%) | Heavy Corr. Area (%) | Total Corrosion Area (%) | Heavy Corr. Area (%) |
|---|---|---|---|---|
| Control 2-#1 | --- | --- | 54 | 12 |
| Inhibitor A-#1 | --- | --- | 49 | 19 |
| Inhibitor B-#2 | --- | --- | 57 | 57 |
| 2%DAS-#1 | 0 | 0 | --- | --- |
| 2%DAS-#2 | 0 | 0 | 0 | 0 |
| 2%DAS-#3 | --- | --- | 0 | 0 |
| 1%DAS-#1 | 0 | 0 | --- | --- |
| 1%DAS-#2 | 0 | 0 | 0 | 0 |
| 1%DAS-#3 | --- | --- | 0 | 0 |
| ½%DAS-#1 | 6 | 0 | --- | --- |
| ½%DAS-#2 | 0 | 0 | 0 | 0 |
| ½%DAS-#3 | --- | --- | 0 | 0 |
| 2%DSS-#1 | 0 | 0 | 0 | 0 |
| 2%DSS-#2 | 0 | 0 | 0.1 | 0 |
| 1%DSS-#1 | --- | --- | 0 | 0 |
| 1%DSS-#2 | 0 | 0 | 0 | 0 |
| 1%DSS-#3 | 0 | 0 | --- | --- |

**Table 5.7  Visual inspection results for the slab specimens.**

| Specimen | Total Corrosion Area (%) | Heavy Corrosion Area (%) |
|---|---|---|
| Control 1-#1 | 54 | 54 |
| Control 1-#2 | 55 | 55 |
| Control 2-#3 conn | 58 | 58 |
| Control 2-#4 unconn | 33 | 33 |
| Inhibitor A-#2 | 3.8 | 3.8 |
| Inhibitor A-#3 | 2.5 | 2.5 |
| Inhibitor B-#2 | 0.3 | 0 |
| Inhibitor B-#3 | 0 | 0 |
| 2%DAS-#1 | 0 | 0 |
| 2%DAS-#2 | 0.1 | 0 |
| 2%DAS-#3 | 0.2 | 0 |
| 1%DAS-#1 | 0.2 | 0 |
| 1%DAS-#2 | 0.2 | 0 |
| 1%DAS-#3 | 0.2 | 0 |
| ½%DAS-#2 | 0.3 | 0 |
| ½%DAS-#3 | 0.6 | 0 |
| 2%DSS-#1 | 0.6 | 0 |
| 2%DSS-#2 | 1.5 | 0 |
| 2%DSS-#3 | 0.5 | 0 |
| 1%DSS-#1 | 2.0 | 0 |
| 1%DSS-#2 | 0.2 | 0 |
| 1%DSS-#3 | 0.2 | 0 |

**Table 5.8  Visual inspection results for the "saw cut" lollipop specimens.**

| Specimen | Total Corrosion Area (%) | Heavy Corrosion Area (%) |
|---|---|---|
| 2%DAS-#2 | 3.0 | 2.7 |
| 1%DAS-#3 | 1.9 | 1.6 |
| 2%DSS-#3 | 0.3 | 0.2 |
| 1%DSS-#3 | 0.2 | 0.0 |

68

**Table 5.9  Visual inspection results for the pre-cracked lollipop specimens.**

| Specimen | Total Corrosion Area (%) | Heavy Corrosion Area (%) |
|---|---|---|
| ½%DAS-#1 | 44 | 44 |
| ½%DAS-#2 | 38 | 38 |
| ½%DAS-#5 (air dry) | 43 | 43 |
| ¼%DAS-#1 | 53 | 53 |
| ¼%DAS-#2 | 57 | 57 |
| 1/8%DAS-#1 | 72 | 72 |
| 1/8%DAS-#2 | 82 | 82 |
| ½%DSS-#1 | 58 | 58 |
| ½%DSS-#2 | 58 | 58 |
| ½%DSS-#5 (air dry) | 41 | 41 |
| ¼%DSS-#1 | 80 | 80 |
| ¼%DSS-#2 | 56 | 56 |
| 1/8%DSS-#1 | 62 | 62 |
| 1/8%DSS-#2 | 93 | 93 |
| Inhibitor A-#1 | 74 | 74 |
| Inhibitor A-#2 | 39 | 39 |
| Inhibitor A-#5 (air dry) | 19 | 19 |
| Inhibitor B-#1 | 57 | 57 |
| Inhibitor B-#2 | 68 | 68 |
| Inhibitor B-#5 (air dry) | 16 | 16 |
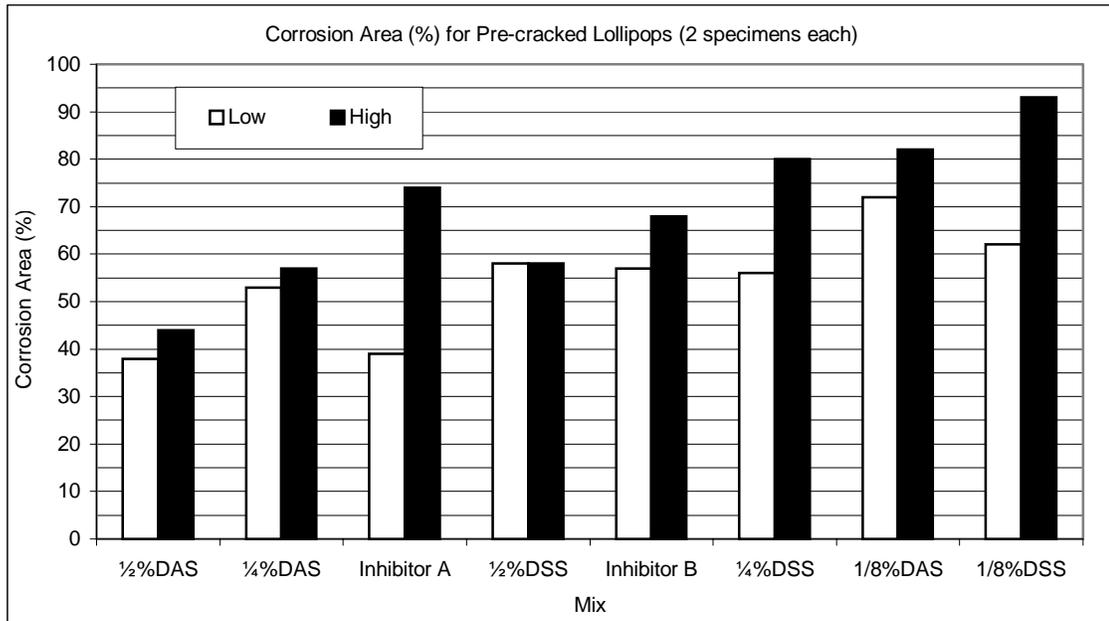


**Figure 5.35  Comparison of corroded area for the pre-cracked lollipop specimens.**

### 5.6.3   Phase II Specimens

These various specimens did not have results as strikingly different as the Phase I specimens did.  Lower concentrations of the DAS and DSS chemicals, a shorter testing period, and a limited amount of concrete cover in the cracked region were used for this phase.  The sides of the rebar labeled as "front" are the sides that were exposed to the preformed crack.  One purpose of the pre-cracked specimens was to see if the DAS and DSS chemicals could prevent the corrosion from spreading around the rebar.  The only significant changes on the surface of the concrete were near the crack openings.  On all exteriors of the specimens some color change due to corrosion was seen (Figures 5.33 and 5.34).  There appeared to be little correlation between what was happening on the surface and the linear polarization data; however, the rebar had more visible results.  The two sides of the ½%DAS and DSS chemicals looked like completely different specimens (Figures 5.31 and 5.32).  There are also some small patches on the "front" sides that were not affected by the corrosion.  Over the 37 week cycling period the chemicals at this concentration protected at least one-half of the bar from the spread of corrosion.  The ¼%DAS and DSS had similar success, both effectively preventing the corrosion from spreading to the "back" side of the bars.  A concentration as low as 1/8% proved ineffective for both chemicals.  At this concentration for both chemicals the two sides were equally corroded, the 1/8%DSS showing a few untouched areas.  Upon visual inspection, the air-dried ½% specimens fared a little better than the oven-dried ones.  The "front" side of the DSS did not have as much corrosion, but there was still a significant amount.  This was also evident in the linear polarization results.

The results for the control, Inhibitor A, and Inhibitor B (Figures 5.31 and 5.32) were similar to those of the Phase I specimens relative to the DAS and DSS specimens.  The "fronts" of Inhibitors A and B were completely covered in corrosion product. The "backs" were not completely clear of corrosion, but they were mostly unaffected.  This would place their effectiveness between that of the ¼% and 1/8% concentrations of DAS and DSS.  This reflected the Phase I outcome as Inhibitors A and B did not perform nearly as well as any of the DAS or DSS specimens with concentrations as low as ½%.  However, air-drying affected the results for Inhibitors A and B significantly.  There was corrosion along the path of the crack, but it did not spread much further than this for either chemical.  The "back" sides were completely clear of corrosion.  The biggest surprise was the surface of the control rebar.  There was not a large area of corroded surface on any of these specimens.  The linear polarization data indicated otherwise.  A possible explanation for this was that the corrosion was more intense, or that the corrosion penetrated deeper towards the core of the rebar.

### 5.6.4   "Saw Cut" Lollipop Specimens

In Phase I, some specimens had a "crack" cut into the surface using a masonry saw.  These specimens were not designed for electrochemical testing, but they followed the same cycling as the other specimens.  For further details on the production of these specimens, please see Reference [48].  These specimens were only inspected visually due to the lack of electrochemical testing.  The difference between these specimens and the

pre-cracked specimens was that these specimens demonstrated the effects when a crack forms during the life of the concrete whereas the pre-cracked specimens produced the effect of a crack during the casting. These "saw cut" specimens had a concrete cover of approximately one-quarter inch or less. The removed bars (Figure 5.30) showed impressive results. After about 18 months of cycling, the bars had only tiny spots of corrosion, which generally formed where there was an air bubble in the concrete, thus reducing the cover to even smaller amounts. There was no spreading of corrosion from these spots as the chemicals apparently contained the corroded area.

## 5.7  CORROSION SUMMARY

The "uncracked" specimens with the DAS and DSS chemicals prevented the chlorides from penetrating the concrete and reaching the rebar in both the lollipop (2- and 3-inch diameter) and slab specimens. Powdered samples of the concrete extracted from the specimens showed that no chlorides had permeated through the concrete over approximately 2 years of harsh, weekly wet/dry cycling. The "saw cut" cylinders, with approximately one-and-a-half years of cycling had no corrosion except for minor areas at air bubbles. In the places where chlorides did penetrate and corrosion began (in some "saw cut" specimens and the dropped 1%DSS specimen), the chemicals prevented any expansion of the corrosion. The chemicals also increased the solution resistance of the concrete considerably.

In Phase I, all of the concentrations of DAS and DSS performed extremely well with regards to corrosion. The 2% concentrations produced excessive strength reduction. Therefore, based on these Phase I results, the ½%DAS or the 1%DSS mix would be selected due to corrosion resistance, strength, and economy. Both protected the rebar as well as the higher concentrations. Lowering the concentration for the DAS specimens is important due to the strong ammonium odor that developed during the mixing and hydration processes.

After including the Phase II results, the DAS and DSS chemical concentrations of ¼% appeared comparable to those of the ½% concentrations. The ½% or ¼% concentrations would then be selected for the same reasons as mentioned above.

Inhibitors A and B delayed the onset of corrosion in the lollipop specimens, but the corrosion still initiated. In both the slab and pre-cracked specimens, the air-drying aided the inhibitors greatly in their corrosion prevention. After nearly two years of cycling the slabs had no corrosion initiation.

## 5.8  OVERALL PERFORMANCE SUMMARY:  DAS AND DSS CHEMICALS

Compared with Inhibitor A and Inhibitor B, the Phase I and Phase II tests indicated that the DSS and DSS chemicals
1.      provided significantly more protection in uncracked concrete (essentially no corrosion in any 2 or 3 in. lollipops or in any slabs after 100 weeks of testing),
2.      provided significantly more protection in cracked concrete (lower corrosion rates and lower corroded areas),
3.      provided significantly better protection against chloride penetration, and

4.        provided very good freezing-and-thawing protection without additional air-entraining admixture.

The DSS chemical was selected in the Phase I research as being the best overall. Phase II test results did not show one of the DSS or DAS chemicals significantly better than the other. Because of the ammonia odor produced when mixing concrete with DAS, the DSS chemical is recommended for use in concrete.

Determining the desired concentration of the chemical depends on how the concrete will be used. Higher concentrations of both DSS and DAS generally outperformed lower concentrations in most of the tests, except for compressive strength, where lower concentrations were better. Except for compressive strength, both DSS and DAS performed better than Inhibitors A and B in all Phase II tests. (It should be noted that Inhibitor B also produced lower compressive strengths, 7% and 15% lower in Phase II and I, respectively, than the Control.) A ½% to 1% concentration gave the best overall performance. In this range, there was a trade off between strength and corrosion protection. Phase I results also showed very good freezing-and-thawing protection with these concentrations.

Although strength can be an important requirement of concrete, the corrosion inhibiting characteristics of the DSS and DAS chemicals in concrete were far better than those of the commercial inhibitors. The sacrifice of some compressive strength is overshadowed by significantly improved corrosion protection provided by these two chemicals compared with the commercial inhibitors. Results from Phases I and II indicate 4,000 psi compressive strength can be achieved with a ½% concentration. For bridge decks, parapets, and pavements, 4,000 psi strength would be more than adequate. Therefore, the ½% concentration is recommended.

# CHAPTER 6.0
# CONCLUSIONS AND RECOMMENDATIONS

## CONCLUSIONS

Several conclusions were made after reviewing all data and results from both Phase I and Phase II of the research performed at the University of Connecticut.

1. Adding either of the new chemicals, DAS or DSS, to the concrete prevented corrosion from initiating in all of the Phase I corrosion monitored specimens.

2. Adding the DAS or DSS chemicals prevented or drastically reduced corrosion in the "saw cut" lollipop specimens (Section 5.6.4).

3. Adding the DAS or DSS chemicals hindered the spreading of corrosion from a preformed crack (Section 5.6.3).

4. Adding the DAS or DSS chemicals created a significant increase in the solution resistance of the concrete for the Phase I specimens (Section 5.4.3).

5. Adding the DAS or DSS chemicals prevented chlorides from penetrating the concrete to the surface of the rebar (Section 5.3).

6. Adding the DAS or DSS chemicals provided the concrete with freeze-thaw durability without the addition of any other admixture [48].

7. Adding the DAS or DSS chemicals lowered the compressive strength of the concrete. With a concentration of about ½ %, the compression strength was still more than adequate for structural use.

8. Adding the DAS or DSS chemicals accelerated the set time (Section 5.2.3).

9. The DAS chemical produced a strong ammonium odor during the casting of these specimens. There were no unusual odors present during the production of the DSS specimens.

10. Overall, the DAS and DSS chemicals performed similarly, but the DSS was judged to be slightly better.

11. Inhibitors A and B delayed, but did not suppress entirely, the initiation of corrosion in the lollipop specimens.

12. Air-drying increased the performance of Inhibitors A and B in the Phase I slab specimens and the Phase II pre-cracked specimens. Corrosion did not initiate in the slabs after two years of testing and the corrosion rate was minimal in the pre-cracked specimens, relative to the performance of the other specimens.

## RECOMMENDATIONS FOR FUTURE RESEARCH

1. Further laboratory work could evaluate the performance of the DAS and DSS chemicals (a) in lower concentrations (½% and ¼%) in whole cylinders, (b) in higher concentrations (2% and 1%) in pre-cracked cylinders, and (c) with bare rebar.

2. Based on the very favorable performance observed in these laboratory tests, it is recommended that the DSS chemical be added to concrete at concentrations of ½% to 1% for field trials with bridge decks, bridge parapets, or "Jersey" barriers.

# APPENDIX A
# SOLUTION RESISTANCE AND CORROSION RATE DATA

This appendix presents $R_s$ (solution resistance) and $1/R_p$ (corrosion rate) versus time data for all of the specimens. In these plots the "corrosion rate" is shown as "$1/R_p$" but it actually is the corrosion rate calculated using the electrode area and the B value (Section 5.5). Each week of time corresponds to one wet-dry cycle. The "$1/R_p$" values were corrected for the IR error. Each plot shows results for individual specimens for each group, for example, for the 2%DSS mix, specimen numbers 1, 2, and 3. Obvious erroneous data points, such as a "spike" along the path of the data or other "bad" data, were not included in the data reduction process. Where data ends, the specimens were removed from testing and cut open to be examined visually.

Solution resistances ($R_s$) versus time results are displayed as follows:

| | |
|---|---|
| 2-inch diameter lollipop specimens | Figures A.1 through A.8 |
| 3-inch diameter lollipop specimens | Figures A.9 through A.16 |
| Slab specimens | Figures A.17 through A.26 |
| Pre-cracked lollipop specimens | Figures A.27 through A.36 |

Corrosion rates ($1/R_p$) versus time results are presented as:

| | |
|---|---|
| 2-inch diameter lollipop specimens | Figures A.37 through A.44 |
| 3-inch diameter lollipop specimens | Figures A.45 through A.52 |
| Slab specimens | Figures A.53 through A.62 |
| Pre-cracked lollipop specimens | Figures A.63 through A.72 |
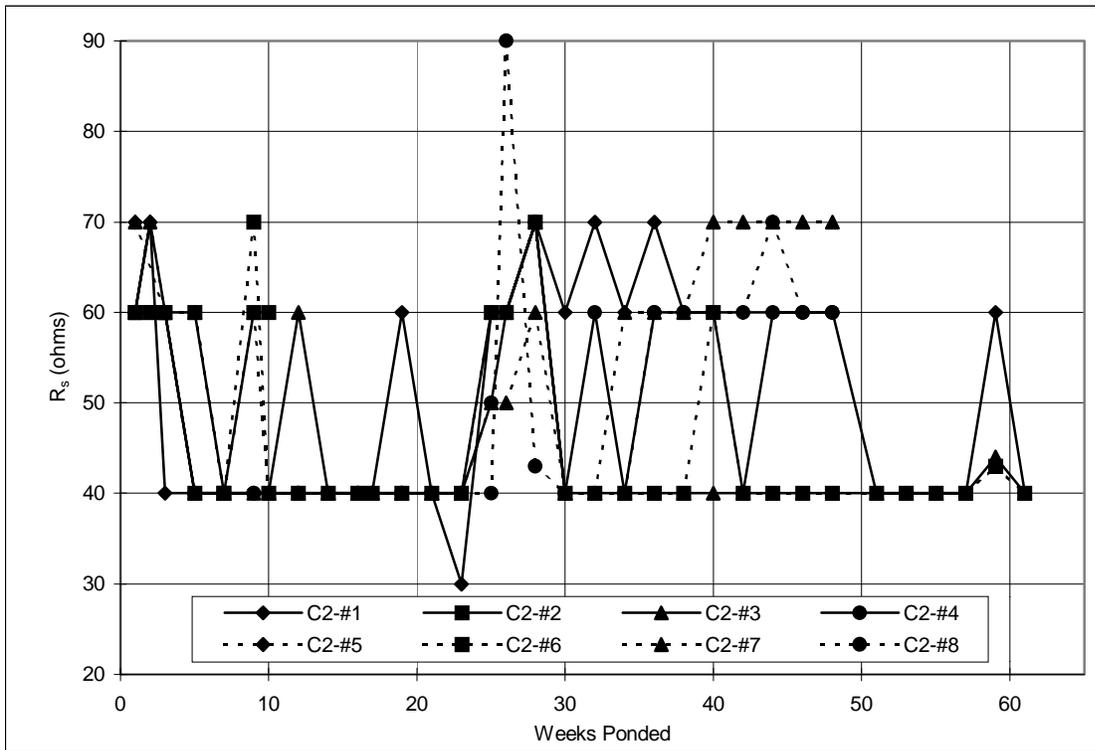
**Figure A.1  Solution resistance, 2-inch diameter lollipops:  Control.**
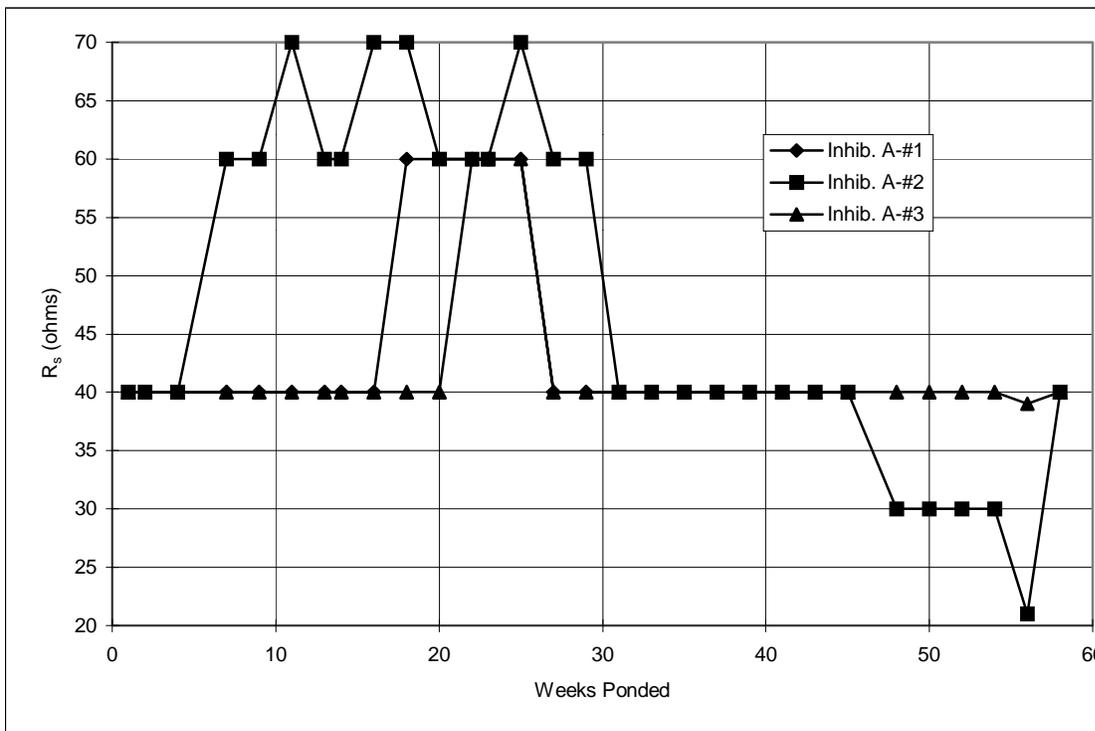


**Figure A.2  Solution resistance, 2-inch diameter lollipops:  Inhibitor A.**

76
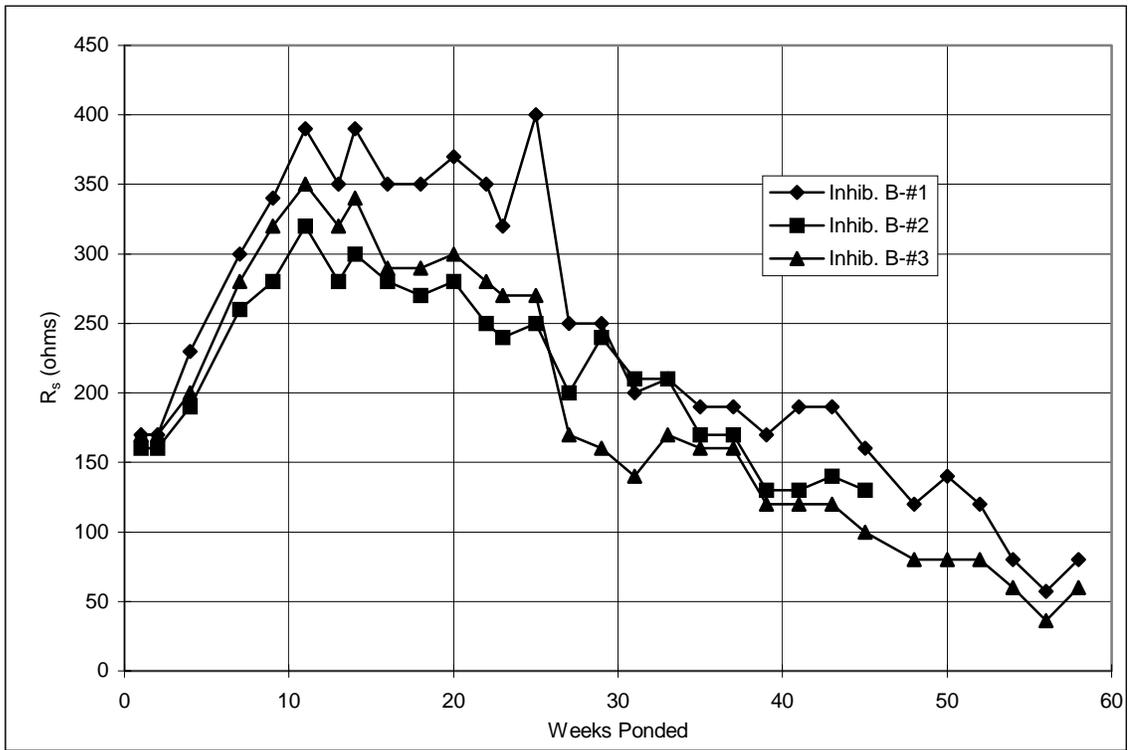
**Figure A.3  Solution resistance, 2-inch diameter lollipops:  Inhibitor B.**
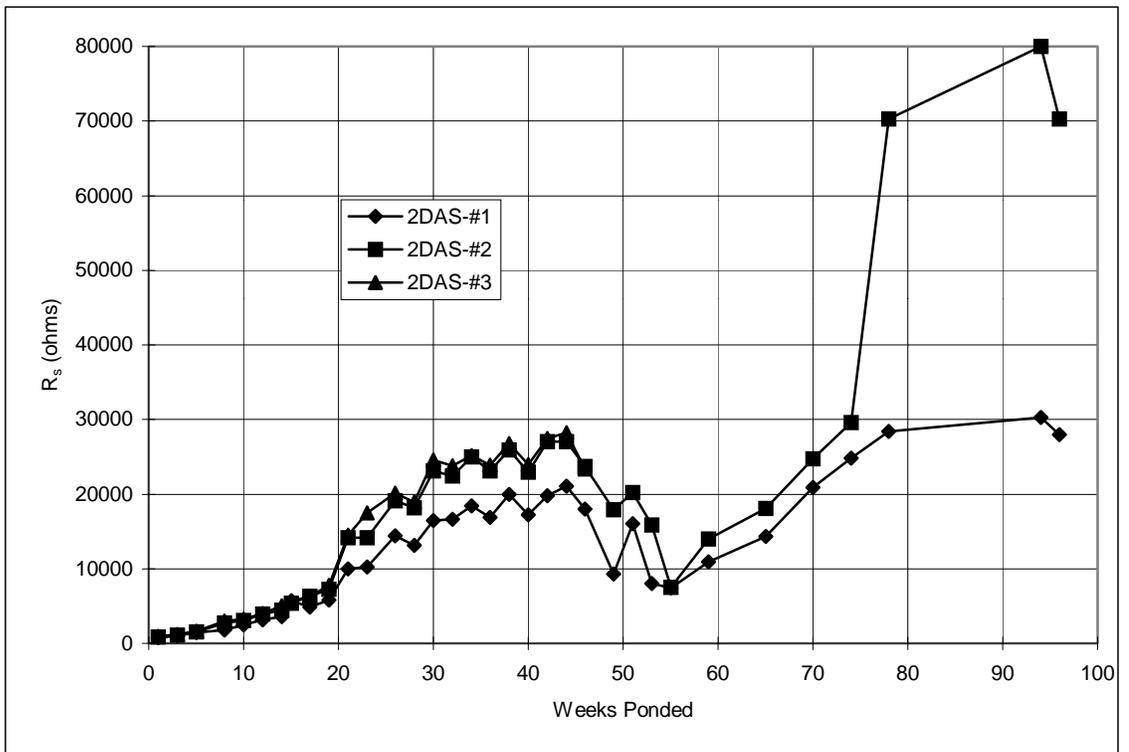


**Figure A.4  Solution resistance, 2-inch diameter lollipops:  2%DAS.**
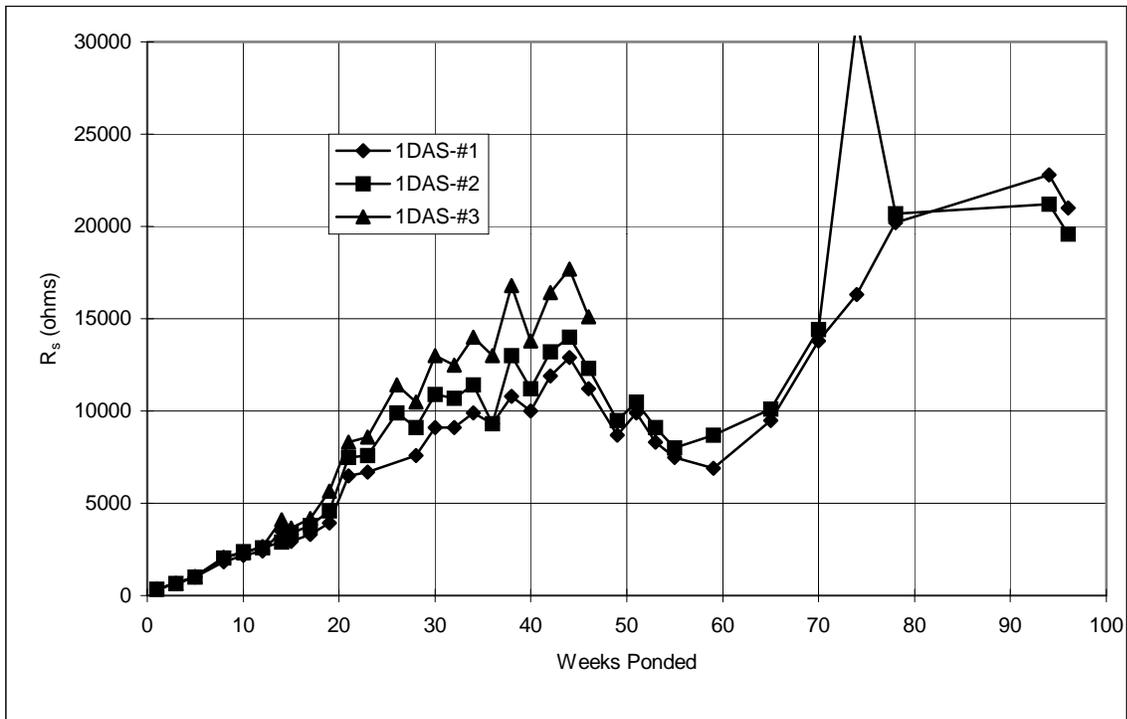
**Figure A.5  Solution resistance, 2-inch diameter lollipop:  1%DAS.**
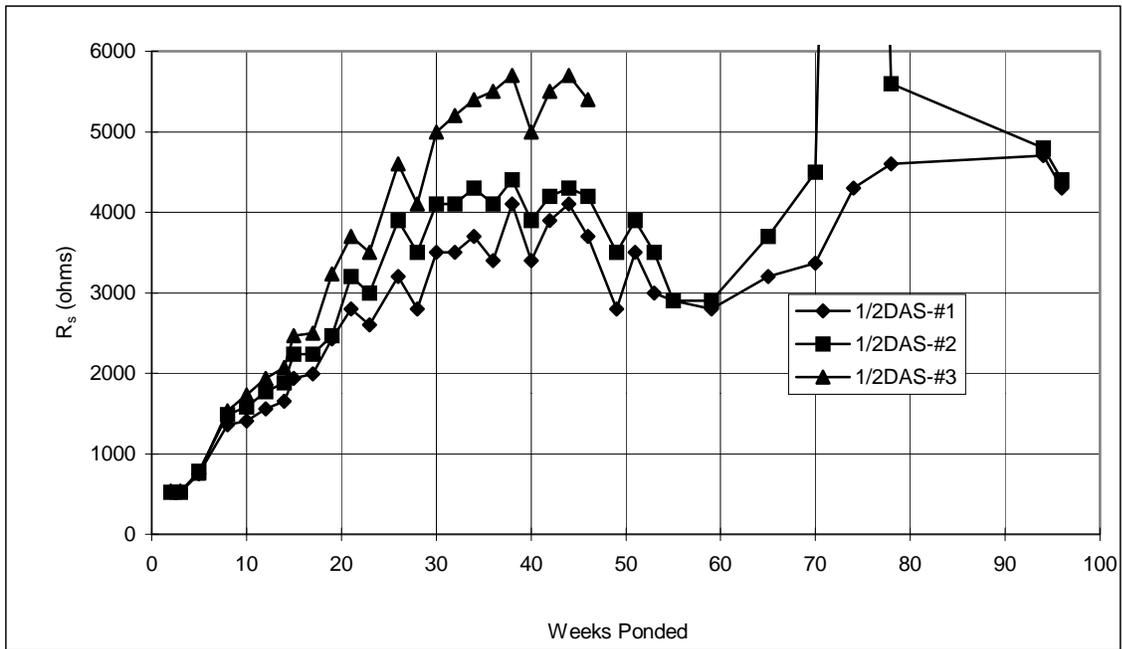


**Figure A.6  Solution resistance, 2-inch diameter lollipops:  ½%DAS.**
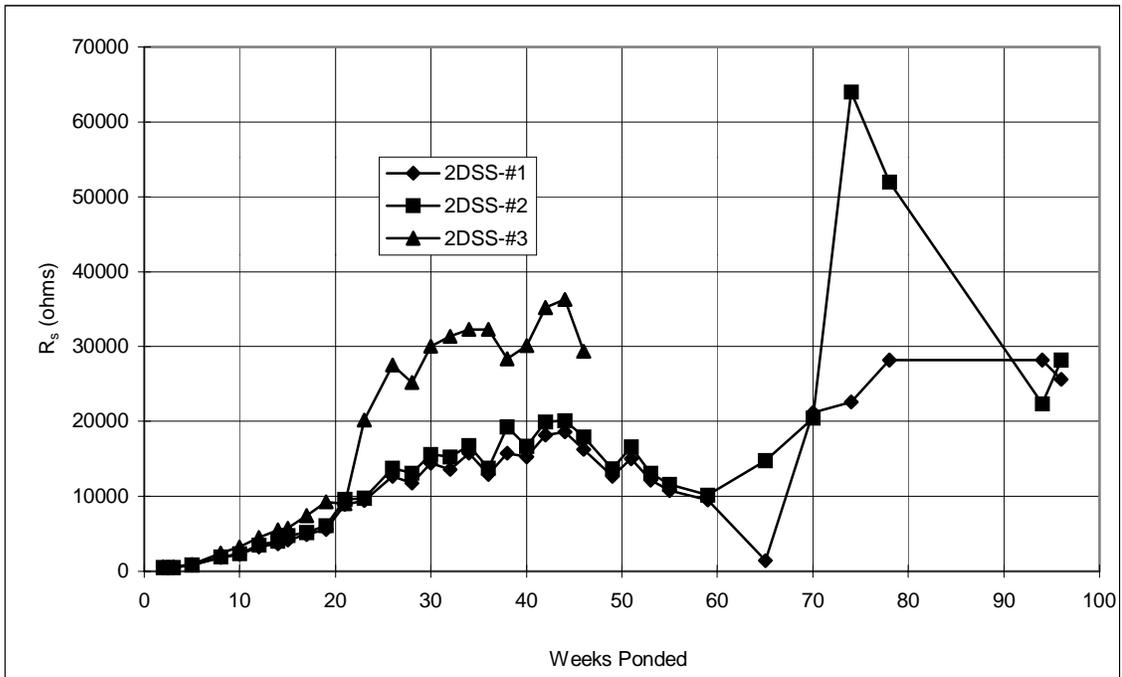
**Figure A.7  Solution resistance, 2-inch diameter lollipops:  2%DSS.**
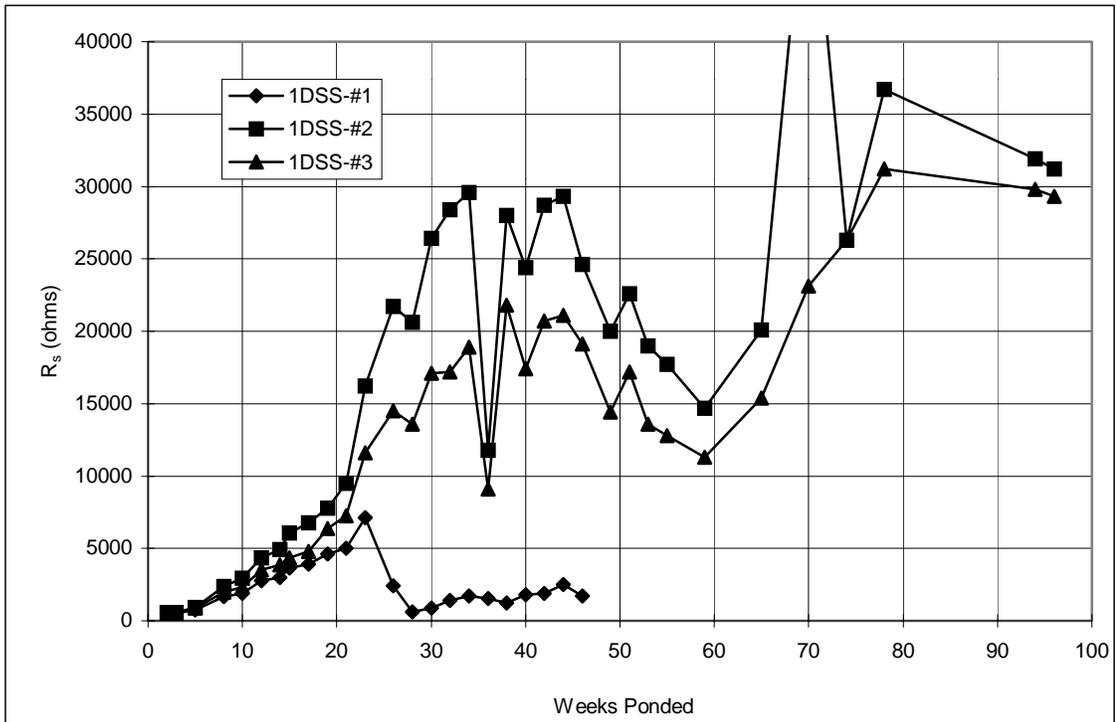


**Figure A.8  Solution resistance, 2-inch diameter lollipops:  1%DSS.**
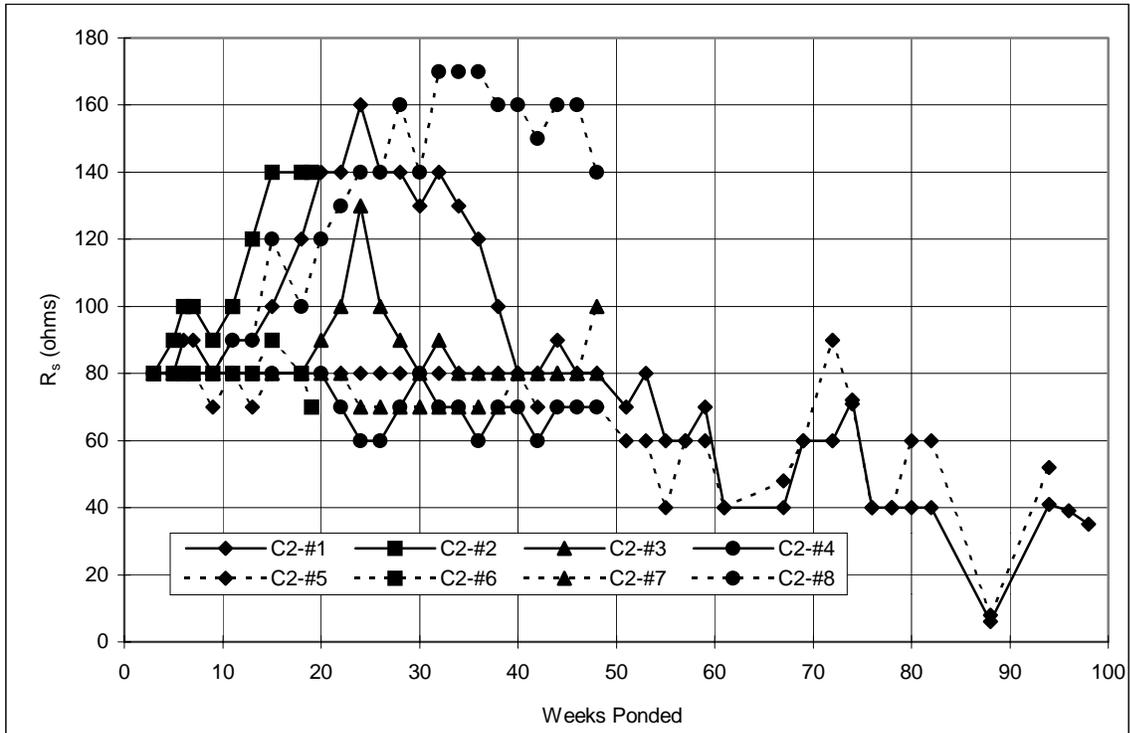
**Figure A.9  Solution resistance, 3-inch diameter lollipops:  Control.**
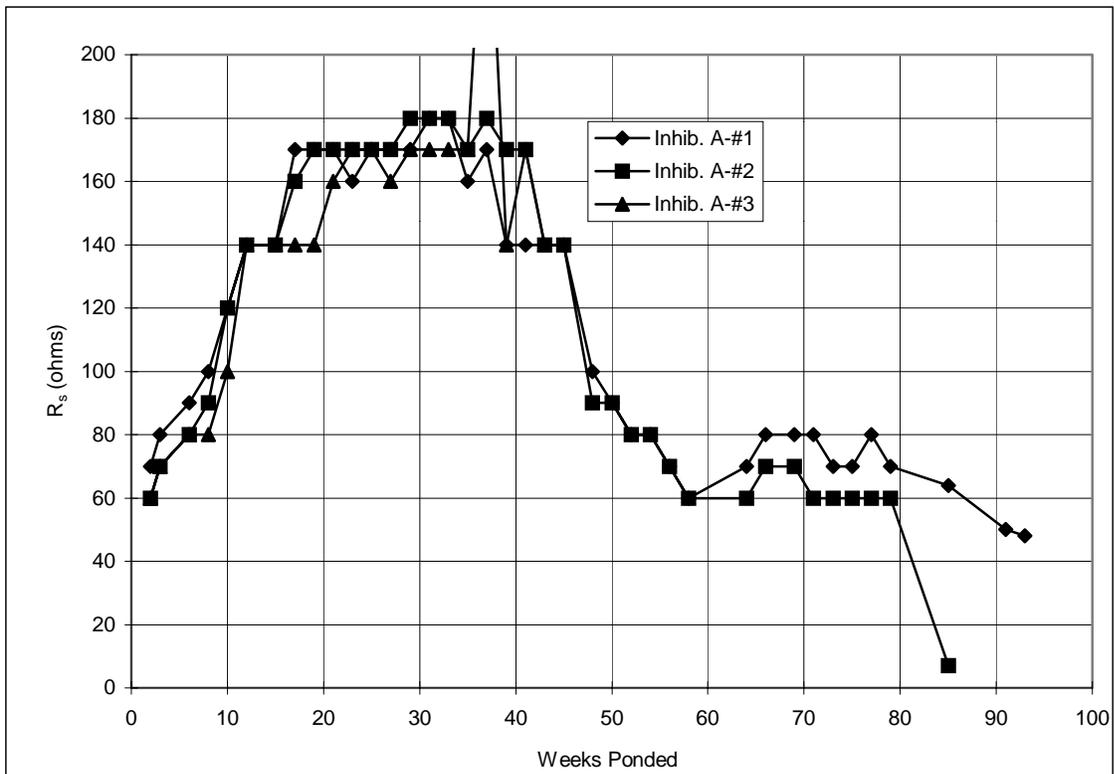


**Figure A.10  Solution resistance, 3-inch diameter lollipops:  Inhibitor A.**
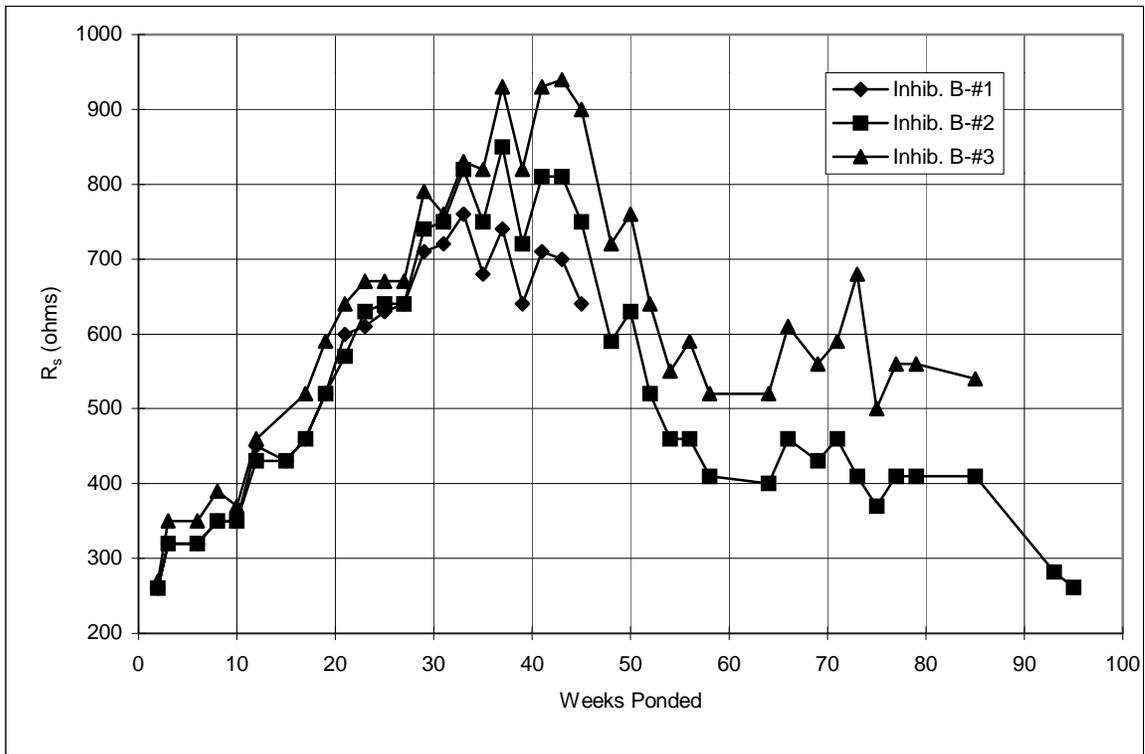
**Figure A.11  Solution resistance, 3-inch diameter lollipops:  Inhibitor B.**
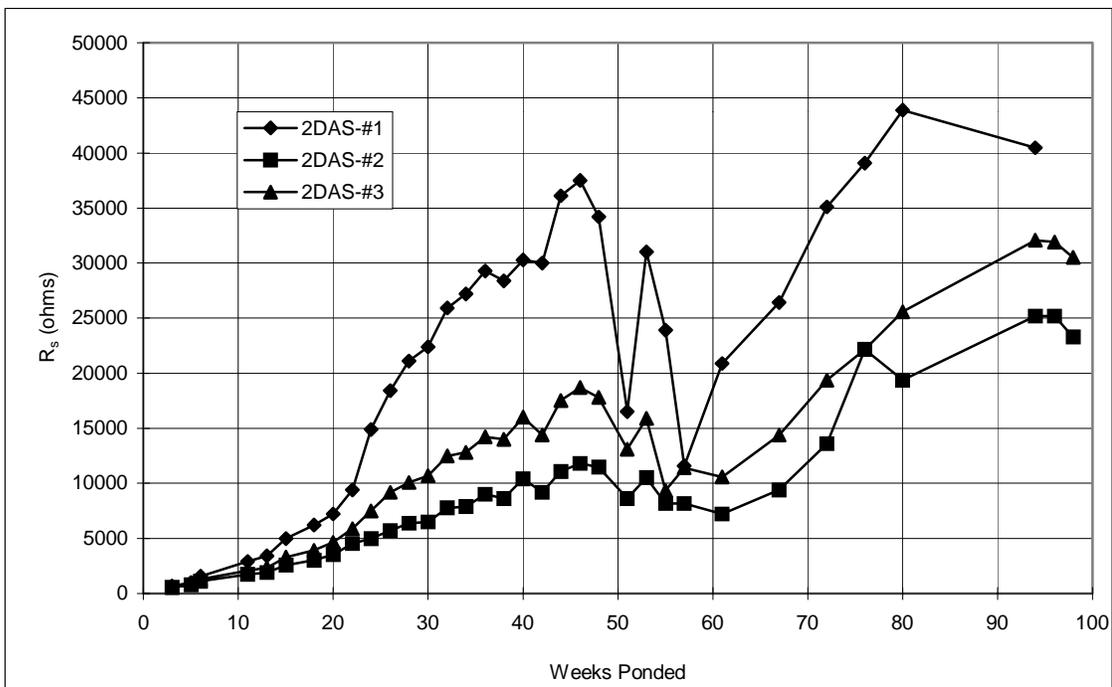


**Figure A.12  Solution resistance, 3-inch diameter lollipops:  2%DAS.**
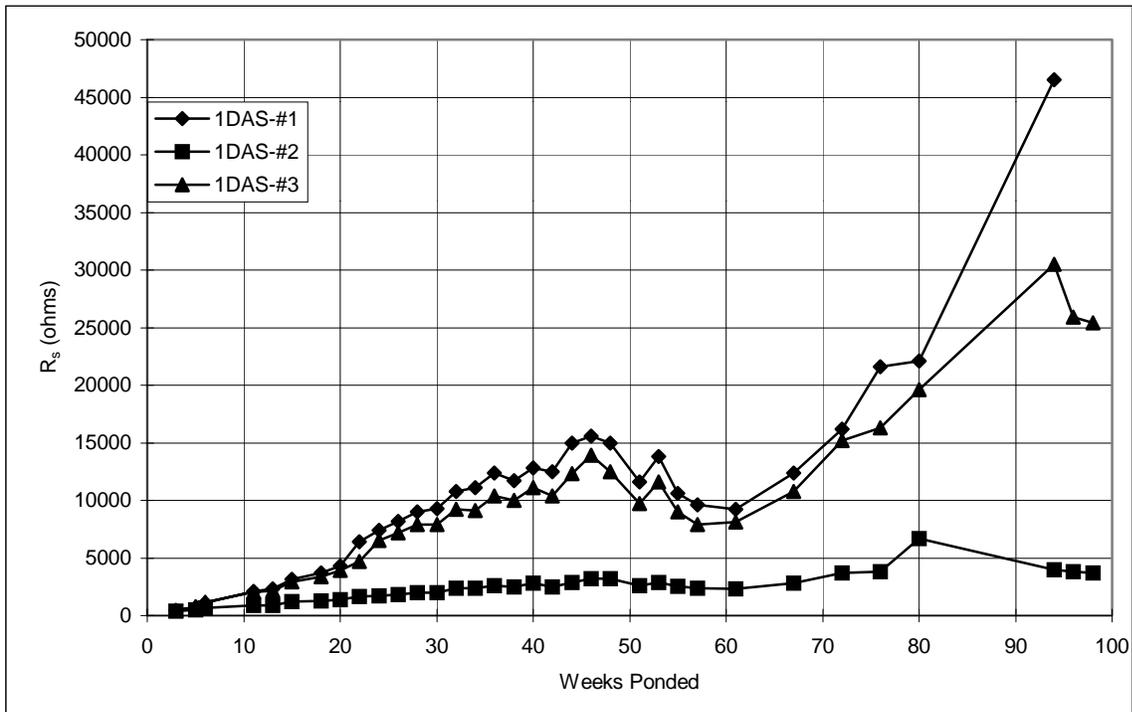
**Figure A.13  Solution resistance, 3-inch diameter lollipops:  1%DAS.**
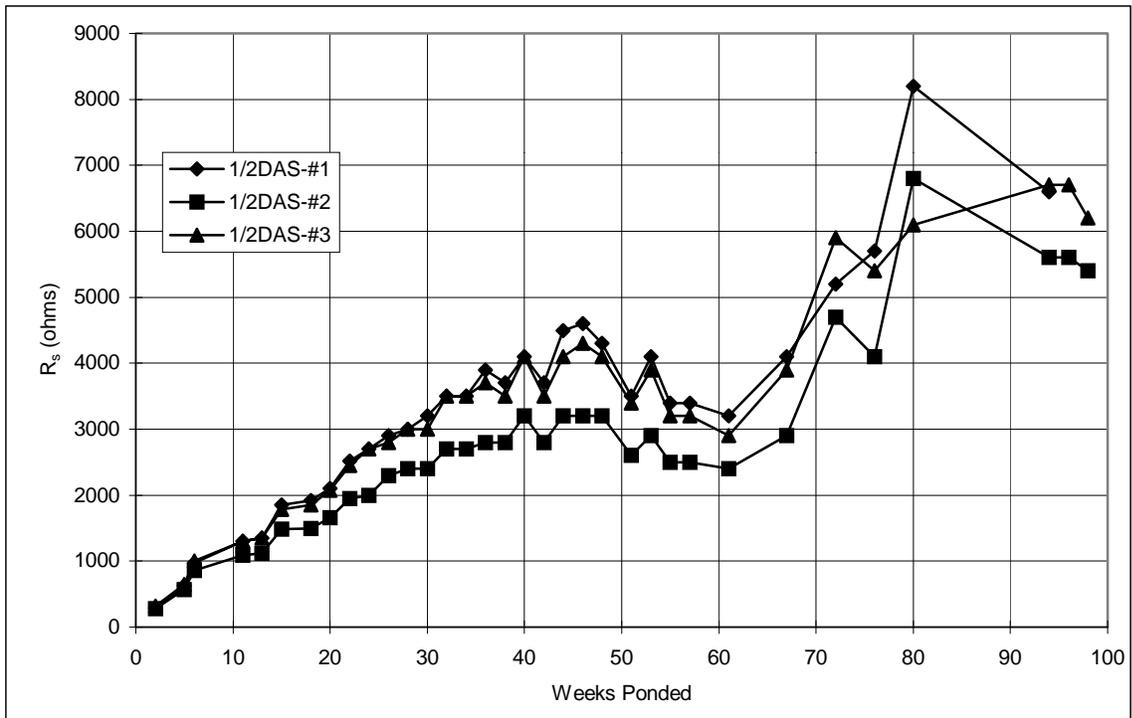


**Figure A.14  Solution resistance, 3-inch diameter lollipops:  ½%DAS.**
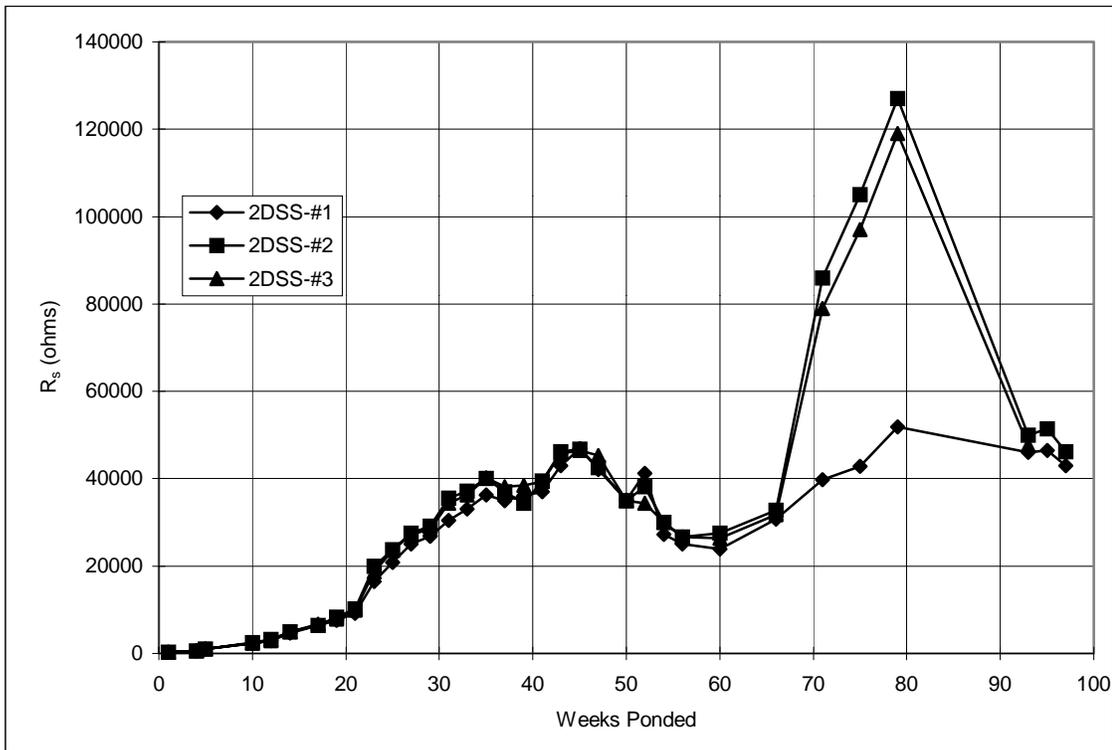
**Figure A.15  Solution resistance, 3-inch diameter lollipops:  2%DSS.**
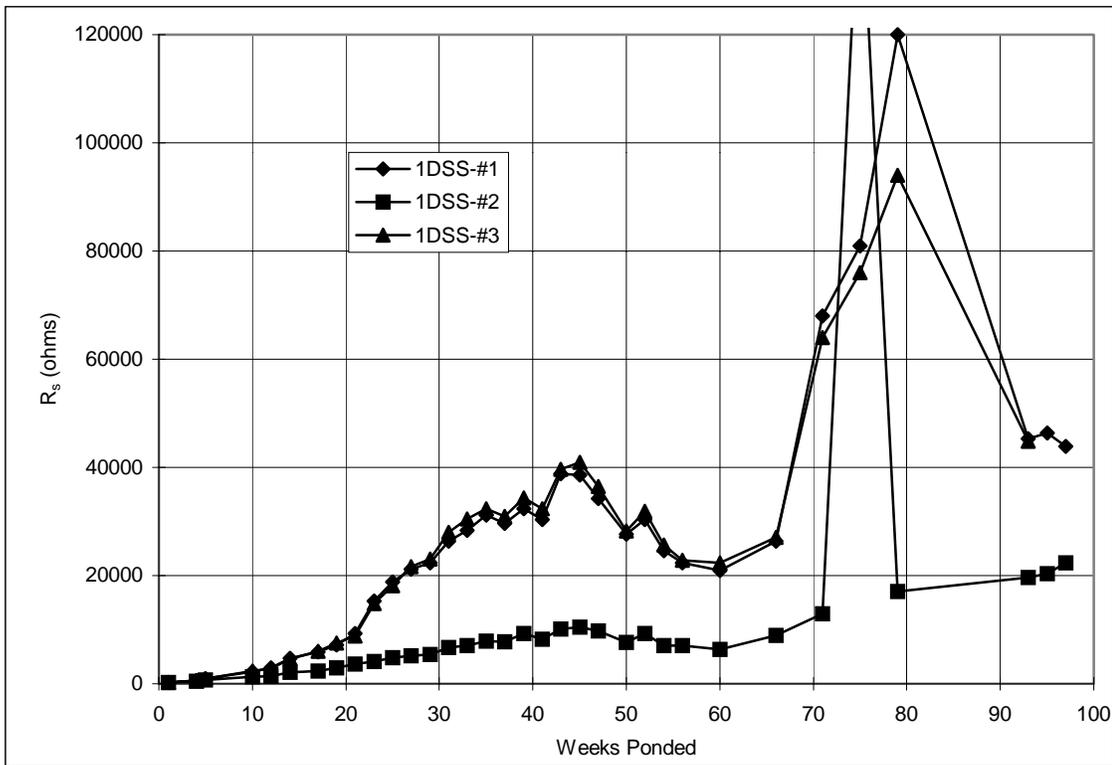


**Figure A.16  Solution resistance, 3-inch diameter lollipops:  1%DSS.**
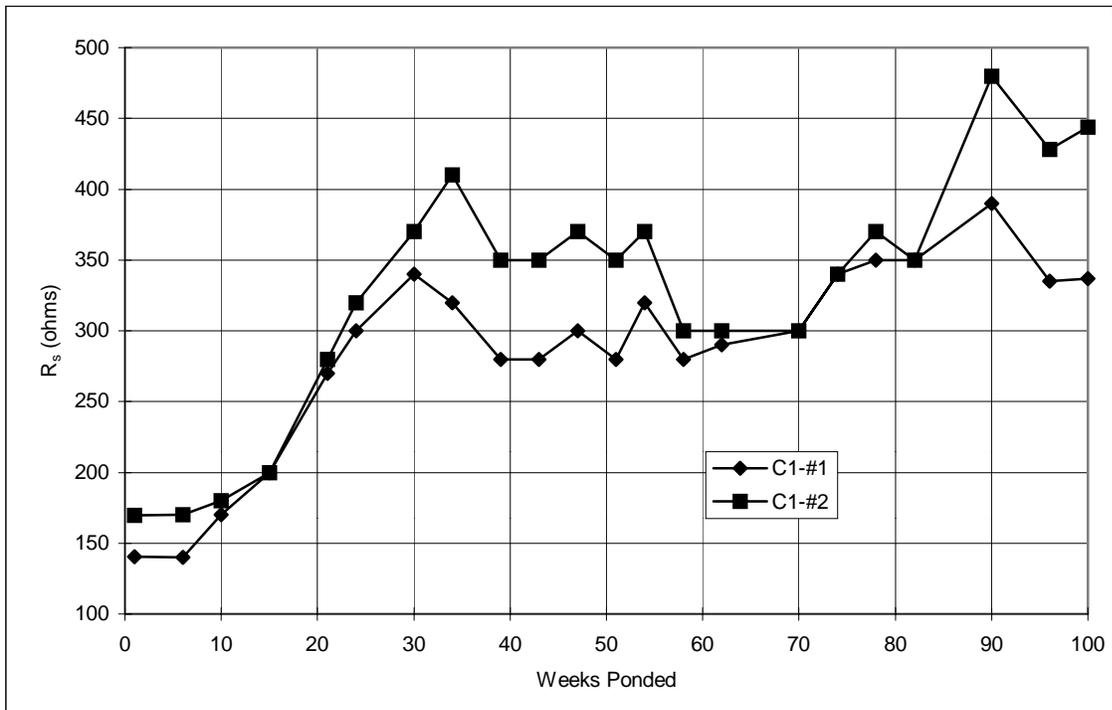
**Figure A.17  Solution resistance, slabs:  Control 1.**
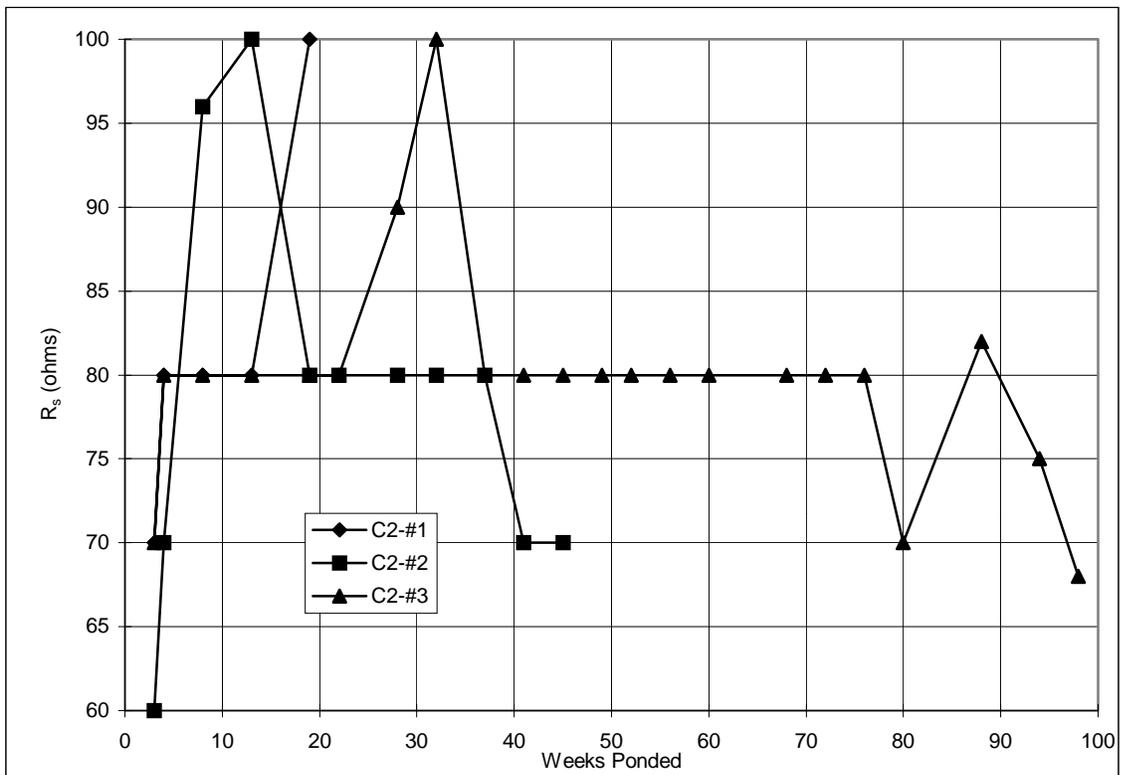


**Figure A.18  Solution resistance, slabs:  Control 2-connected.**
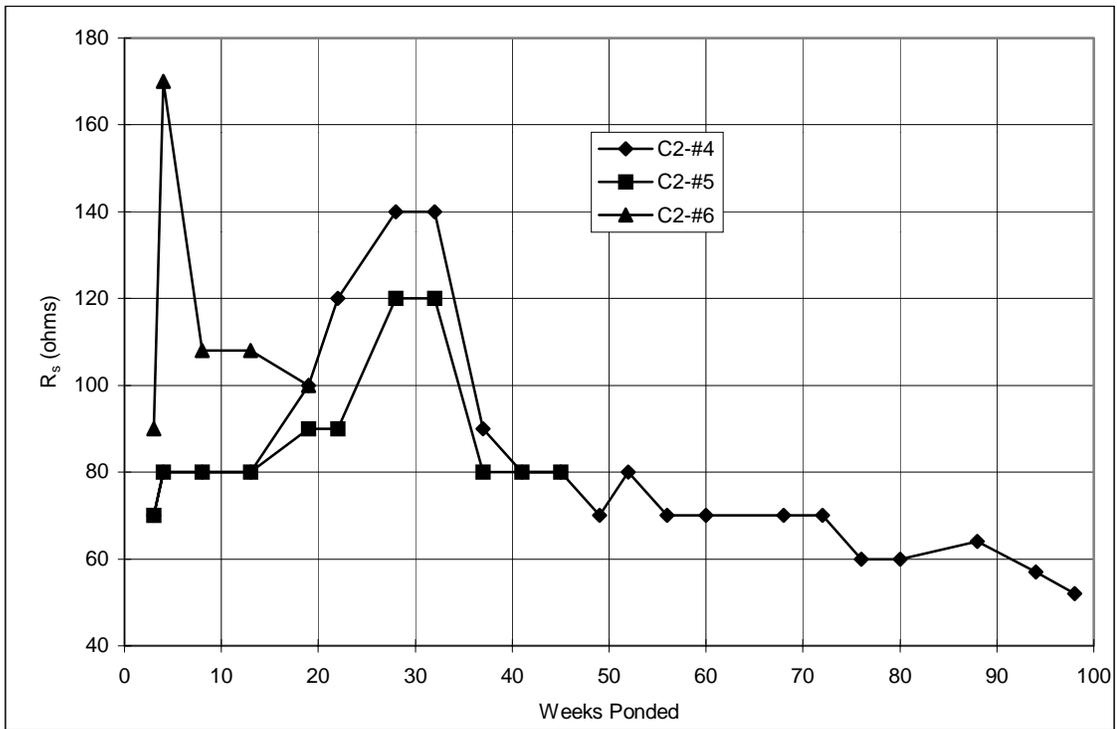
**Figure A.19  Solution resistance, slabs:  Control 2-unconnected.**
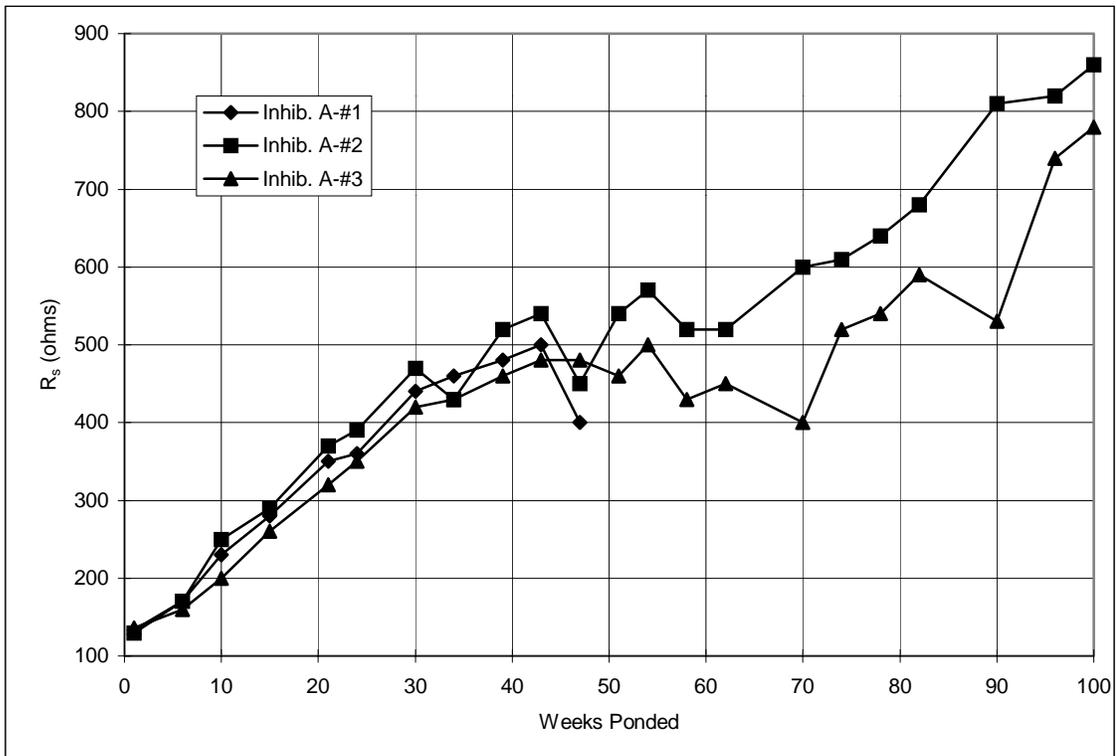


**Figure A.20  Solution resistance, slabs:  Inhibitor A.**
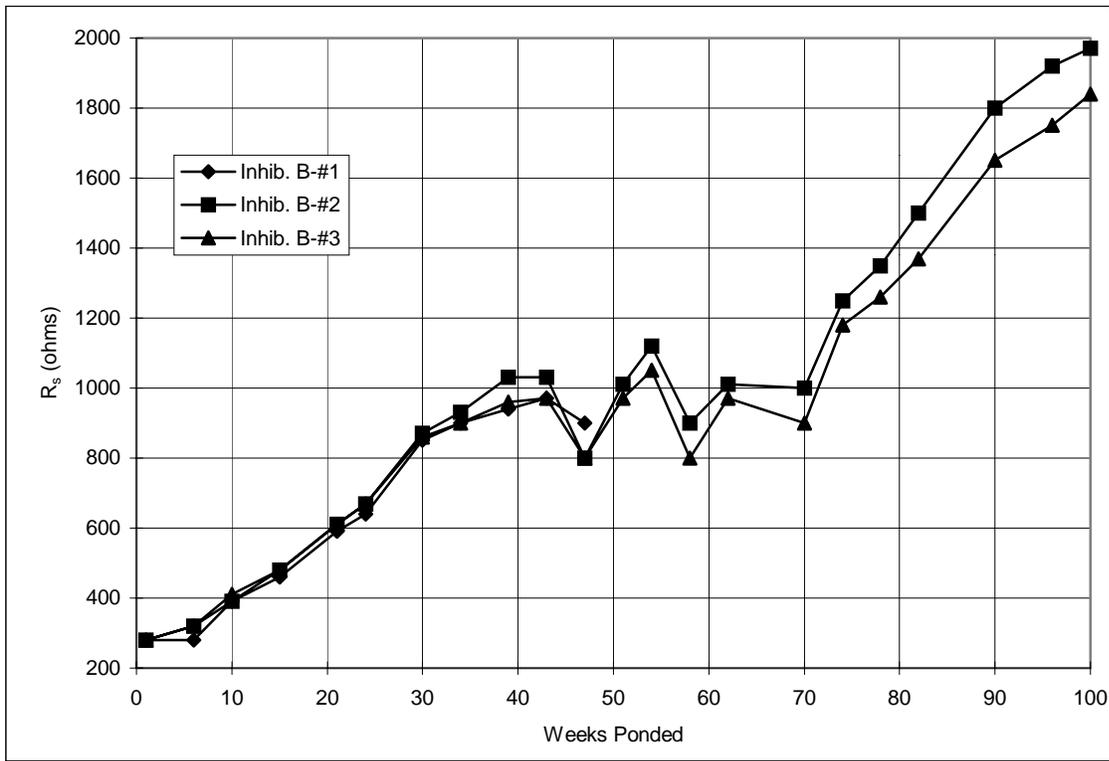
**Figure A.21  Solution resistance, slabs:  Inhibitor B.**
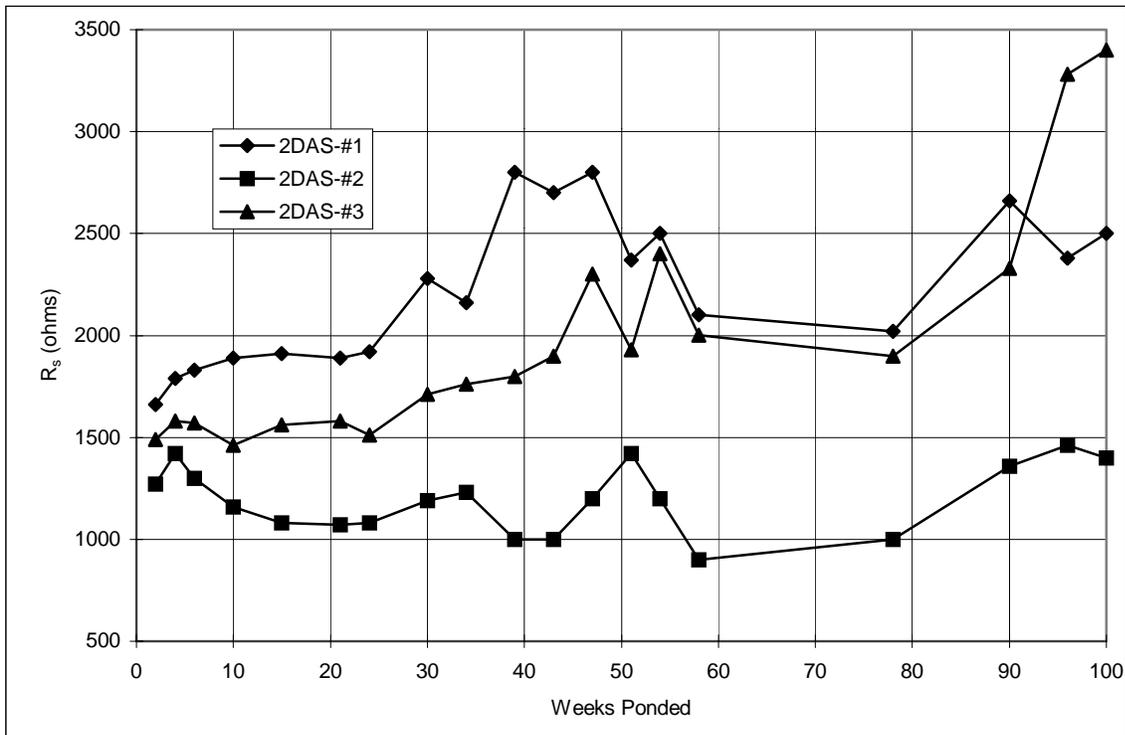


**Figure A.22  Solution resistance, slabs:  2%DAS.**
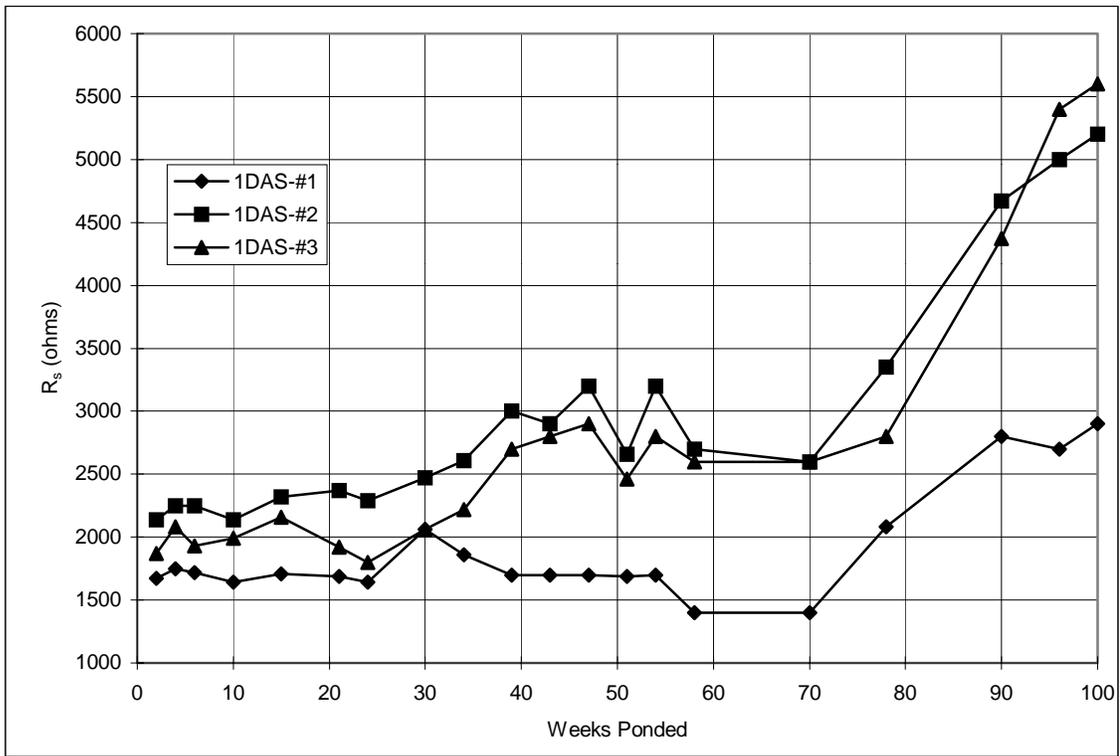
**Figure A.23  Solution resistance, slabs:  1%DAS.**
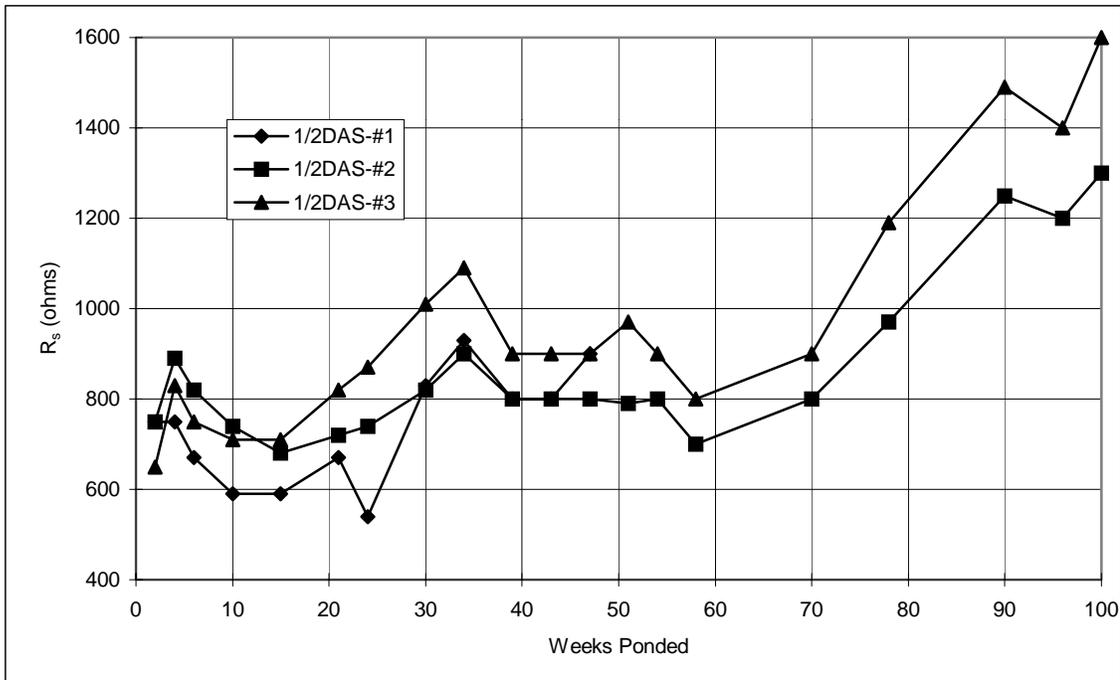


**Figure A.24  Solution resistance, slabs:  ½%DAS.**
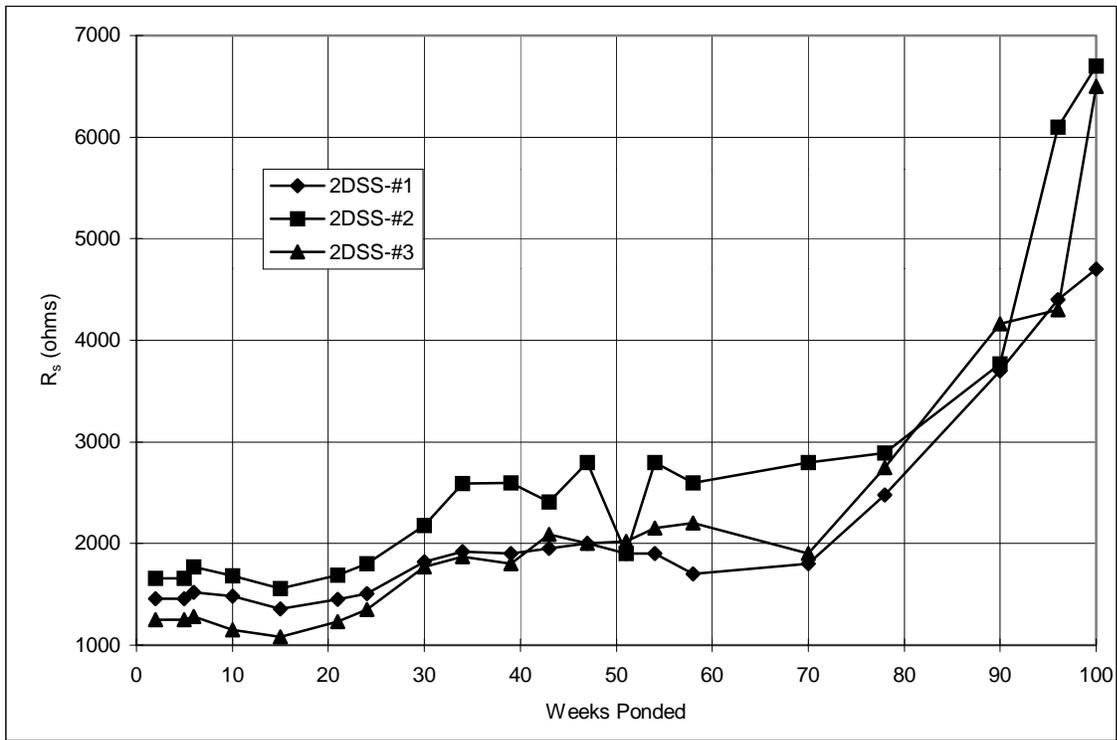
**Figure A.25  Solution resistance, slabs:  2%DSS.**
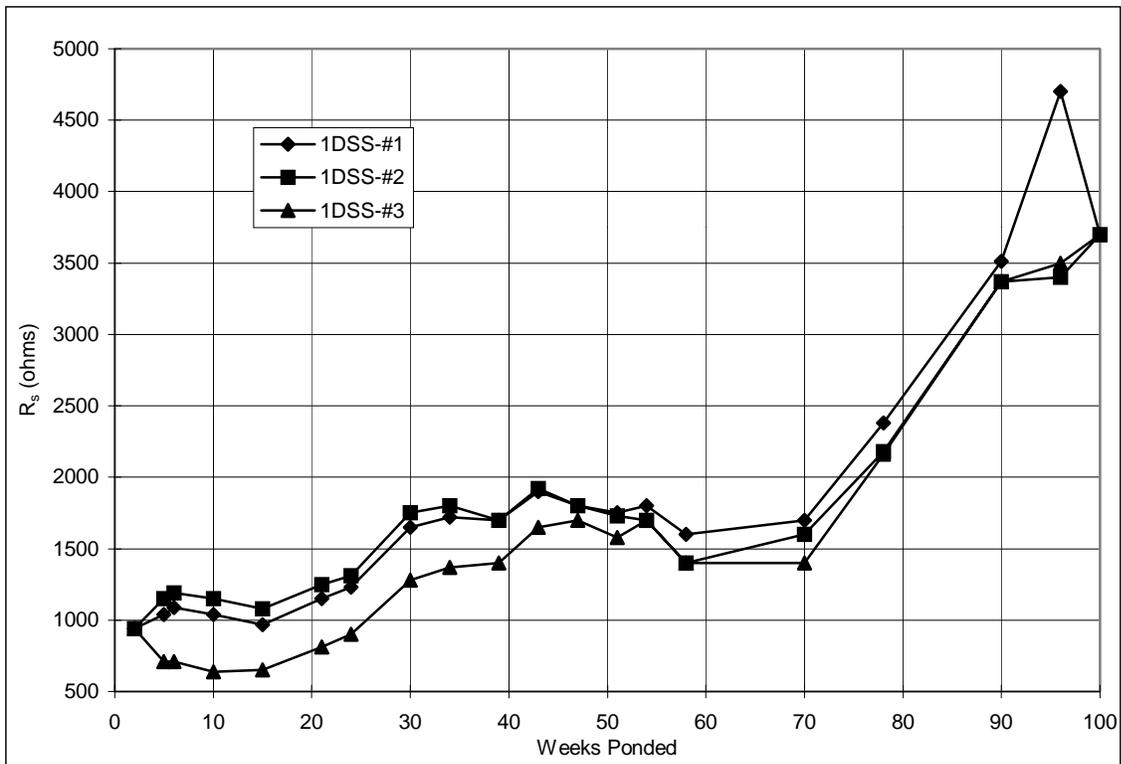


**Figure A.26  Solution resistance, slabs:  1%DSS.**
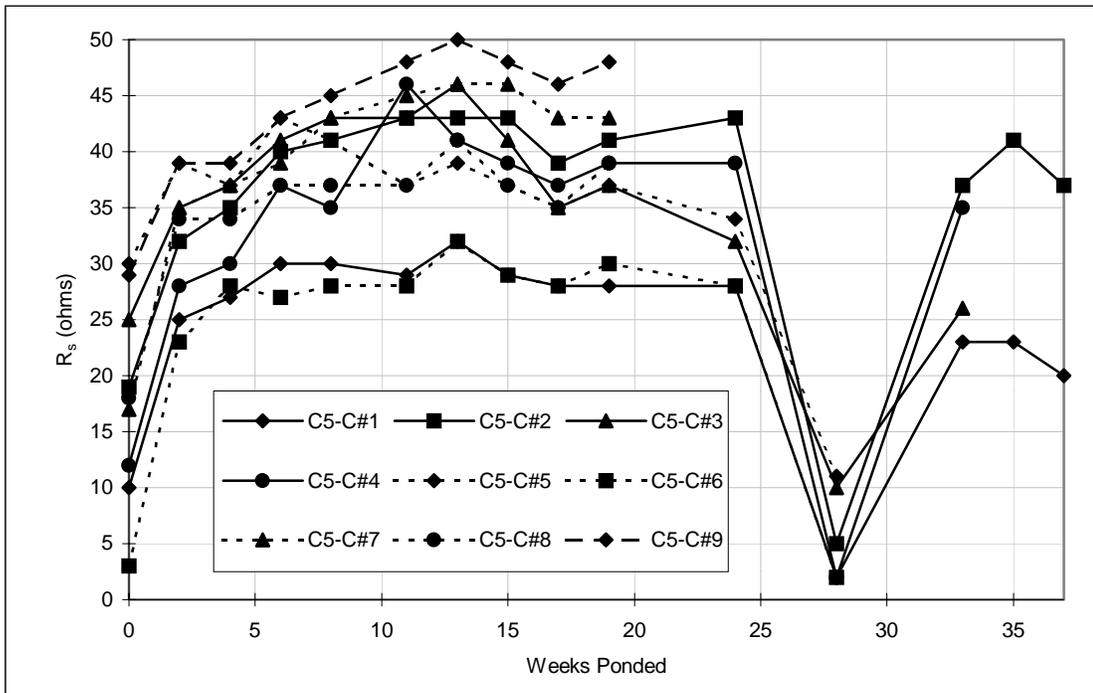
**Figure A.27  Solution resistance, pre-cracked lollipops:  Control, specimens 1-9.**
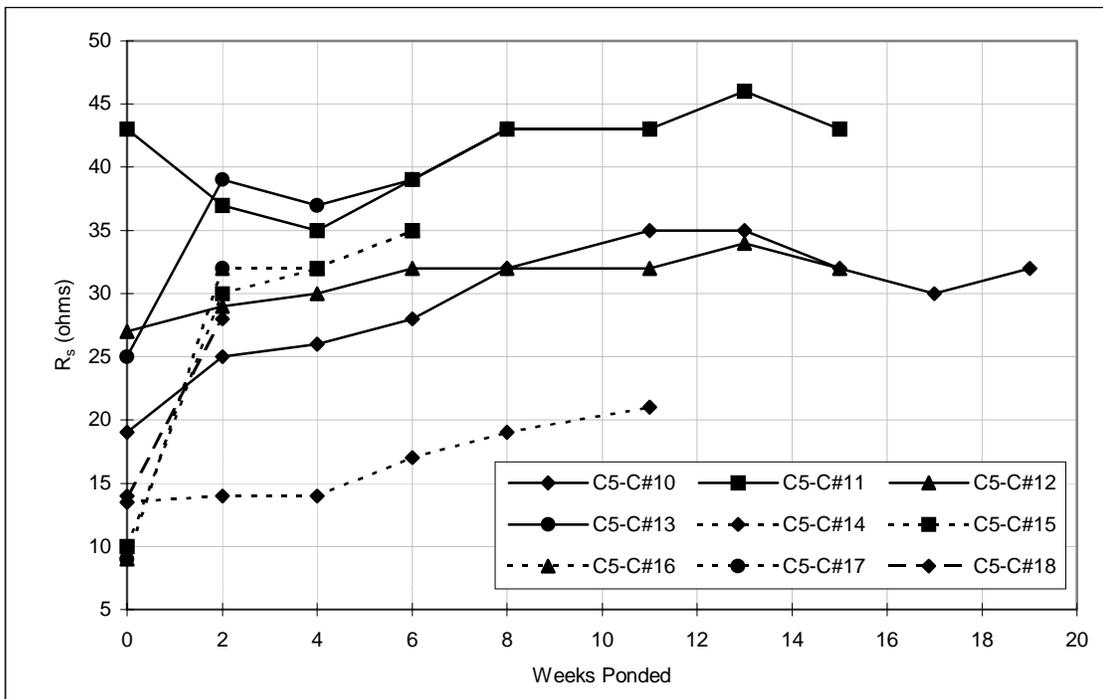


**Figure A.28  Solution resistance, pre-cracked lollipops:  Control, specimens 10-18.**
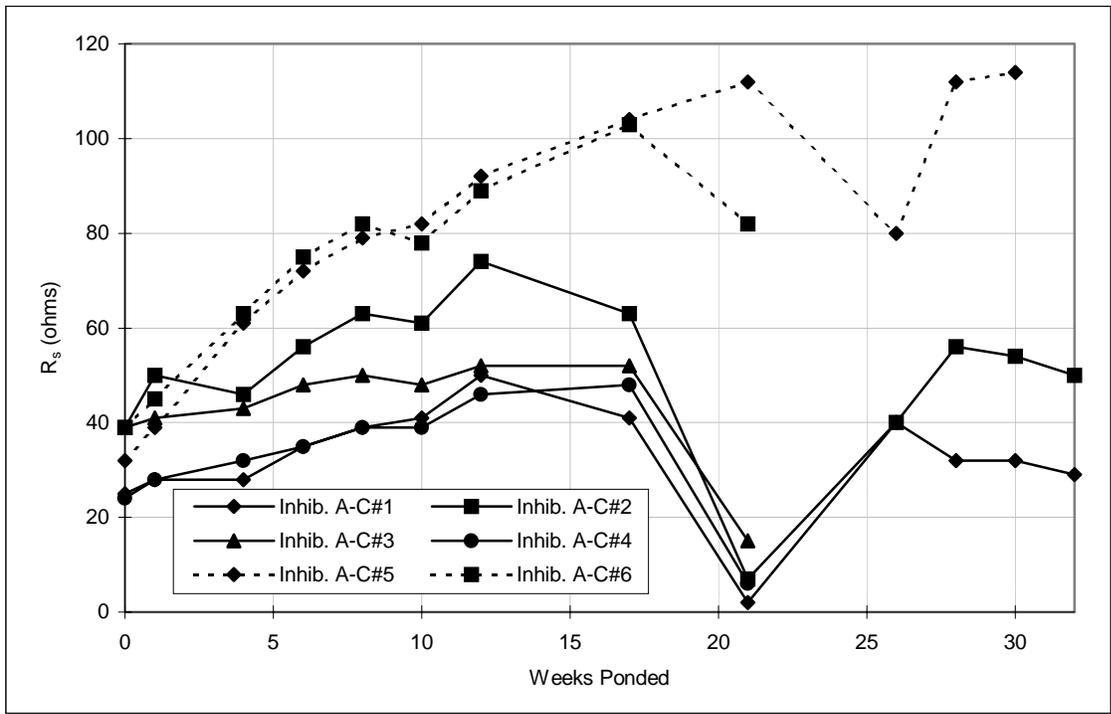
**Figure A.29  Solution resistance, pre-cracked lollipops:  Inhibitor A.**
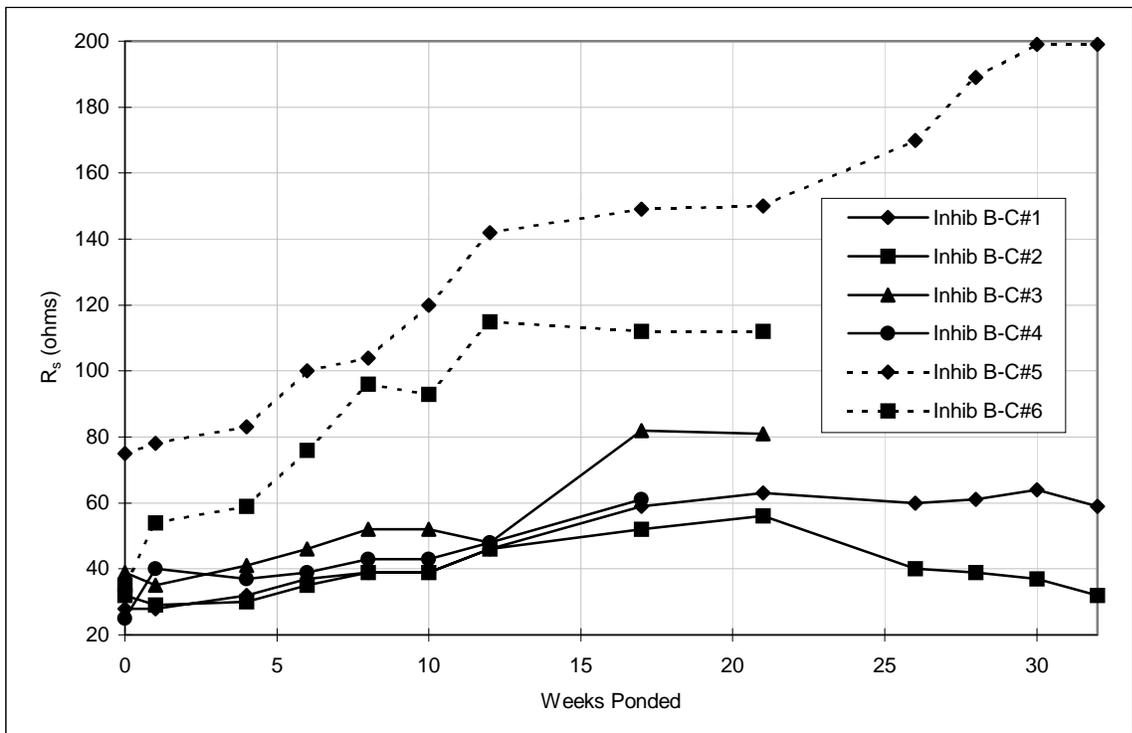


**Figure A.30  Solution resistance, pre-cracked lollipops:  Inhibitor B.**
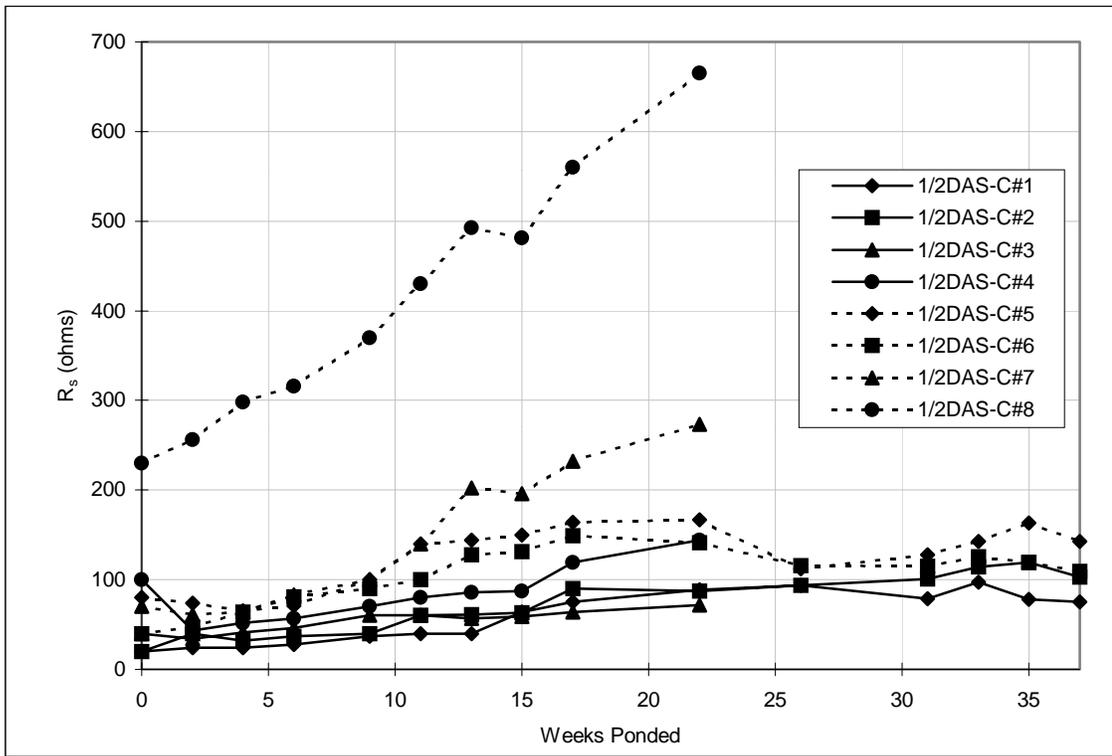
**Figure A.31  Solution resistance, pre-cracked lollipops:  ½%DAS.**
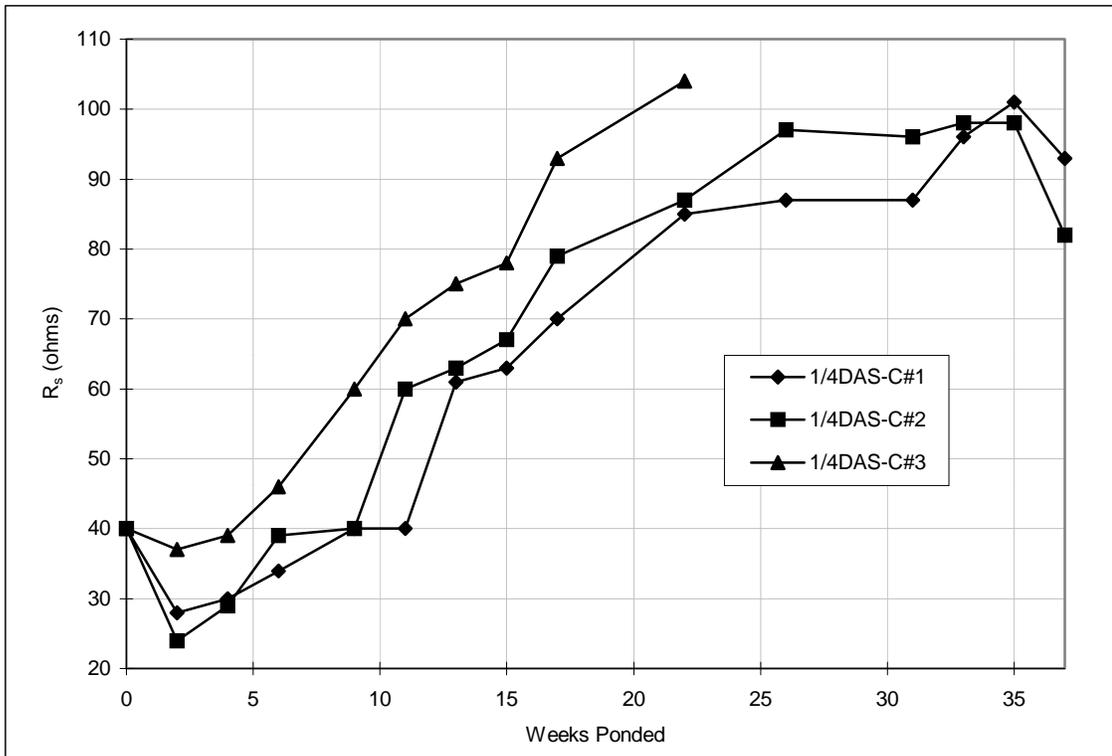


**Figure A.32  Solution resistance, pre-cracked lollipops:  ¼%DAS.**
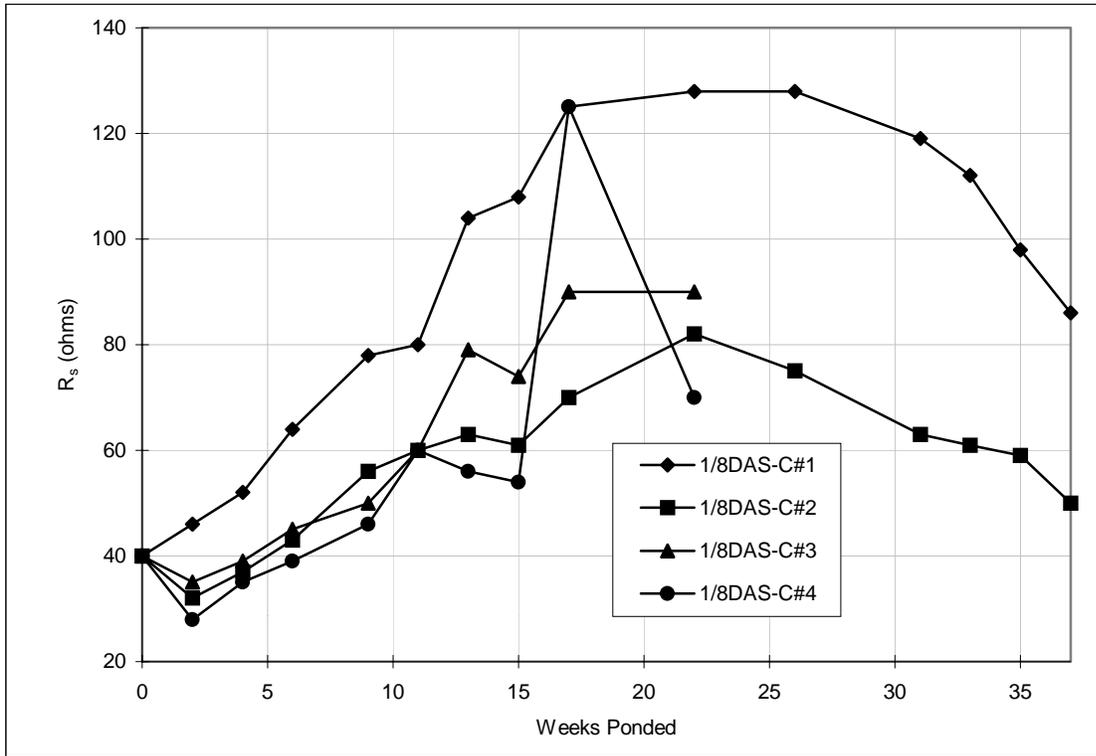
**Figure A.33  Solution resistance, pre-cracked lollipops:  1/8%DAS.**
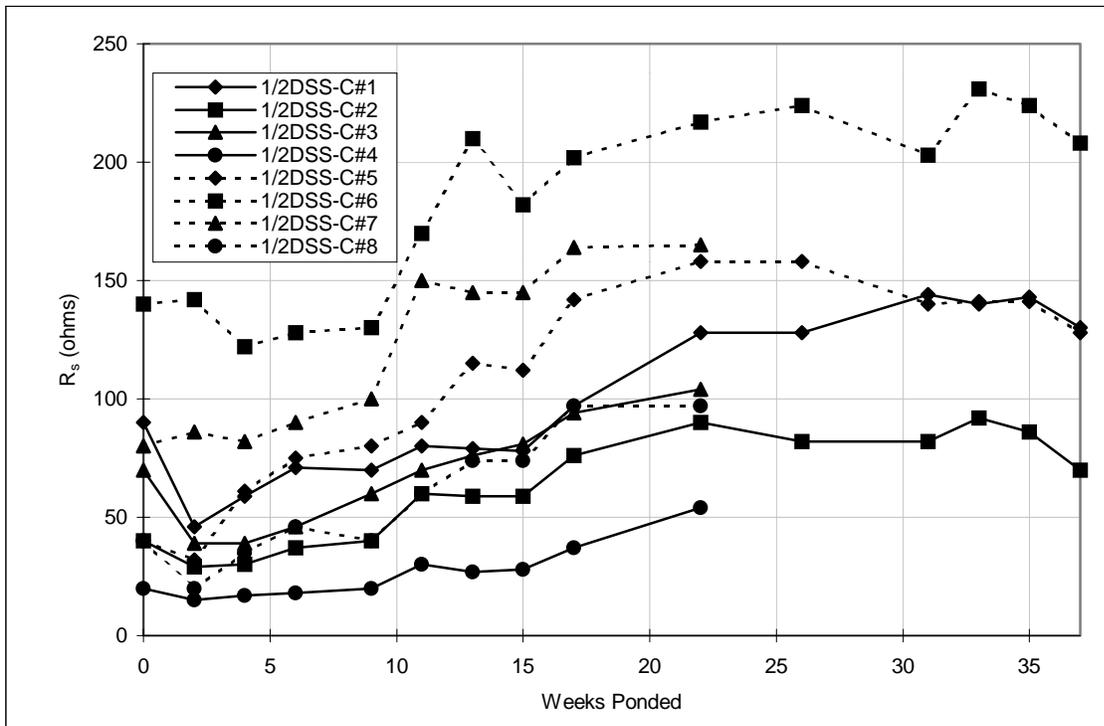


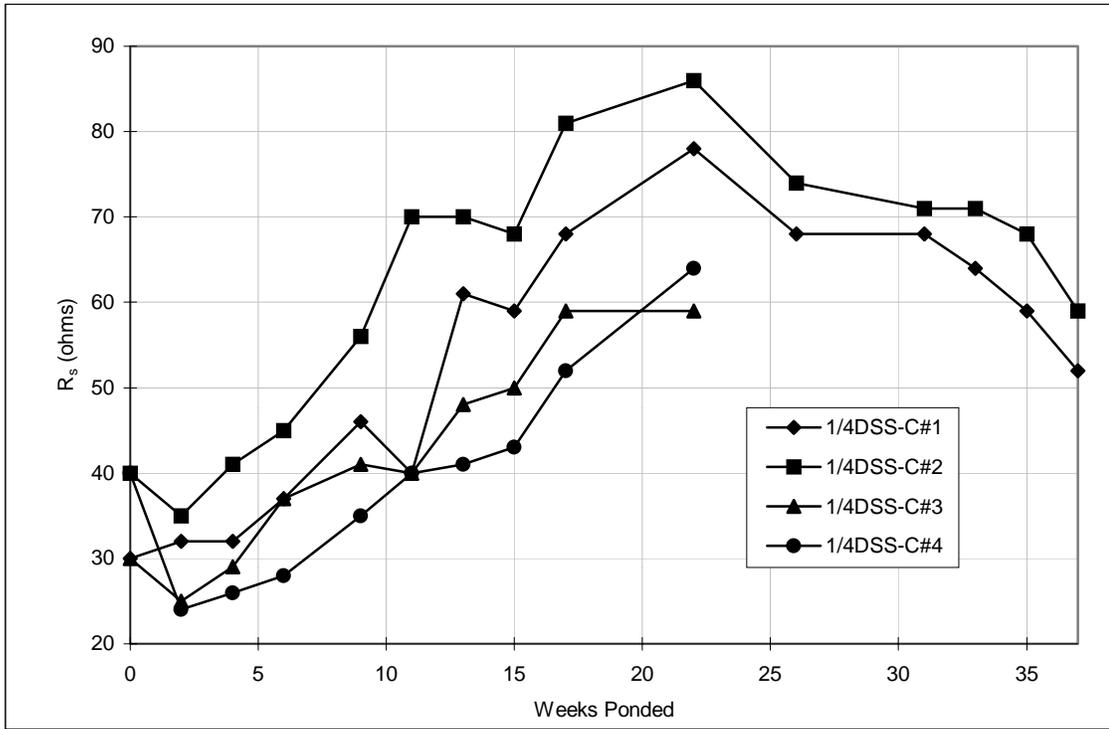**Figure A.34  Solution resistance, pre-cracked lollipops:  ½%DSS.**

**Figure A.35  Solution resistance, pre-cracked lollipops:  ¼%DSS.**
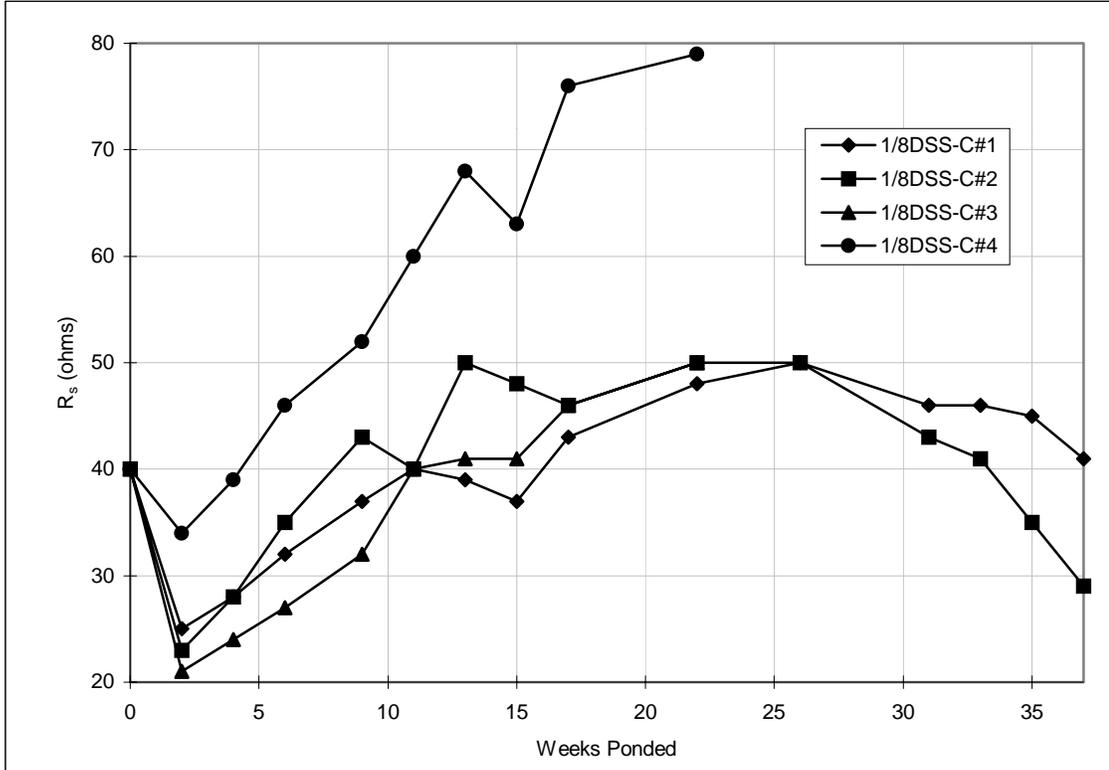


**Figure A.36  Solution resistance, pre-cracked lollipops:  1/8%DSS.**
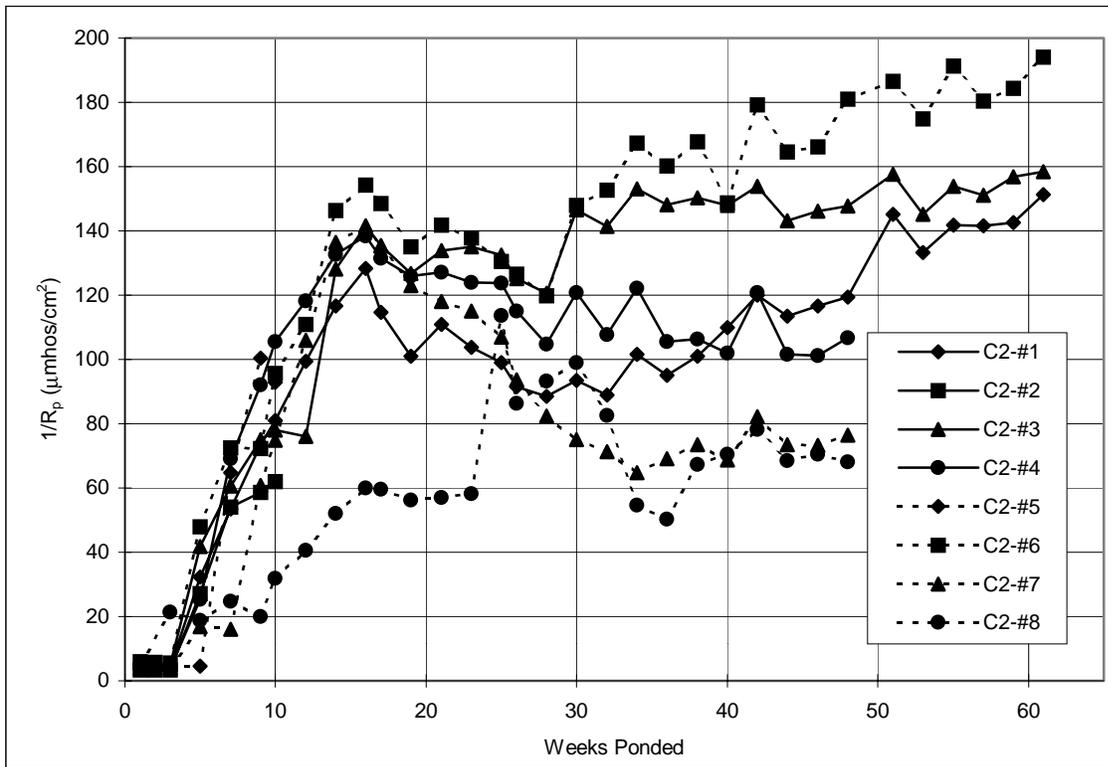
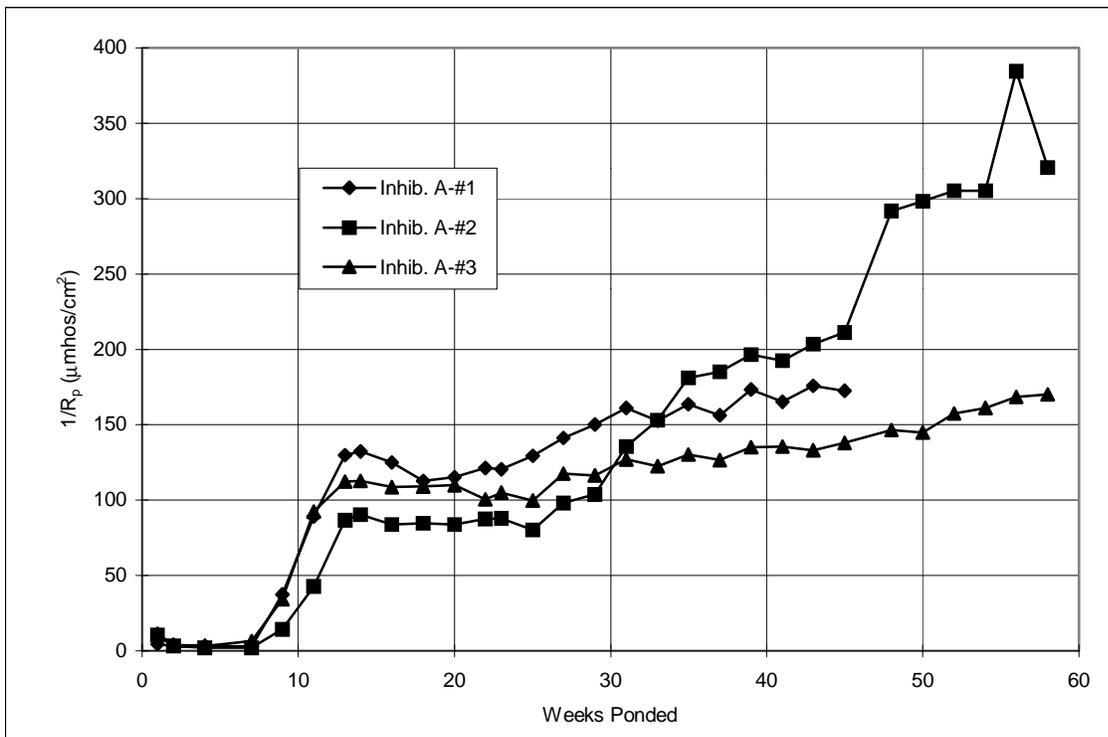**Figure A.37  Corrosion rate, 2-inch diameter lollipops:  Control.**
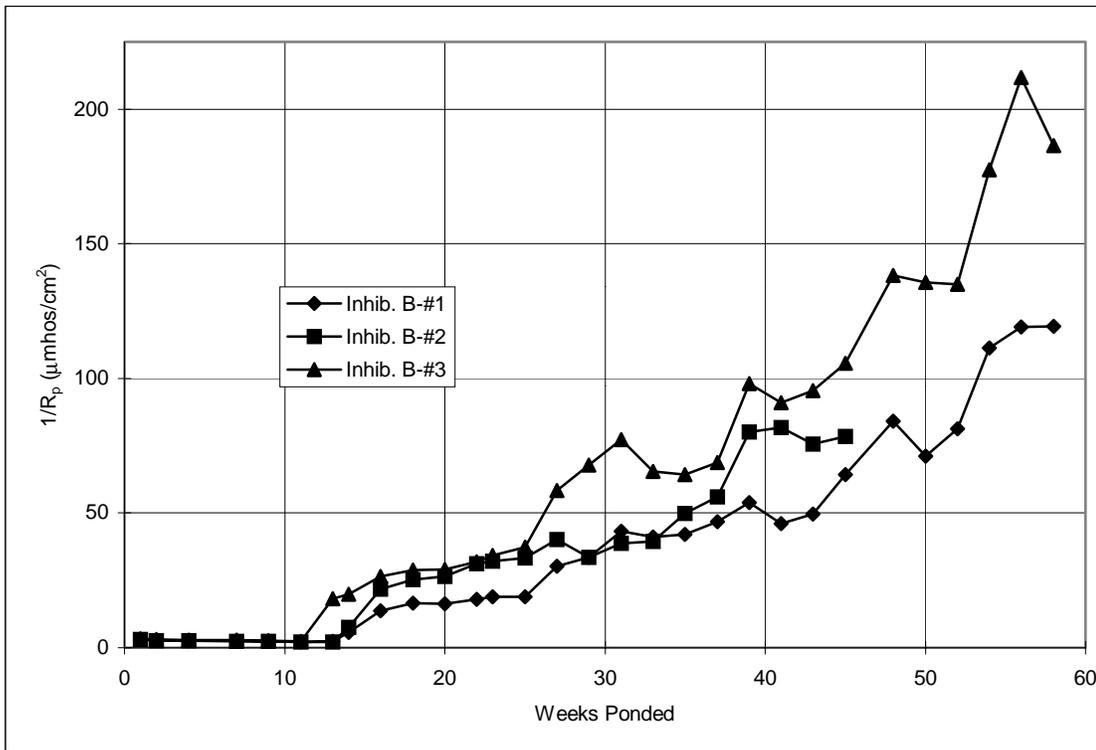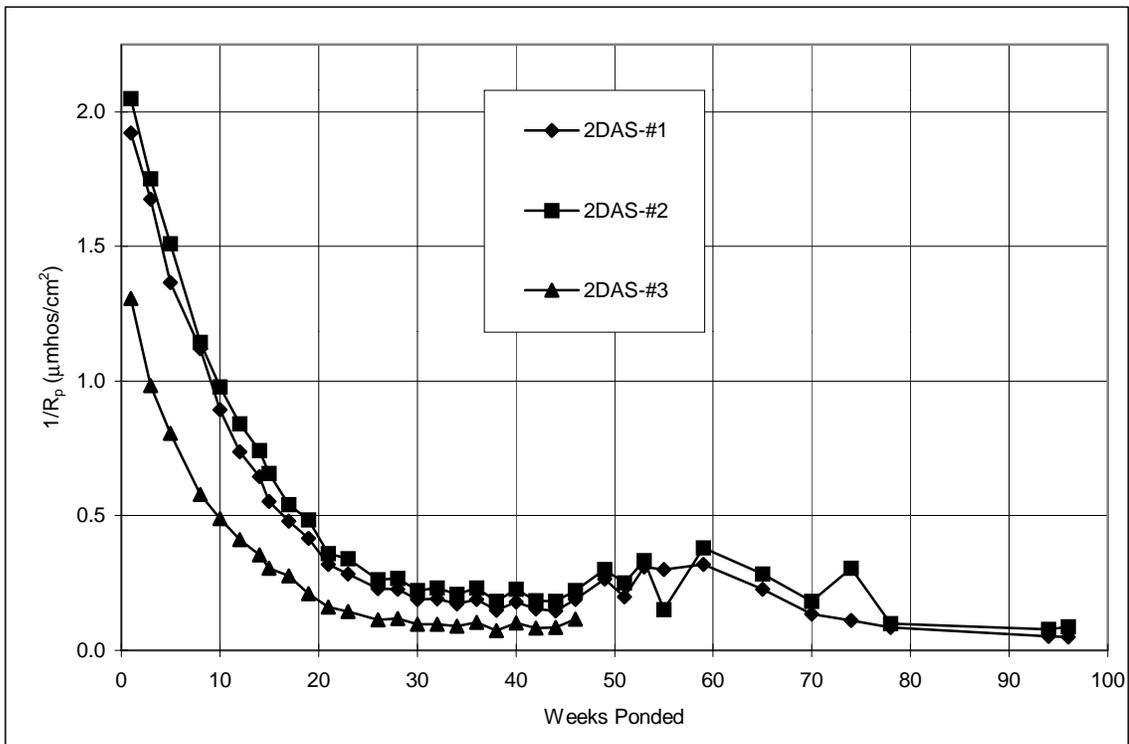


**Figure A.38  Corrosion rate, 2-inch diameter lollipops:  Inhibitor A.**

94

**Figure A.39  Corrosion rate, 2-inch diameter lollipops:  Inhibitor B.**



**Figure A.40  Corrosion rate, 2-inch diameter lollipops:  2%DAS.**

**Figure A.41  Corrosion rate, 2-inch diameter lollipop:  1%DAS.**



**Figure A.42  Corrosion rate, 2-inch diameter lollipops:  ½%DAS.**

**Figure A.43  Corrosion rate, 2-inch diameter lollipops:  2%DSS.**



**Figure A.44  Corrosion rate, 2-inch diameter lollipops:  1%DSS.**

**Figure A.45  Corrosion rate, 3-inch diameter lollipops:  Control.**
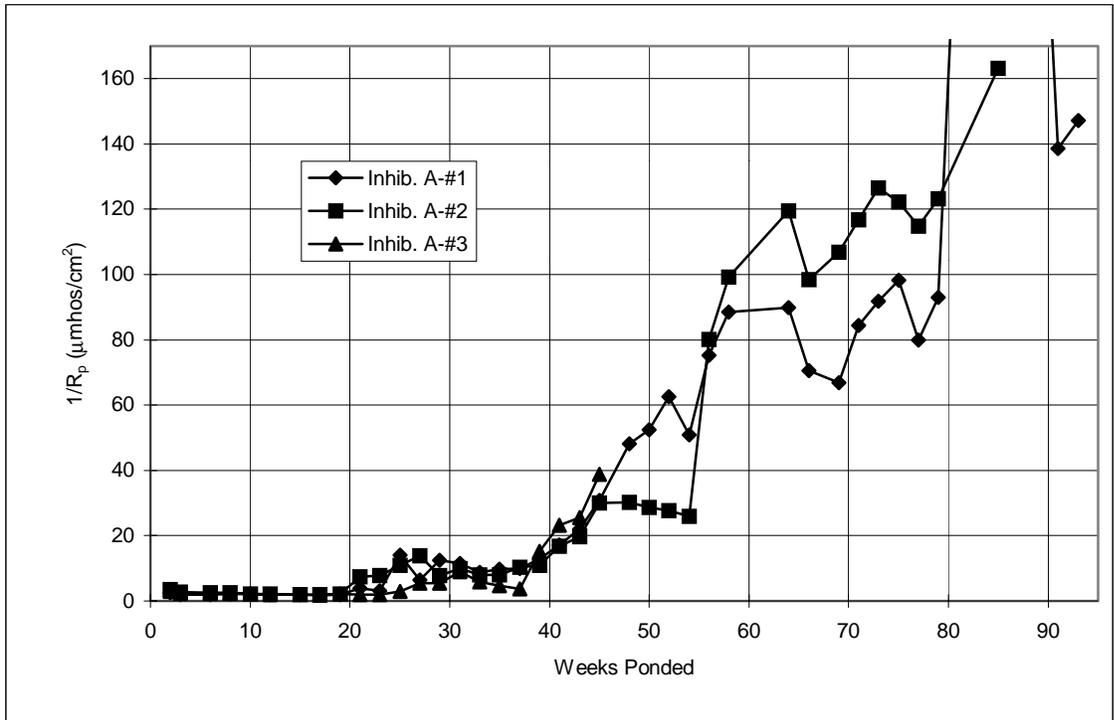


**Figure A.46  Corrosion rate, 3-inch diameter lollipops:  Inhibitor A.**
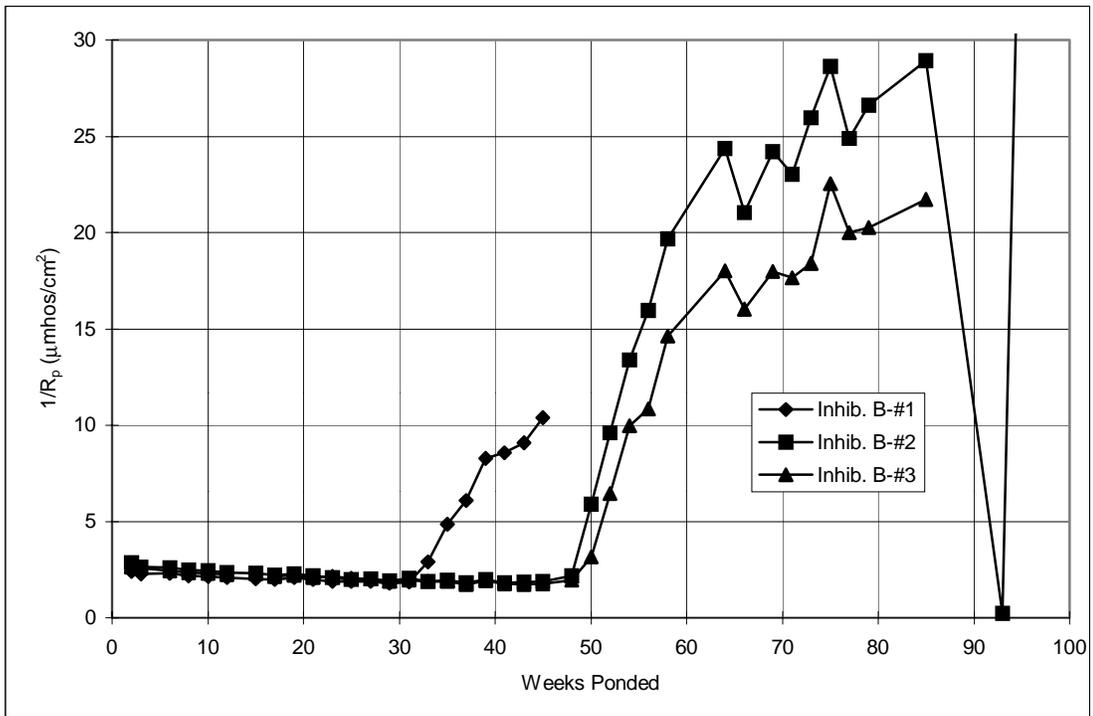
**Figure A.47  Corrosion rate, 3-inch diameter lollipops:  Inhibitor B.**
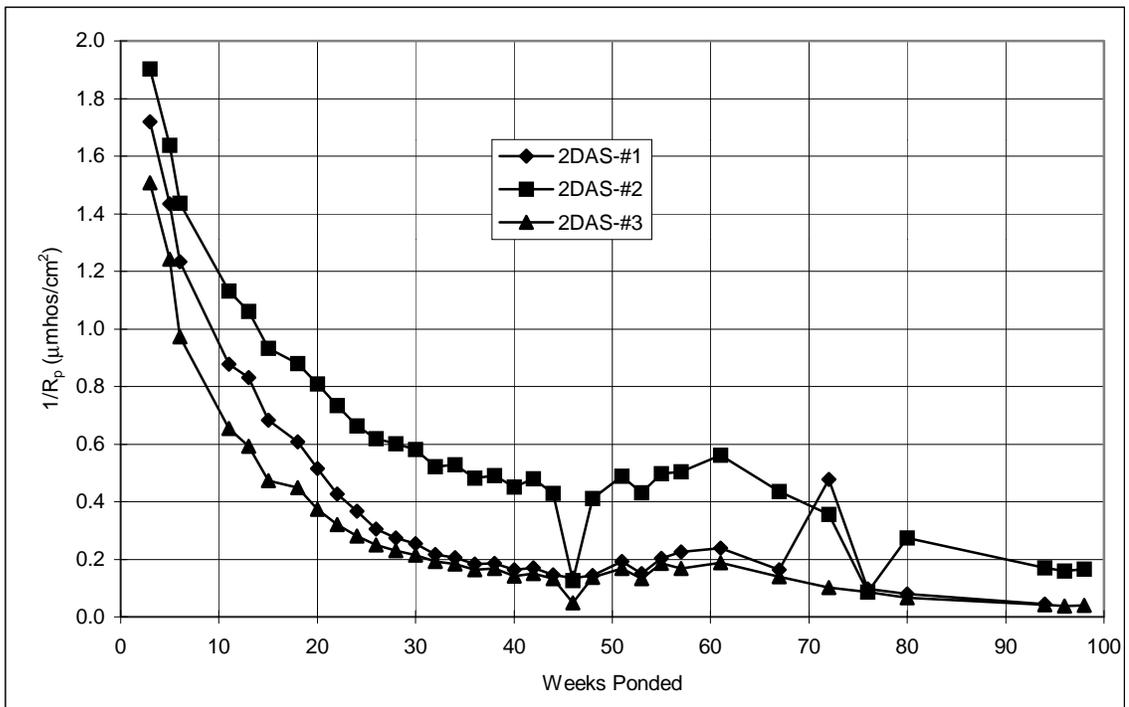


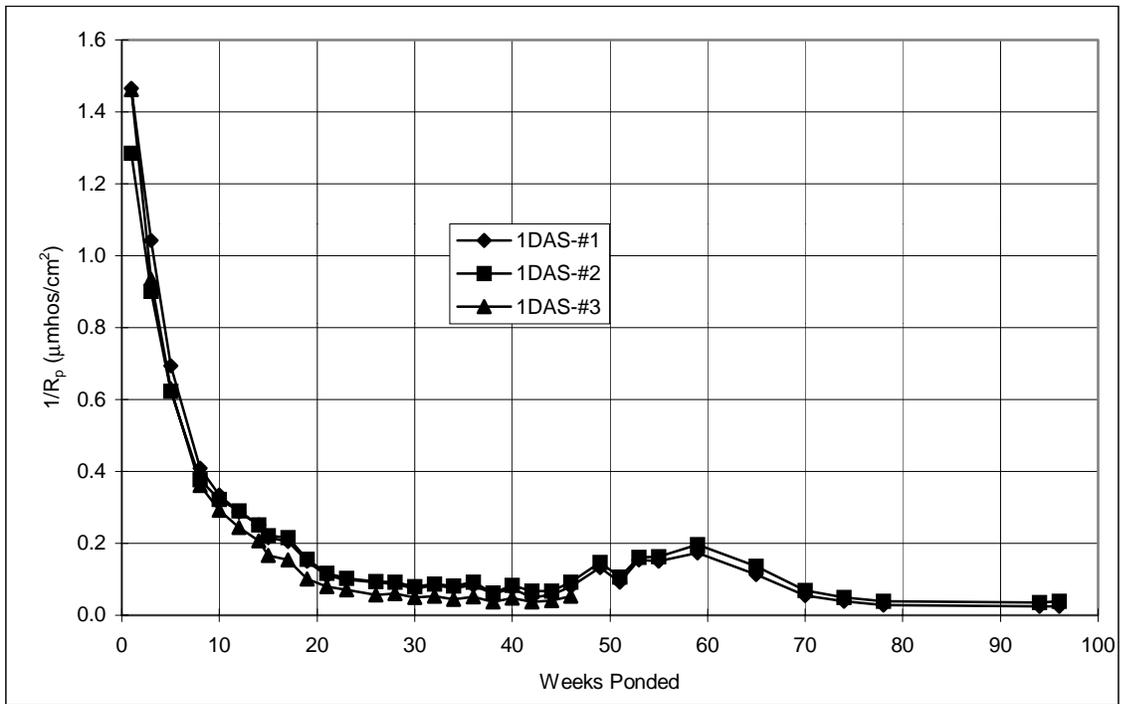**Figure A.48  Corrosion rate, 3-inch diameter lollipops:  2%DAS.**
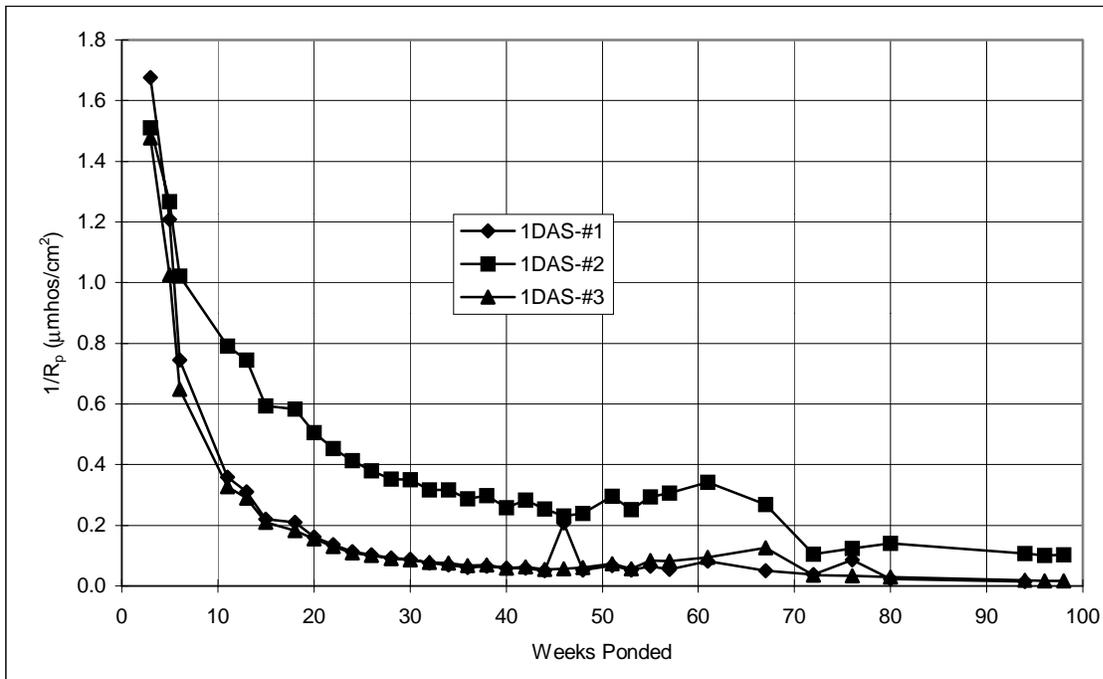
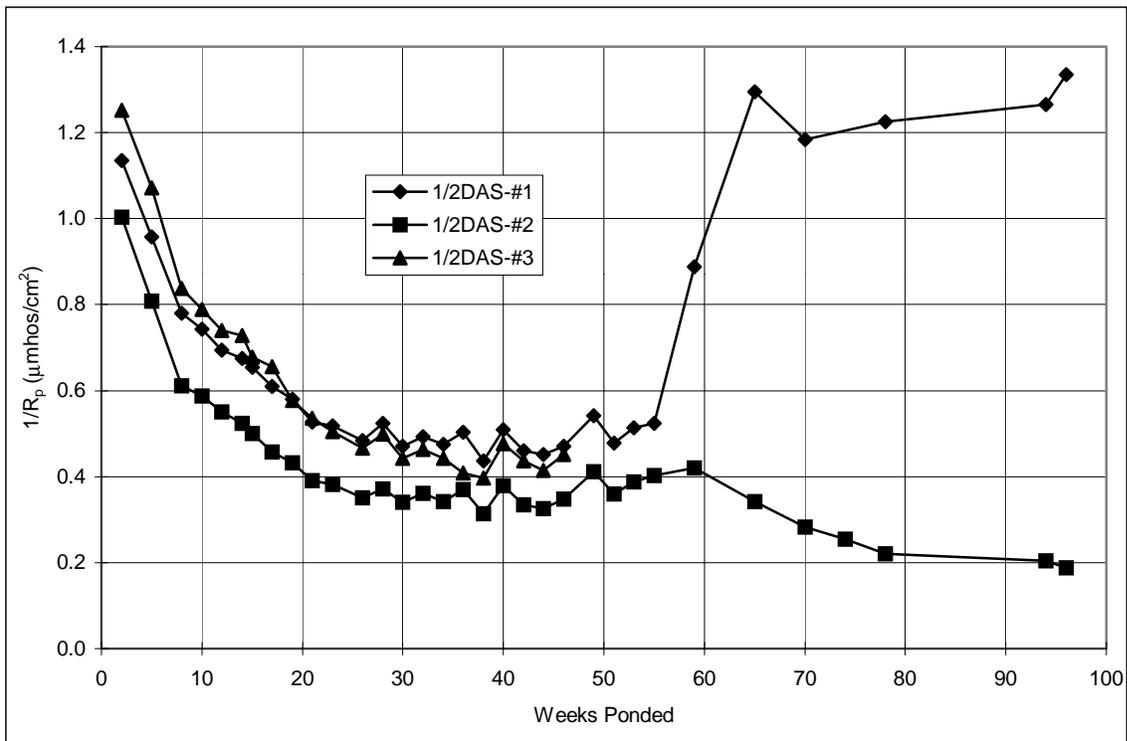**Figure A.49  Corrosion rate, 3-inch diameter lollipops:  1%DAS.**
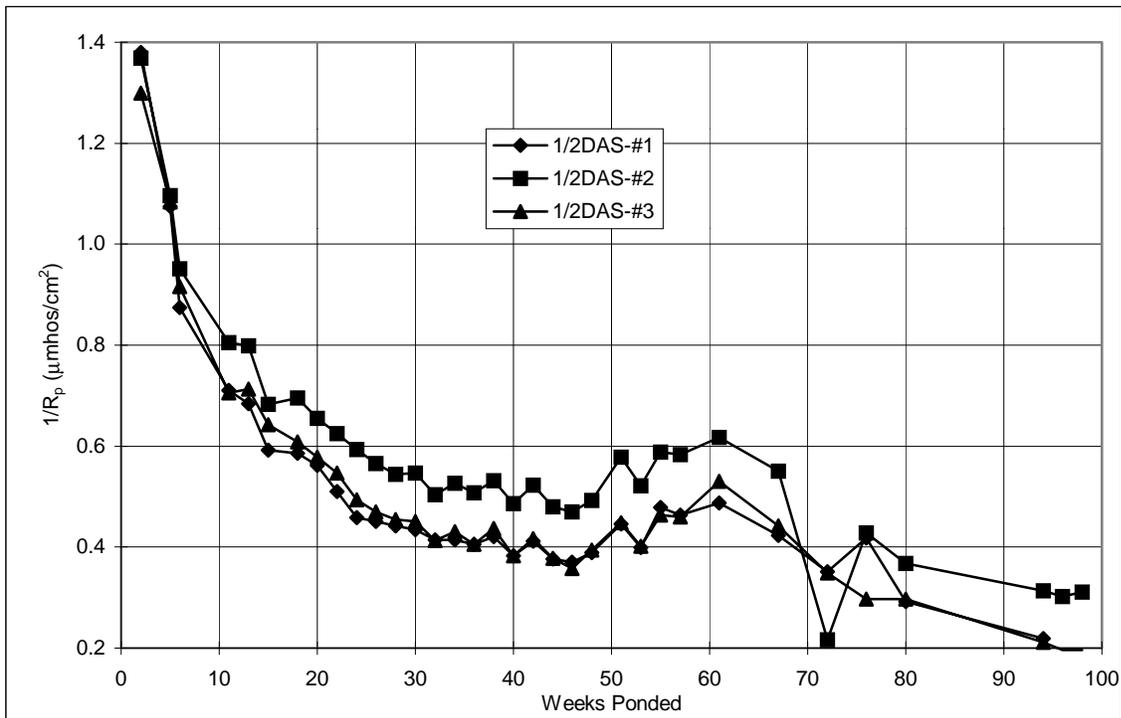


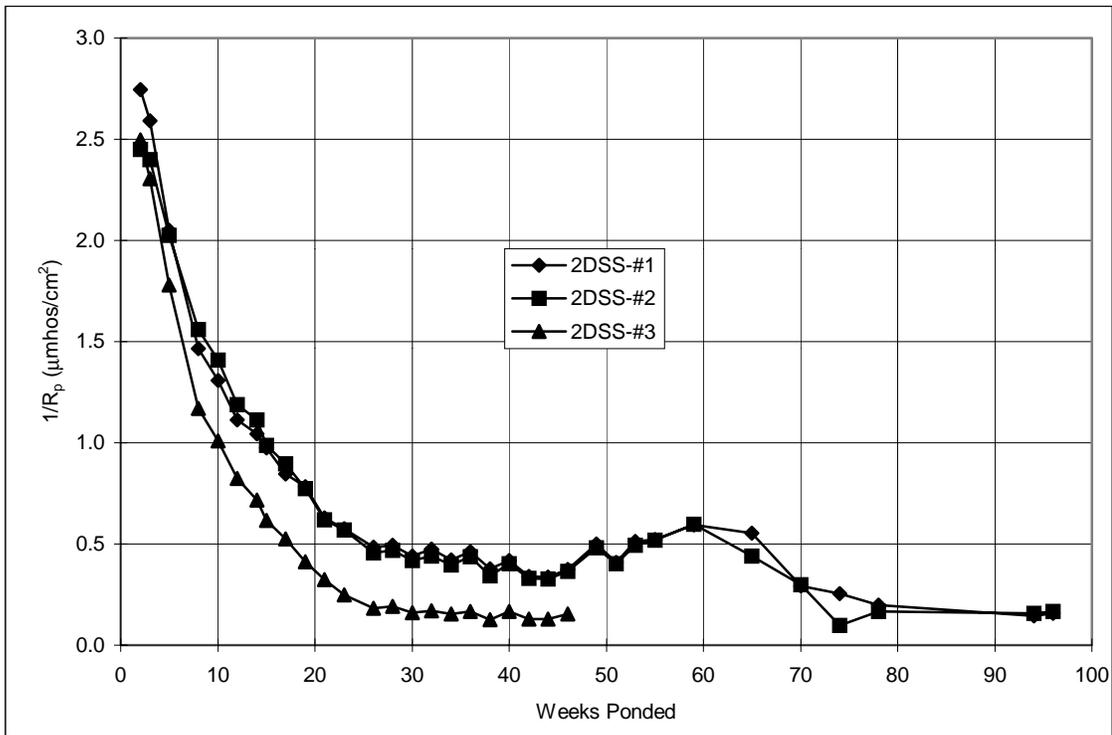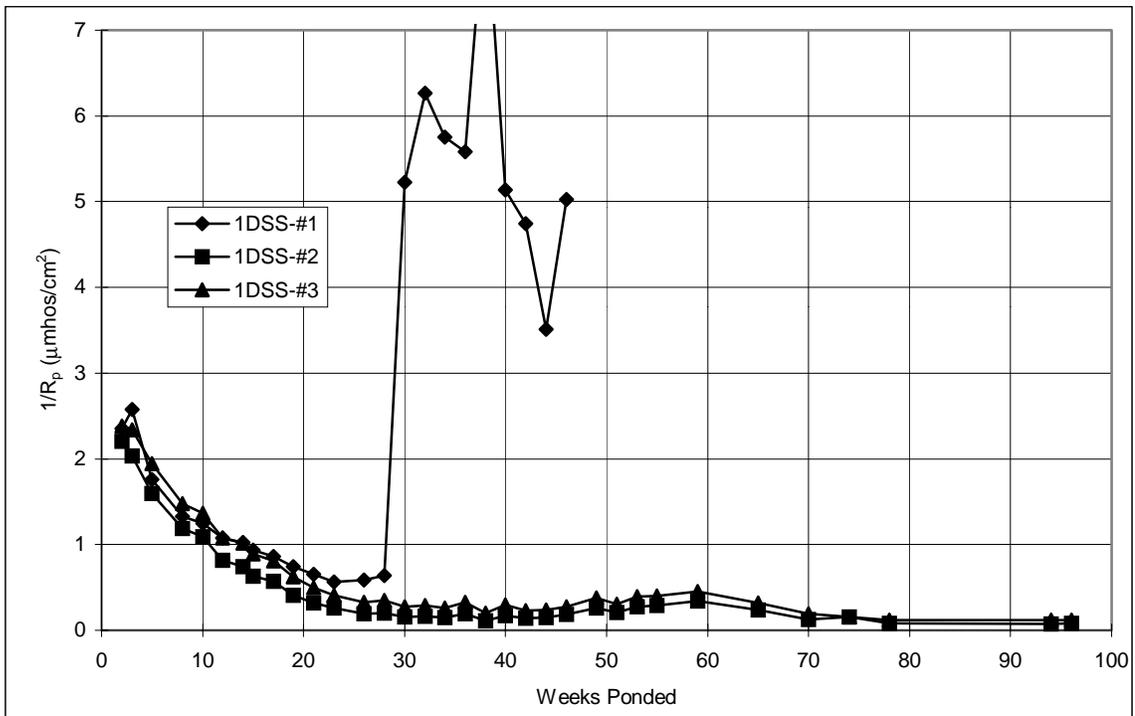**Figure A.50  Corrosion rate, 3-inch diameter lollipops:  ½%DAS.**

100
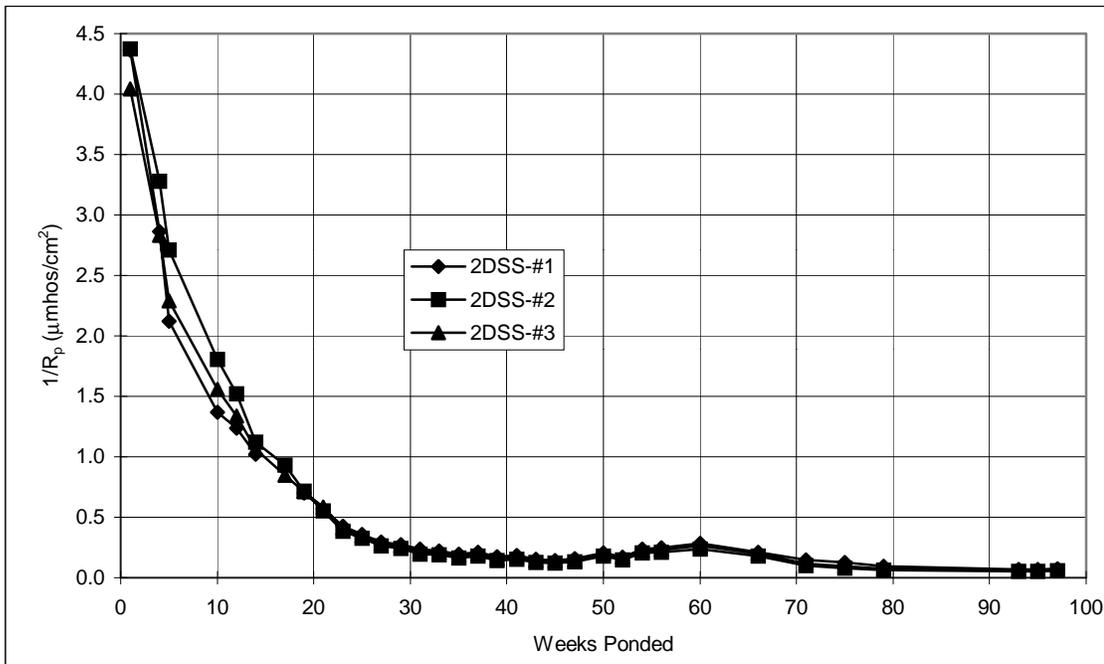
**Figure A.51  Corrosion rate, 3-inch diameter lollipops:  2%DSS.**
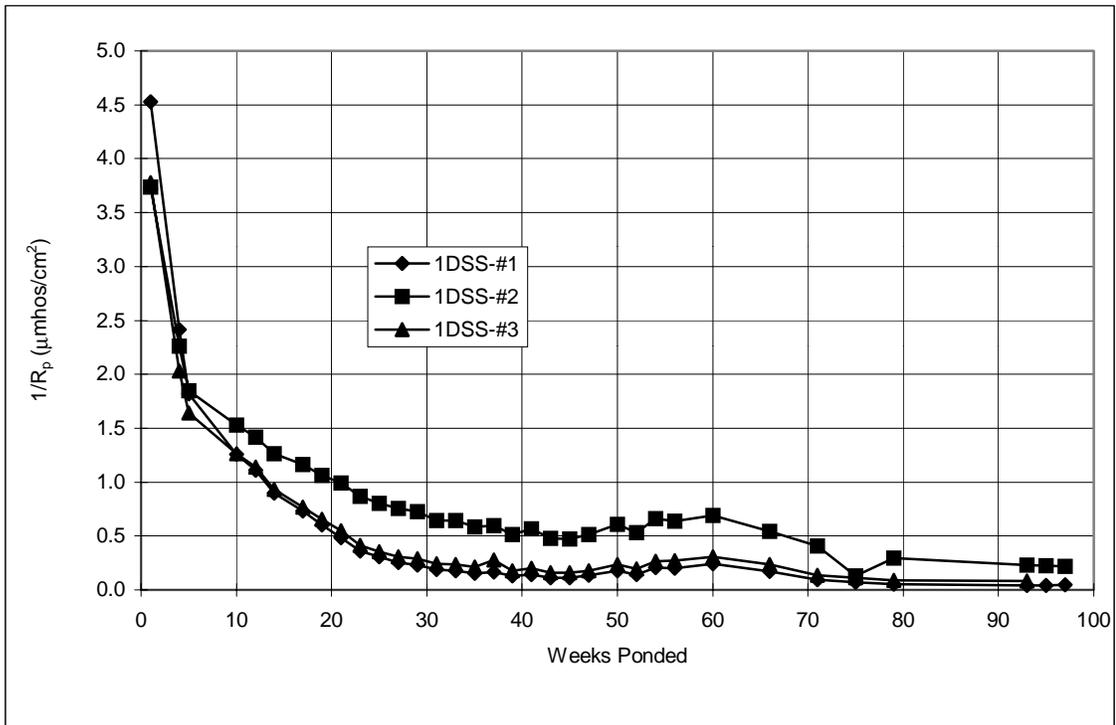


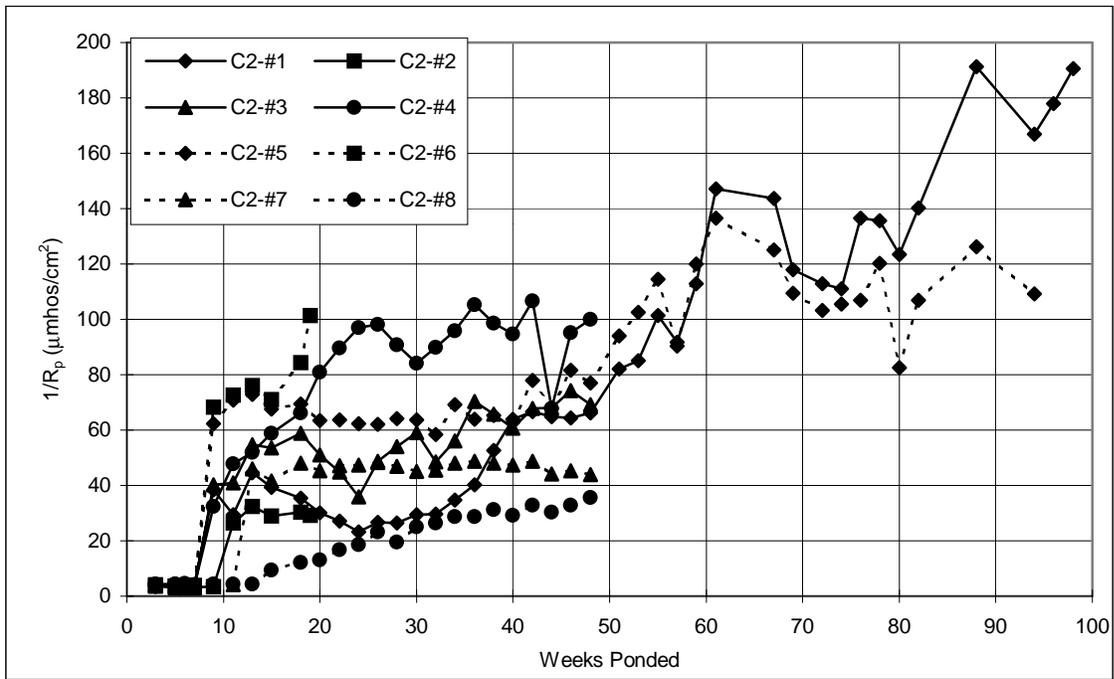**Figure A.52  Corrosion rate, 3-inch diameter lollipops:  1%DSS.**
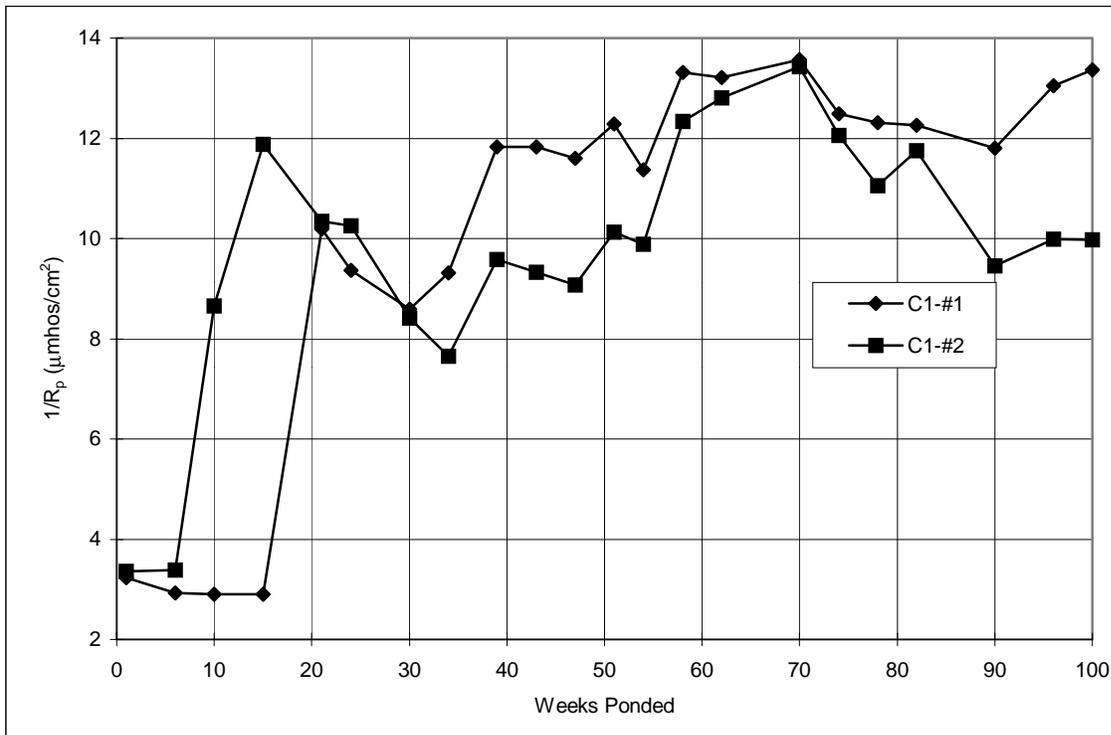
**Figure A.53  Corrosion rate, slabs:  Control 1.**
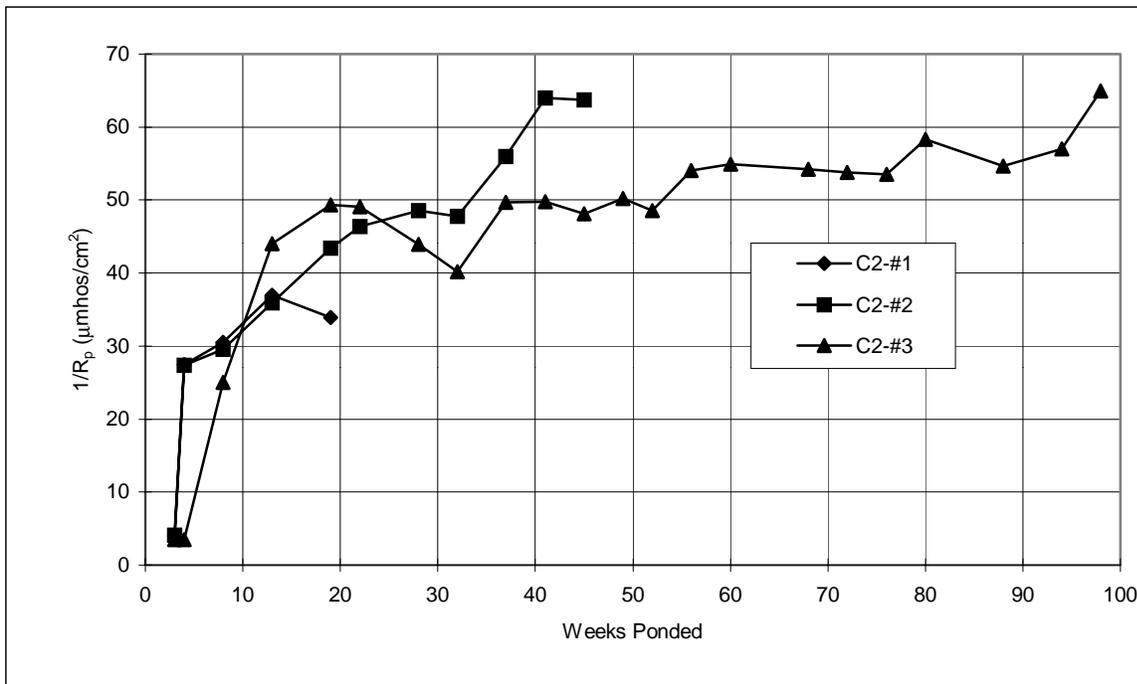


**Figure A.54  Corrosion rate, slabs:  Control 2-connected.**

102
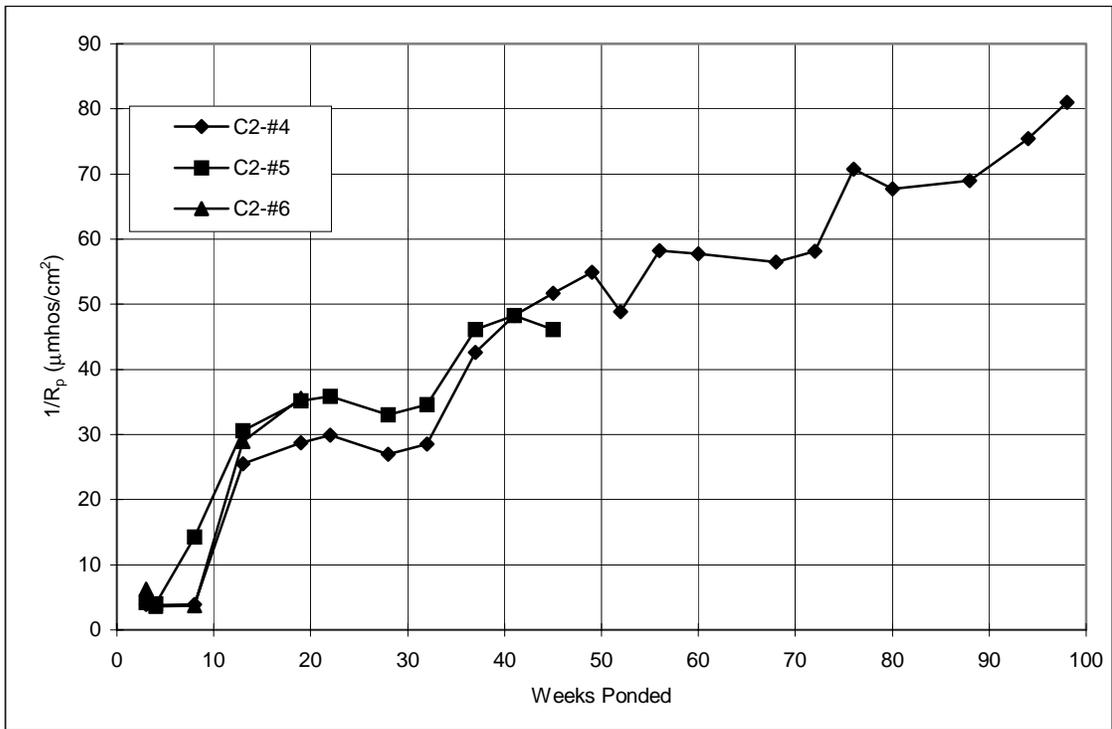
**Figure A.55  Corrosion rate, slabs:  Control 2-unconnected.**
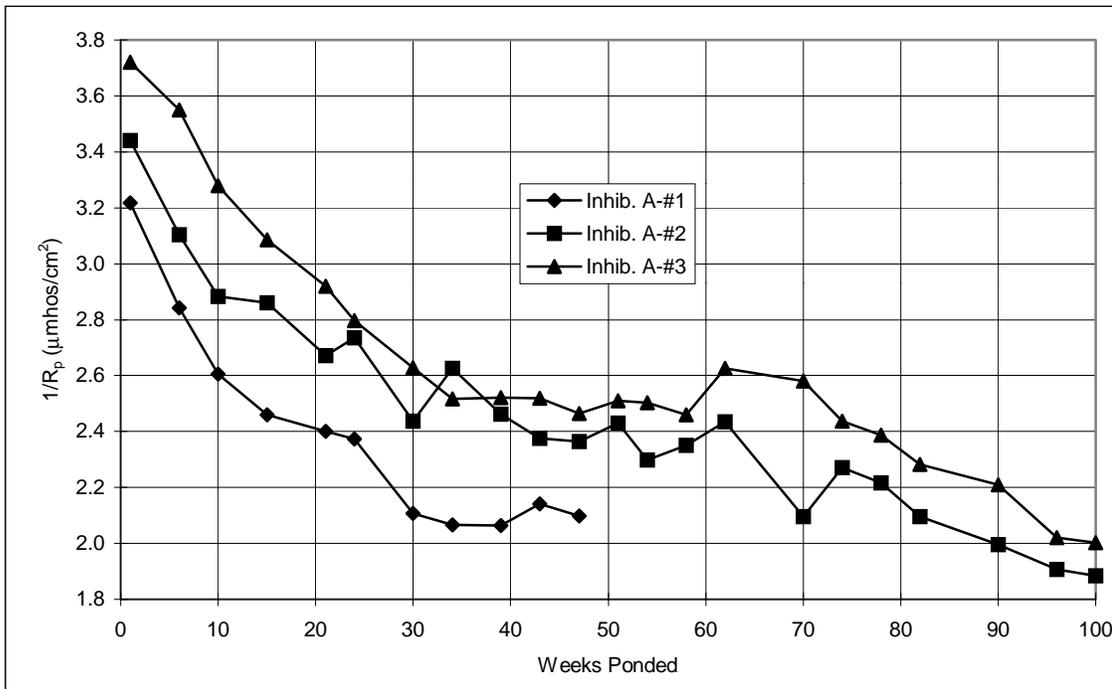


**Figure A.56  Corrosion rate, slabs:  Inhibitor A.**
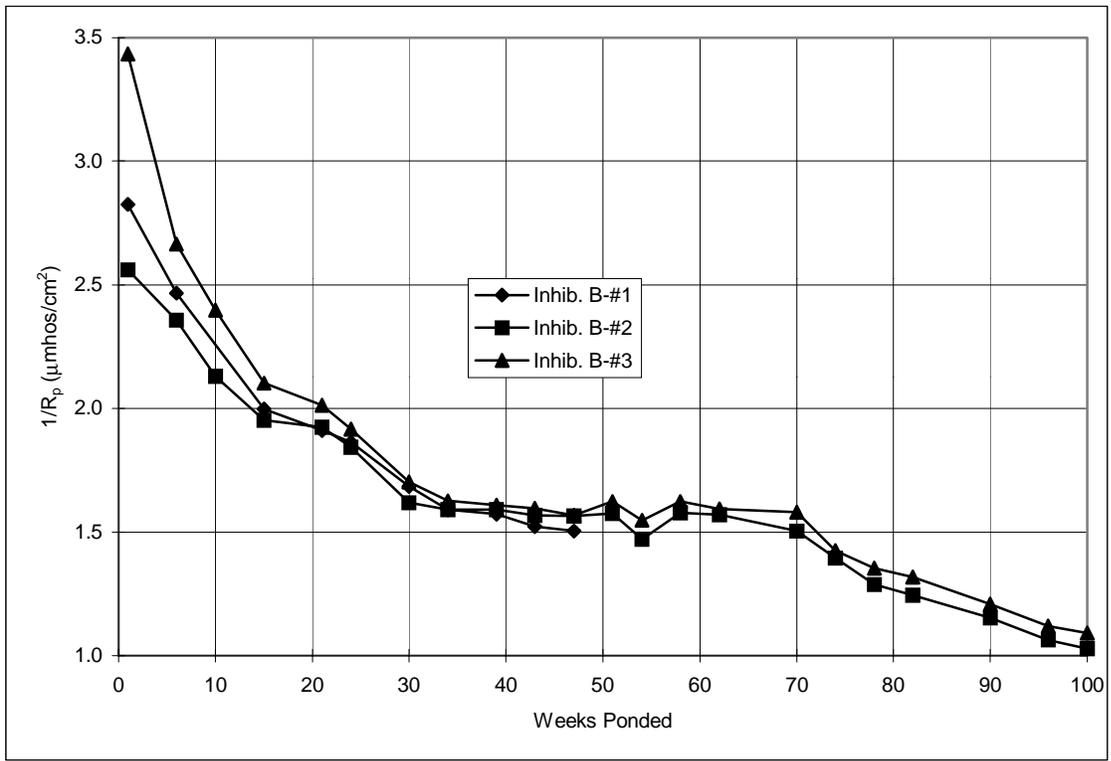
**Figure A.57  Corrosion rate, slabs:  Inhibitor B.**
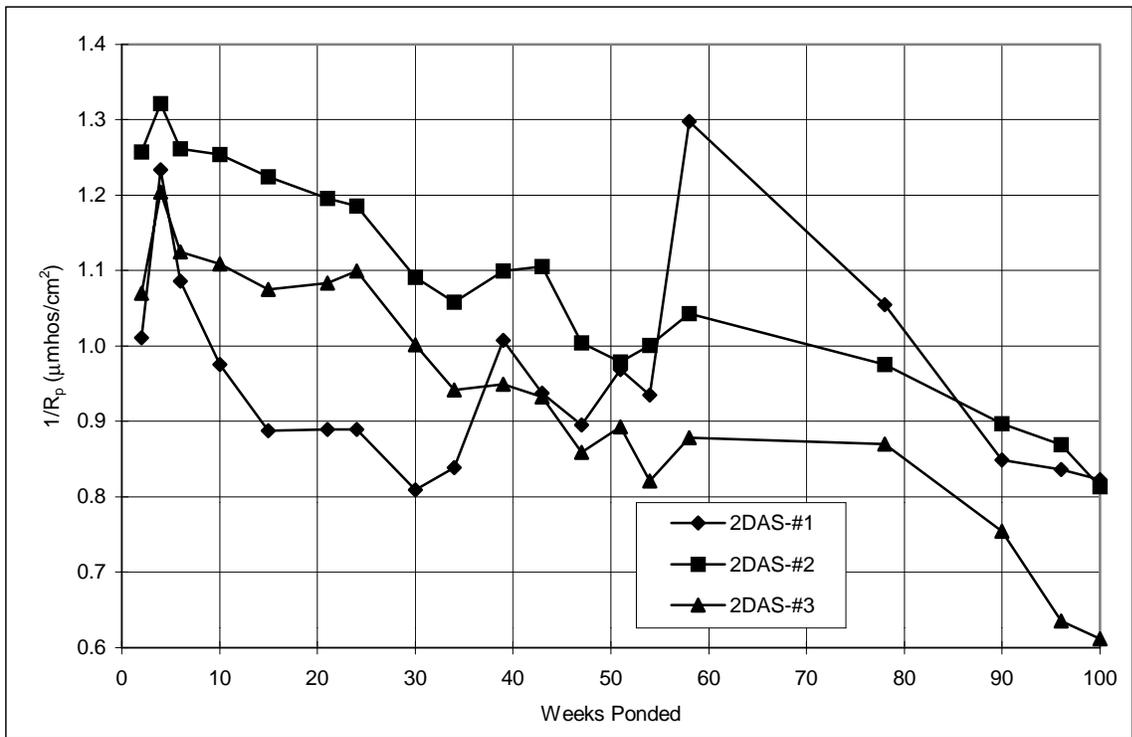


**Figure A.58  Corrosion rate, slabs:  2%DAS.**

104
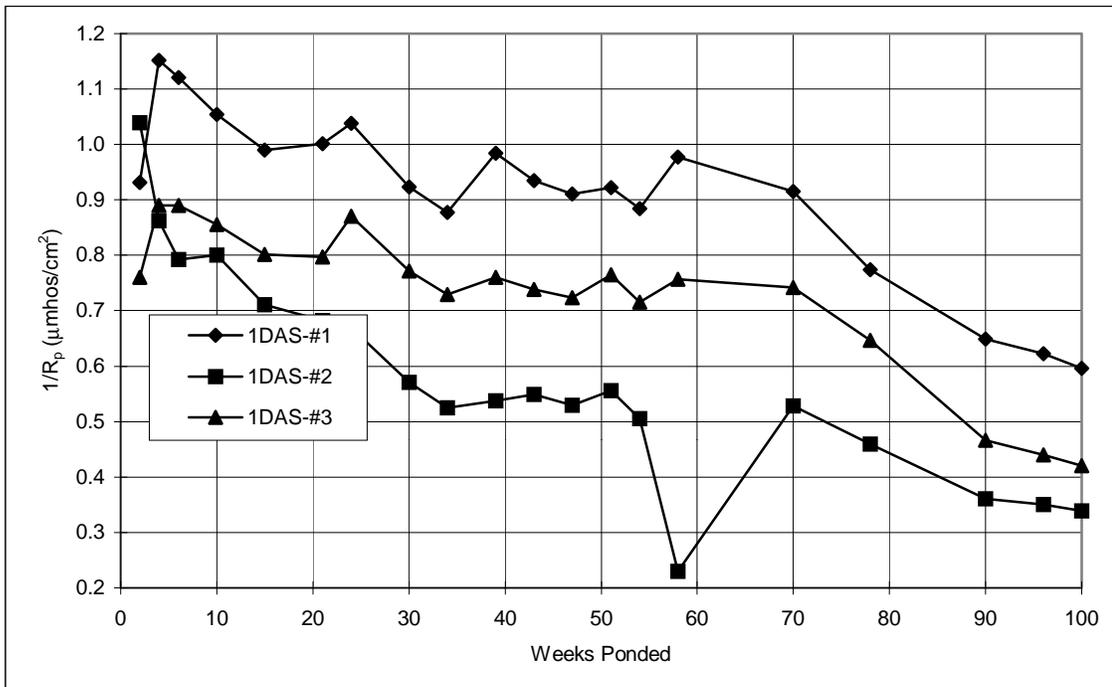
**Figure A.59  Corrosion rate, slabs:  1%DAS.**
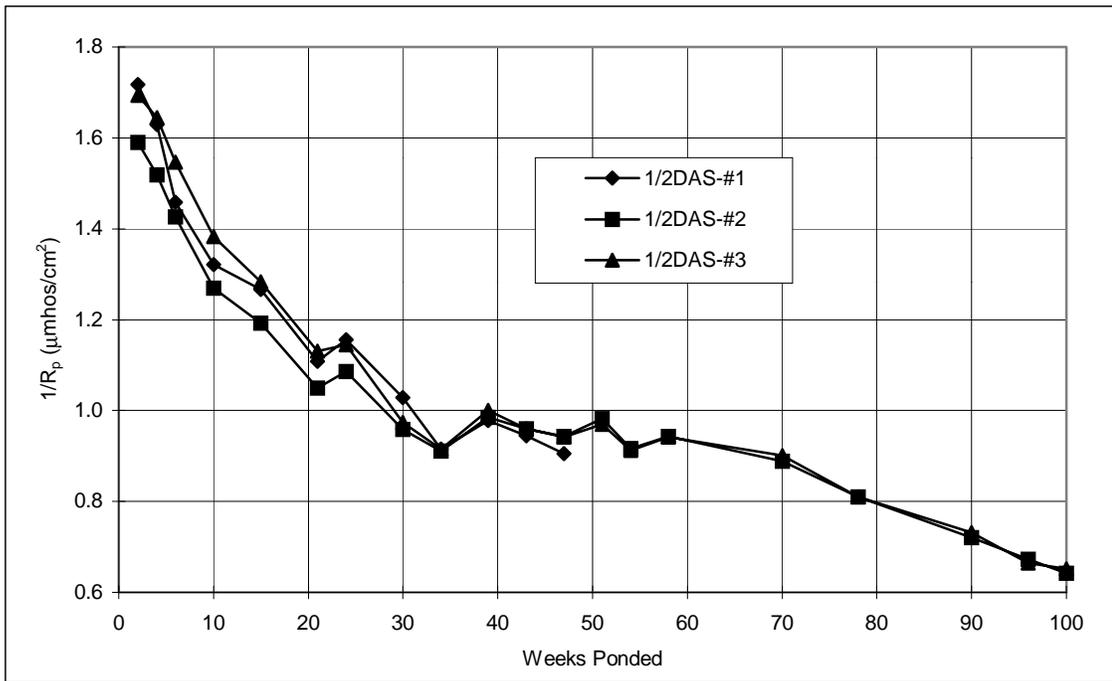


**Figure A.60  Corrosion rate, slabs:  ½%DAS.**
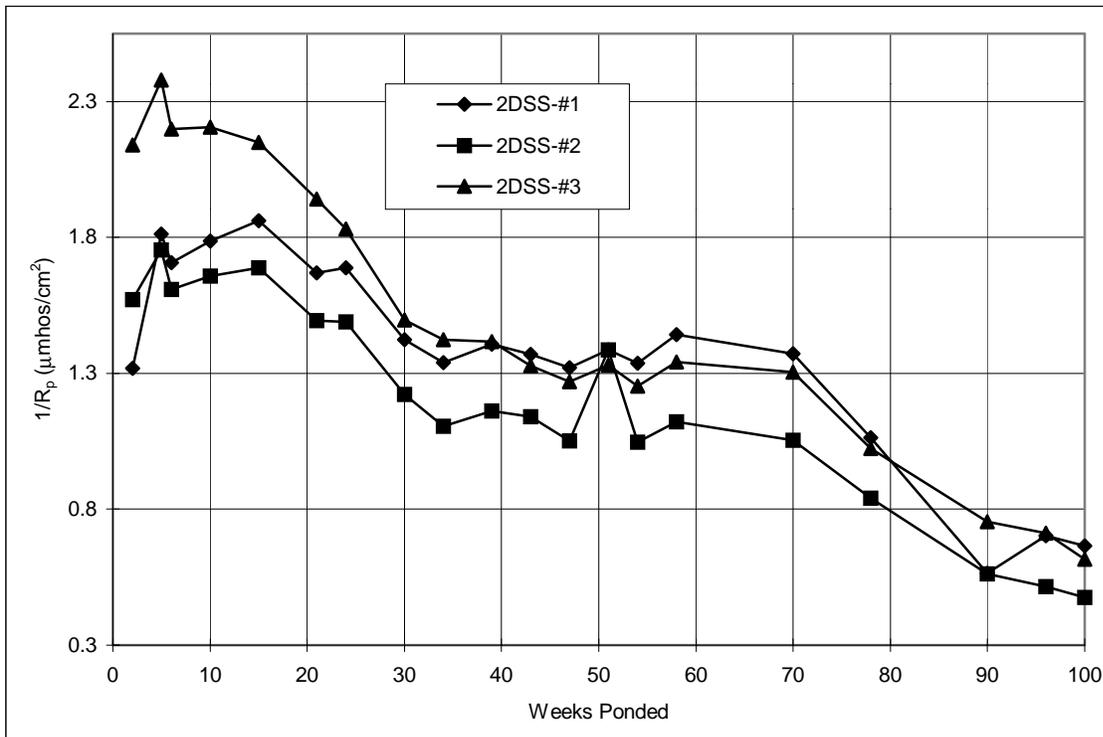
**Figure A.61 Corrosion rate, slabs: 2%DSS.**
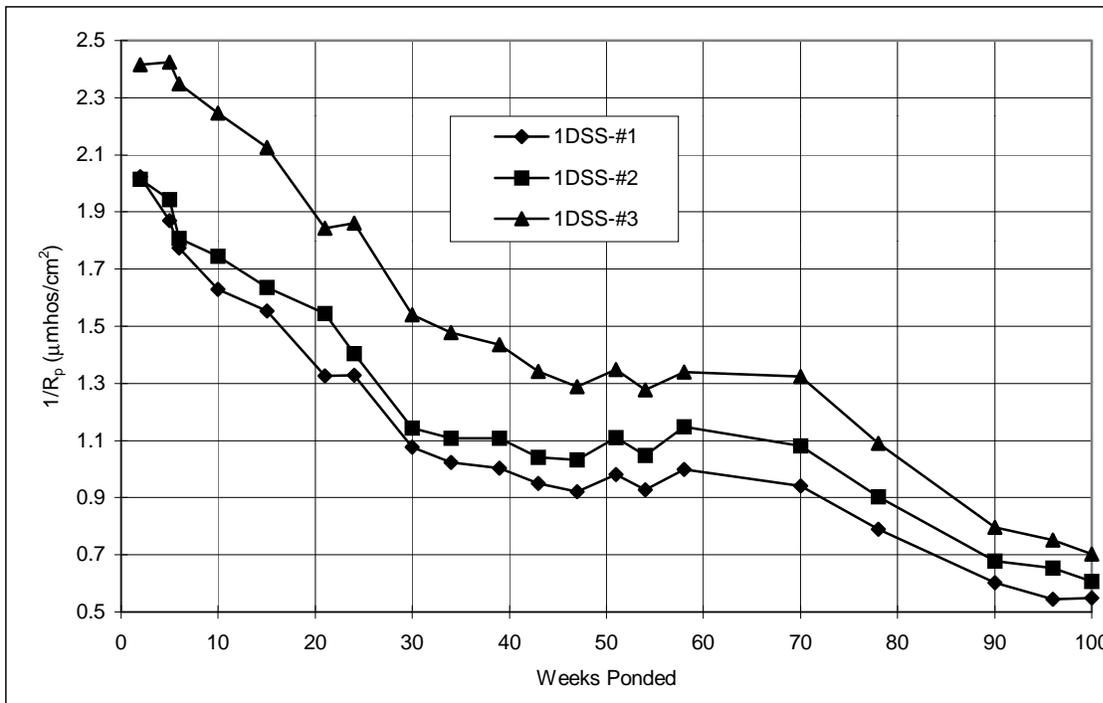


**Figure A.62 Corrosion rate, slabs: 1%DSS.**

106
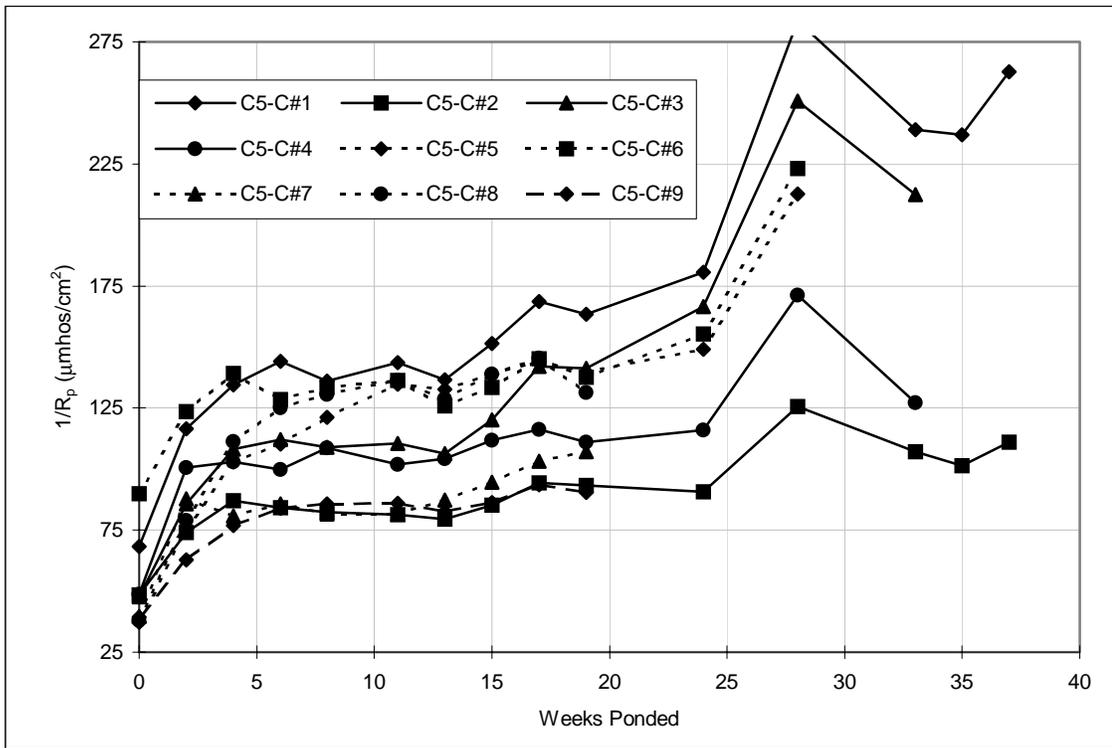
**Figure A.63  Corrosion rate, pre-cracked lollipops:  Control, specimens 1-9.**
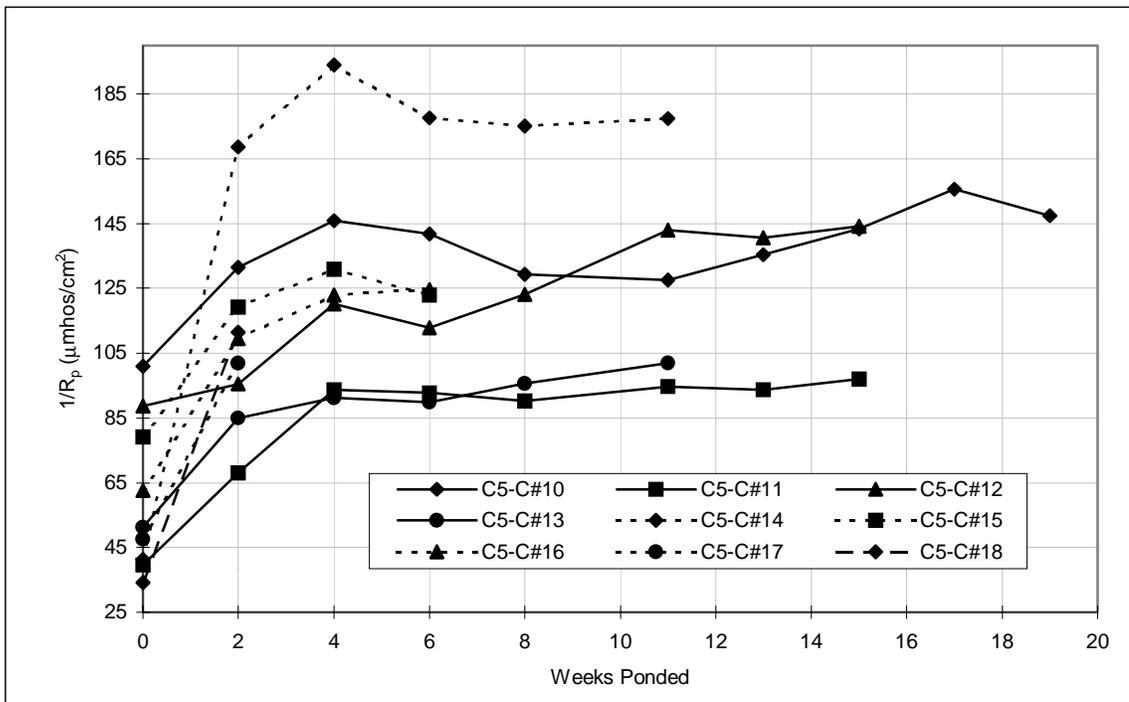


**Figure A.64  Corrosion rate, pre-cracked lollipops:  Control, specimens 10-18.**
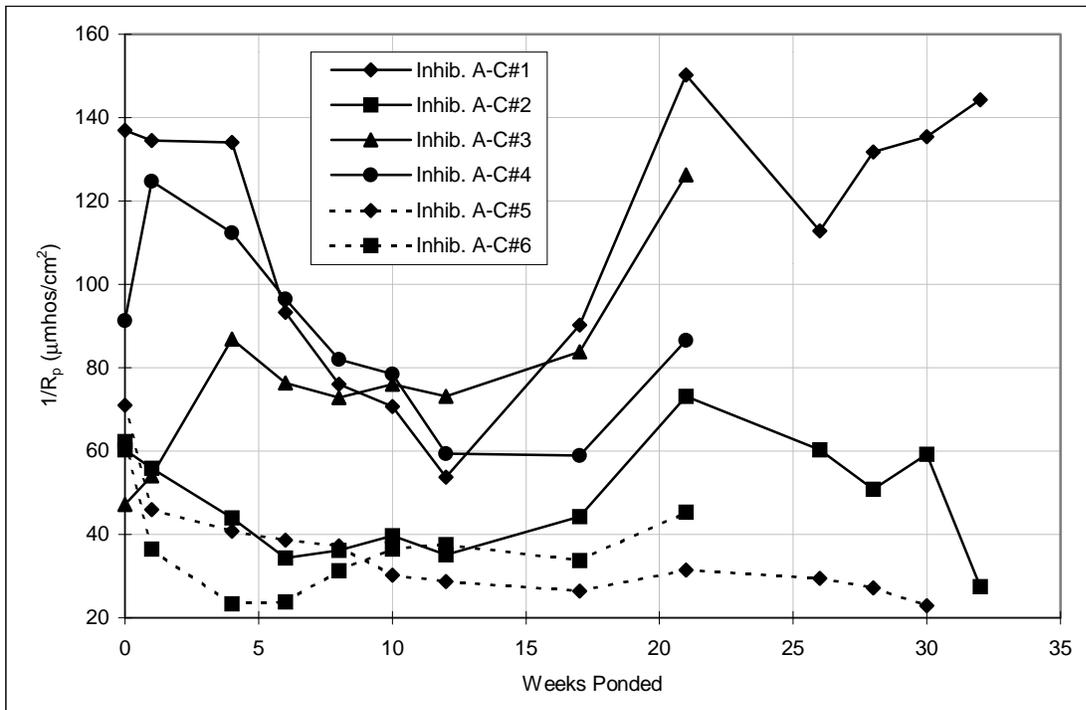
**Figure A.65  Corrosion rate, pre-cracked lollipops:  Inhibitor A.**
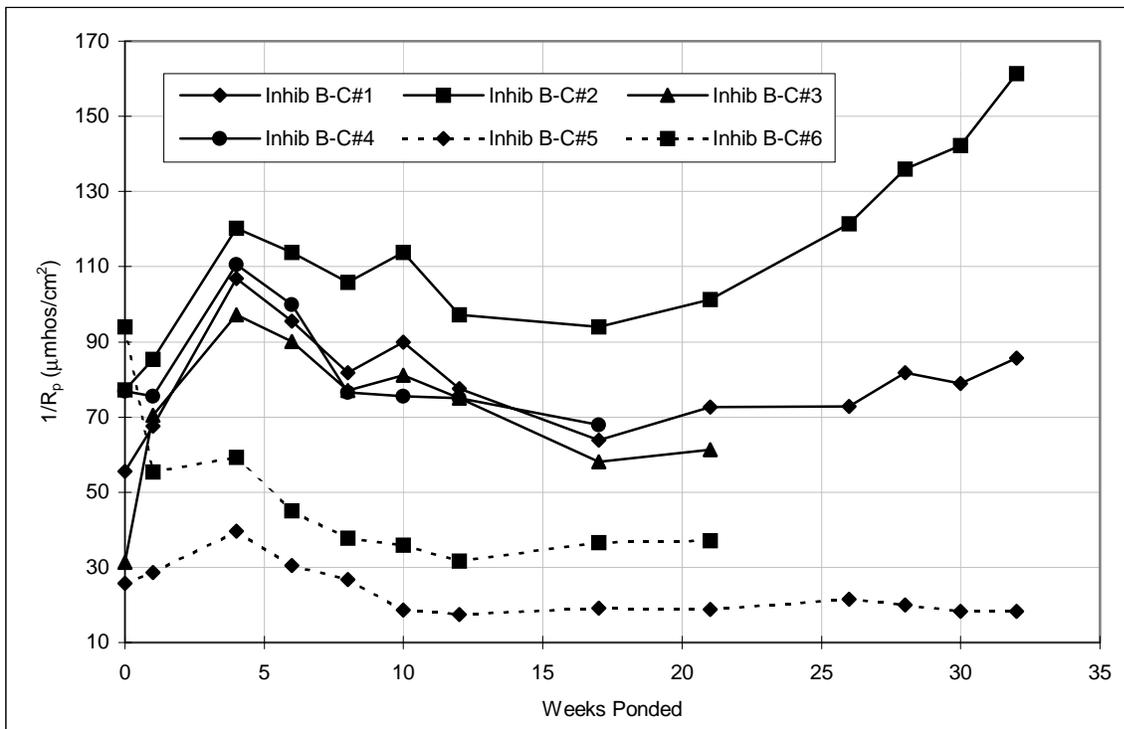


**Figure A.66  Corrosion rate, pre-cracked lollipops:  Inhibitor B.**
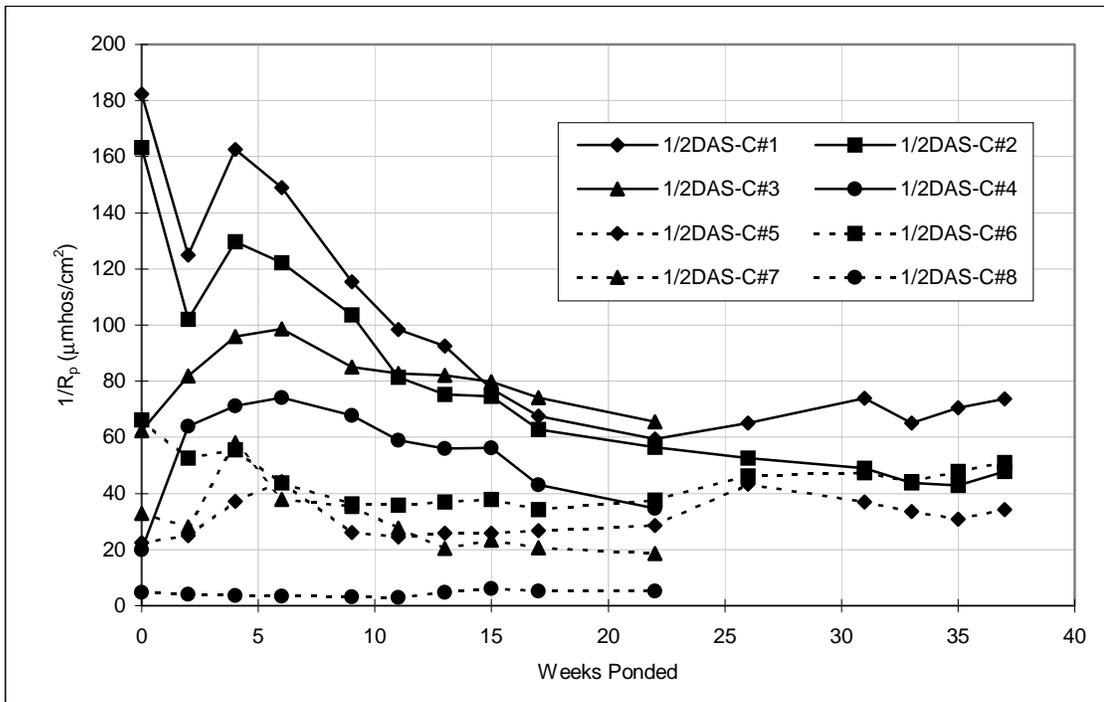
**Figure A.67 Corrosion rate, pre-cracked lollipops: ½%DAS.**
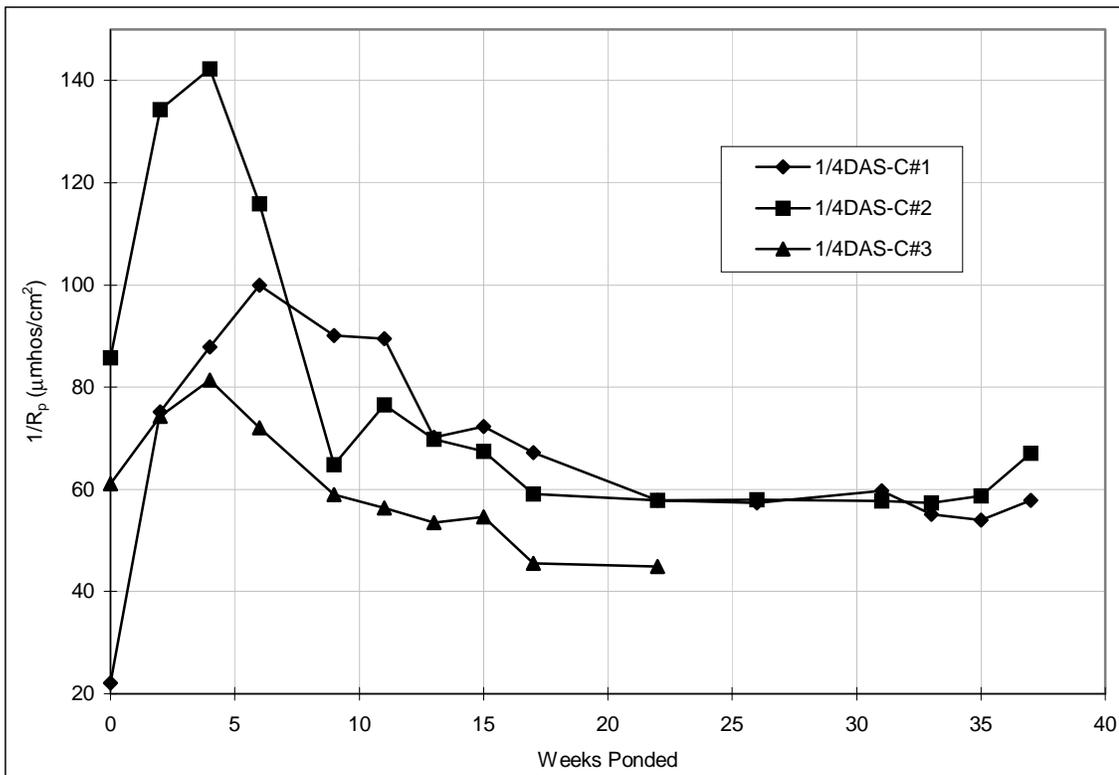


**Figure A.68 Corrosion rate, pre-cracked lollipops: ¼%DAS.**
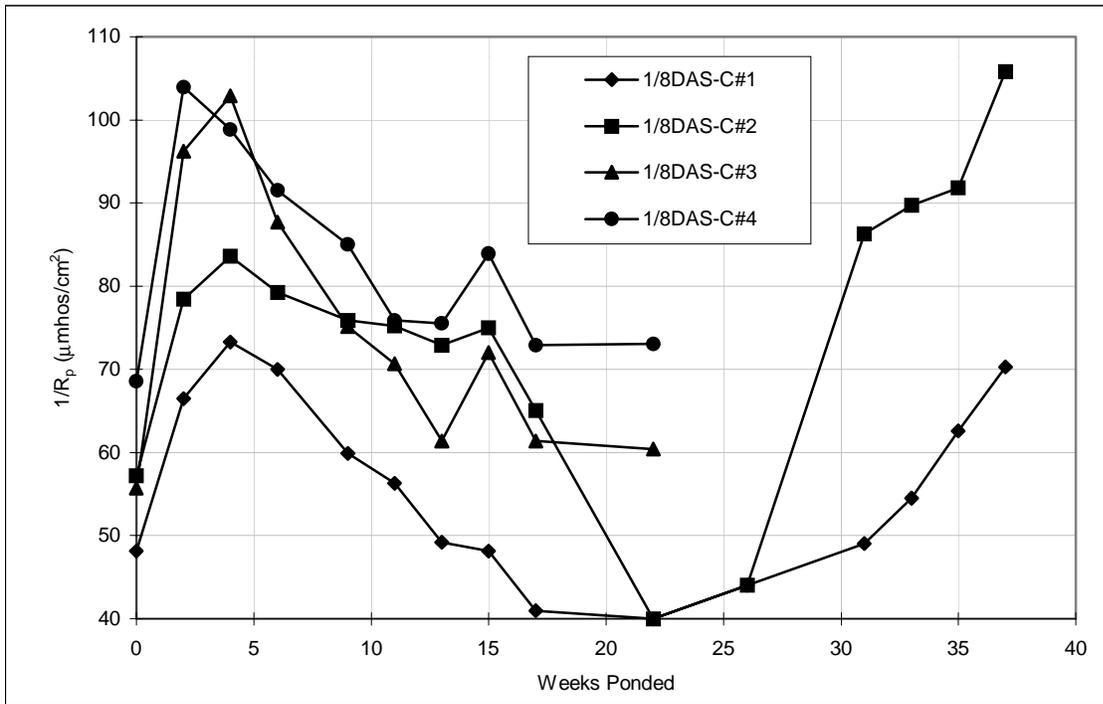
**Figure A.69  Corrosion rate, pre-cracked lollipops:  1/8%DAS.**
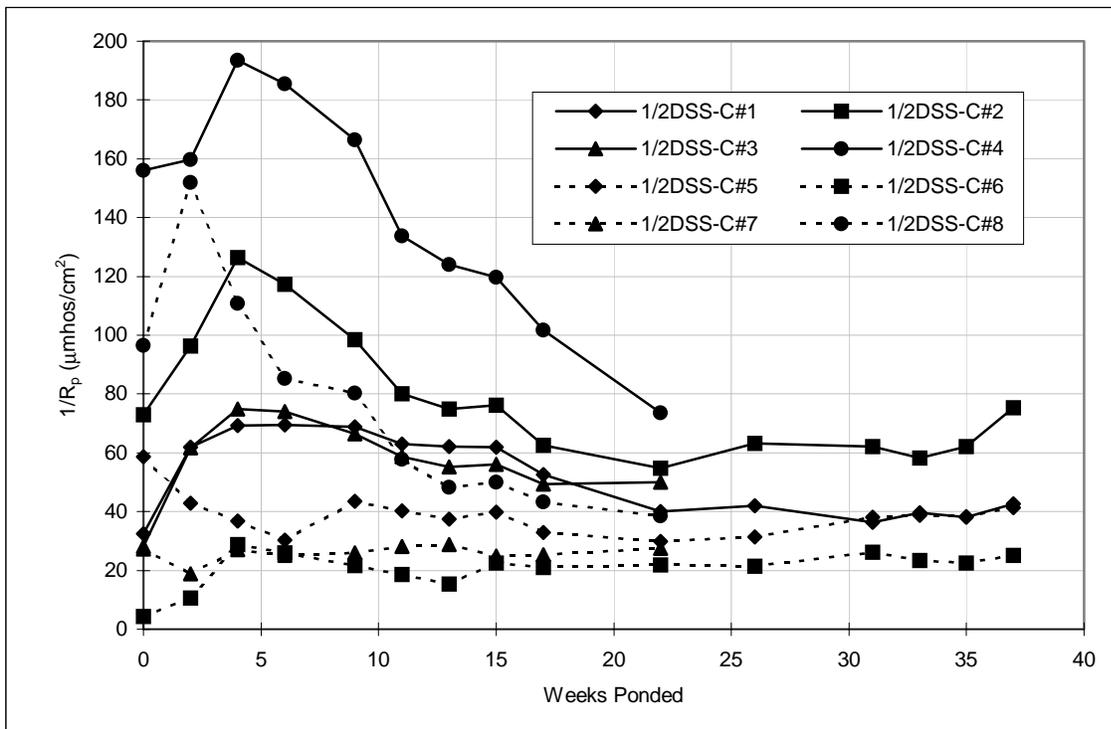


**Figure A.70  Corrosion rate, pre-cracked lollipops:  ½%DSS.**
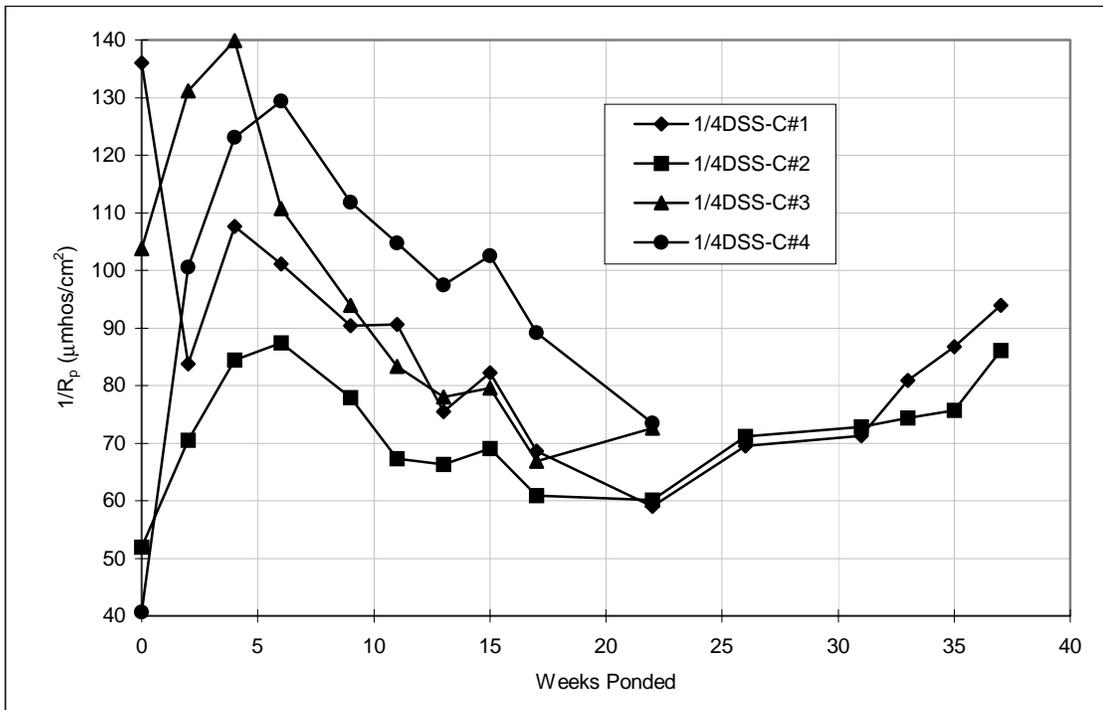
**Figure A.71 Corrosion rate, pre-cracked lollipops: ¼%DSS.**
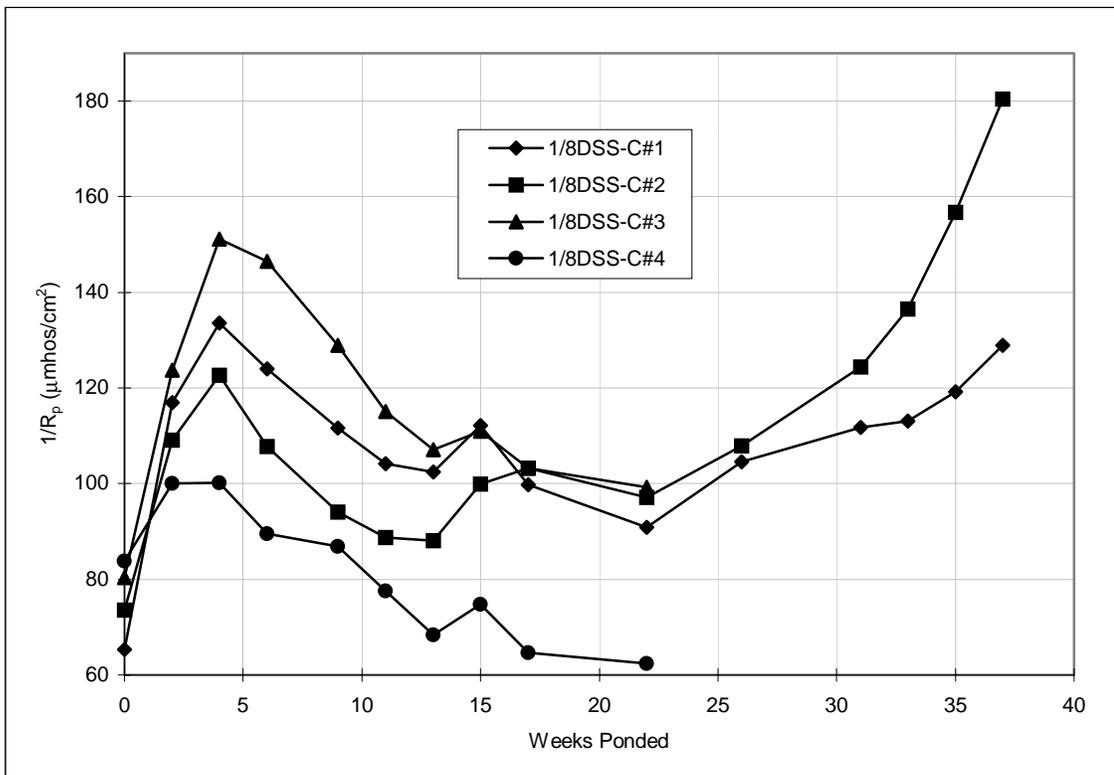


**Figure A.72 Corrosion rate, pre-cracked lollipops: 1/8%DSS.**

# APPENDIX B
# MATERIAL SAFETY DATA SHEETS

Material Safety Data Sheet  *Diammonium Salt*  **K-12 DAS**

## 1. PRODUCT IDENTIFICATION

| | |
|---|---|
| MANUFACTURER'S NAME | The Humphrey Chemical Company, Inc., A *Cambrex* Company |
| ADDRESS | 45 Devine Street North Haven, Connecticut 06473 |
| TELEPHONE | (203) 281-0012 |
| EMERGENCY TELEPHONE NUMBER | (800) 424-9300 (Chemtrec) |
| TRADE/CHEMICAL NAME | Diammonium tetrapropenyl succinate (K-12 DAS) |
| CHEMICAL FAMILY | Alkenyl succinate ammonium |
| PRODUCT USE | Concrete additive (experimental use only) |
| PIN NUMBER | None |
| WHMIS CLASS | D2B |
| ORIGINAL DATE | November 4, 1996 |
| REVISION DATE | November 4, 1996 |

## 2. COMPOSITION/INFORMATION ON COMPONENTS

| COMPONENT NAME | CAS NUMBER | WEIGHT % |
|---|---|---|
| Ammonium tetrapropenyl succinate | K-12 DAS | 100% |

## 3. HAZARDS IDENTIFICATION

**EMERGENCY OVERVIEW**
Tan transparent, near odorless liquid.

**BREATHING**
May cause some degree of respiratory tract irritation.

**SKIN CONTACT**
Not expected to be a primary skin irritant.

**EYE CONTACT**
Moderate to strong eye irritant.

**SWALLOWING**
Not expected to be toxic by ingestion.

**LONG TERM HEALTH EFFECTS**
Not known.

**CONDITIONS AGGRAVATED BY EXPOSURE**
Not known.

## 4. FIRST AID MEASURES

**BREATHING**
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Seek immediate medical attention.

**SKIN**
Wash with soap and water. Get medical attention if irritation develops or persists.

**EYE**
Immediately flush eyes with plenty of water for at least 15 minutes, holding eyelids apart. Get medical attention if irritation or other symptoms occur.

**SWALLOWING**
Get immediate medical attention. Never give anything by mouth to an unconscious person.

## 5. FIRE FIGHTING MEASURES

| | |
|---|---|
| FLASH POINT | N/A |
| METHOD | N/A |
| LOWER EXPLOSIVE LIMIT | N/A |
| UPPER EXPLOSIVE LIMIT | N/A |
| AUTO IGNITION TEMPERATURE | N/A |
| EXTINGUISHING MEDIA | $CO_2$, dry chemical, alcohol foam |

page 1 of 4 (K-12 DAS)

113

## 5. FIRE FIGHTING MEASURES ...continued

**FIREFIGHTING PROCEDURES**
Evacuate area and fight fire from safe distance. Wear pressure-demand self-contained breathing apparatus (MSHA/NIOSH-approved or equivalent) and full protective gear.

**SPECIAL FIREFIGHTING PROCEDURES**
As with any fire, toxic gases, vapors and fumes can be generated. Use pressure-demand self-contained breathing apparatus (MSHA/NIOSH approved or equivalent) and full protective gear. Using water can cause frothing with increasing fire intensity.

**UNUSUAL FIRE AND EXPLOSION HAZARDS**
None known.

**SENSITIVITY TO EXPLOSION**
None expected by mechanical impact or static discharge.

**CONDITIONS OF FLAMMABILITY**
This product is a solution of water and organic compounds. Organic residues will burn after water has evaporated.

## 6. ACCIDENTAL RELEASE MEASURES

**GENERAL**
This material should be prevented from contaminating soil or from entering sewerage and drainage systems and bodies of water. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Prevent skin and eye contact. See Section 8 Exposure Controls/Personal Protection.

**SMALL SPILL**
Absorb spill with inert material (e.g. dry sand, earth). Place in a chemical waste container.

**LARGE SPILL**
Shut off leak, if safe to do so. Clean up spills immediately, observing precautions in Protective Equipment section. Contain spilled liquid with sand or earth. Retain all contaminated water for removal and treatment.

## 7. HANDLING AND STORAGE

**HANDLING**
Use with adequate ventilation. Avoid contact with skin and eyes. Wash thoroughly after handling. Follow all MSDS/label precautions even after container is emptied because they may retain product residues.

**STORAGE**
Keep container closed when not in use.

## 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

**EXPOSURE LEVELS**
None established.

**ENGINEERING CONTROLS**
Use process enclosures, local exhaust ventilation or other engineering controls to control sources of dust, mist or vapor.

**RESPIRATORY PROTECTION**
Use a NIOSH/MSHA approved air purifying respirator as needed to control exposure. Consult with respirator manufacturer to determine respirator selection, use and limitations. Use a positive pressure air supplied respirator for uncontrolled releases or when air purifying

respirator limitations may be exceeded. Follow respiratory protection program requirements (OSHA 1910.134 and ANSI Z88.2) for all respirator use.

**EYE/FACE PROTECTION**
Use safety glasses. Where contact with the eyes is likely, use chemical goggles. Use a face shield as needed.

**SKIN PROTECTION**
Use impervious gloves. Use clean protective body-covering clothing as needed to minimize contact with clothing and skin.

## 9. PHYSICAL AND CHEMICAL PROPERTIES

| | | | |
|---|---|---|---|
| **APPEARANCE** | Tan transparent liquid | **pH** | 5-9 |
| **ODOR** | slight ammonia | **SOLUBILITY IN WATER** | Miscible |
| **INITIAL BOILING POINT** | N/A | **FREEZING/MELTING POINT** | N/A |
| **FINAL BOILING POINT** | N/A | **OCTANOL/WATER PARTITION COEFFICIENT** | N/A |
| **SPECIFIC GRAVITY**(relative to water) | N/A | **ODOR THRESHOLD** | N/A |
| **VAPOR DENSITY** (relative to air) | Heavier | **VISCOSITY** | >water |
| **VAPOR PRESSURE** (mm Hg) | N/A | **EVAPORATION RATE**(relative to n-Butylacetate) | N/A |

page 2 of 4 (K-12 DAS)

114

## 10. STABILITY AND REACTIVITY

**STABLE**
Yes

**STRONG OXIDIZER**
No

**HAZARDOUS POLYMERIZATION**
Not prone to hazardous polymerization.

**INCOMPATIBILITY**
Can react vigorously with oxidizing materials, alkali metals.

**CONDITIONS TO AVOID**
Keep from contact with oxidizing materials.

**HAZARDOUS DECOMPOSITION PRODUCTS**
Products of incomplete combustion may include $CO$, $CO_2$, $NH_3$ and dense smoke.

## 11. TOXICOLOGICAL INFORMATION

**TOXICOLOGY**
Not available.

## 12. ECOLOGICAL INFORMATION

**ECOTOXICOLOGICAL INFORMATION**
Not available.

**CHEMICAL FATE**
Not available, but slow oxidation is anticipated.

## 13. WASTE DISPOSAL

**WASTE DISPOSAL**
Dispose of in accordance with all federal, state and local regulations.

**CONTAINER DISPOSAL**
Dispose of in accordance with all federal, state and local regulations.

## 14. TRANSPORT INFORMATION

DOT Shipping Name:    :    Not  regulated  (Disodium  succinate tetrapropenyl)
Hazard Class:
Packing Group:
UN/NA No.:
DOT Labels:
Subsidiary Label:
DOT Placards:

IMO Shipping Name:      Not regulated
Hazard Class:
Packing Group:
UN No.:
IMO Labels:
Subsidiary Label:

IATA Shipping Name:      Not regulated
Hazard Class:
Packing Group:
UN No.:
IATA Labels:
Subsidiary Label:

## 15. REGULATORY INFORMATION

SARA 311/312 Chronic Health Hazard:          no
SARA 311/312 Acute Health Hazard:            yes
SARA 311/312 Fire Hazard:                    no
SARA 311/312 Sudden Pressure:                no
SARA 311/312 Reactivity Hazard:              no

**TSCA**
This substance is not listed on the public inventory under TSCA. This substance is for R&D/experimental use only and can be used only under the supervision of a technically qualified individual.

**DSL/EINECS**
This substance is not listed on the Canadian Domestic Substances List, nor is it listed on the European Inventory of Existing Commercial Chemical Substances.

**WHMIS**
This MSDS was prepared in accordance with Canadian Controlled Product Regulations. This product meets hazard class criterion D2B.

## 15. REGULATORY INFORMATION

...continued

| | | Section 302 Extremely Hazardous | | |
|---|---|---|---|---|
| Ingredients | CAS# | Weight % | TPQ |
| | | **None** | |

| | | CERCLA Hazardous Substances | | |
|---|---|---|---|---|
| Ingredients | CAS# | Weight % | RQ |
| | | **None** | |

| | | Section 313 Toxic Chemicals | |
|---|---|---|---|
| Ingredients | CAS# | Weight % |
| | | **None** | |

| | NJ Environmental Hazardous Substance List | |
|---|---|---|
| Ingredients | CAS# |
| | **None** |

| | | California Proposition 65 Ingredients | |
|---|---|---|---|
| Ingredients | CAS# | Weight % |
| | | **None** | |

## 16. OTHER INFORMATION

**NFPA HAZARD RATINGS**

| | |
|---|---|
| Health: | 2 |
| Fire: | 1 |
| Reactivity: | 0 |
| Specific Hazard: | None |

**HMIS HAZARD RATING**

| | |
|---|---|
| Health: | 2 |
| Fire: | 1 |
| Reactivity: | 0 |

**MSDS REVISIONS**

2/23/95; Added WHMIS class (sect 1); Revised SARA hazard class and added DSL, EINECS and WHMIS information (sect 15); Revised HMIS and NFPA ratings (sect 16).

**NOTE**

This information is furnished without warranty, expressed or implied, except that it is accurate to the best knowledge of The Humphrey Chemical Company, Inc. The data on this sheet relates only to the specific material designated herein. The Humphrey Chemical Company, Inc. assumes no legal responsibility for the use or reliance upon these data.

116

Material Safety Data Sheet

# K-12 DSS

## 1. PRODUCT IDENTIFICATION

| | |
|---|---|
| MANUFACTURER'S NAME | The Humphrey Chemical Company, Inc., |
| | A *Cambrex* Company |
| ADDRESS | 45 Devine Street |
| | North Haven, Connecticut 06473 |
| TELEPHONE | (203) 281-0012 |
| | |
| EMERGENCY TELEPHONE NUMBER | (800) 424-9300 (Chemtrec) |
| TRADE/CHEMICAL NAME | Disodium tetrapropenyl succinate (K-12 DSS) |
| CHEMICAL FAMILY | Alkenyl succinate sodium salt |
| PRODUCT USE | Concrete additive (experimental use only |
| PIN NUMBER | None |
| WHMIS CLASS | D2B |
| ORIGINAL DATE | October 17, 1996 |
| REVISION DATE | October 17, 1996 |

## 2. COMPOSITION/INFORMATION ON COMPONENTS

| COMPONENT NAME | CAS NUMBER | WEIGHT % |
|---|---|---|
| Disodium tetrapropenyl succinate | k-12 dss | 100% |

## 3. HAZARDS IDENTIFICATION

**EMERGENCY OVERVIEW**
Tan transparent, near odorless liquid.

**BREATHING**
May cause some degree of respiratory tract irritation.

**SKIN CONTACT**
Not expected to be a primary skin irritant.

**EYE CONTACT**
Moderate to strong eye irritant.

**SWALLOWING**
Not expected to be toxic by ingestion.

**LONG TERM HEALTH EFFECTS**
Not known.

**CONDITIONS AGGRAVATED BY EXPOSURE**
Not known.

## 4. FIRST AID MEASURES

**BREATHING**
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Seek immediate medical attention.

**SKIN**
Wash with soap and water. Get medical attention if irritation develops or persists.

**EYE**
Immediately flush eyes with plenty of water for at least 15 minutes, holding eyelids apart. Get medical attention if irritation or other symptoms occur.

**SWALLOWING**
Get immediate medical attention. Never give anything by mouth to an unconscious person.

## 5. FIRE FIGHTING MEASURES

| | |
|---|---|
| FLASH POINT | N/A |
| METHOD | N/A |
| LOWER EXPLOSIVE LIMIT | N/A |
| UPPER EXPLOSIVE LIMIT | N/A |
| AUTO IGNITION TEMPERATURE | N/A |
| EXTINGUISHING MEDIA | $CO_2$, dry chemical, alcohol foam |

page 1 of 4 (K-12 DSS)

117

## 5. FIRE FIGHTING MEASURES

**FIREFIGHTING PROCEDURES**
Evacuate area and fight fire from safe distance. Wear pressure-demand self-contained breathing apparatus (MSHA/NIOSH-approved or equivalent) and full protective gear.

**SPECIAL FIREFIGHTING PROCEDURES**
As with any fire, toxic gases, vapors and fumes can be generated. Use pressure-demand self-contained breathing apparatus (MSHA/NIOSH approved or equivalent) and full protective gear. Using water can cause frothing with increasing fire intensity.

**UNUSUAL FIRE AND EXPLOSION HAZARDS**
None known.

**SENSITIVITY TO EXPLOSION**
None expected by mechanical impact or static discharge.

**CONDITIONS OF FLAMMABILITY**
This product is a solution of water and organic compounds. Organic residues will burn after water has evaporated.

## 6. ACCIDENTAL RELEASE MEASURES

**GENERAL**
This material should be prevented from contaminating soil or from entering sewerage and drainage systems and bodies of water. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Prevent skin and eye contact. See Section 8 Exposure Controls/Personal Protection.

**SMALL SPILL**
Absorb spill with inert material (e.g. dry sand, earth). Place in a chemical waste container.

**LARGE SPILL**
Shut off leak, if safe to do so. Clean up spills immediately, observing precautions in Protective Equipment section. Contain spilled liquid with sand or earth. Retain all contaminated water for removal and treatment.

## 7. HANDLING AND STORAGE

**HANDLING**
Use with adequate ventilation. Avoid contact with skin and eyes. Wash thoroughly after handling. Follow all MSDS/label precautions even after container is emptied because they may retain product residues.

**STORAGE**
Keep container closed when not in use.

## 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

**EXPOSURE LEVELS**
None established.

**ENGINEERING CONTROLS**
Use process enclosures, local exhaust ventilation or other engineering controls to control sources of dust, mist or vapor.

**RESPIRATORY PROTECTION**
Use a NIOSH/MSHA approved air purifying respirator as needed to control exposure. Consult with respirator manufacturer to determine respirator selection, use and limitations. Use a positive pressure air supplied respirator for uncontrolled releases or when air purifying

respirator limitations may be exceeded. Follow respiratory protection program requirements (OSHA 1910.134 and ANSI Z88.2) for all respirator use.

**EYE/FACE PROTECTION**
Use safety glasses. Where contact with the eyes is likely, use chemical goggles. Use a face shield as needed.

**SKIN PROTECTION**
Use impervious gloves. Use clean protective body-covering clothing as needed to minimize contact with clothing and skin.

## 9. PHYSICAL AND CHEMICAL PROPERTIES

| | | | |
|---|---|---|---|
| APPEARANCE | Tan transparent liquid | pH | 5-9 |
| ODOR | Near odorless | SOLUBILITY IN WATER | Miscible |
| INITIAL BOILING POINT | N/A | FREEZING/MELTING POINT | N/A |
| FINAL BOILING POINT | N/A | OCTANOL/WATER PARTITION COEFFICIENT | N/A |
| SPECIFIC GRAVITY(relative to water) | N/A | ODOR THRESHOLD | N/A |
| VAPOR DENSITY (relative to air) | Heavier | VISCOSITY | >water |
| VAPOR PRESSURE (mm Hg) | N/A | EVAPORATION RATE(relative to n-Butylacetate) | N/A |

page 2 of 4 (K-12 DSS)

118

## 10. STABILITY AND REACTIVITY

**STABLE**
Yes

**STRONG OXIDIZER**
No

**HAZARDOUS POLYMERIZATION**
Not prone to hazardous polymerization.

**INCOMPATIBILITY**
Can react vigorously with oxidizing materials, alkali metals and amines.

**CONDITIONS TO AVOID**
Keep from contact with oxidizing materials.

**HAZARDOUS DECOMPOSITION PRODUCTS**
Products of incomplete combustion may include CO, $CO_2$, dense smoke.

## 11. TOXICOLOGICAL INFORMATION

**TOXICOLOGY**
Not available.

## 12. ECOLOGICAL INFORMATION

**ECOTOXICOLOGICAL INFORMATION**
Not available.

**CHEMICAL FATE**
Not available, but slow oxidation is anticipated.

## 13. WASTE DISPOSAL

**WASTE DISPOSAL**
Dispose of in accordance with all federal, state and local regulations.

**CONTAINER DISPOSAL**
Dispose of in accordance with all federal, state and local regulations.

## 14. TRANSPORT INFORMATION

**DOT Shipping Name:** Not regulated (Disodium succinate tetrapropenyl)
**Hazard Class:**
**Packing Group:**
**UN/NA No.:**
**DOT Labels:**
**Subsidiary Label:**
**DOT Placards:**

**IMO Shipping Name:** Not regulated
**Hazard Class:**
**Packing Group:**
**UN No.:**
**IMO Labels:**
**Subsidiary Label:**

**IATA Shipping Name:** Not regulated
**Hazard Class:**
**Packing Group:**
**UN No.:**
**IATA Labels:**
**Subsidiary Label:**

## 15. REGULATORY INFORMATION

SARA 311/312 Chronic Health Hazard: no
SARA 311/312 Acute Health Hazard: yes
SARA 311/312 Fire Hazard: no
SARA 311/312 Sudden Pressure: no
SARA 311/312 Reactivity Hazard: no

**TSCA**
This substance is not listed on the public inventory under TSCA. This substance is for R&D/experimental use only and can be used only under the supervision of a technically qualified individual.

**DSL/EINECS**
This substance is not listed on the Canadian Domestic Substances List, nor is it listed on the European Inventory of Existing Commercial Chemical Substances.

**WHMIS**
This MSDS was prepared in accordance with Canadian Controlled Product Regulations. This product meets hazard class criterion D2B.

## 15. REGULATORY INFORMATION
... continued

**Section 302 Extremely Hazardous**

| Ingredients | CAS# | Weight % | TPQ |
|---|---|---|---|
| | **None** | | |

**CERCLA Hazardous Substances**

| Ingredients | CAS# | Weight % | RQ |
|---|---|---|---|
| | **None** | | |

**Section 313 Toxic Chemicals**

| Ingredients | CAS# | Weight % |
|---|---|---|
| | **None** | |

**NJ Environmental Hazardous Substance List**

| Ingredients | CAS# |
|---|---|
| | **None** |

**California Proposition 65 Ingredients**

| Ingredients | CAS# | Weight % |
|---|---|---|
| | **None** | |

## 16. OTHER INFORMATION

**NFPA HAZARD RATINGS**

| | |
|---|---|
| Health: | 2 |
| Fire: | 1 |
| Reactivity: | 0 |
| Specific Hazard: | None |

**HMIS HAZARD RATING**

| | |
|---|---|
| Health: | 2 |
| Fire: | 1 |
| Reactivity: | 0 |

**MSDS REVISIONS**
2/23/95; Added WHMIS class (sect 1); Revised SARA hazard class and added DSL, EINECS and WHMIS information (sect 15); Revised HMIS and NFPA ratings (sect 16).

**NOTE**
This information is furnished without warranty, expressed or implied, except that it is accurate to the best knowledge of The Humphrey Chemical Company, Inc. The data on this sheet relates only to the specific material designated herein. The Humphrey Chemical Company, Inc. assumes no legal responsibility for the use or reliance upon these data.

120

# REFERENCES

[1]     Walker, H. C., "Parking Structure Durability," *Concrete International*, Vol. 20, No.7, July, 1998, pp. 53-54.

[2]     McDonald, D. B., "Design Options for Corrosion Protection," *Concrete 95 Toward Better Concrete Structures,* Brisbane, Australia, 4-7 September 1995, pp.1-9.

[3]     Clear, K. C., Hartt, W. H., McIntyre, J., Lee, S. K., "Performance of Epoxy-Coated Reinforcing Steel in Highway Bridges," National Cooperation Highway Research Program Report No. 370, Transportation Research Board, 1995.

[4]     Berke, N. S.  "Corrosion Inhibitors in Concrete," *Concrete International*, Vol. 13, No. 7, July 1991, pp. 24-27.

[5]     Craig, R. J., Wood, L. E., "Effectiveness of Corrosion Inhibitors and Their Influence on the Physical Properties of Portland Cement Mortars," *Highway Research Record*, No. 328, 1970, pp. 77-88.

[6]     Trepanier, S. M., "Effectiveness of Corrosion Inhibitors in Reinforced Concrete," *Masters Thesis - Queen's University*, Kingston, Ontario, Canada, April, 1994.

[7]     Fontana, M. G., *Corrosion Engineering*, 3rd Ed., McGraw Hill, Inc., New York, NY, 1986.

[8]     Scannell, W. T., Sohanghpurwala, A. A., Islam, M., "Assessment of Physical Conditions of Concrete Bridge Components," *FHWA - SHRP Showcase*, US Department of Transportation Federal Highway Administration, July, 1996.

[9]     Lorentz, T., French, C., Leon, R. T., "Corrosion of Coated and Uncoated Reinforcing Steel in Concrete," *Structural Engineering Report No. 92-03*, University of Minnesota Center of Transportation Studies, May, 1992.

[10]    Uhlig, H. H., Revie, R. W., *Corrosion and Corrosion Control - An Introduction to Corrosion Science and Engineering*, 3rd Ed., Wiley & Sons, NewYork, 1985.

[11]    Kruger, J., "The Nature of the Passive Film on Iron and Ferrous alloys," *Corrosion Science*, Vol. 29, No. 2/3, pp. 149-162, 1989

[12]    Toney, M. F., Davenport, A. J., Oblonsky, L. J., Ryan, M. P., Viturs, C. M., "Atomic Structure of the Passive Oxide Film Formed on Iron," *Physical Review Letters*, Vol. 79, No. 21, pp. 4282-4285, 1997.

[13]    Locke, C. E., "Corrosion of Steel in Portland Cement Concrete: Fundamental Studies," *ASTM Special Technical Publication- Symposium on Corrosion Effects of Stray Currents and The Techniques For Evaluating Corrosion of Rebars in Concrete*, STP 906, Nov. 28, 1984, Willamsburg, VA, pp. 5-13.

[14]    Zhang, H., Wheat, H. G., Sennour, M. L., Carrasquillo, R. L., "Corrosion of Steel Bars in Concrete Containing Different Chemical and Mineral Admixtures," *Material Performance*, December 1992, pp. 37-43.

[15]    Borgard, B., Warren, C., Somayaji, S., Heidersbach, R., "Mechanisms of Corrosion of Steel in Concrete," *ASTM Special Technical Publication Symposium on Corrosion Rates of Steel in Concrete*, STP 1065, August 1990, Baltimore, MD, pp. 174-187.

[16]    Berke, N. S., Stark, P., "Calcium Nitrate As An Inhibitor: Evaluation And Testing For Corrosion Resistance," *Concrete International*, September 1985, pp. 42-47.

[17]    Cho, H., Lee, H., Hansen, C., "Laboratory Experiment to Measure Corrosion of Rebar Embedded in Concrete with Corrosion Inhibiting Admixtures," *Transportation Research Board 76 Annual Meeting*, Washington, DC, Jan. 12-16, 1997, Paper No. 97-1304.

[18]    Pfeifer, D. W., Landgren, J. R., Zoob, A., "Protective Systems for New Prestressed and Substructure Concrete," *Report No. FHWA-RD-86-193*, Federal Highway Administration, April 1987.

[19]    Clear, K. C., "Time-To-Corrosion of Reinforcing Steel in Concrete Slabs," *Report No. FHWA-RD-76-70*, Federal Highway Administration, April 1976.

[20]    ASTM C 876 - 91, "Standard Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete," *Annual Book of ASTM Standards*, 1991, pp. 434-439.

[21]    Pfeifer, D. W., Landgren, J. R., Krauss, P. D., "Investigation For CRSI on CRSI-Sponsored Corrosion Studies At Kenneth C. Clear Inc., WJE No. 911461," *CRSI Performance Research: Epoxy Coated Reinforcing Steel*, CRSI Final Report - June, 1992.

[22]    ASTM G-109- 92, "Standard Test Method for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to Chloride Environments," *Annual Book of ASTM Standards*, 1992, pp. 452-455.

[23]   Nami, C. K., Farrington, S. A., Bobrowski, G. S., "OrganicBased Corrosion Inhibiting Admixture for Reinforced Concrete," *Concrete International*, Vol. 14, April 1992, pp. 45-51.

[24]   Stern, M., "A Method For Determining Corrosion Rates From Linear Polarization Data," *Corrosion*, Vol. 14, September 1958, pp. 440t-444t.

[25]   Hack, H. P., Moran, P. J., Scully, J. R., "Influence of Electrolyte Resistance on Electrochemical Measurements and Procedures to Minimize or Compensate for Resistance Errors," *ASTM Special Technical Publication Symposium - The Measurements and Correction of Electrolyte Resistance in Electrochemical Tests*, STP 1056, May, 1988, Baltimore, MD, pp. 5-26.

[26]   EG&G Princeton Applied Research, *Model 273A Potentiostat/Galvanostat User's Guide*, 1993.

[27]   Ehrhart, W. C., "IR Drop in Electrochemical Corrosion Studies - Part 2: A Multiple Method IR Compensation System," *ASTM Special Technical Publication Symposium - TheMeasurements and Correction of Electrolyte Resistance in Electrochemical Tests*, STP 1056, May, 1988, Baltimore, MD, pp. 78-94.

[28]   McDonald, D. B., Pfeifer, D. W., Krauss, P. D., Sherman, M. R., "Test Methods For New Breeds of Reinforcing Bars," *Corrosion and Corrosion Protection of Steel in Concrete, Proceedings of International Conference held at the University of Sheffield*, 24 - 28 July, 1994, pp. 1155-1171.

[29]   Taylor, S. R., Mason, S. E., Cella, P. A., Clemena, G. G., "An Investigation of New Inhibitors to Mitigate Rebar Corrosion in Concrete," *Report No. FHWA/VA-96-R24,* Virginia Transportation Research Council, April 1996.

[30]   Berke, N. S., Stark, P., "Calcium Nitrate As An Inhibitor: Evaluation And Testing For Corrosion Resistance," *Concrete International*, September 1985, pp. 42-47.

[31]   Berke, N. S., Shen, D. F., Sundberg, K. M., "Comparison of the Polarization Resistance Technique to the Macrocell Corrosion Technique," *ASTM Special Technical Publication Symposium on Corrosion Rates of Steel in Concrete*, June 28, 1988 n 1065 August 1990, Baltimore, MD, pp. 38-51.

[32]   Nmai, C. K., Bury, M. A., Farzam, H., "Corrosion Evaluation of a Sodium Thiocyanate-Based Admixture," *Concrete International*, Vol. 16, April 1994, pp. 22-25.

[33]    Gu, P., Elliot, S., Hristova, R., Beaudion, J. J., Brousseau, R., Baldock, B., "A Study of Corrosion Inhibitor Performance in Chloride Contaminated Concrete by Electrochemical Impedance Spectroscopy," *ACI Material Journal*, Sept.-Oct. 1997.

[34]    McDonald, D. B., Pfeifer, D. W., Krauss, P. D., "The Corrosion Performance of Reinforcing Steel in Concretes Containing Calcium Nitrate, Rheocrete 222 and Rheocrete 222+ During 48-weeks of In-Concrete Testing for Master Builders, Inc," *WJE Report No. 951286*, Wiss, Janney, Elstner Associated, Inc, July 1997.

[35]    Tourney, P., Berke, N. S., "A Call for Standardized Tests for Corrosion Inhibiting Admixtures," *Concrete International*, April 1993, pp. 57-62.

[36]    Gannon, E. J., Cady, P. D., "Condition Evaluation of Concrete Bridges Relative to Reinforcement Corrosion - Vol. 1: State-of-the-Art of Existing Methods," *SHRP - S-323*, Strategic Highway Research Council, Washington, DC, 1993.

[37]    Cady, P. D., Flis, J., Sehgal, A., Li, D., Kho, Y. T., Sabol, S., Pickering, H., Osseo-Asare, K., "Condition Evaluation of Concrete Bridges Relative to Reinforcement Corrosion - Vol. 2: Method for Measuring the Corrosion Rate of Reinforcing Steel," *SHRP -S-324*, Strategic Highway Research Council, Washington, DC, 1992.

[38]    Gannon, E. J., Cady, P. D., "Condition Evaluation of Concrete Bridges Relative to Reinforcement Corrosion - Vol. 8: Procedure Manual," *SHRP -S-330*, Strategic Highway Research Council, Washington, DC, 1992.

[39]    State of Connecticut, Department of Transportation, *Standard Specification for Roads, Bridges and Incidental Construction, Form 814*, 1988.

[40]    Portland Cement Association, *Design and Control of Concrete Mixtures*, 13th Ed., Kosmatka, S. H., Panarese, W. C.,  Editors, 1988.

[41]    State of Connecticut, Department of Transportation, *Bridge Design Manual,* 2nd Ed., 1985 with Revision 3 Dated 10/89.

[42]    Shigen, L., Gressert, D. G., Frantz, G. C., Stephens, J. E., "Durability and Bond of High Performance Concrete and Repaired Portland Cement Concrete," *Report No. JHR 97-257*, Joint Highway Research Council of the University of Connecticut and the Connecticut Department of Transportation, June, 1997.

[43]    ASTM C 260 - 94  "Standard Test Method for Air-Entraining Admixtures for Concrete," *Annual Book of ASTM Standards*, 1994, pp. 156-158.

[44]     ASTM C 143 - 90a  "Standard Test Method for Slump of Hydraulic Cement Concrete," *Annual Book of ASTM Standards*, 1990, pp. 88-90.

[45]     ASTM C 231 - 91b  "Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method," *Annual Book of ASTM Standards*, 1991, pp. 134-141.

[46]     Berke, N. S., Shen, D. F., Sundberg, K. M., "Comparison of the Current Interruption and Electrochemical Impedance Techniques in the Determination of Corrosion Rates of Steel in Concrete," *ASTM Special Technical Publication Symposium - The Measurements and Correction of Electrolyte Resistance in Electrochemical Tests*, STP 1056, May, 1988, Baltimore, MD, pp. 191-201.

[47]     Ahmad, S. H., Zia, P., "High Performance Concretes for Highway Applications," *International Workshop on High Performance Concrete- ACI SP-159*, Bangkok, Thailand, November 1994, pp. 335-349.

[48]     Allyn, M., "Corrosion of Steel Reinforcement Embedded in Concrete:  The Evaluation of Two Prototype Concrete Corrosion Inhibitors," Master of Science thesis, The University of Connecticut, Storrs, CT, 1998.

[49]     ASTM C 403 / C 403M – 95  "Standard Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance," *Annual Book of ASTM Standards*, 1995, pp. 217-221.

[50]     Murray, V. E., "Tests of Prestressed Concrete Bridge Beams, Part II – Chloride Penetration in the Bridge Beams," *Report No. JHR 91-198b*, Joint Highway Research Council of the University of Connecticut and the Connecticut Department of Transportation, 1987.

[51]     Popov, Branko N., "Evaluation of Economical and Efficient Method for Corrosion Protection of Concrete," *Report No. FHWA-SC-98-01*, Center for Electrochemical Engineering, Department of Chemical Engineering, University of South Carolina, September, 1998.

[52]     Goodwin, P. D., "Corrosion of Steel Reinforcement Embedded in Concrete:  The Evaluation of Two Prototype Concrete Corrosion Inhibitors – Phase II," Master of Science thesis, The University of Connecticut, Storrs, CT, 2000.