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**SUBJECT:** Evaluation of Doped Polyaniline as a Carbon Steel Protective Coating Using Electrochemical Impedance Spectroscopy

**RELATED DOCUMENTATION:** DM-MSL Report 91-3436  
DM-MSL Report 94-2112  
DM-MSL Report 94-2028  
LO-MSD Report 94-2082

## 1.0 SUMMARY

Electrochemical Impedance Spectroscopy (EIS) was used to evaluate the performance of two doped polyanilines (PAN's) in the emeraldine base form (EB) as protective coatings for carbon steel under immersion in a 3.55% sodium chloride (NaCl) solution. Coatings A and B consisted of EB doped with tetracyanoethylene (TCE) and with p-toluenesulfonic acid (PTSA), respectively. Corrosion potential as well as Bode and Nyquist plots of the data were obtained for each coating after various immersion times. The equivalent circuit  $R_c(C_c [R_c(QR_a)])$  provided a satisfactory fit for the EIS data. The variation with immersion time of the equivalent circuit parameters  $R_c$  and  $C_c$  was recorded and analyzed. Both coatings exhibited similar behavior except for an initial decrease in  $C_c$  observed for coating B. The results showed that EIS can be used as a technique to evaluate the progress in the development and optimization of conductive polymers as protective coatings for carbon steel.

## 2.0 FOREWORD

2.1 Electrically conductive polymers have attracted a great deal of interest since their discovery about two decades ago. Soon after their discovery, it became clear that their unique properties could be used in several technological applications, such as the development of a new class of superconductors, light-emitting plastics, polymer-based switching devices, and sensing devices. The sheer volume of fundamental and applied research in this field makes it inevitable that conductive polymers will find an increasing range of applications.<sup>1</sup>

- 2.2 PAN has attracted much attention as a unique electrically conductive polymer.<sup>2</sup> Many publications revealed that this material exhibits unusual electrical and optical properties. The reversibility of these properties, combined with its good environmental stability and its low cost of production, makes this polymer suitable for the development of the aforementioned technological applications.<sup>3</sup> Another possible application of PAN involves its use in protecting metals and semiconductors from corrosion.<sup>4,5</sup> Investigations aimed at following the improvement of this application are justified.
- 2.3 In the mid 1980's, researchers at the Kennedy Space Center (KSC) in Florida became interested in PAN's as protective coatings for metallic surfaces. Research has been ongoing for over 20 years at KSC to find coating materials to protect launch site structures and equipment from the extremely corrosive conditions present at the launch complexes. The combination of proximity to the Atlantic Ocean and acidic combustion products from solid rocket boosters results in corrosive stresses unique to KSC.
- 2.4 Extensive coating testing at KSC lead to the conclusion that inorganic zinc-rich primers (ZRP's) significantly outperformed organic ZRP in the marine atmosphere of Florida. This was partially attributed to the increased conductivity of the inorganic ZRP coating film. The materials typically used to produce the organic zinc-rich films (e.g., epoxies, vinyls, etc.) produced an undesirable insulating effect on the zinc particles. This effect resulted in decreased galvanic activity of the zinc particles for protection of the carbon steel substrate. On the other hand, the organic ZRP had one advantage in that they allowed for less than perfect surface preparation on steel to achieve performance. The organic polymers provided better adhesion to marginally prepared substrates than the inorganic materials. This result led researchers at KSC to ask the question: "Why don't we use conductive organic materials to formulate these zinc coatings to get the best of both worlds?" The idea being that the conductive organic vehicle would provide both the increased conductivity needed for superior galvanic protection of the steel substrate and would allow better adhesion to less than perfect surface preparation. Hence the work on conductive organic polymers and the search for materials that would allow the production of a new generation of protective coatings based on this technology began.
- 2.5 The main object of this work was to use EIS to study the performance of a recently developed PAN in two doped forms as corrosion protective coatings for carbon steel immersed in a 3.55% NaCl solution.

### 3.0 MATERIALS AND METHODS

- 3.1 Specimens were flat steel coupons 1.59 cm (5/8 in) coated on one side by spraying a 5% EB solution in n-methylpyrrolidone (NMP) about 900 times in 25 spray increments using a Badger airbrush. The samples were dried between sprayings

by placing them in an 86 °C oven. Each was dried for 12 hours at 86 °C and the thickness of the coating was determined to be approximately 3 mils (25.4 μm) thick. The EB has an inherent viscosity measurement in concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) of 0.66 dL/g at 30 °C. Two types of coated samples were prepared by doping the EB coated coupons with either a 0.1 M tetrahydrofuran (THF) solution of TCE at 0 °C (coating A) or 0.1 M PTSA at room temperature (coating B) for 24 hours. The samples were removed from solution, rinsed with THF, and air dried. The flat specimen holder in the electrochemical cell is designed such that the exposed surface area is 1 cm<sup>2</sup>.

- 3.2 A Model 378 Electrochemical Impedance system manufactured by EG&G Princeton Applied Research Corporation was used for all EIS measurements. The system includes: (1) the Model 273 Computer-Controlled Potentiostat/Galvanostat; (2) the Model 5210 Computer-Controlled Lock-In Amplifier; (3) the Model M388 Electrochemical Impedance Software including circuit modeling routines. Data were gathered in the frequency range from 100 kHz to 0.01 Hz. Three experiments were performed in a sequence covering the entire frequency range, and the data were automatically merged and saved. The frequency ranges for the three experiments were 100 kHz to 5 Hz, 10 Hz to 0.1 Hz, and 0.1 Hz to 0.01 Hz. The AC amplitude used for the experiments was 10 mV.
- 3.3 The electrochemical cell included a saturated calomel reference electrode (SCE), two graphite rod counter electrodes, the sample working electrode, and a bubbler/vent tube. Each sample was studied at different immersion times in an aerated 3.55% NaCl solution. All solutions were prepared using deionized water. Aeration with dry air was maintained throughout the test. The corrosion potential, Bode magnitude (showing the logarithm of the modulus of the impedance,  $\log |Z|$ , as a function of the logarithm of frequency), and Nyquist plots (showing the negative of the imaginary impedance as a function of real impedance) of the data were obtained for each coating after various immersion times. The impedance data were analyzed using the Equivalent Circuit computer simulation program by B. A. Boukamp.<sup>6</sup>

#### 4.0 RESULTS AND DISCUSSION

- 4.1 The variation of the corrosion potential with immersion time in a 3.55% NaCl solution for the two coatings included in this investigation is presented in Figure 1. Assuming that these coatings may display a mechanism of corrosion protection involving a galvanic as well as a barrier effect,<sup>7,8</sup> the change in the corrosion potential with immersion time is a useful indicator of the galvanic mechanism of protection for the coating. The corrosion potential of steel in a 3.55% NaCl solution quoted in various standards and codes of practice is -0.780 V versus SCE.<sup>9</sup> Below this potential, the corrosion of the steel is reduced. Coating B does not achieve this potential at all.

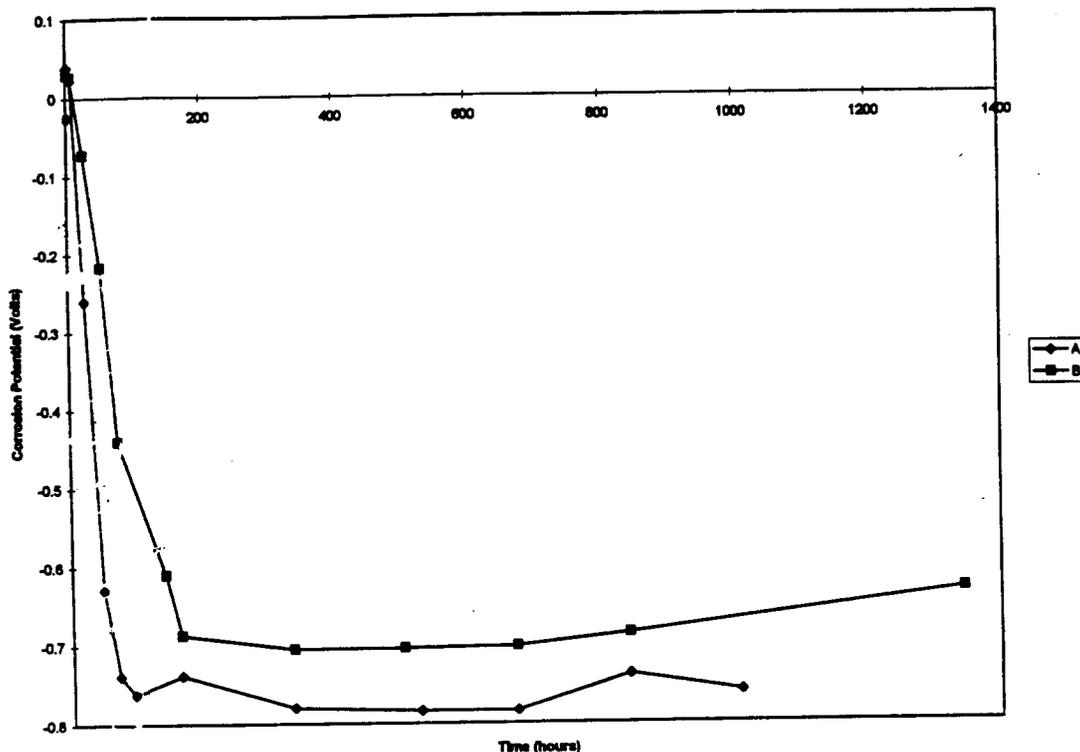


Figure 1. - Corrosion potential for PAN's as a function of immersion time in 3.55% NaCl

Coating A requires 336 hours of immersion to reach this potential, and then never drops below this value. Y. Wei *et al.*<sup>10</sup> have recently reported similar trends in the corrosion potential of hydrochloric acid-doped PAN coated on carbon steel under conditions similar to those used in this investigation. The corrosion potential results suggest that the rest potential of the conductive films makes them more noble than the steel they are expected to protect. Further development is needed to reduce the rest potential of the films below that of carbon steel in order to improve their galvanic mechanism of protection.

4.2 Bode magnitude plots for coating A are shown in Figures 2 and 3 for selected immersion times. The resistive region of the plots (characterized by a plateau) shows that the impedance increases during the first 24 hours of immersion in the electrolyte (Figure 2) before it starts to decrease (Figure 3). Coating B exhibited similar behavior, with the difference that the decrease in the impedance was observed after 72 hours of immersion. Bode magnitude plots at all the immersion times included in this investigation are shown in appendixes A and B for coatings A and B, respectively. The decrease in impedance is an indication of the deterioration of the protective qualities of the film. Visual observation revealed the appearance of blisters on both films after 48 hours of immersion.

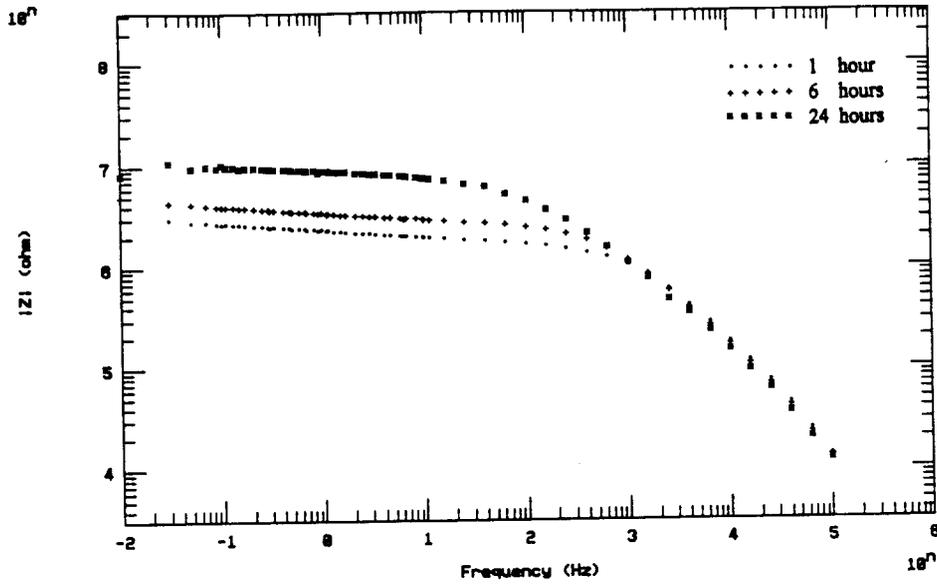


Figure 2 - Bode magnitude plots for coating A at selected immersion times in 3.55% NaCl

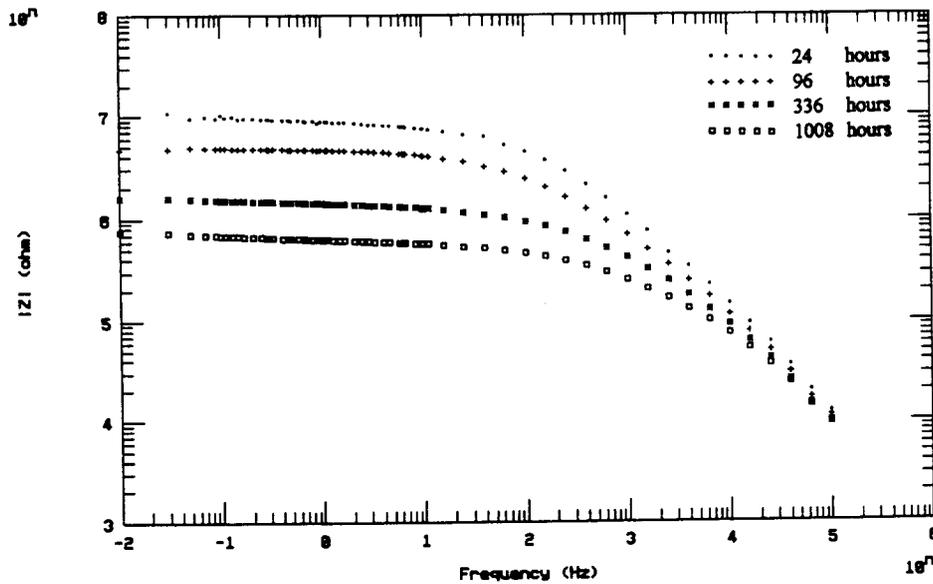


Figure 3 - Bode magnitude plots for coating A at selected immersion times in 3.55% NaCl

4.3 The Nyquist plots for coatings A and B were similar to those obtained in this laboratory for ZRP's<sup>11-13</sup> and to those reported by other authors for organic coatings on steel substrates.<sup>14</sup> Coatings A and B exhibited Nyquist plots with a semicircle in the high frequency region and what appears to be a diffusion tail (or the beginning of a second semicircle) in the low frequency region during the early immersion times (Figures 4 and 5). The low frequency tail was not observed for coating A between the 24 and 168 hours of immersion (Figure 6), nor for coating B between the 72 and 336 hours of immersion. Nyquist plots at all the immersion

times included in this investigation are shown in appendixes A and B for coatings A and B, respectively. A single Nyquist response is expected as defects that develop after immersion become blocked with corrosion products.<sup>14</sup> The change of impedance data as a function of immersion time can be used in the future to further identify the mechanisms by which conductive polymers exert their corrosion protective effect.

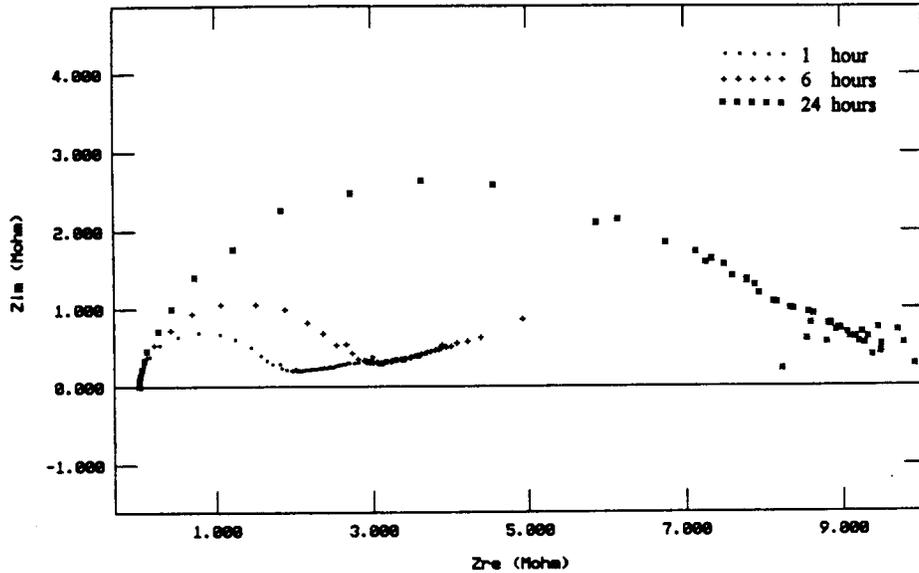


Figure 4 - Nyquist plots for coating A at selected immersion times in 3.55% NaCl

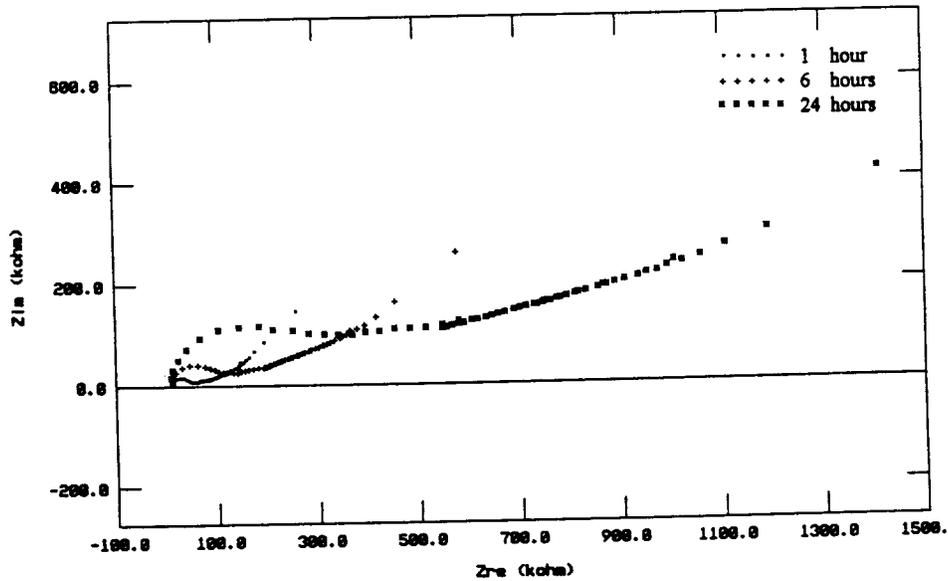


Figure 5 - Nyquist plots for coating B at selected immersion times in 3.55% NaCl

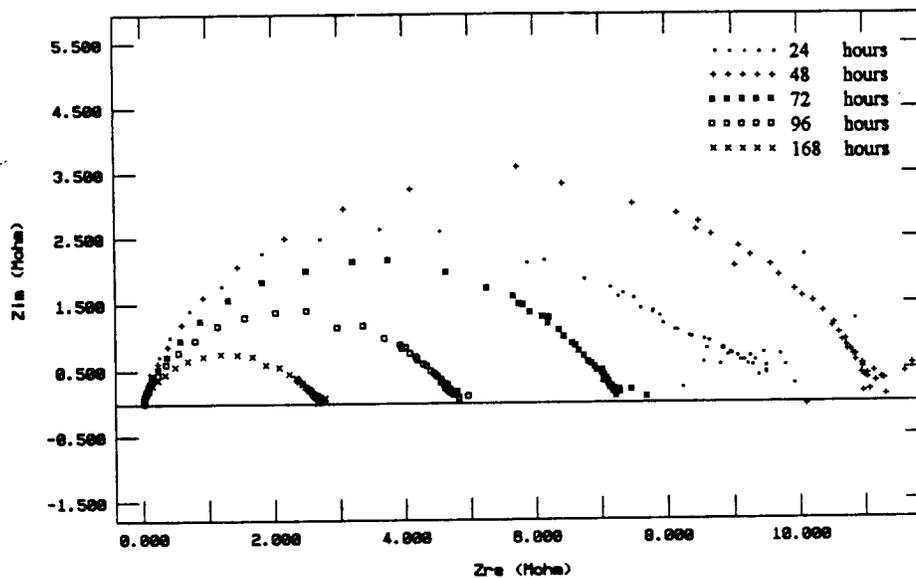


Figure 6 - Nyquist plots for coating A at selected immersion times in 3.55% NaCl

4.4 To extract information from the EIS data generated in this investigation, the impedance spectra were analyzed using a computer simulation program.<sup>6</sup> The Equivalent Circuit program allows the design of a theoretical model of the impedance experiment by mathematically simulating an electronic network. The program generates a theoretical impedance plot for the equivalent circuit designed, overlays this plot on the experimental results, performs a statistical analysis of the fit of the model to the data, and automatically adjusts the component values in the model to optimize its fit to the data. The equivalent circuit  $R_e(C_c [R_c(QR_a)])$  shown in Figure 7 provided a satisfactory fit for the data (see simulation curves in Figures 8 and 9). The letters R, C, and Q denote a resistor, capacitor, and a constant phase element (CPE), respectively. The CPE is described by the following equation:

$$Y = Y_o(j\omega)^n$$

where Y represents the admittance (the inverse of the impedance, Z), n determines the dependence of Y on the frequency f,  $Y_o$  is the CPE constant, and j is the imaginary number ( $j^2 = -1$ ). The CPE represents a resistance for  $n = 0$ , a capacitance for  $n = 1$ , a Warburg diffusion for  $n = 0.5$ , and an induction for  $n = -1$ . The physical meaning of a CPE has been discussed by many authors and related to surface heterogeneity.<sup>15,16</sup>  $R_e$  represents the electrolyte resistance,  $C_c$  is the capacitance of the coating, and  $R_c$  is the resistance of the coating. The impedance of the parallel arrangement ( $R_a$  and Q) has been related to the faradaic reaction at the metal-solution interface. The contribution to the impedance of the system by these terms is currently unknown.

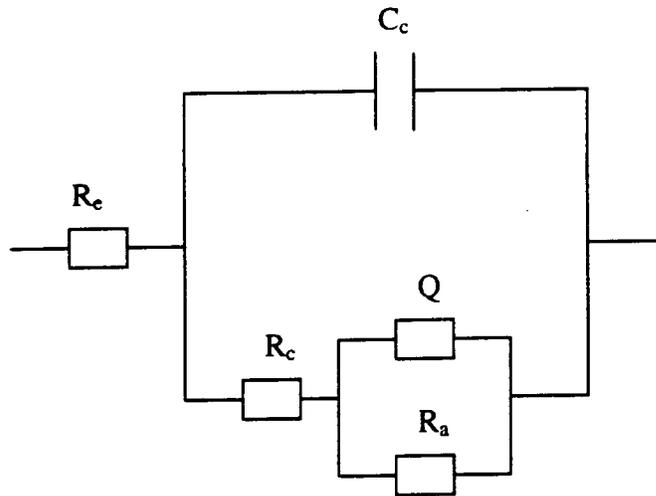


Figure 7 - Equivalent circuit for doped polyanilines

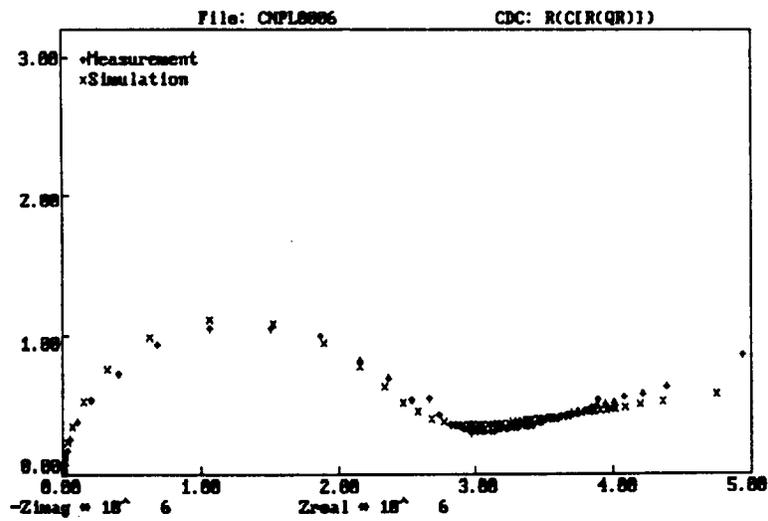


Figure 8 - Nyquist plot for coating A after 6 hours of immersion in 3.55% NaCl

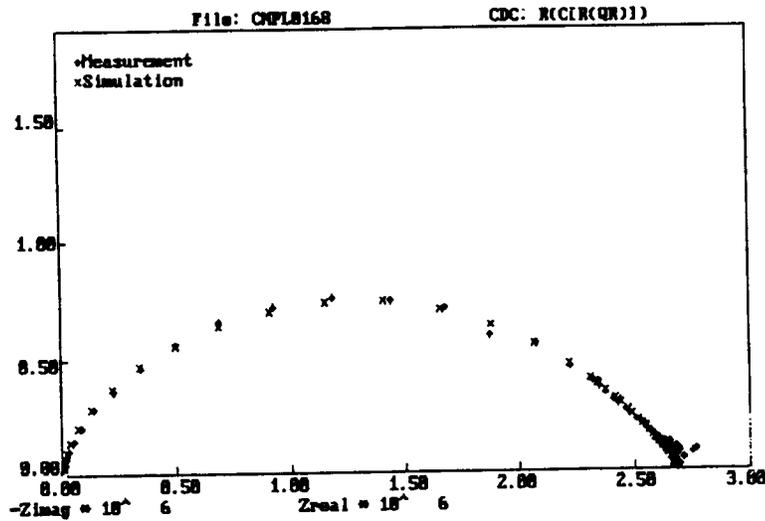


Figure 9 - Nyquist plot for coating A after 168 hours of immersion in 3.55% NaCl

TABLE 1 - Parameters from Equivalent Circuit of Doped Polyaniline for Coating A

Immersion Time, Hours	$R_e$ , kohms	$C_c$ , pfarads	$R_c$ , kohms	$Y_c$ , mho $\times 10^{-9}$	n	$R_a$ , Mohms
1	9.80	101	970	389	0.17	44.7
6	9.97	109	827	328	0.09	(A)
24	9.18	122	1580	8.80	0.45	8.25
48	7.43	120	1020	3.16	0.61	10.3
72	8.68	143	1010	4.67	0.61	6.30
96	8.98	149	699	6.03	0.61	4.09
168	8.12	164	507	9.87	0.59	2.18
336	7.99	182	243	35.7	0.47	1.28
528	8.23	191	166	53.6	0.43	1.04
672	8.14	200	138	64.5	0.43	8.35
840	7.33	211	108	82.2	0.40	7.20
1008	8.59	201	78	88.6	0.41	6.07

<sup>(A)</sup> Not available

TABLE 2 - Parameters from Equivalent Circuit of Conductive Polymers for Coating B

Immersion Time, Hours	$R_c$ , kohms	$C_c$ , pfarads	$R_c$ , kohms	$Y_o$ , mho $\times 10^{-9}$	n	$R_a$ , Mohms
.1	11.37	231	31	8720	0.24	4.47
6	10.63	160	67	3270	0.22	4.47
24	9.18	122	1580	8.80	0.45	8.25
48	9.59	136	970	173	0.30	2.79
72	9.29	140	546	7.97	0.48	1.03
144	8.31	154	493	8.92	0.52	6.98
168	8.67	159	299	15.7	0.47	4.68
336	8.72	190	299	426	0.22	3.12
504	8.42	194	182	1260	0.18	3.12
672	8.28	206	231	1840	0.24	3.12
840	8.43	211	206	2140	0.24	3.12
1344	7.58	210	117	1490	0.14	3.11

4.5 The parameters obtained by simulating the data with the equivalent circuit in Figure 7 are presented in Tables 1 and 2 for coatings A and B, respectively. The variation of  $R_c$  with time of immersion is characterized initially by a steep increase followed by a gradual decrease (Figure 10). On the basis of similarities obtained with EIS spectra of inorganic ZRP's,<sup>11-13</sup> it can be hypothesized that during the early stages of immersion,  $R_c$  reflects the galvanic mechanism of protection involving the oxidation of the PAN's. An initial increase in  $R_c$  may be indicative of a decrease in the galvanic activity of the coating, since its rate of oxidation and  $R_c$  are inversely related. After this period, the resistance is equivalent to the magnitude of the film resistance.  $R_c$  represents the extent of ionic conduction through the coating in an electrolytic environment. Its decrease over time is indicative of increased ionic conductivity for the coating and lower protective properties as a result of electrolyte penetration.<sup>18,19</sup> This behavior is similar to that of the more typical non-conductive organic coating that protects by barrier formation alone, and is in contrast to the behavior of inorganic ZRP's exhibiting a galvanic as well as a barrier mechanism of protection for the steel.<sup>7,8</sup>

4.6 The variation of  $C_c$  with time of immersion for coatings A and B is shown in Figure 11. The initial step decrease in the capacitance for coating B is similar to that observed previously for inorganic ZRP's.<sup>12,13</sup> However, this effect is short-lived and followed by an increase in the coating capacitance that can be

associated with the water uptake of the coating. Figures 8 and 9 also show that, except for the initial decrease in  $C_c$  for coating B, the two coatings behave similarly despite the fact that the doping agents were different. This finding suggests that EIS can be used to test the performance of PAN's when different doping substances or processes are tested.

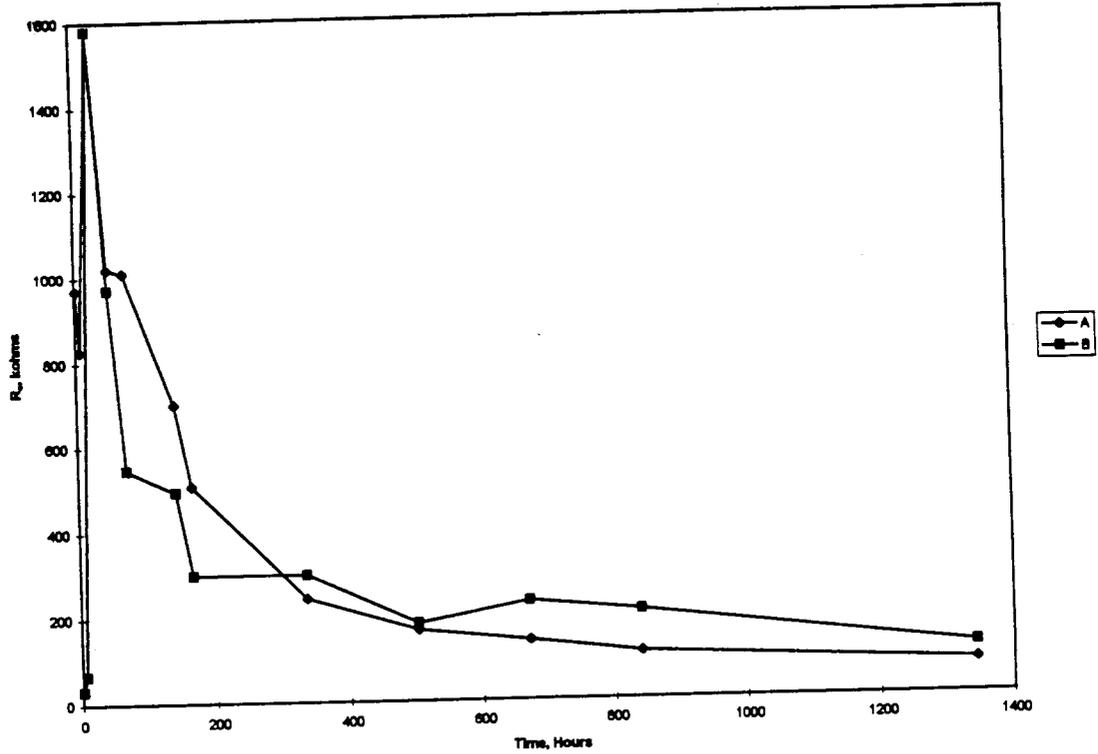


Figure 10 -  $R_c$  as a function of immersion time in 3.55% NaCl

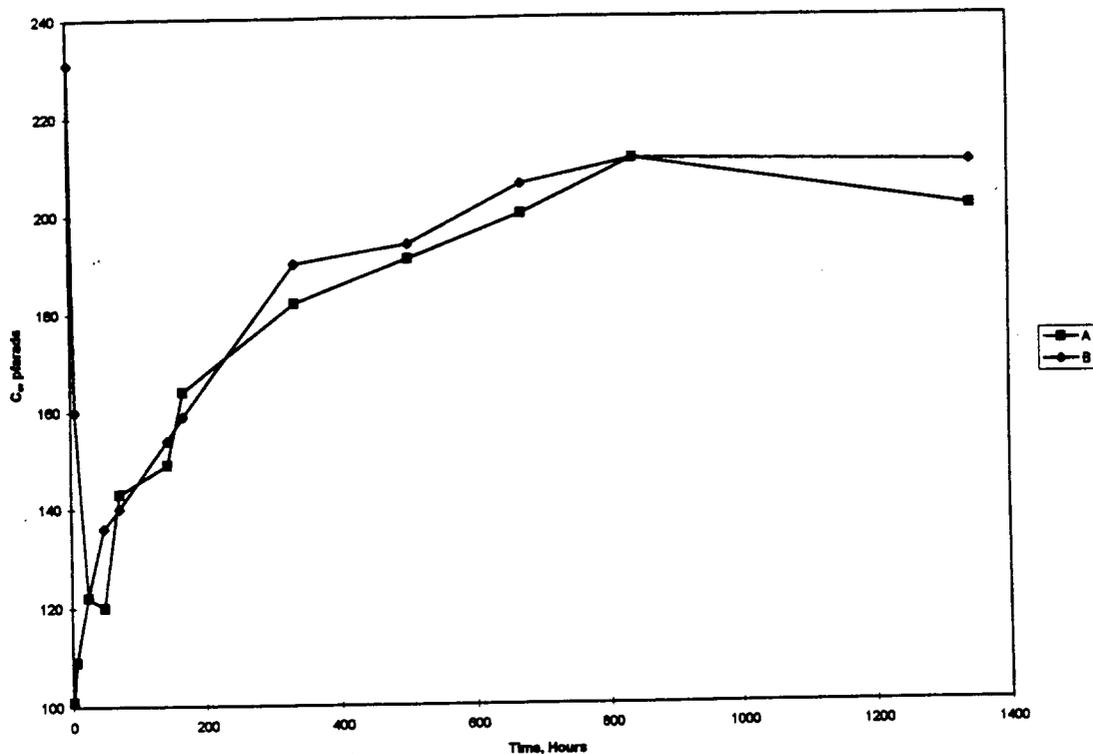


Figure 11 -  $C_c$  as a function of immersion time in 3.55% NaCl

4.6 J. Walaszkowski *et al.*<sup>20,21</sup> recently reported the use of EIS to investigate the electrical and electrochemical properties of conductive composites based on ethylene-propylene-diene-monomer (EPDM). They used the equivalent circuit  $R(Q(R(RQ)))$  to obtain parameters from their EIS data. This data was used to follow the progress in the development of conductive polymers as corrosion protective coatings for stainless steel. The Nyquist and Bode plots for the two coatings at different immersion times were similar to those observed for inorganic ZRP's.<sup>11-13</sup> In fact, a good fit of the PAN data is obtained with the same equivalent circuit that was used to interpret the EIS of ZRP's,<sup>12,13</sup> but the equivalent circuit described in this work provides for a better fit of the data.

## 5.0 CONCLUSIONS

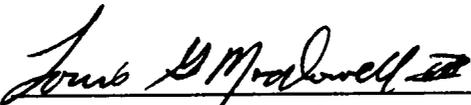
5.1 EIS is an excellent investigative technique to monitor the progress in the development of conductive polymer coatings for the protection of carbon steel or other metals in corrosive environments. Changes in the EIS spectra can be used to monitor the performance of these coatings and to investigate their mechanism of protection.

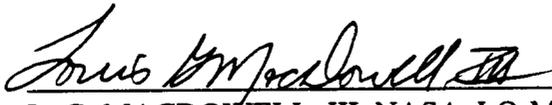
5.2 The equivalent circuit  $R_c(C_c [R_c(QR_a)])$  provided a satisfactory fit for the EIS data of doped PAN's. The corrosion potential and the initial variation of  $R_c$  with exposure to the corrosive environment are parameters indicative of the galvanic

mechanism of protection. The capacitance of the coating is related to the barrier mechanism of protection,<sup>7,8</sup> and its variation with time is related to the uptake of water by the coating. A gradual increase in the capacitance with immersion time in the electrolyte indicates penetration of the coating by the electrolyte. An increase in capacitance after a long-lasting plateau has been reported for epoxy coatings and attributed to detachment of the organic coating from the substrate resulting from loss of adhesion.<sup>22</sup> This behavior was not observed for the two coatings included in this investigation, possibly due to the short immersion time.

## 6.0 ACKNOWLEDGMENT

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APPENDIX A

NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE PLOTS FOR  
COATING A IN 3.55% NaCl

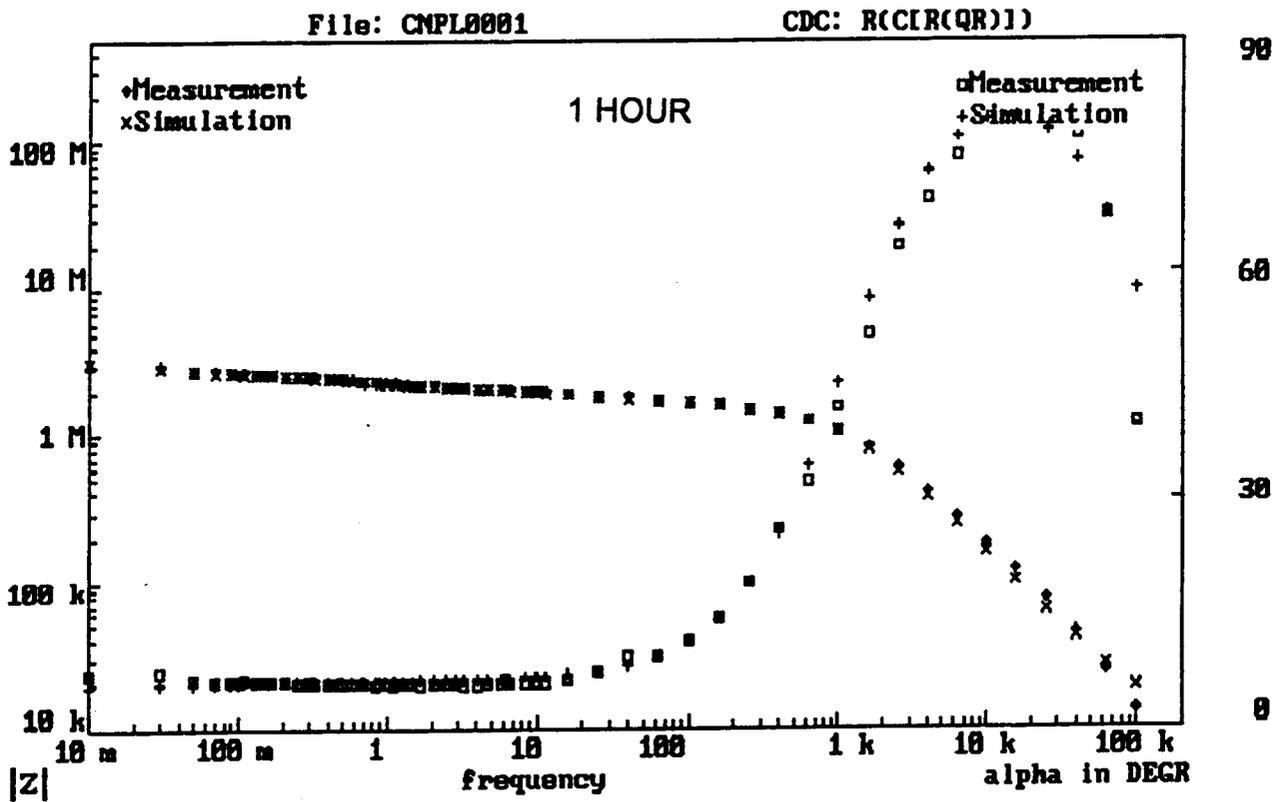
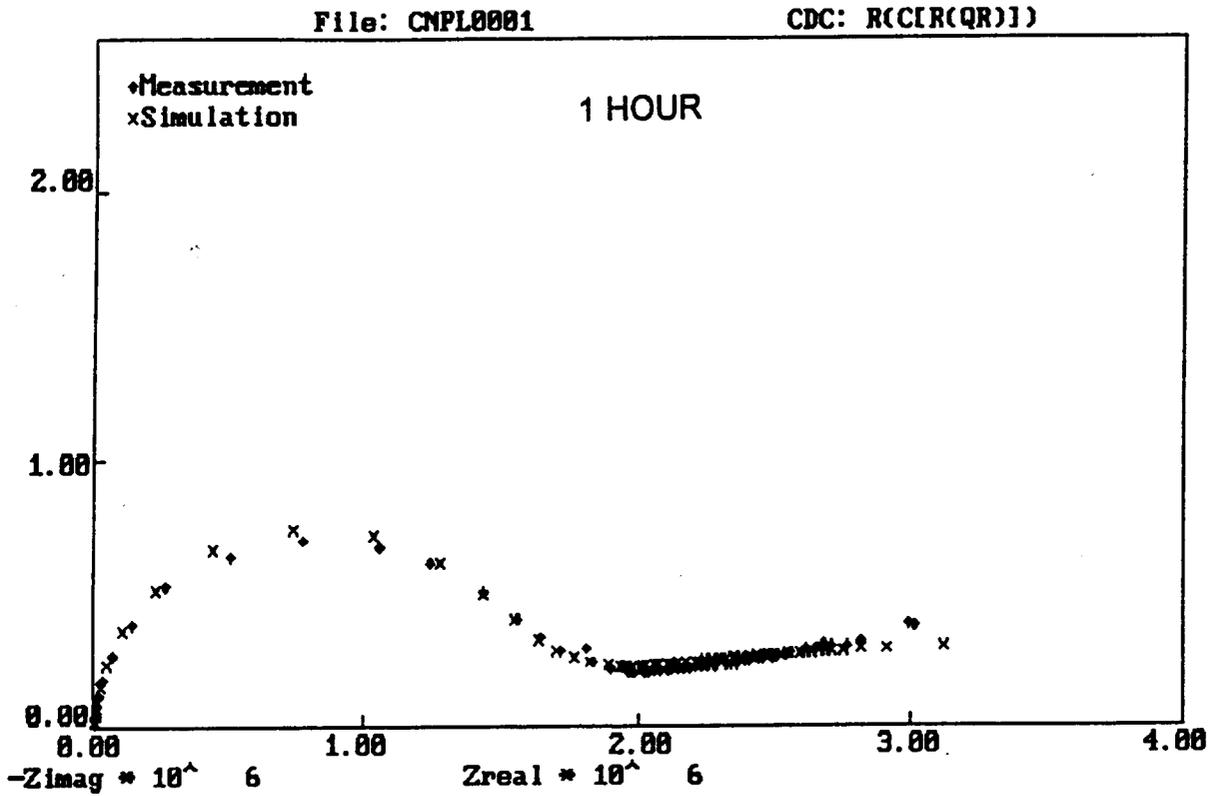


FIGURE A-1. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 1 HOUR IMMERSION IN NaCl

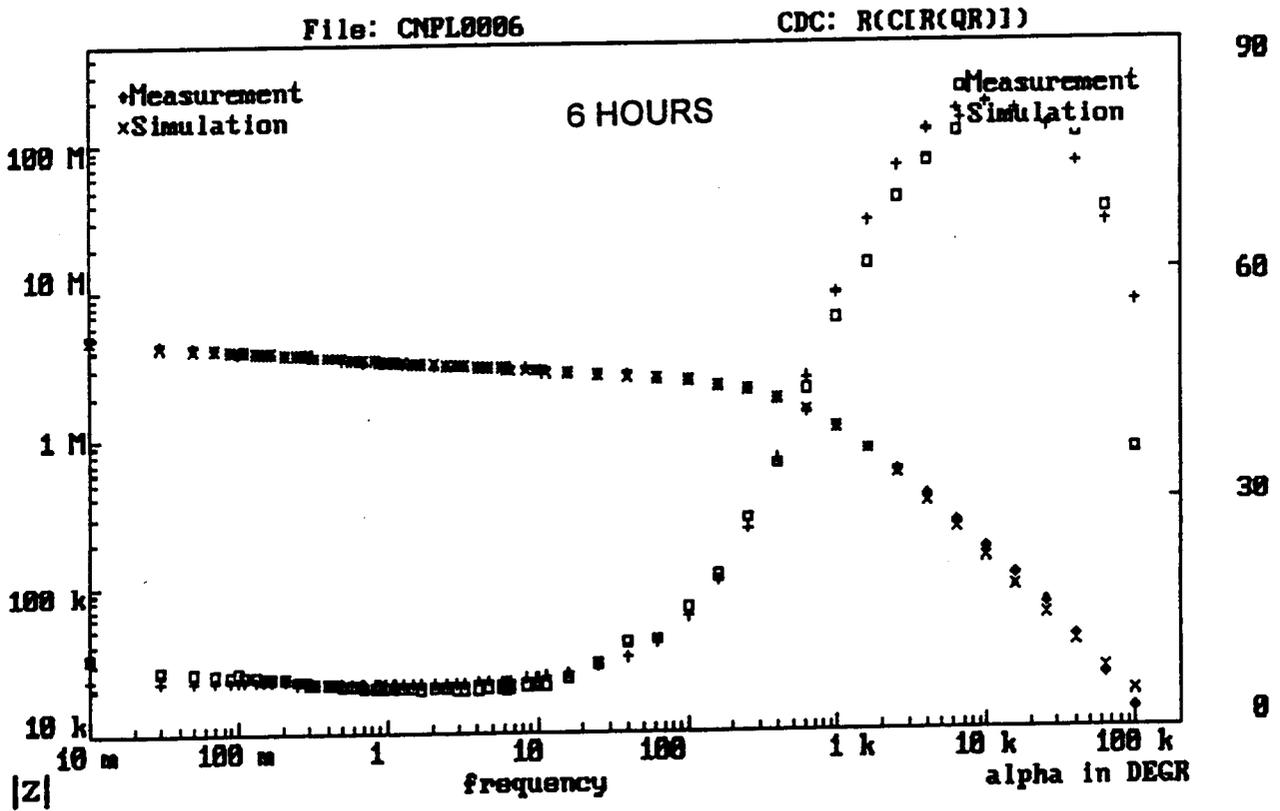
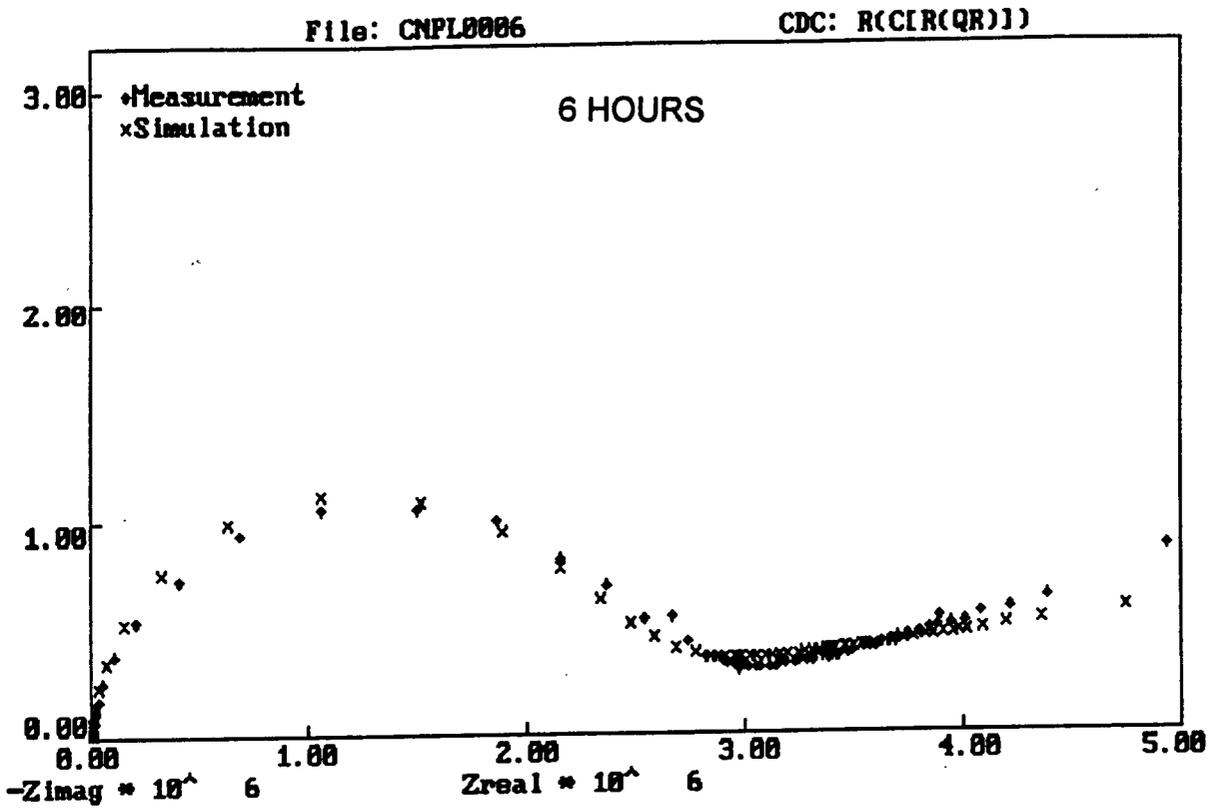


FIGURE A-2. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 6 HOURS IMMERSION IN NaCl

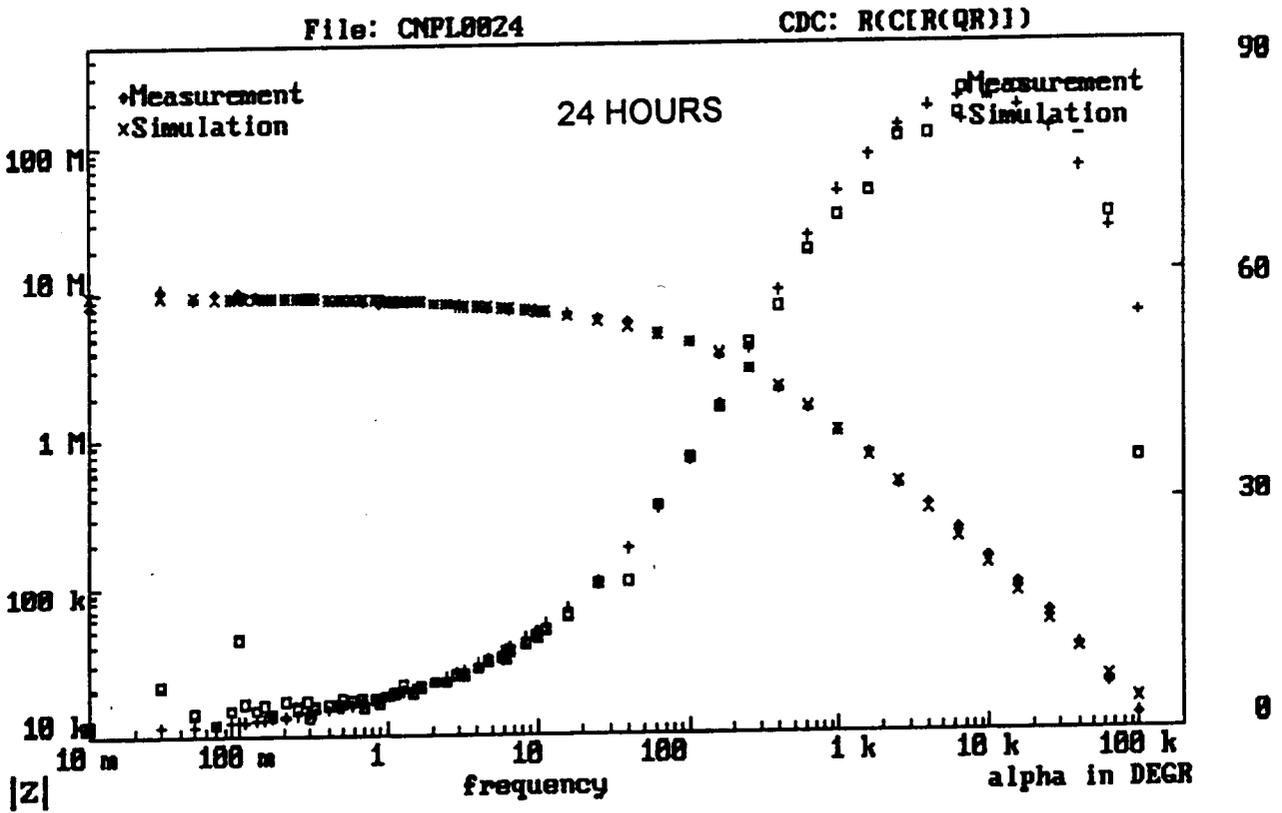
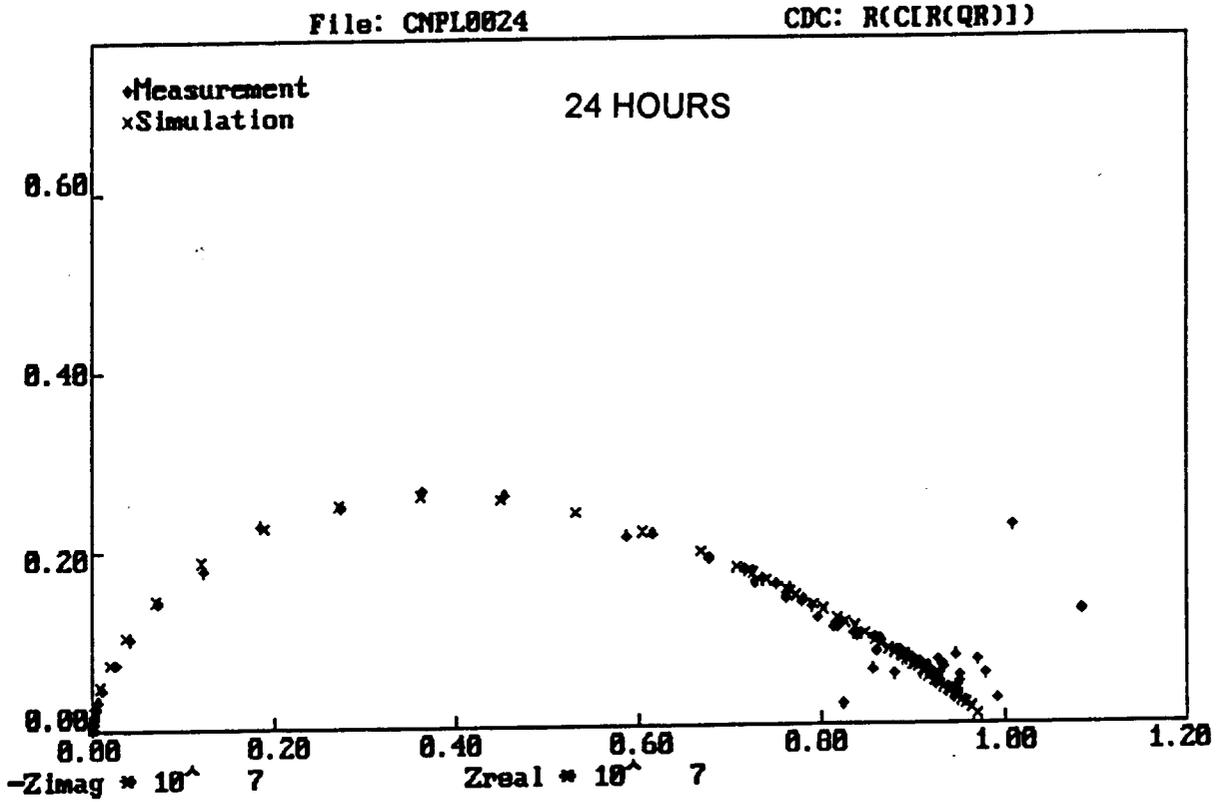


FIGURE A-3. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 24 HOURS IMMERSION IN NaCl

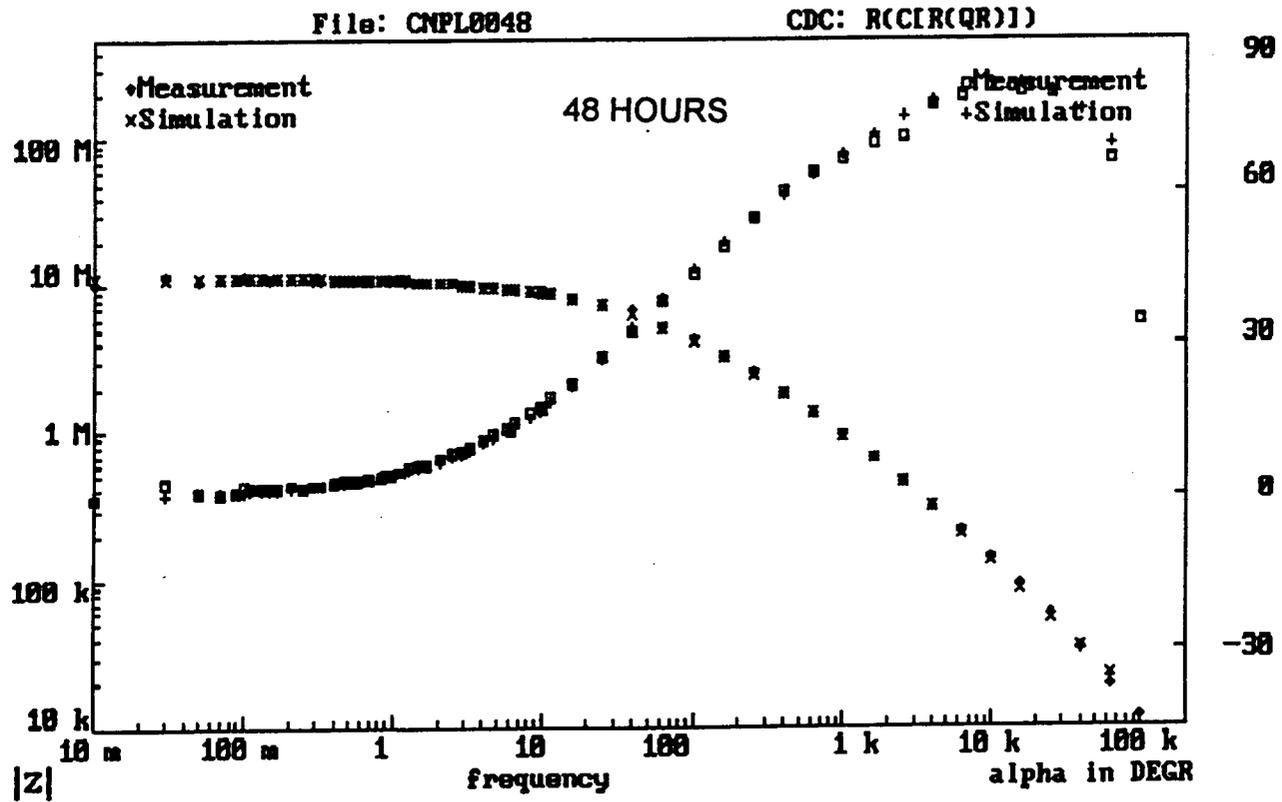
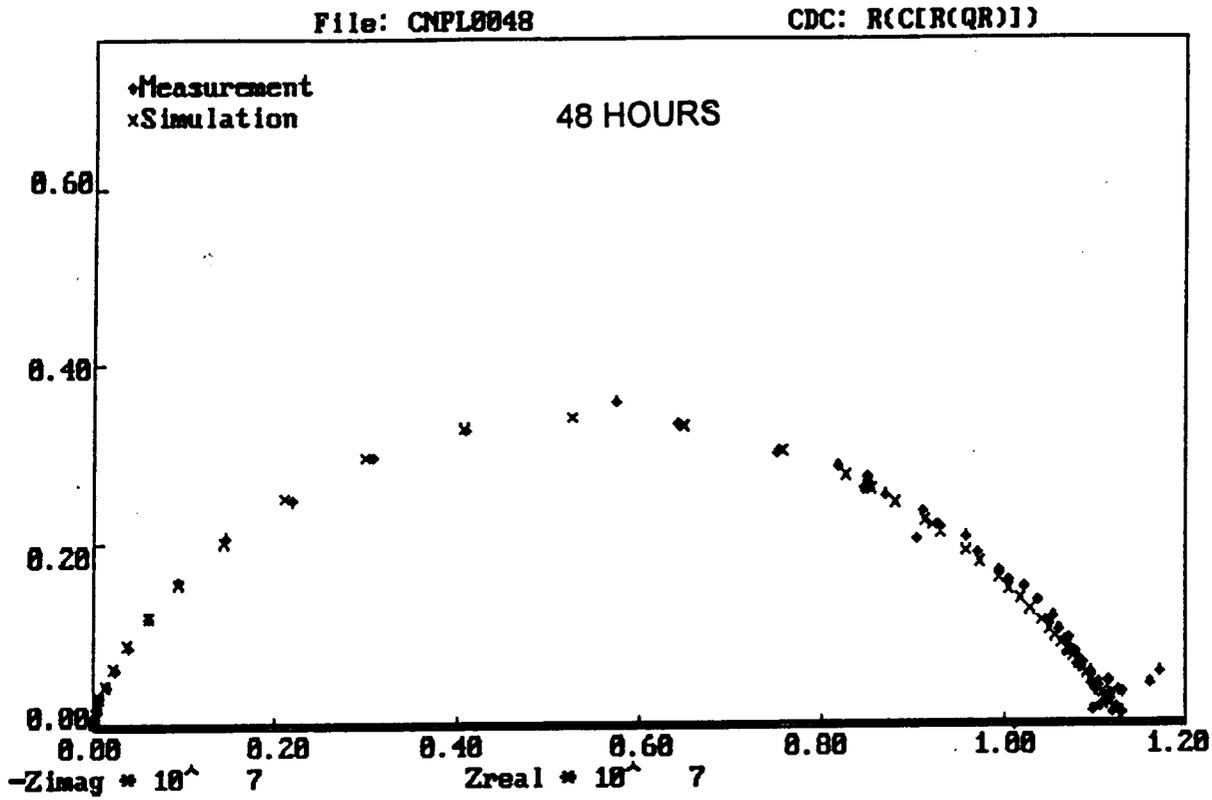


FIGURE A-4. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 48 HOURS IMMERSION IN NaCl

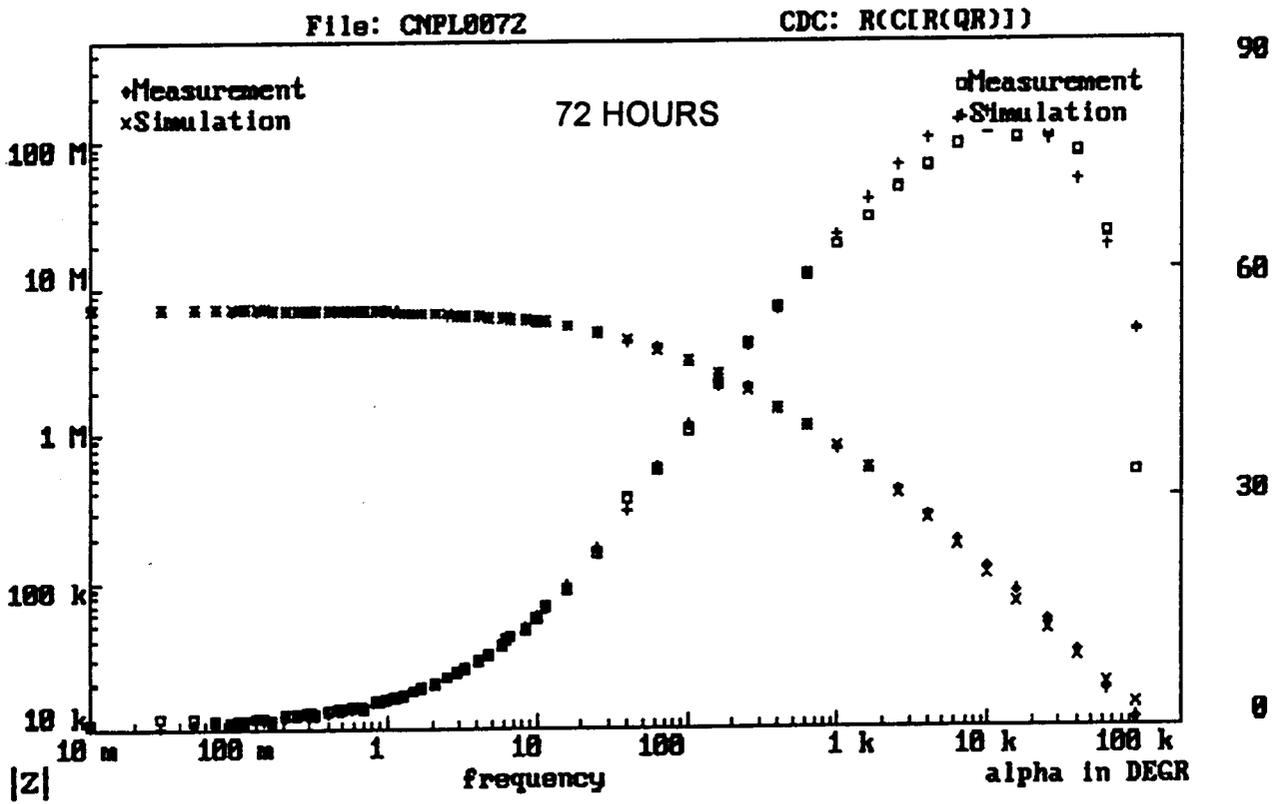
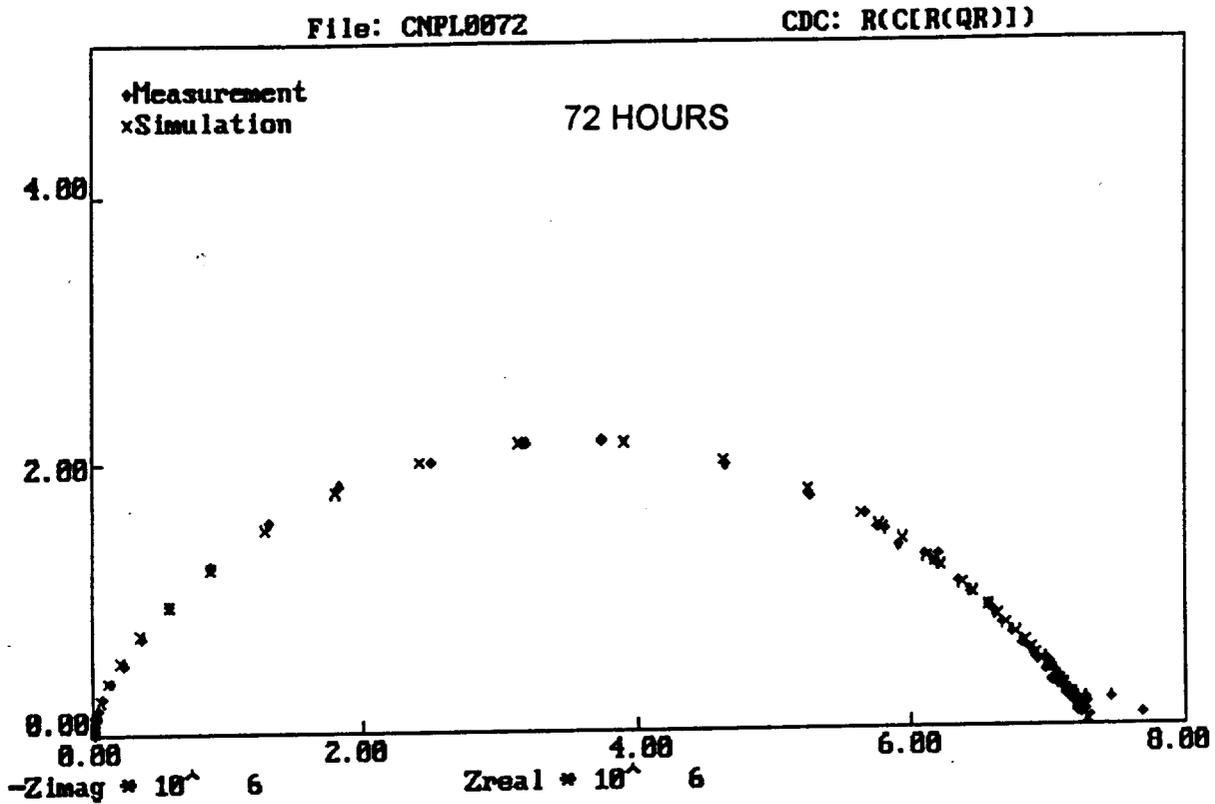


FIGURE A-5. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 72 HOURS IMMERSION IN NaCl

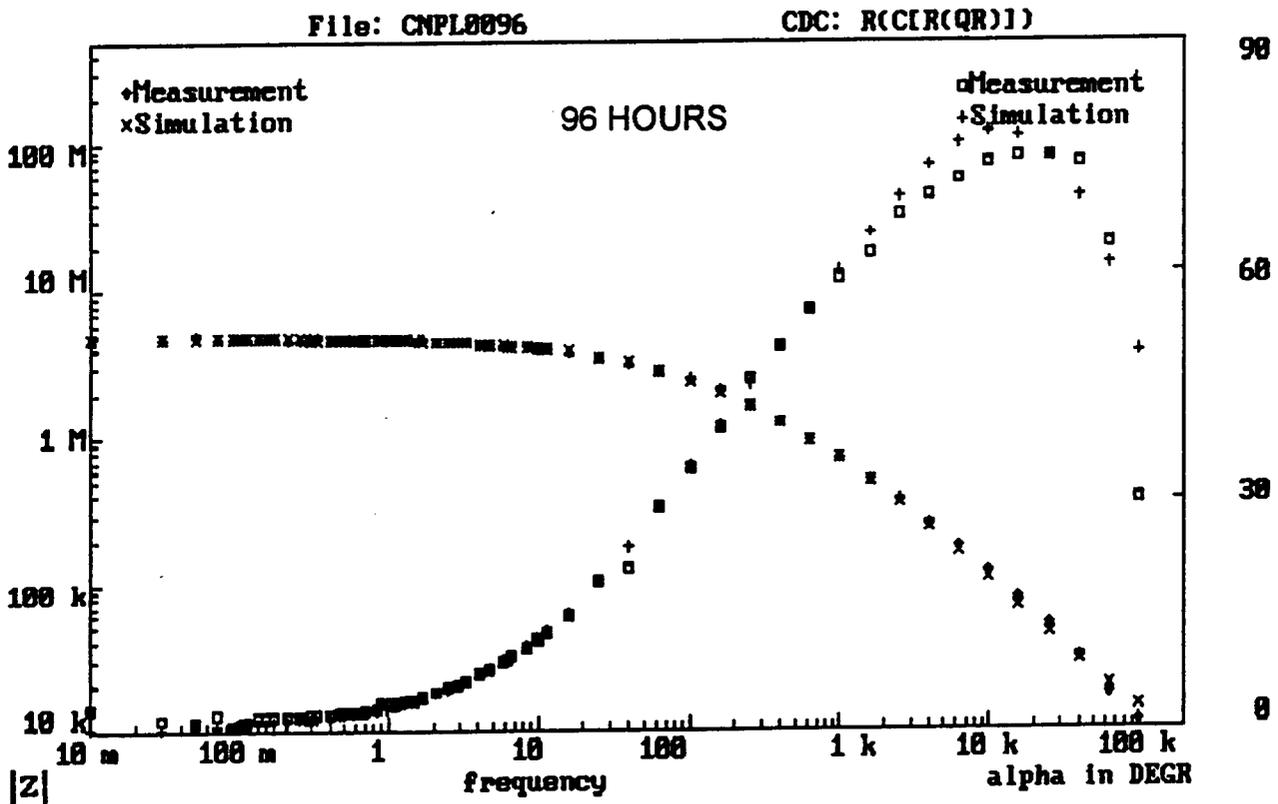
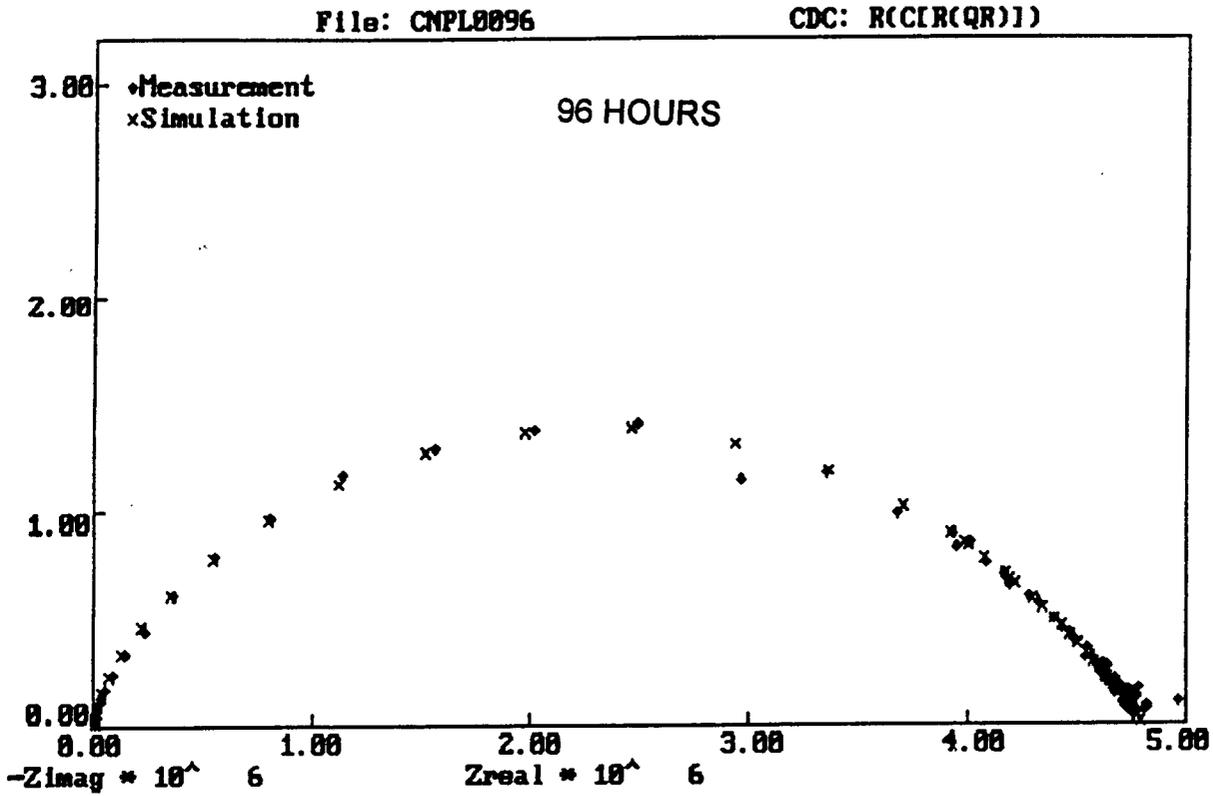


FIGURE A-6. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 96 HOURS IMMERSION IN NACL

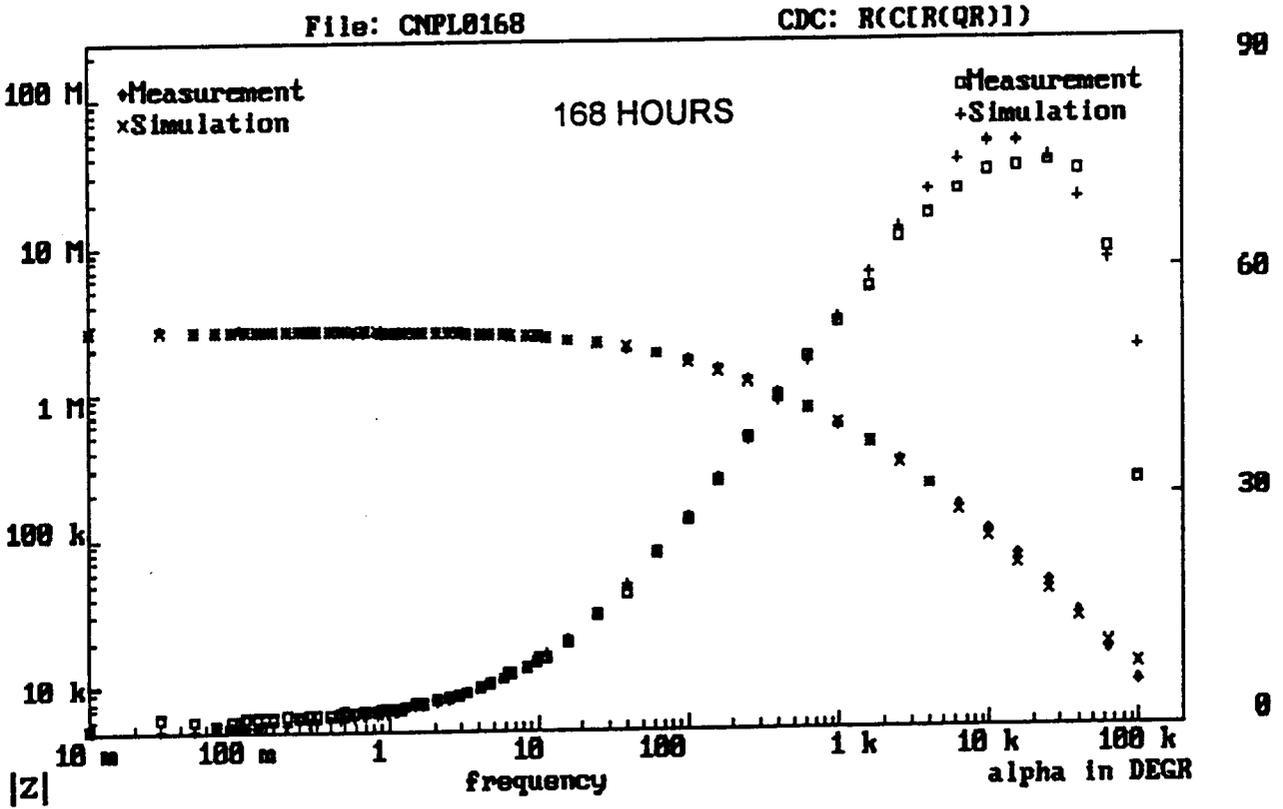
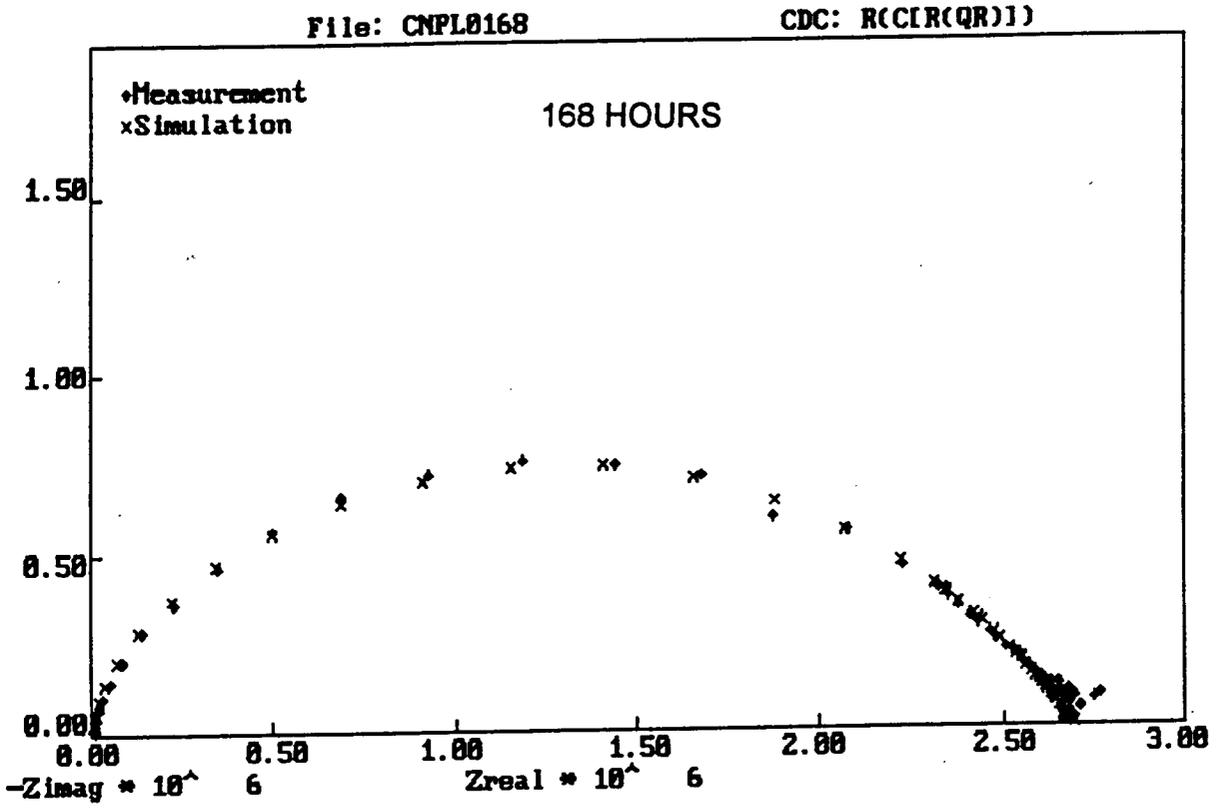


FIGURE A-7. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 168 HOURS IMMERSION IN NACL

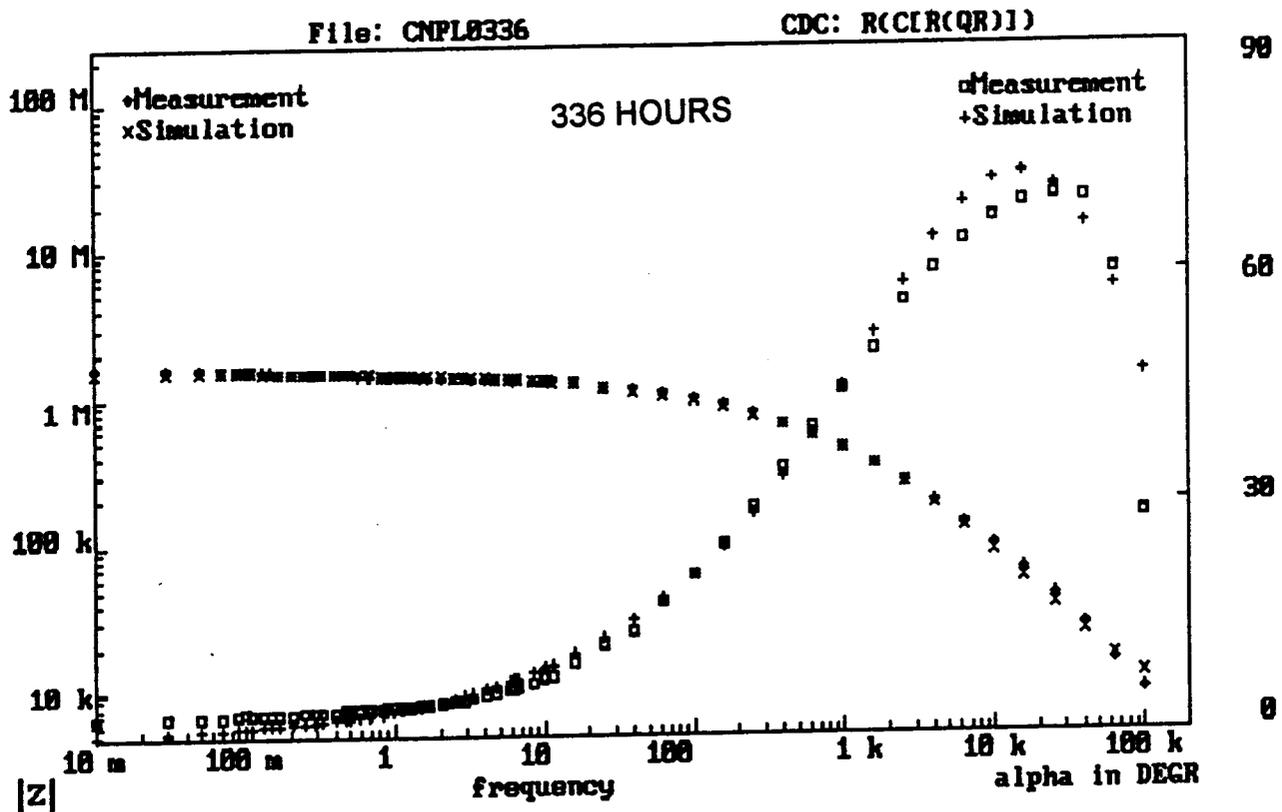
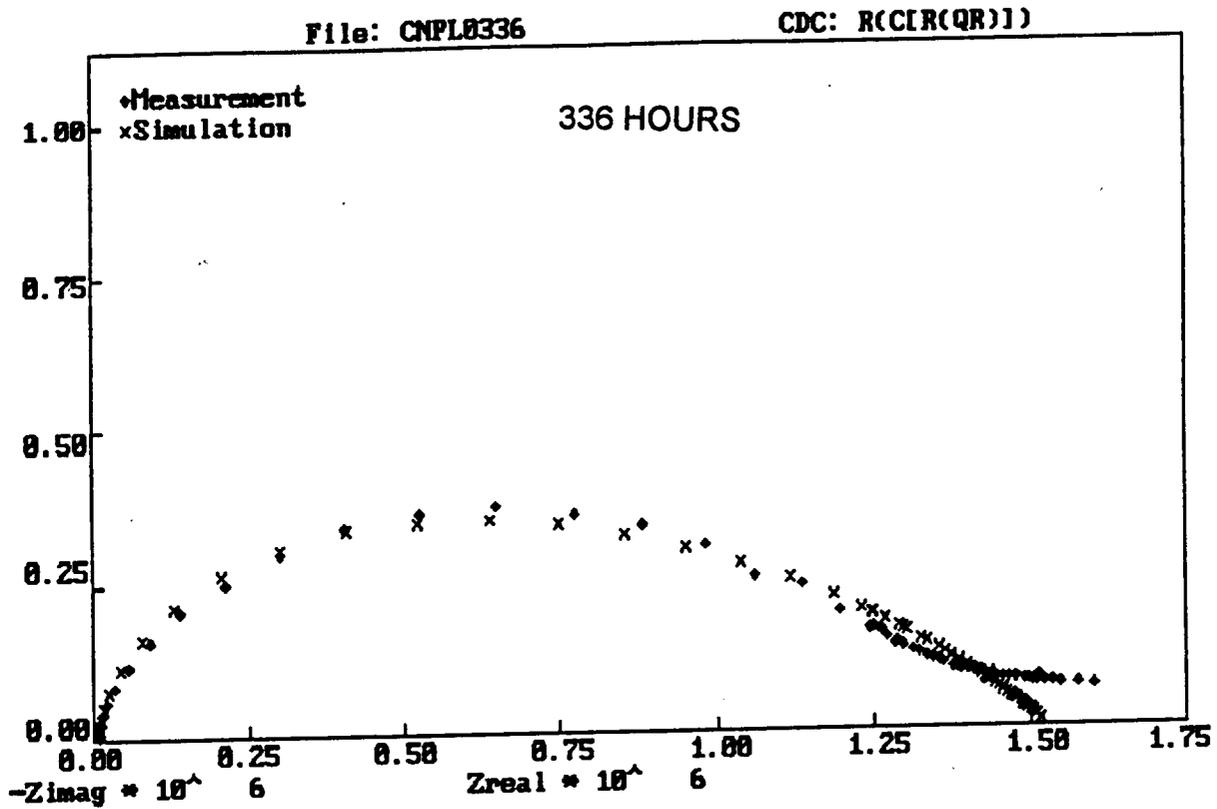


FIGURE A-8. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 336 HOURS IMMERSION IN NaCl

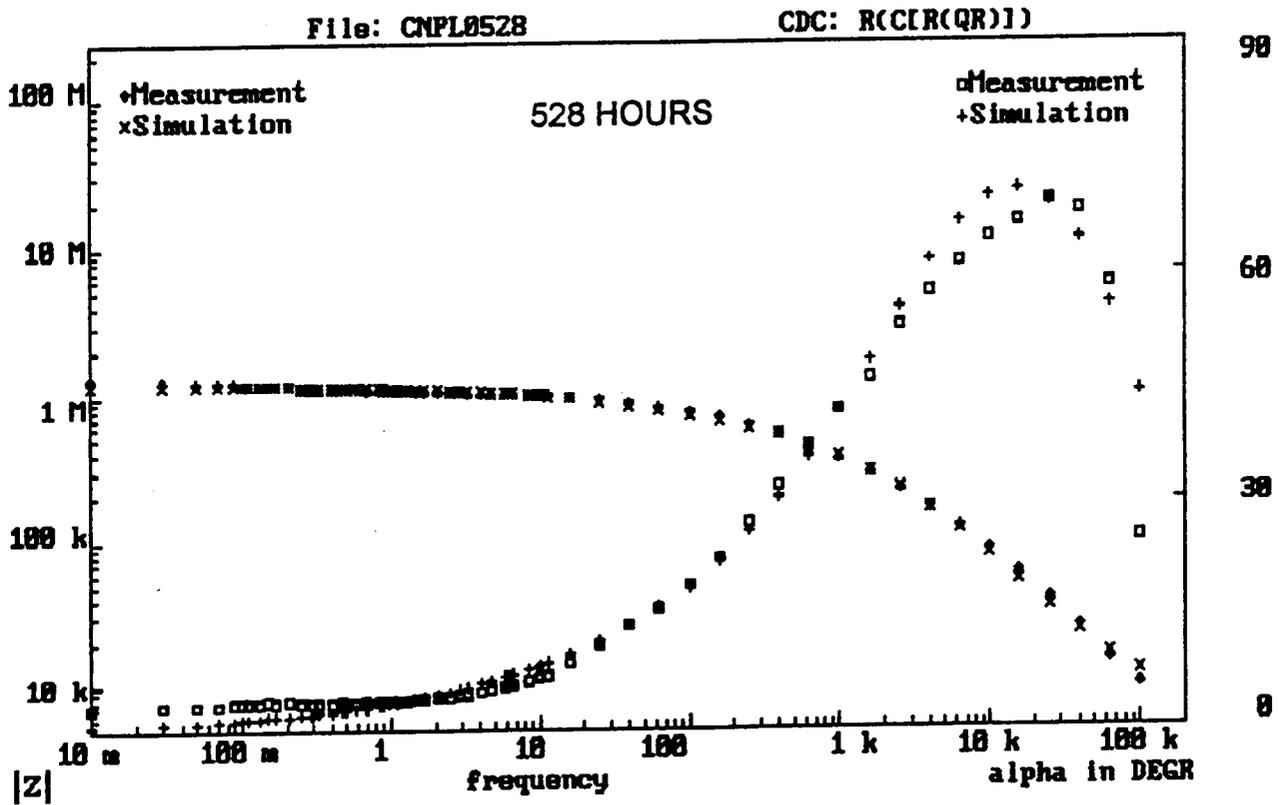
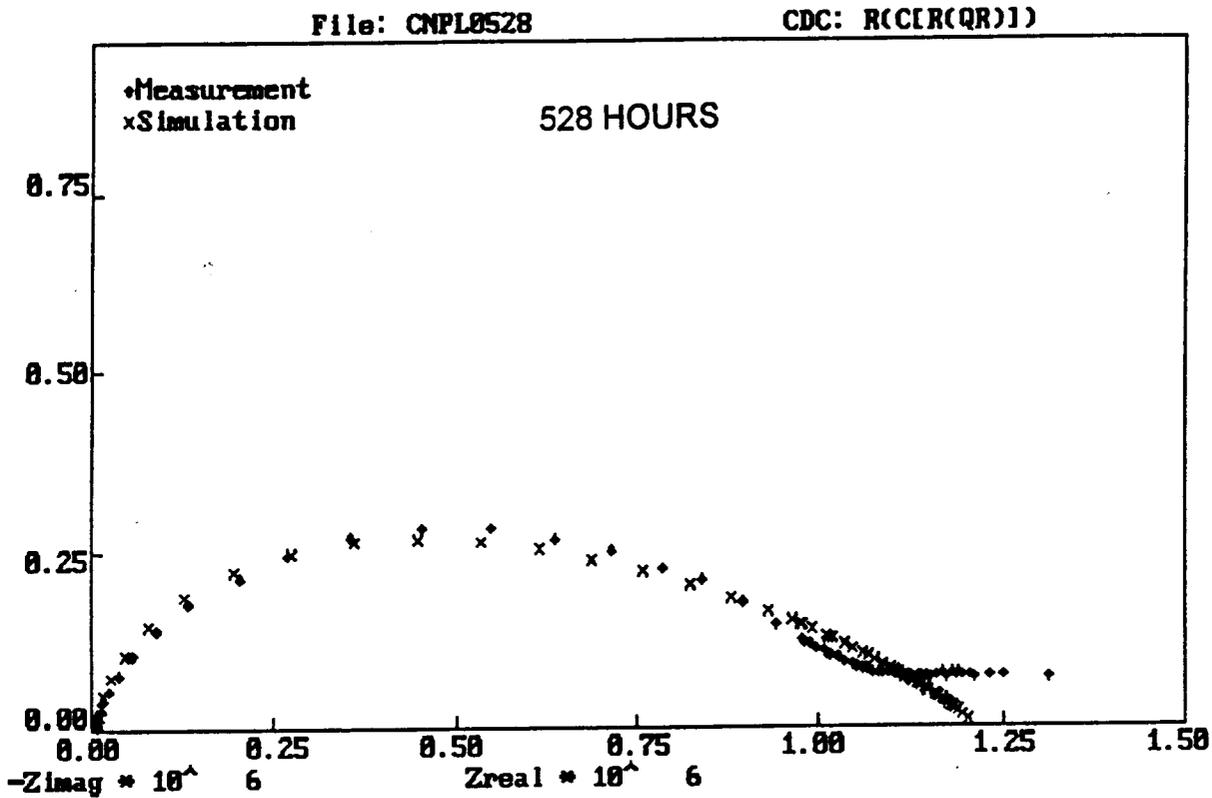


FIGURE A-9. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 528 HOURS IMMERSION IN NACL

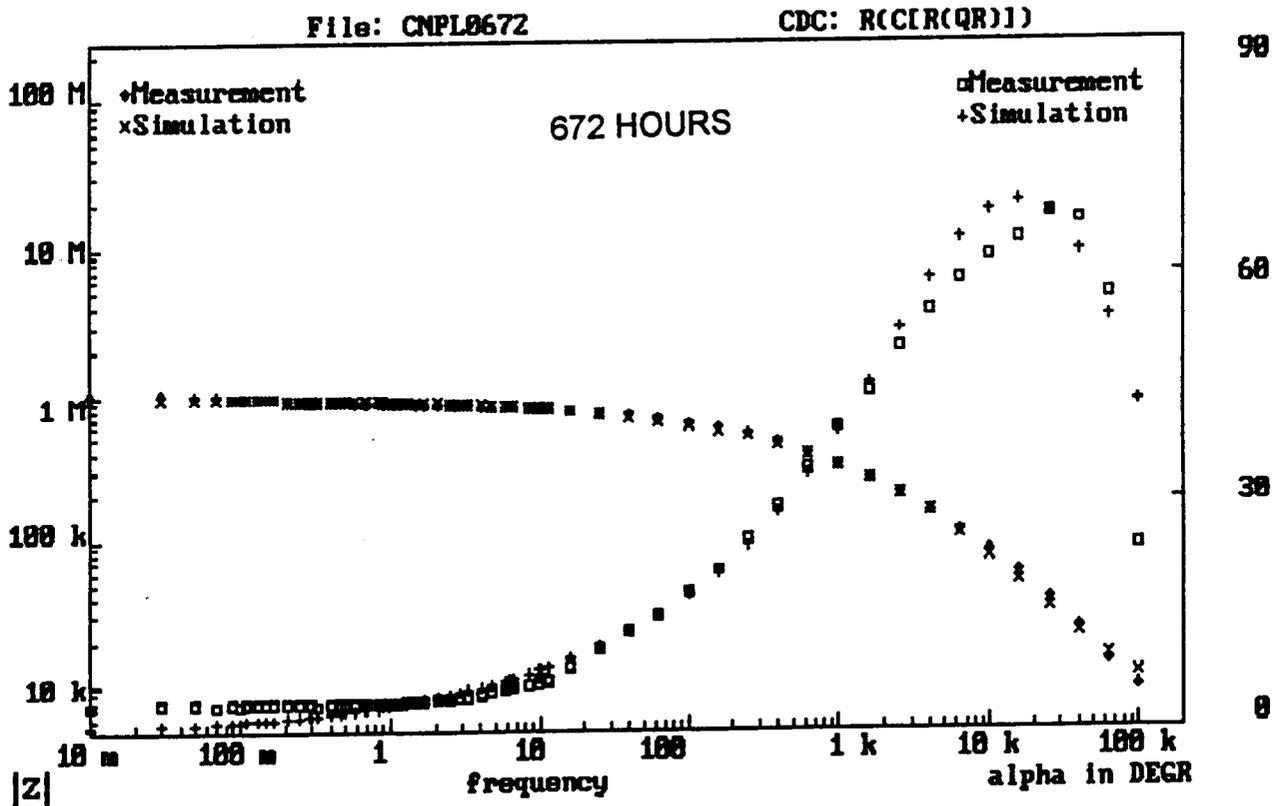
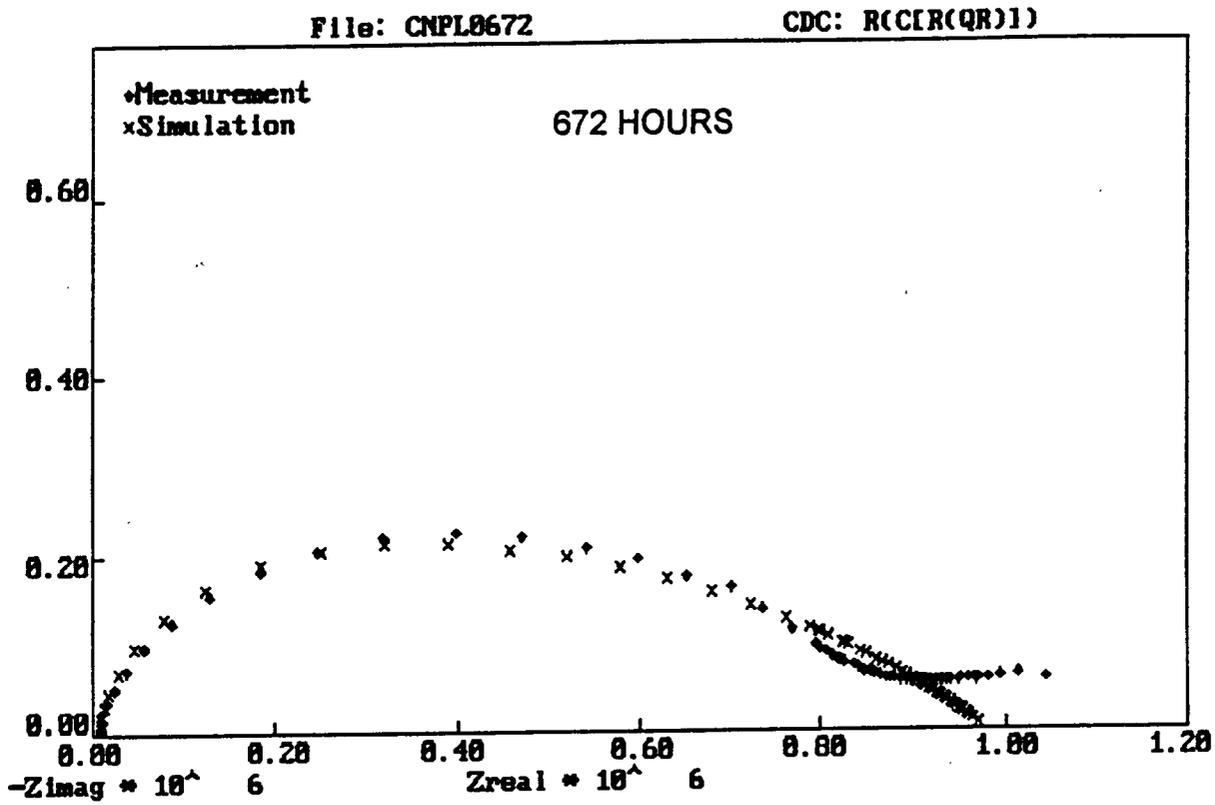


FIGURE A-10. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 672 HOURS IMMERSION IN NA CL

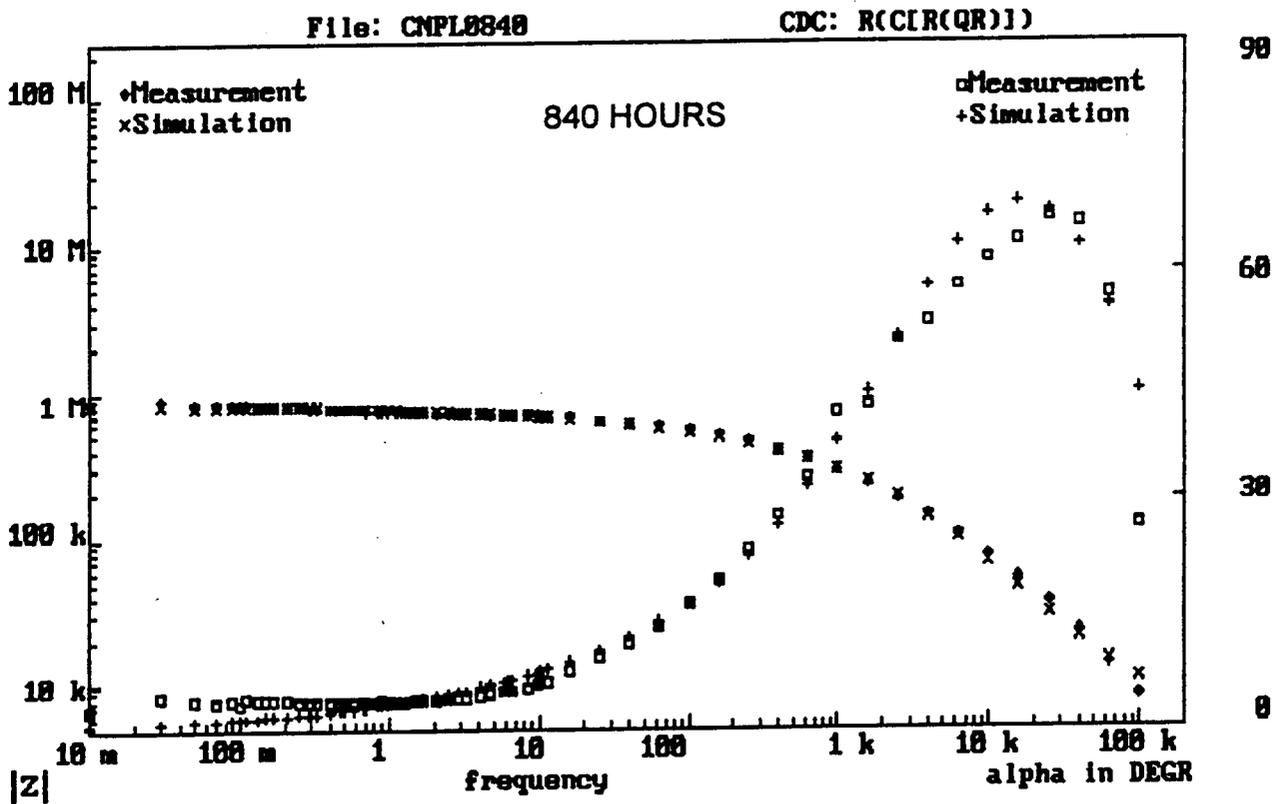
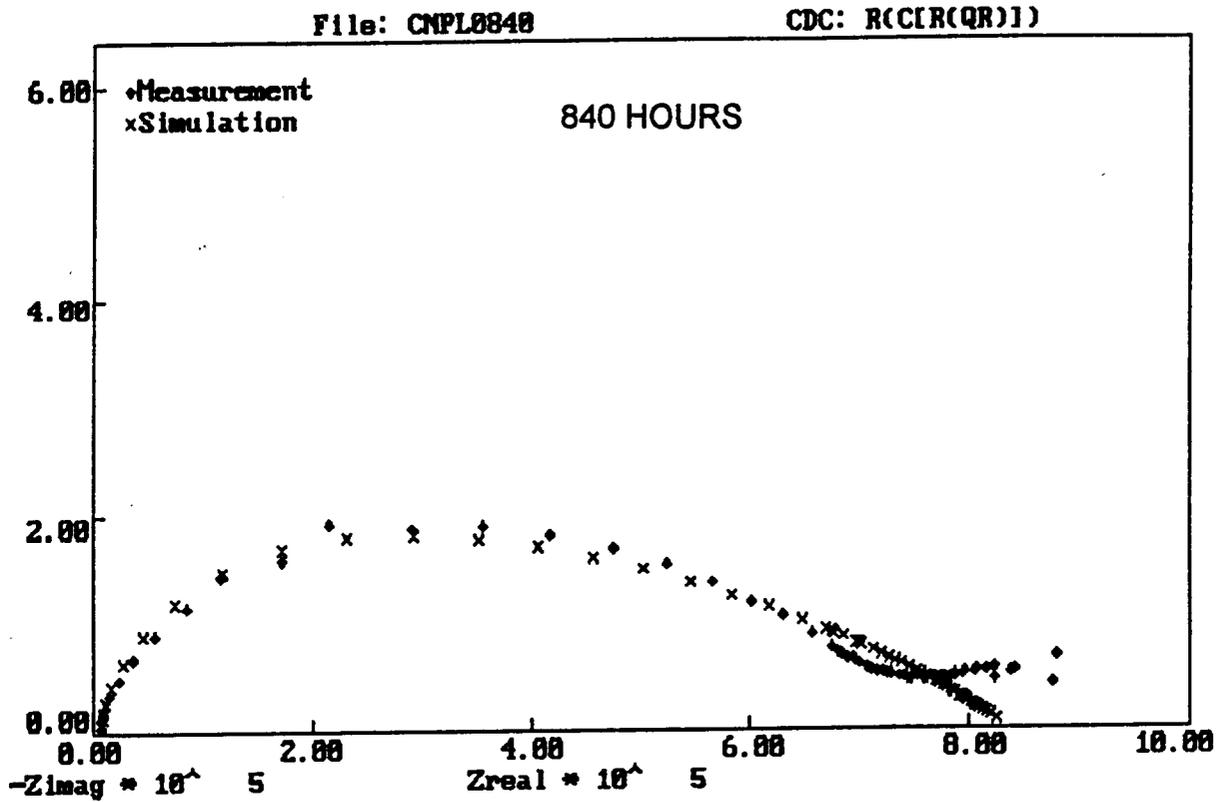


FIGURE A-11. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 840 HOURS IMMERSION IN NACL

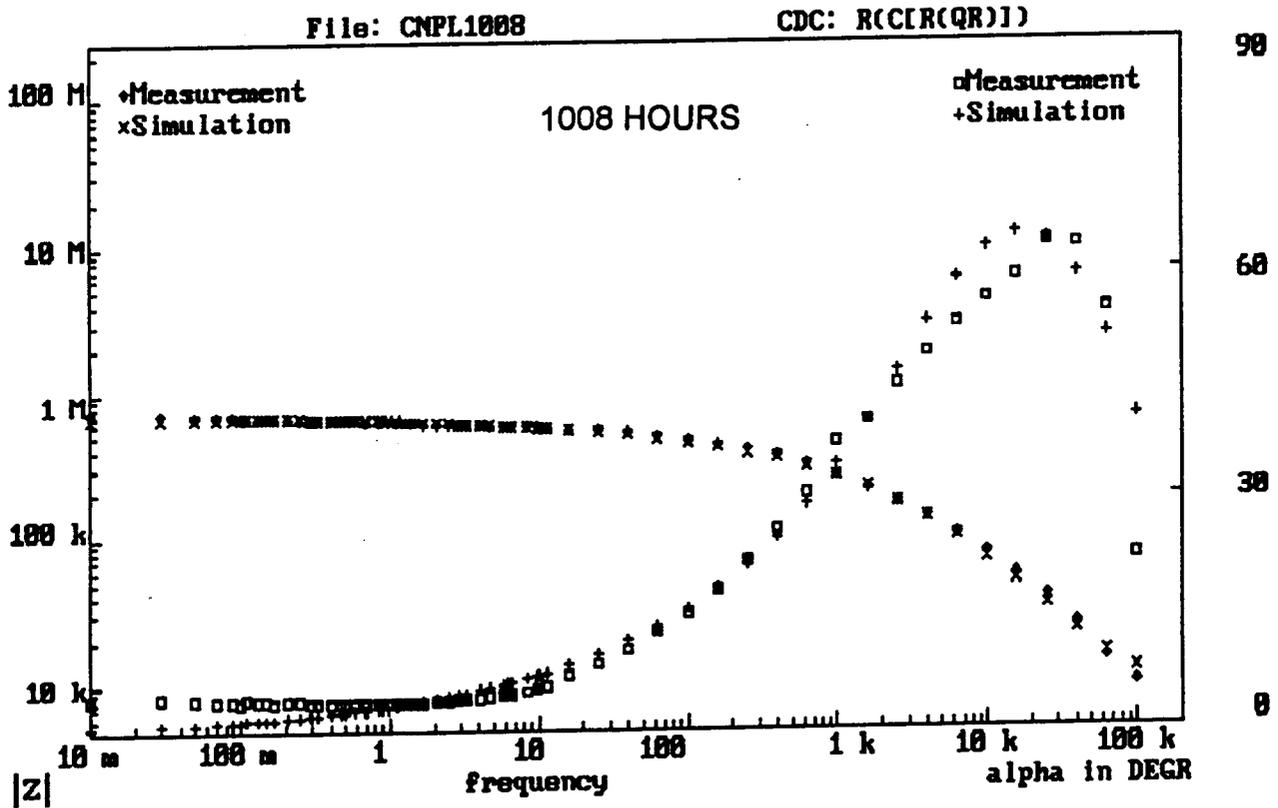
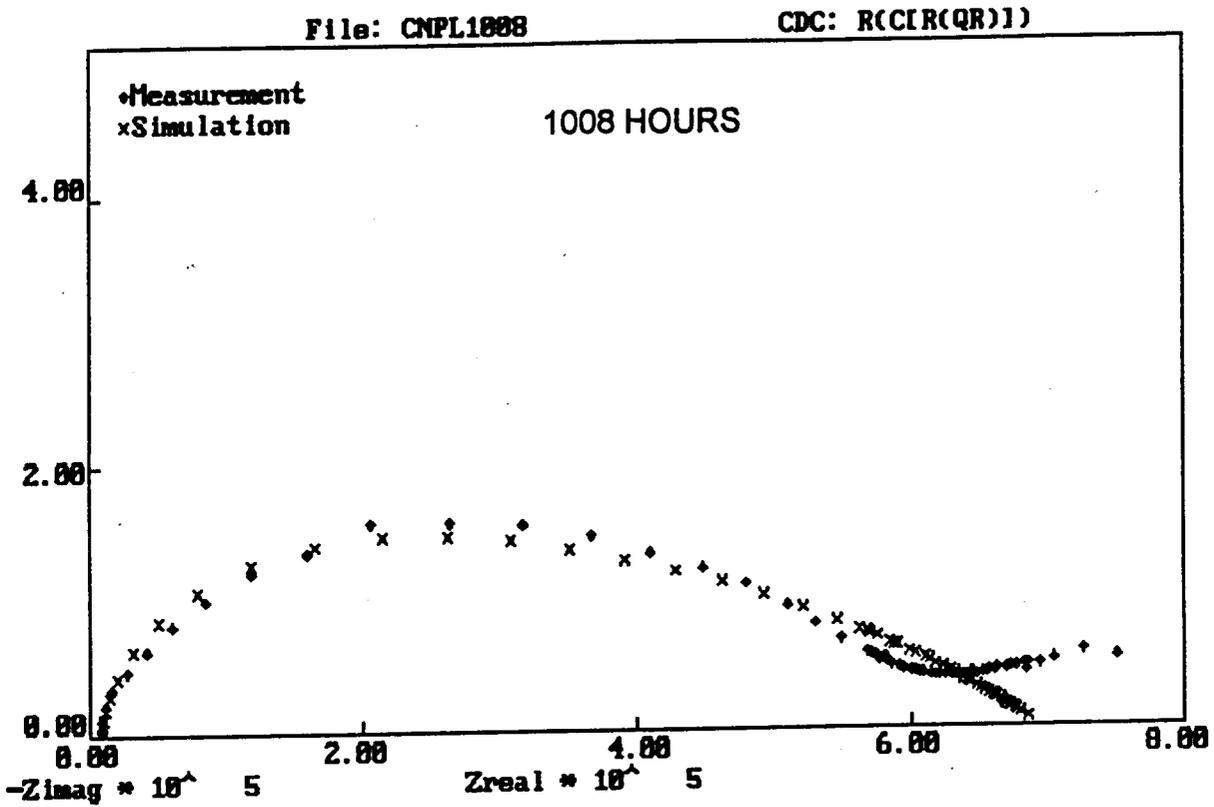


FIGURE A-12. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING A AFTER 1008 HOURS IMMERSION IN NACL

**APPENDIX B**

**NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE PLOTS FOR  
COATING B IN 3.55% NaCl**

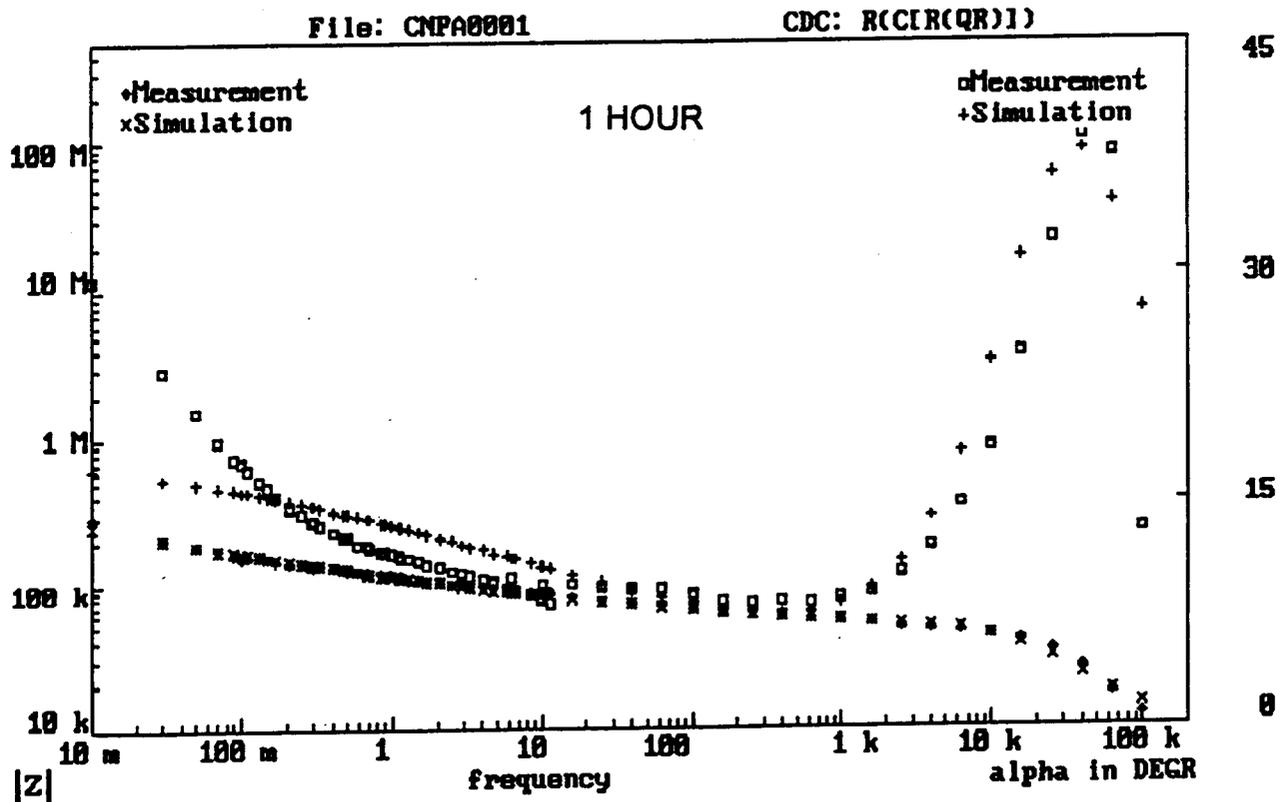
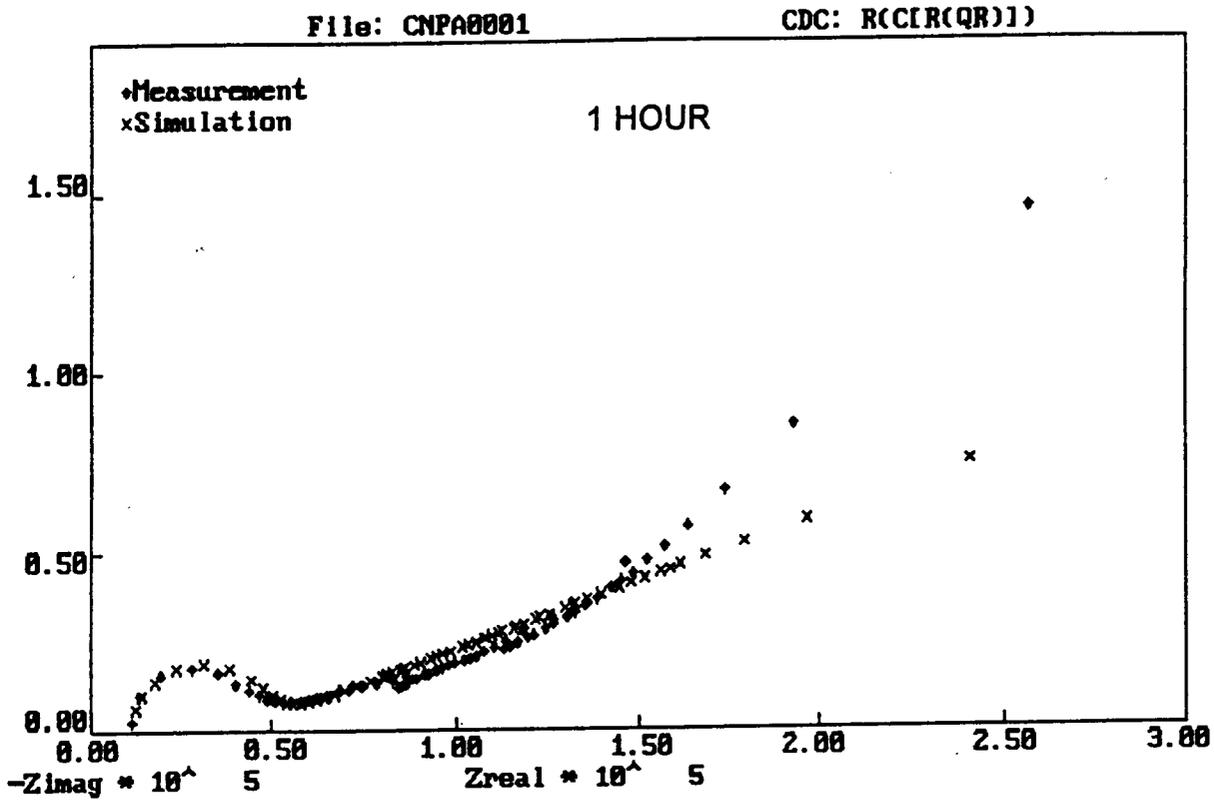


FIGURE B-1. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 1 HOUR IMMERSION IN NA CL

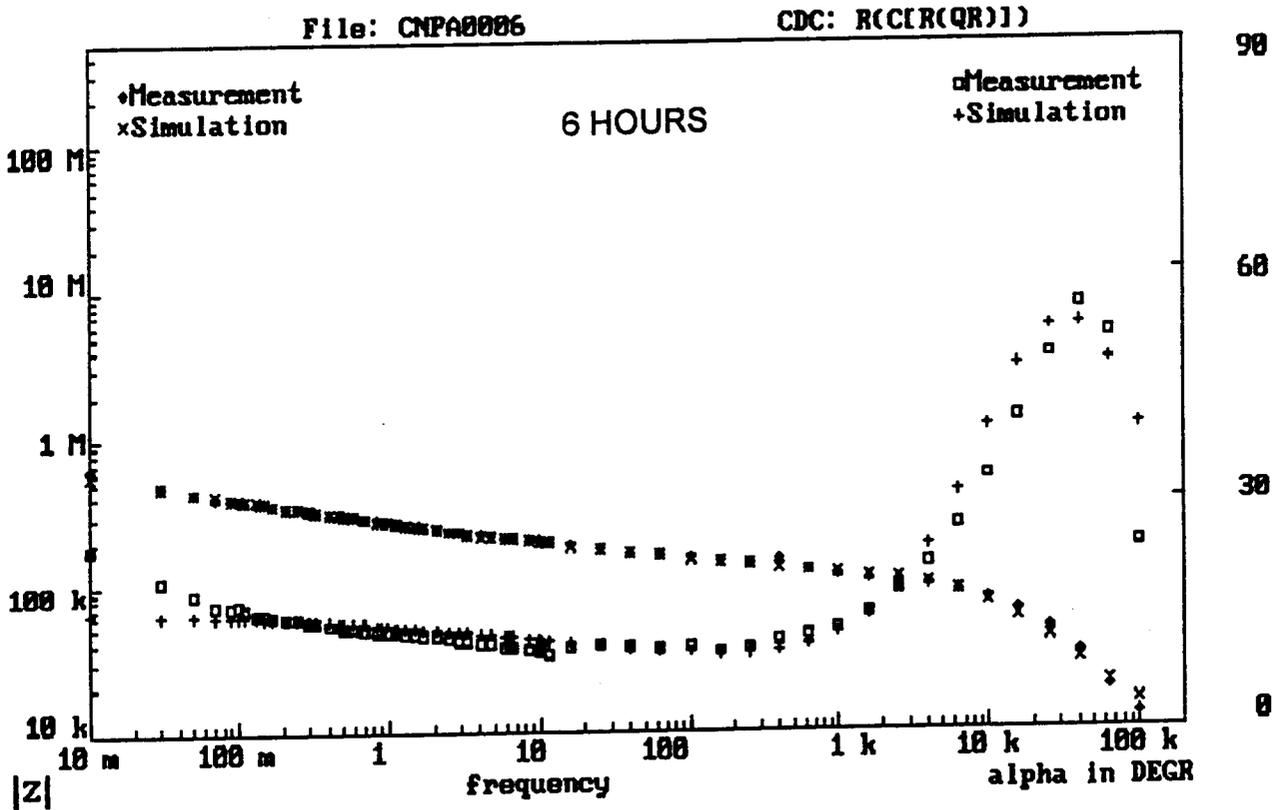
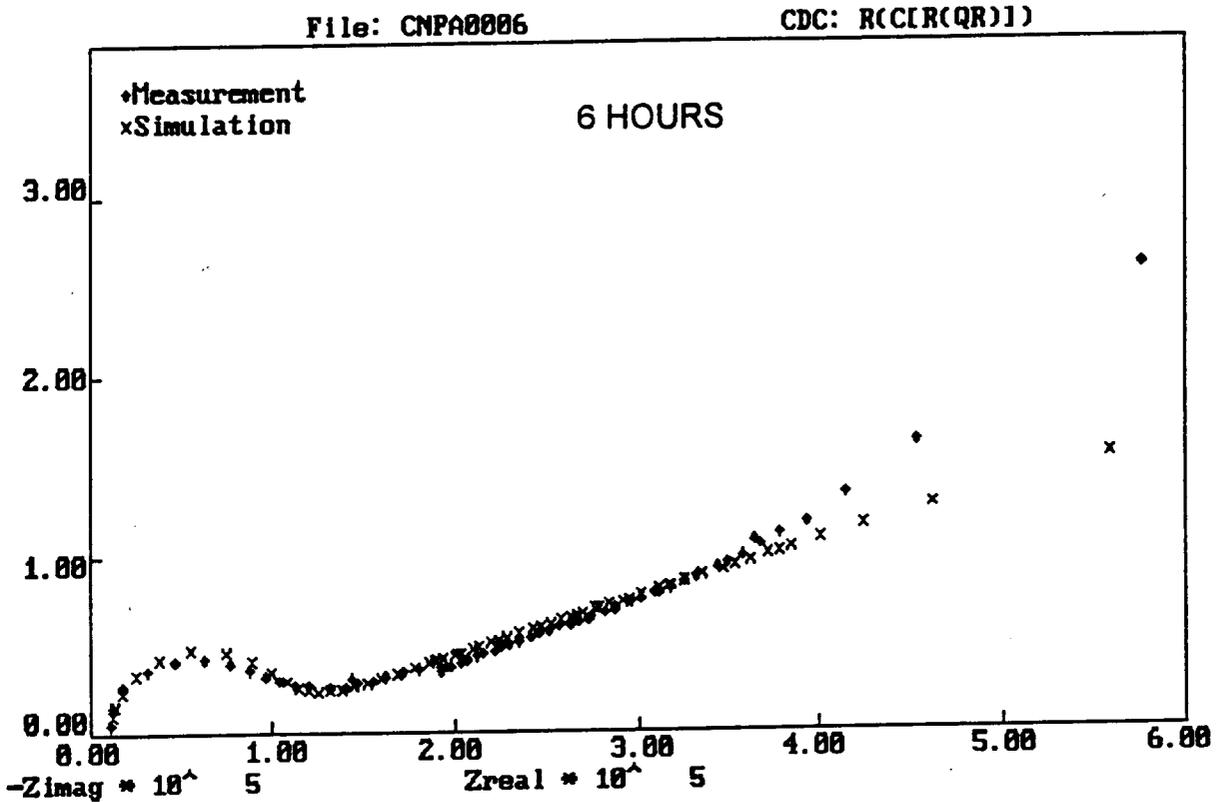


FIGURE B-2. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 6 HOURS IMMERSION IN NaCl

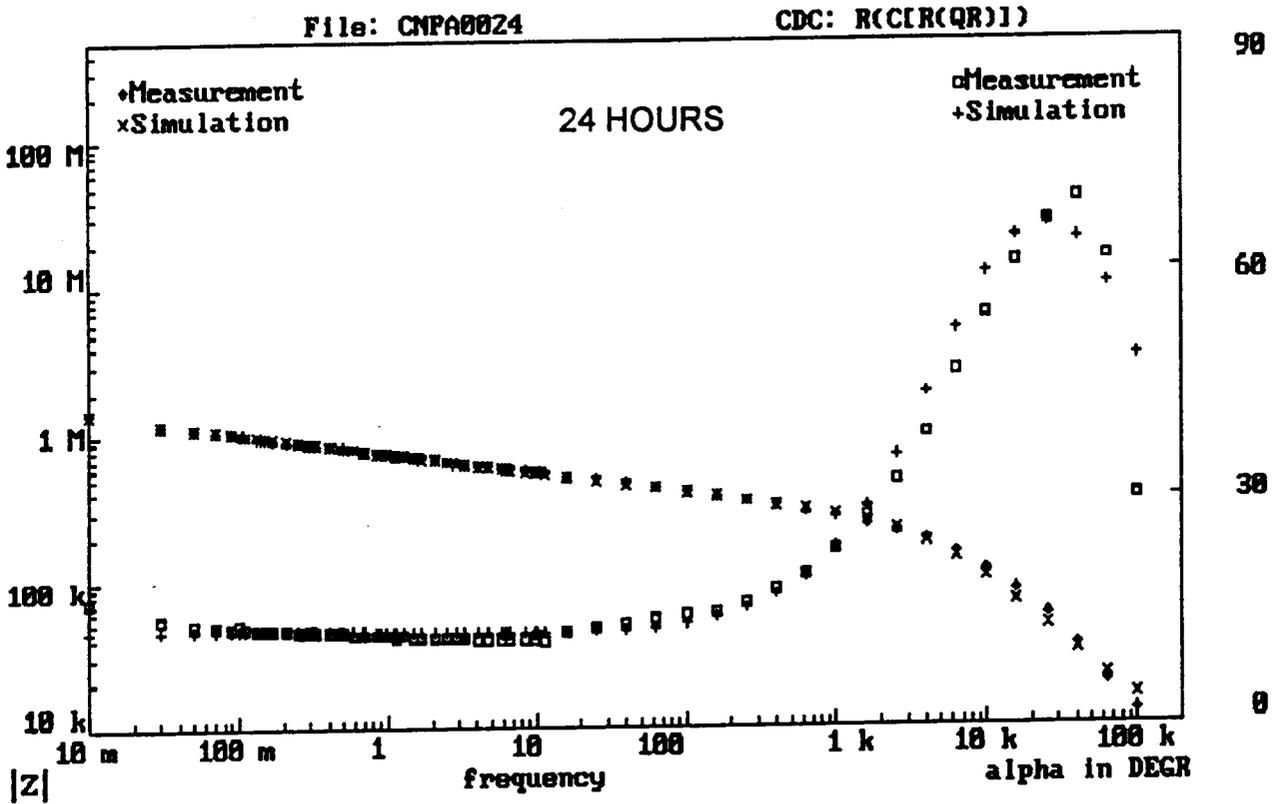
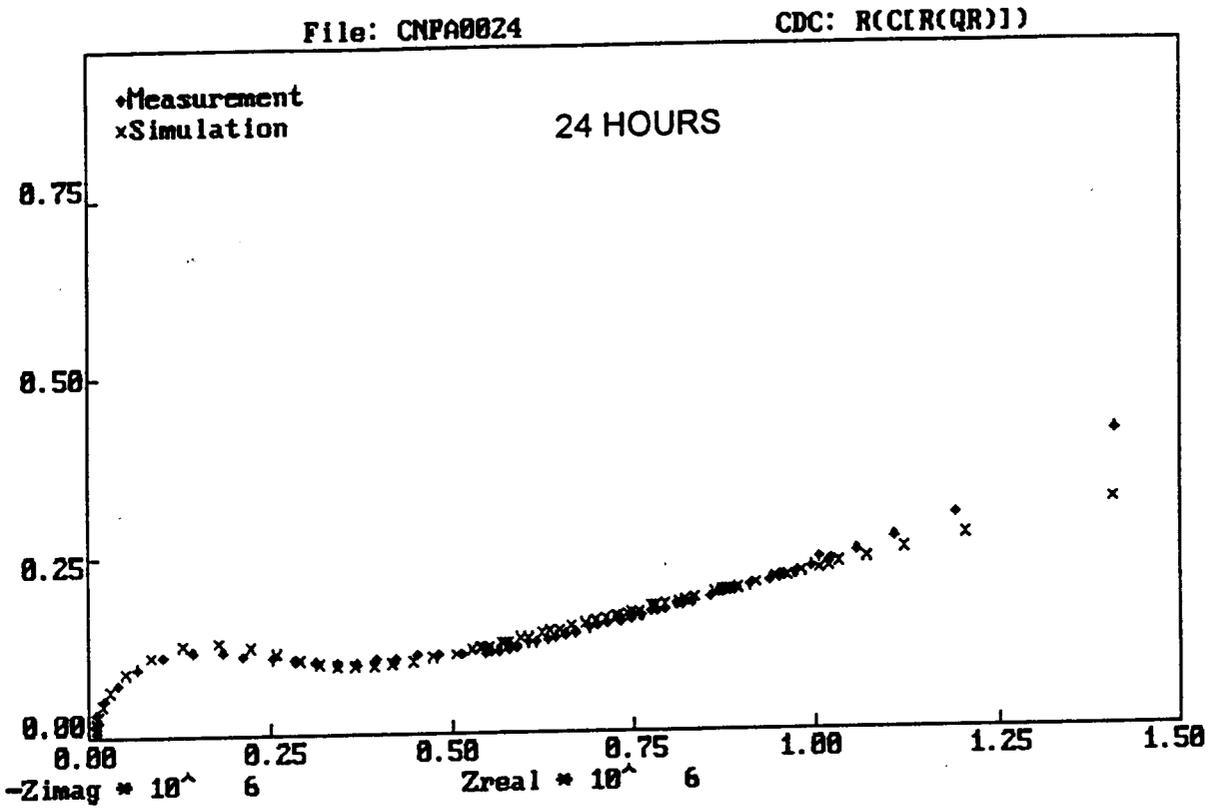


FIGURE B-3. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 24 HOURS IMMERSION IN NACL

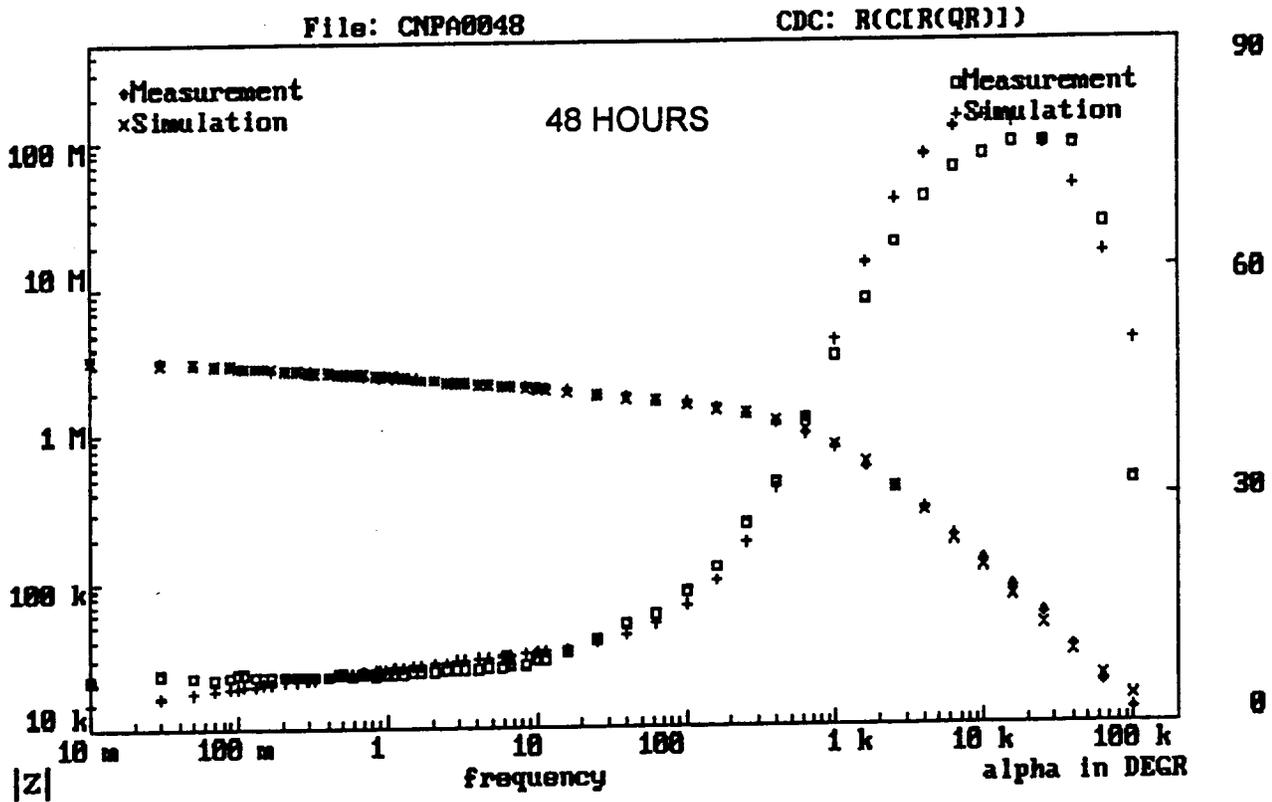
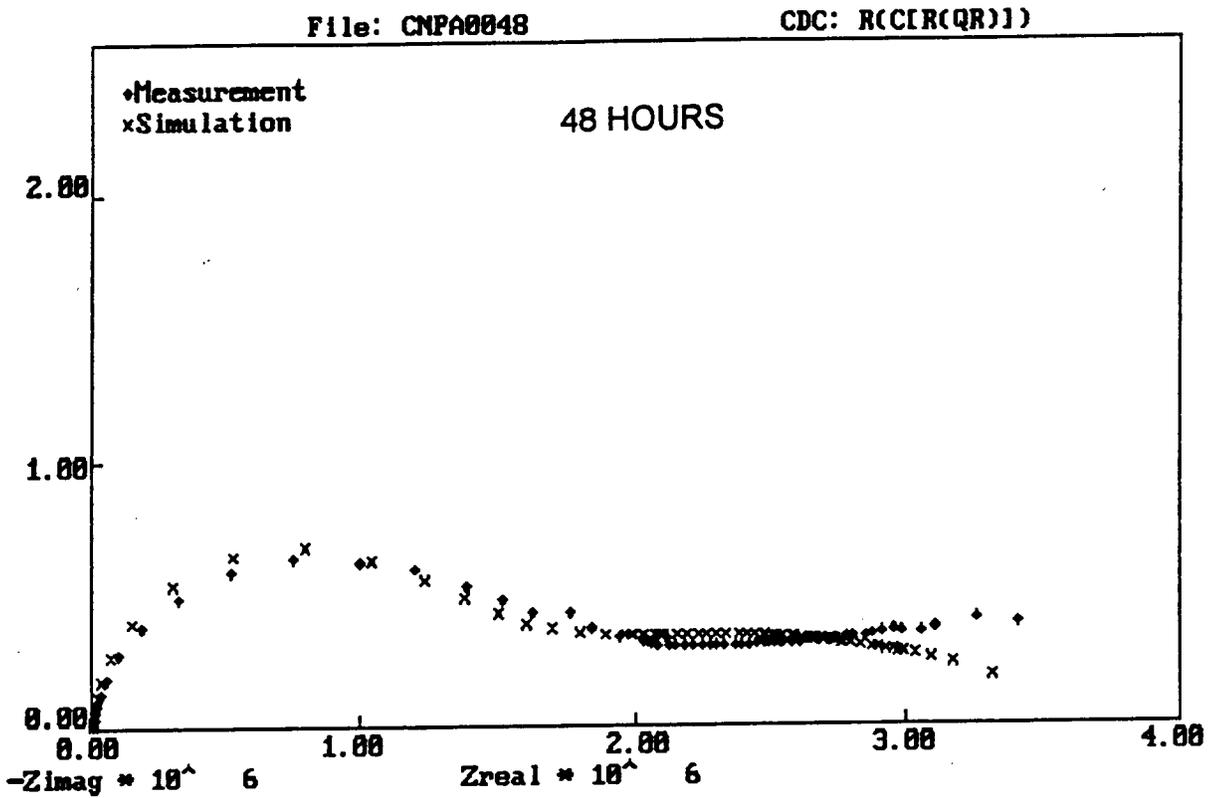


FIGURE B-4. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 48 HOURS IMMERSION IN NaCl

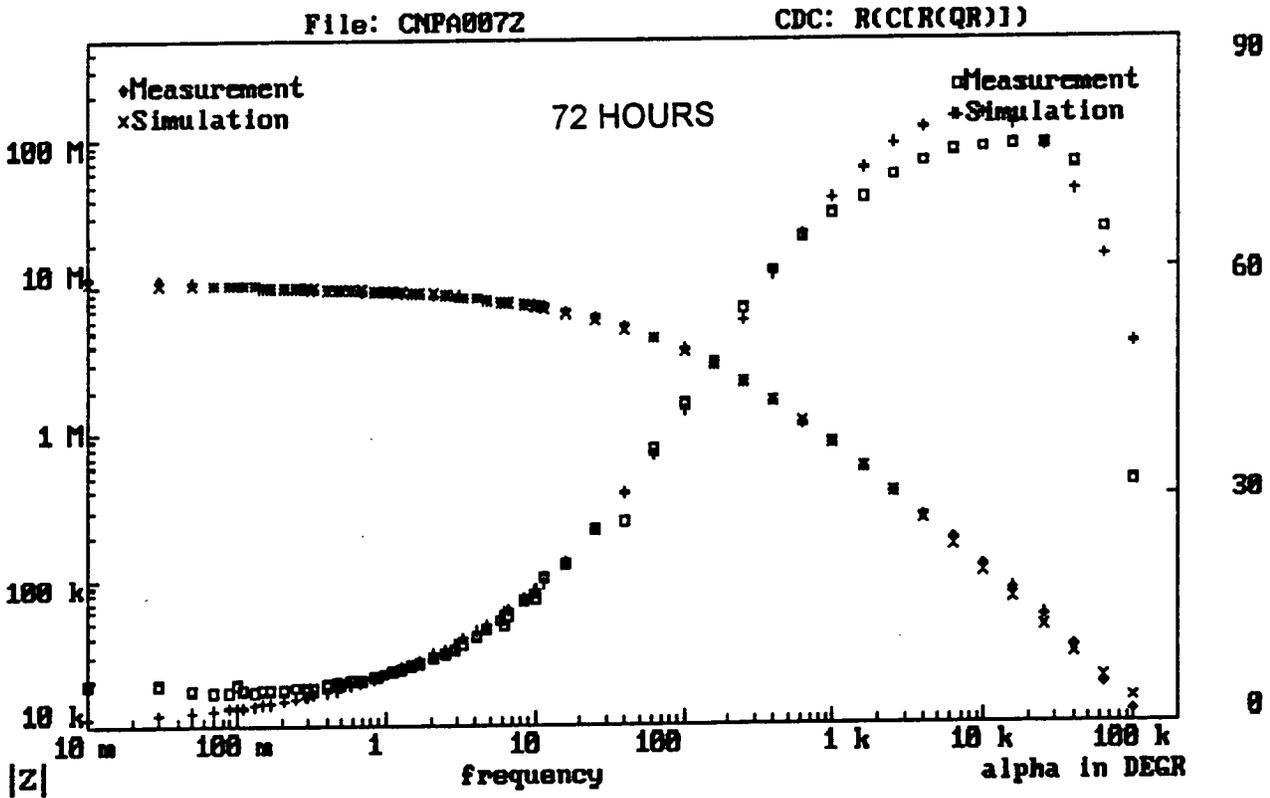
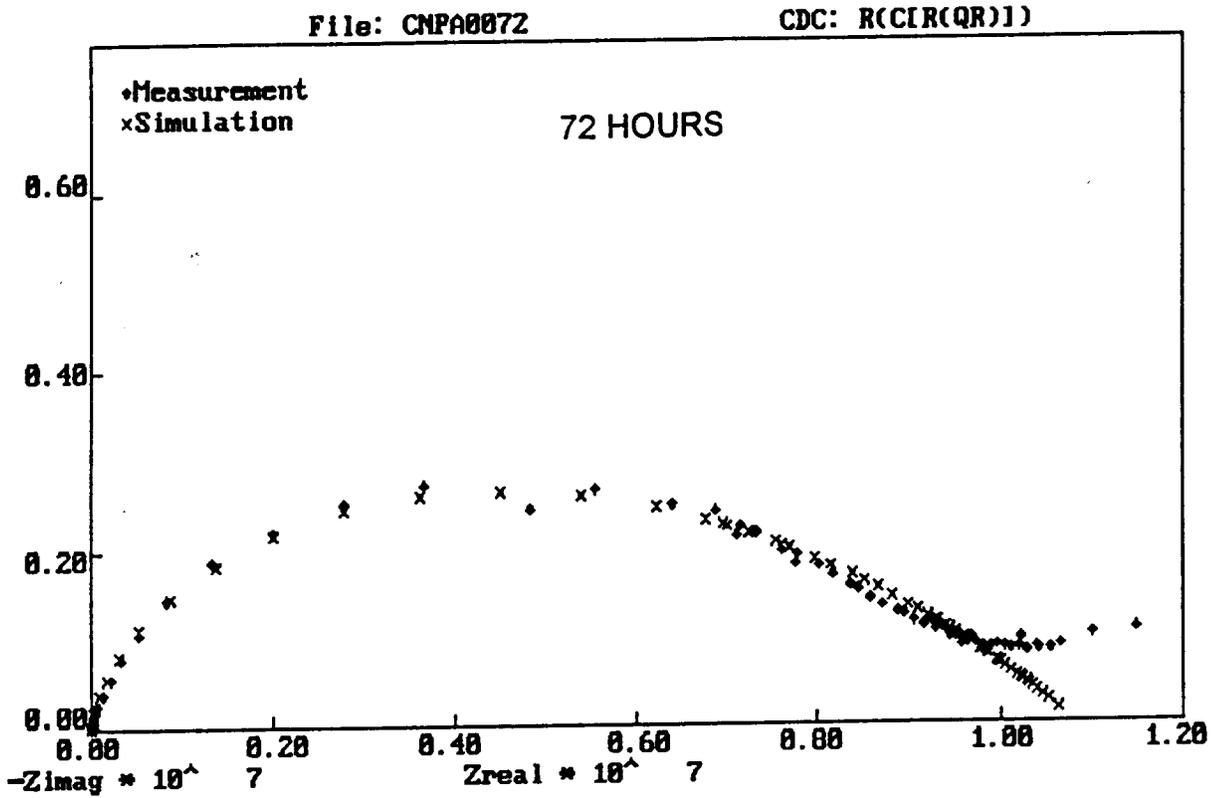


FIGURE B-5. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 72 HOURS IMMERSION IN NaCl

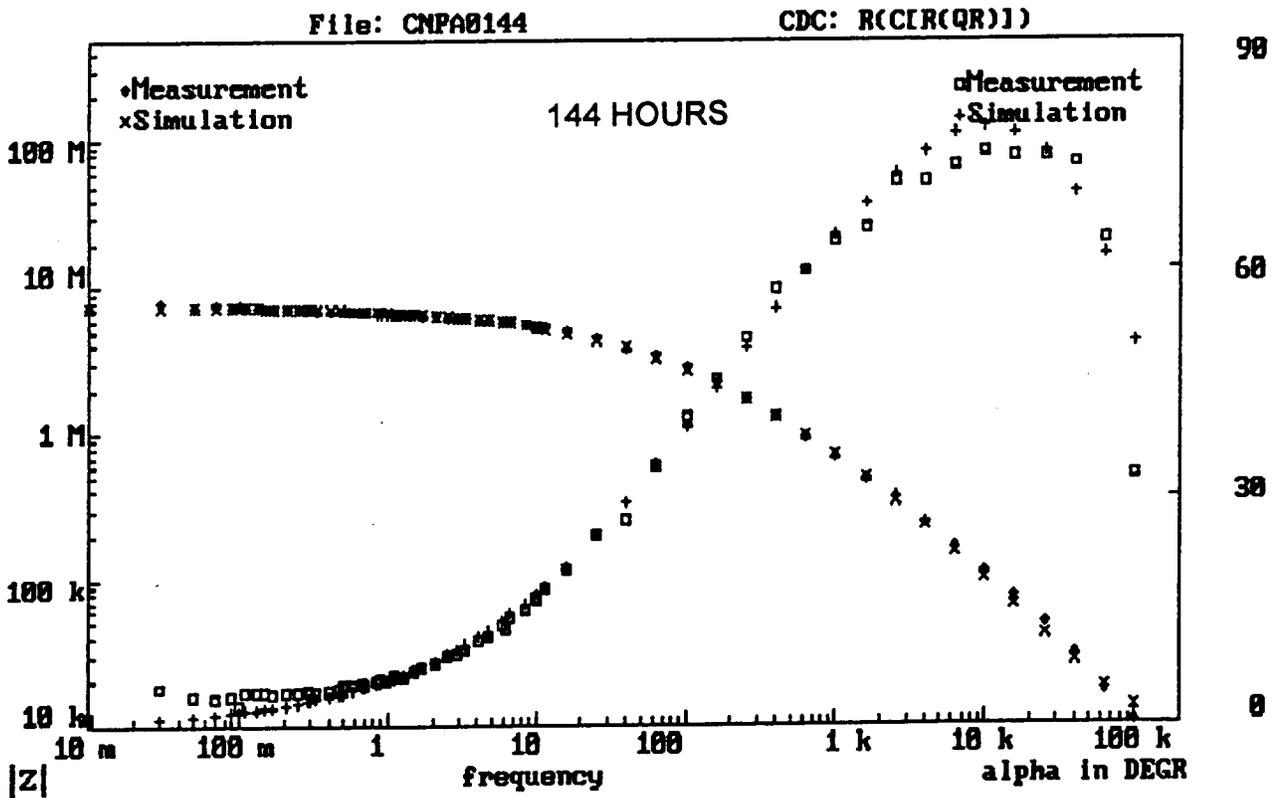
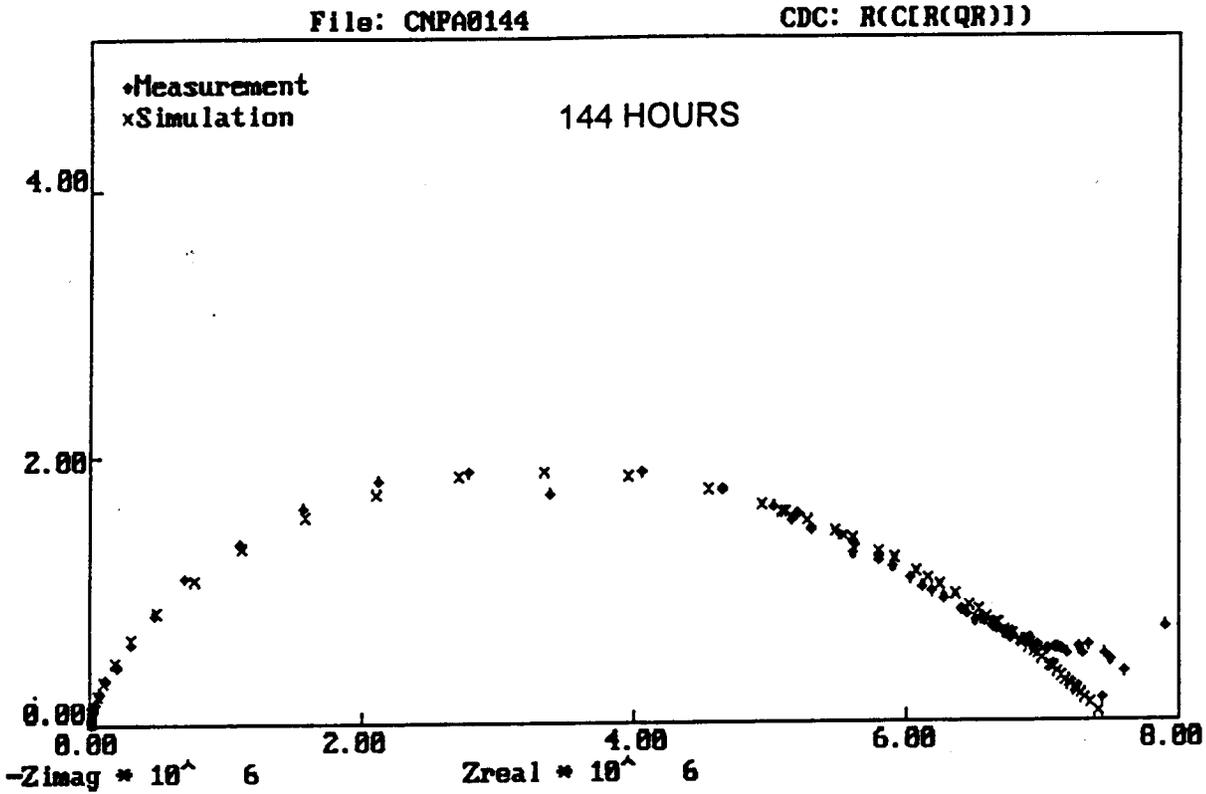


FIGURE B-6. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 144 HOURS IMMERSION IN NaCl

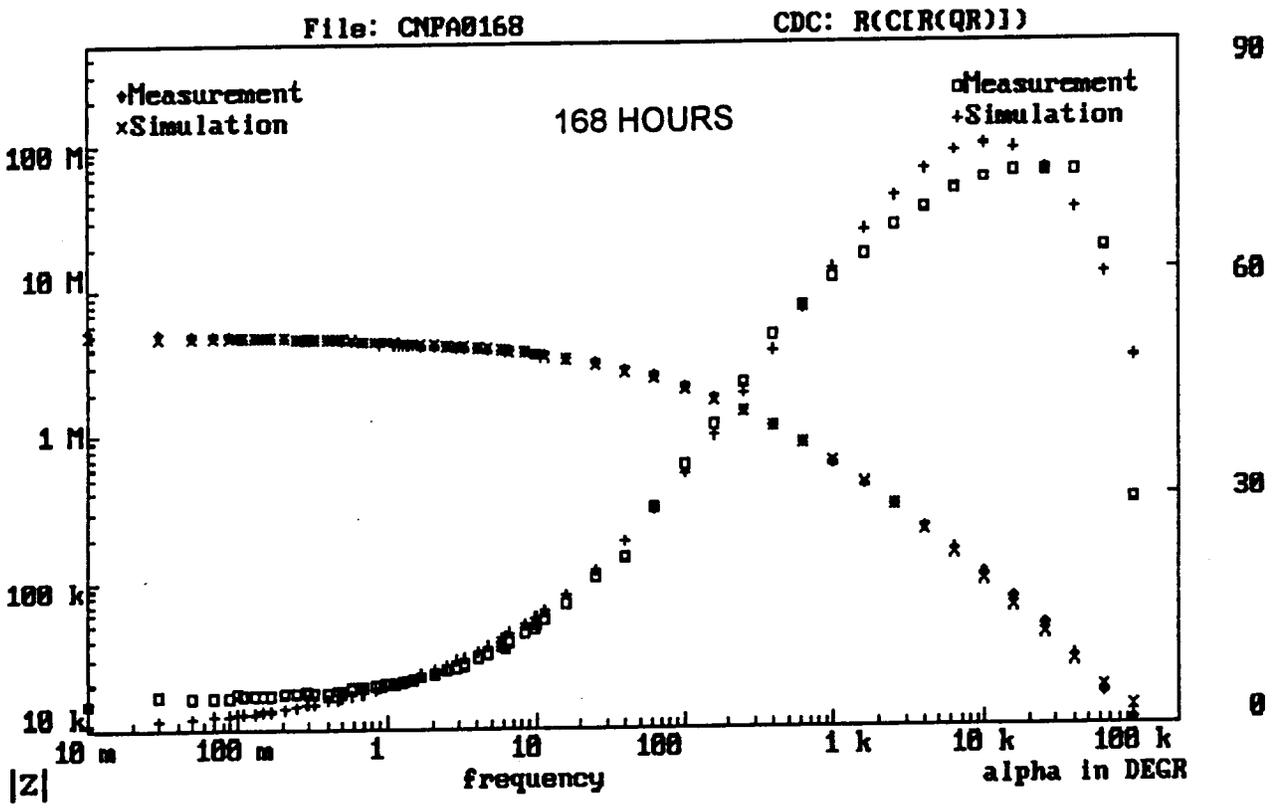
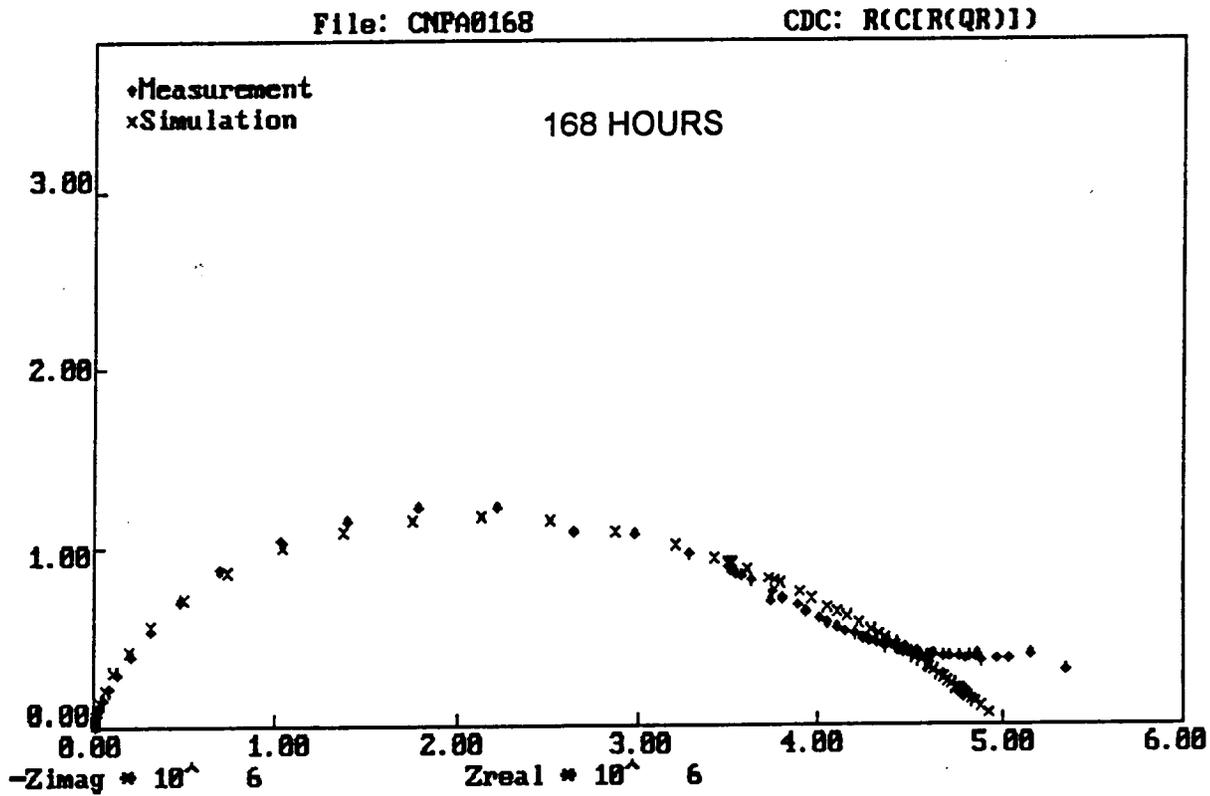


FIGURE B-7. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 168 HOURS IMMERSION IN NACL

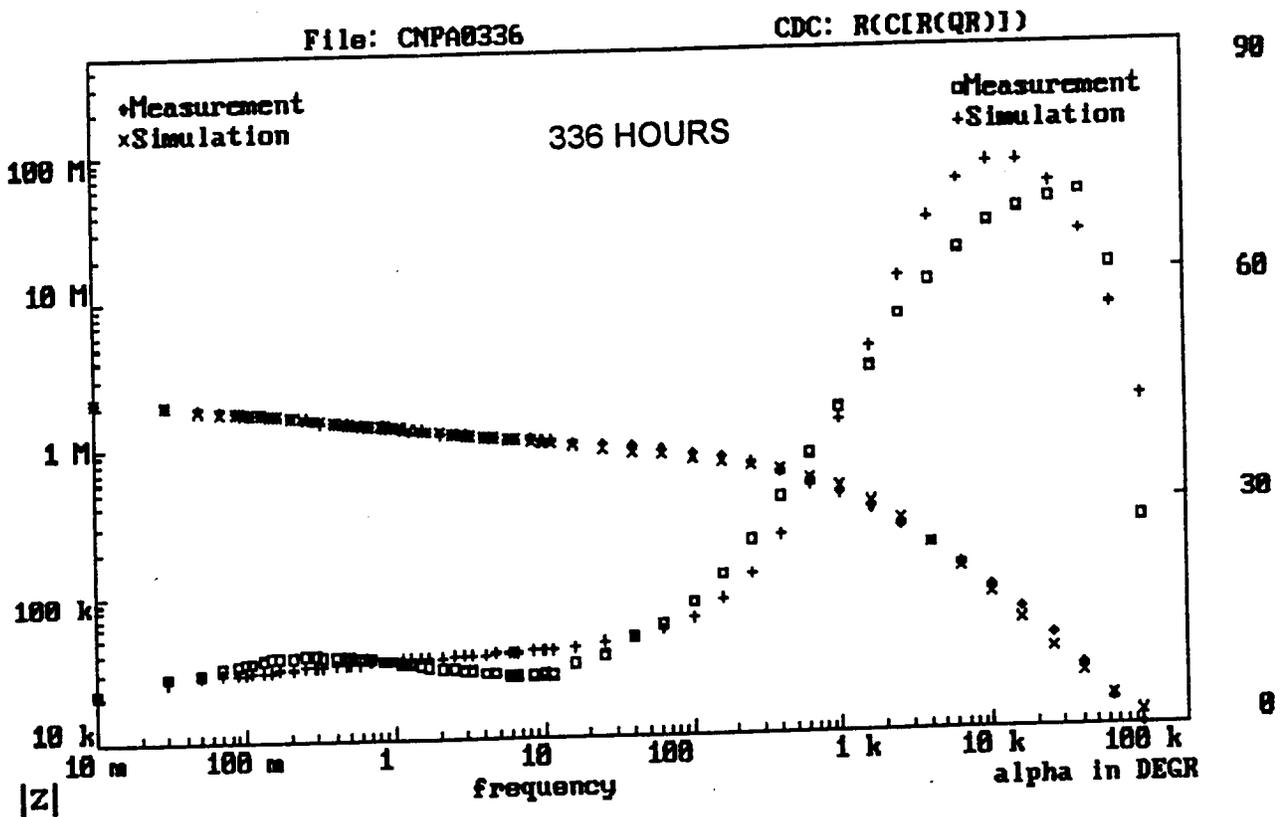
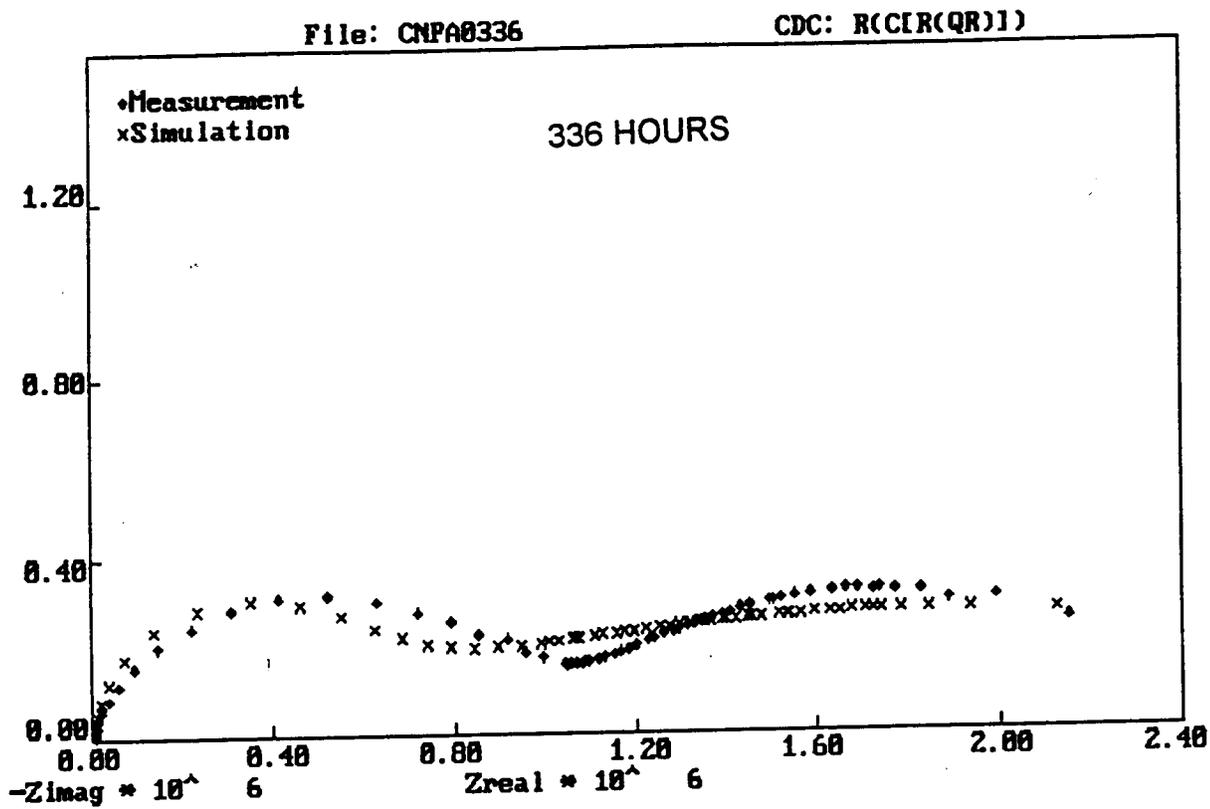


FIGURE B-8. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 336 HOURS IMMERSION IN NaCl

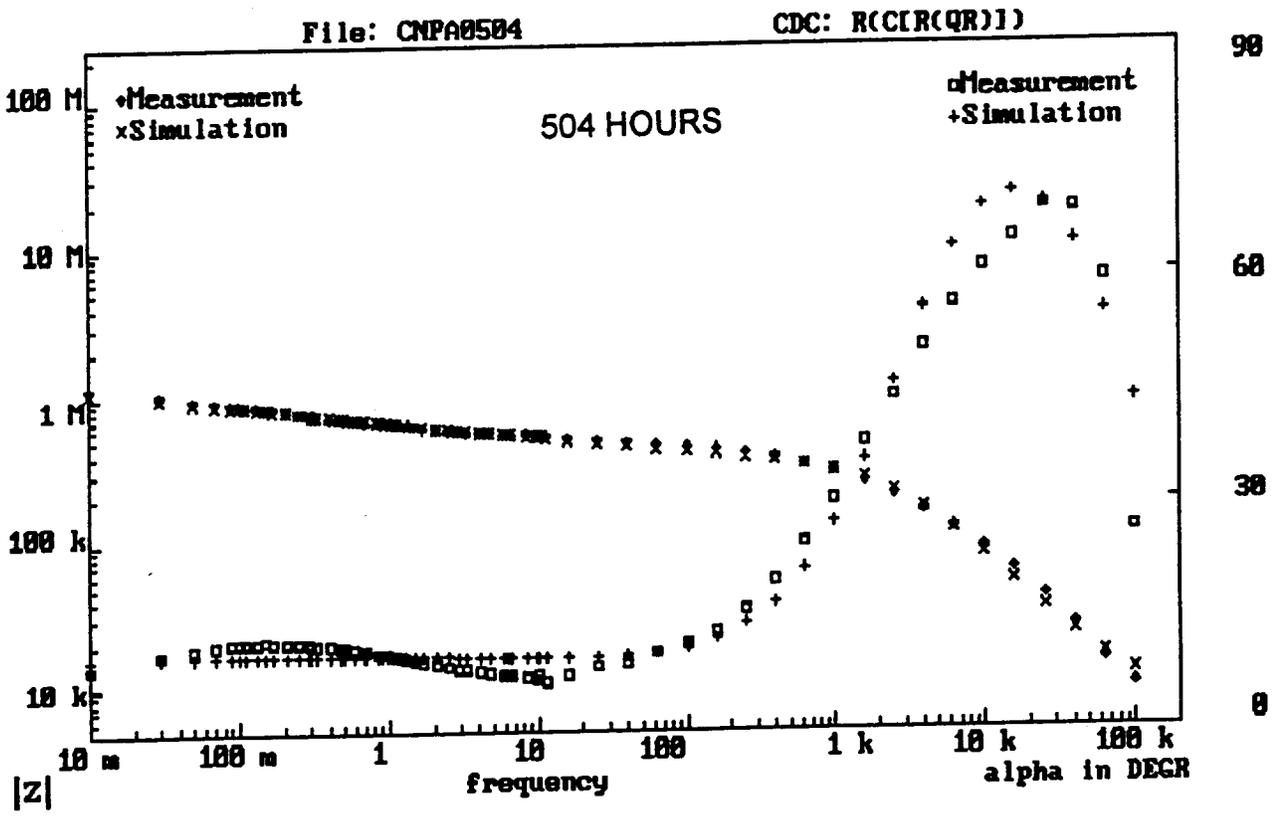
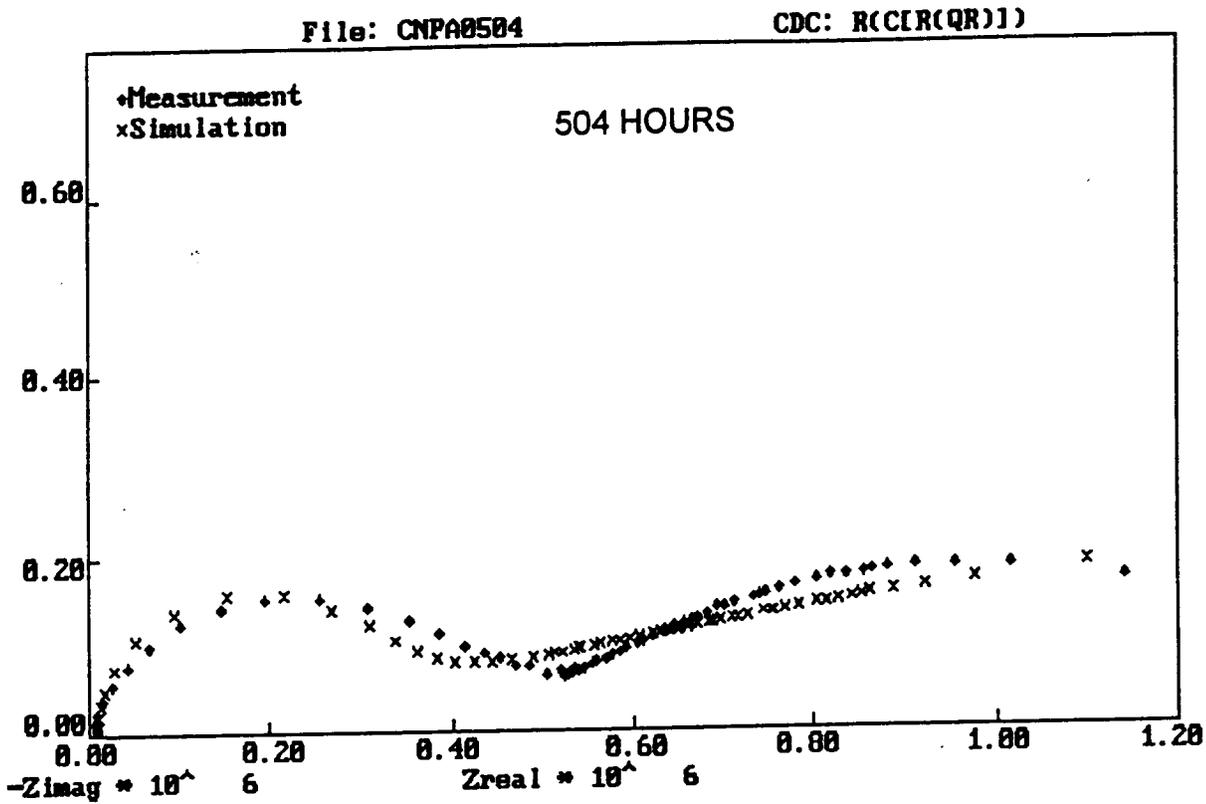


FIGURE B-9. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 504 HOURS IMMERSION IN NACL

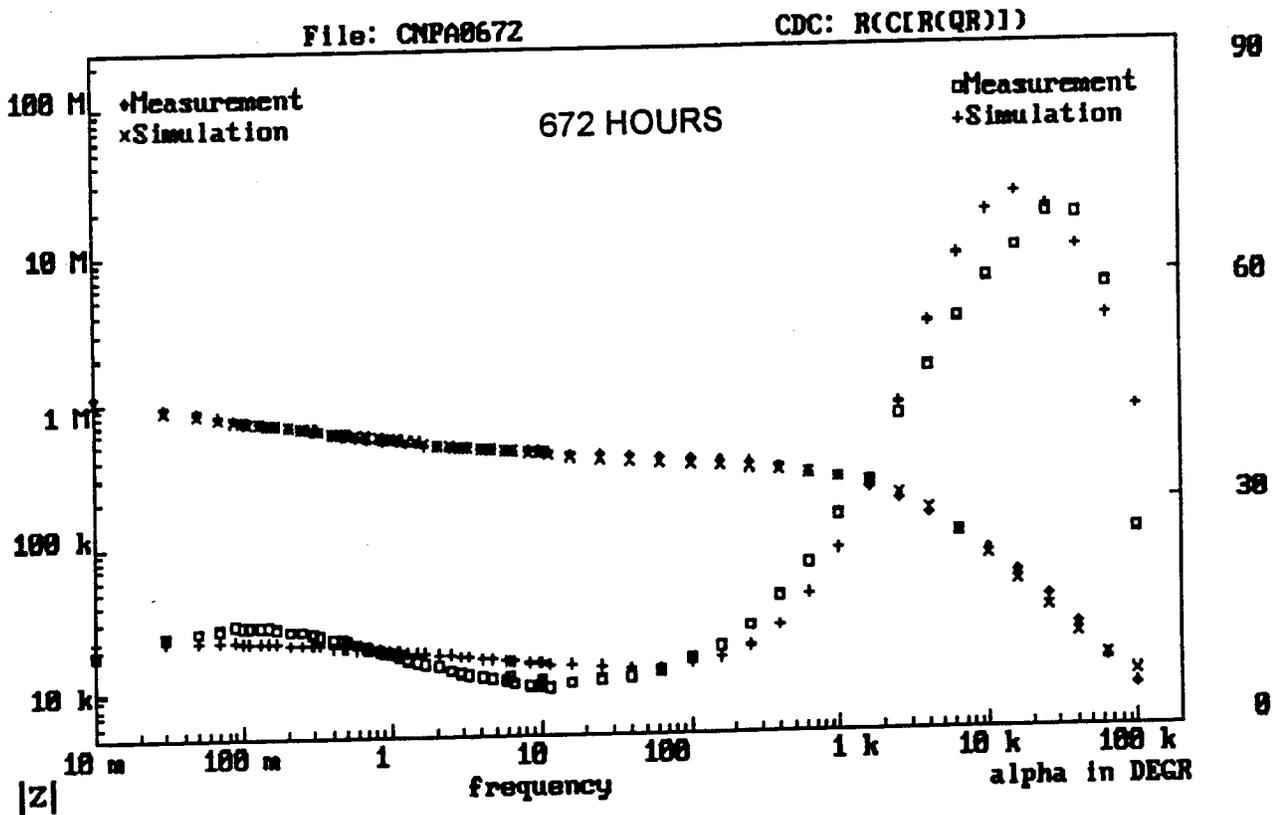
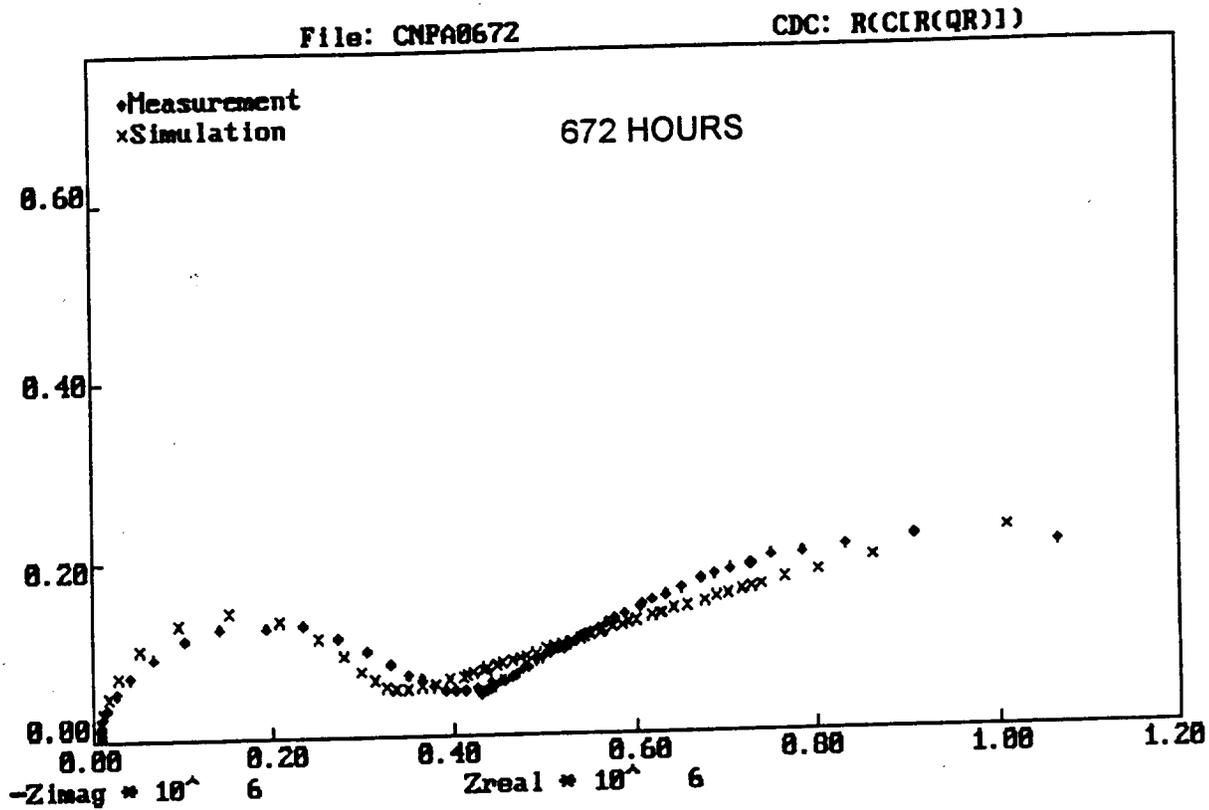


FIGURE B-10. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 672 HOURS IMMERSION IN NACL

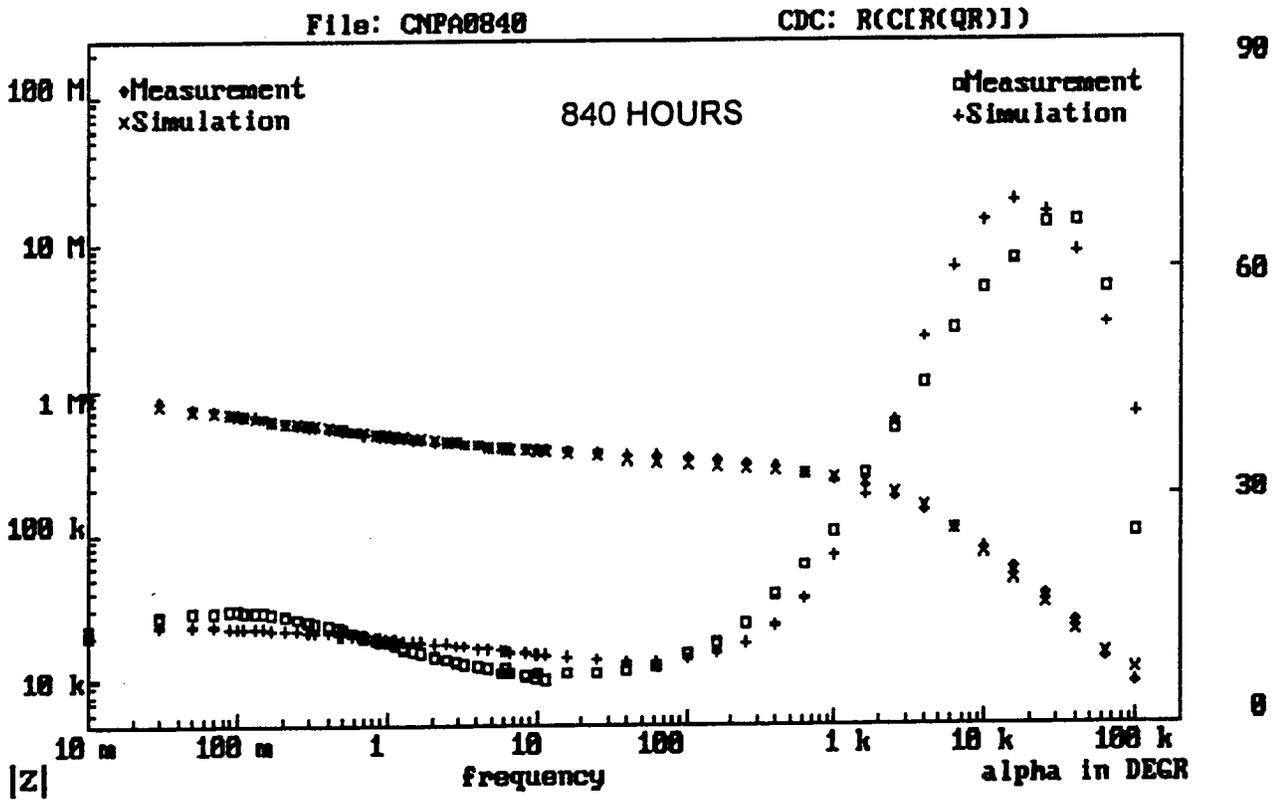
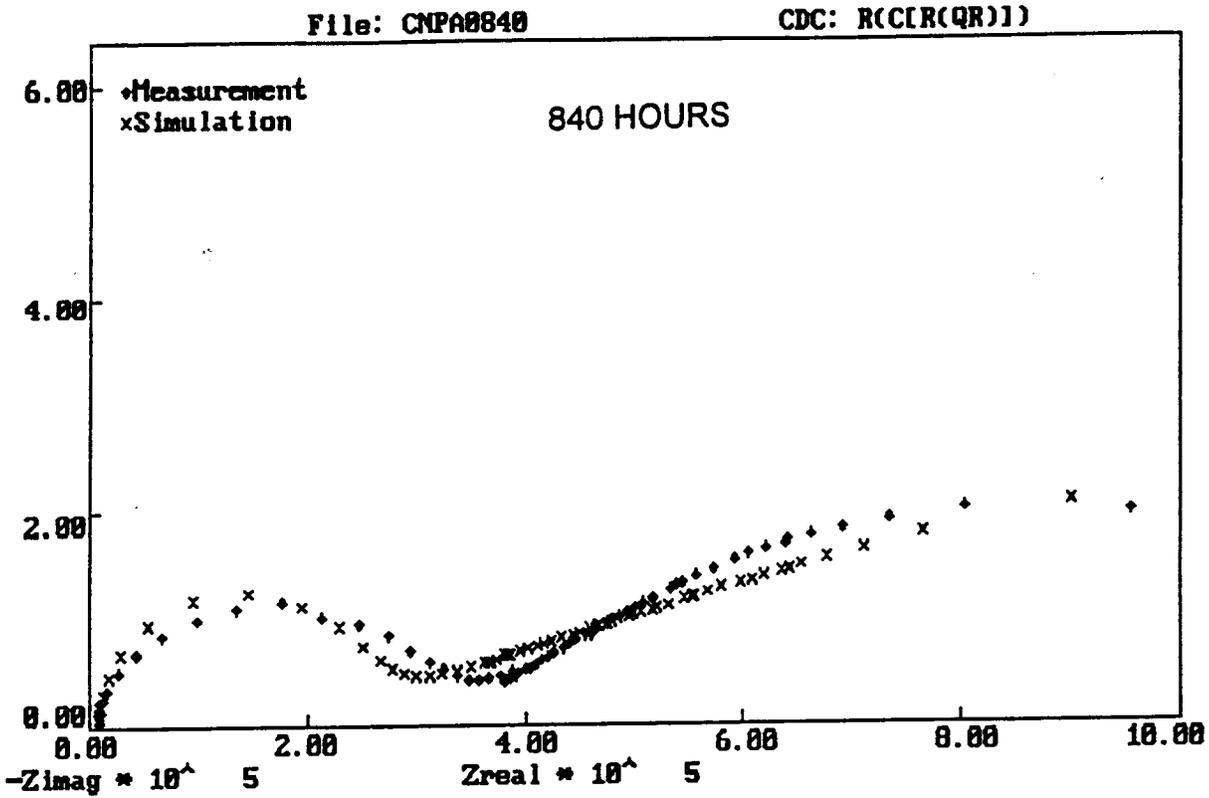


FIGURE B-11. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 840 HOURS IMMERSION IN NAACL

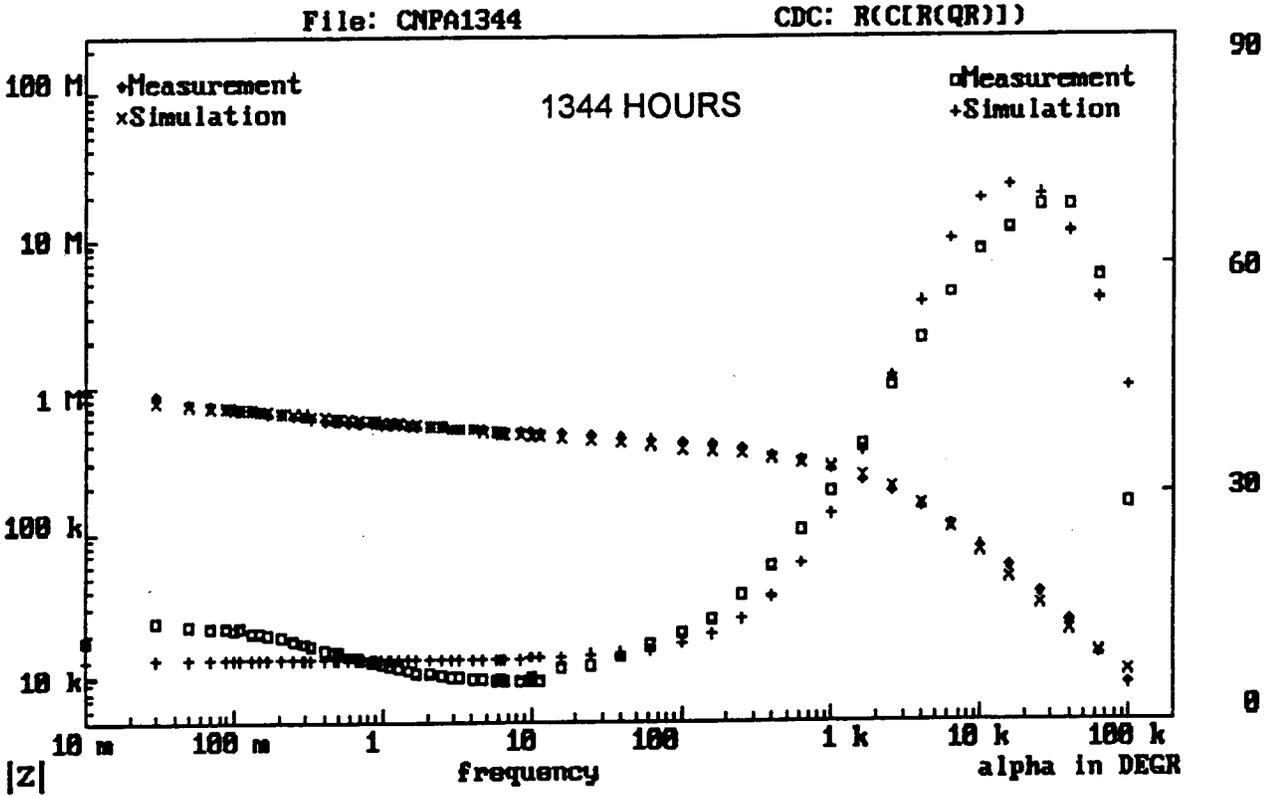
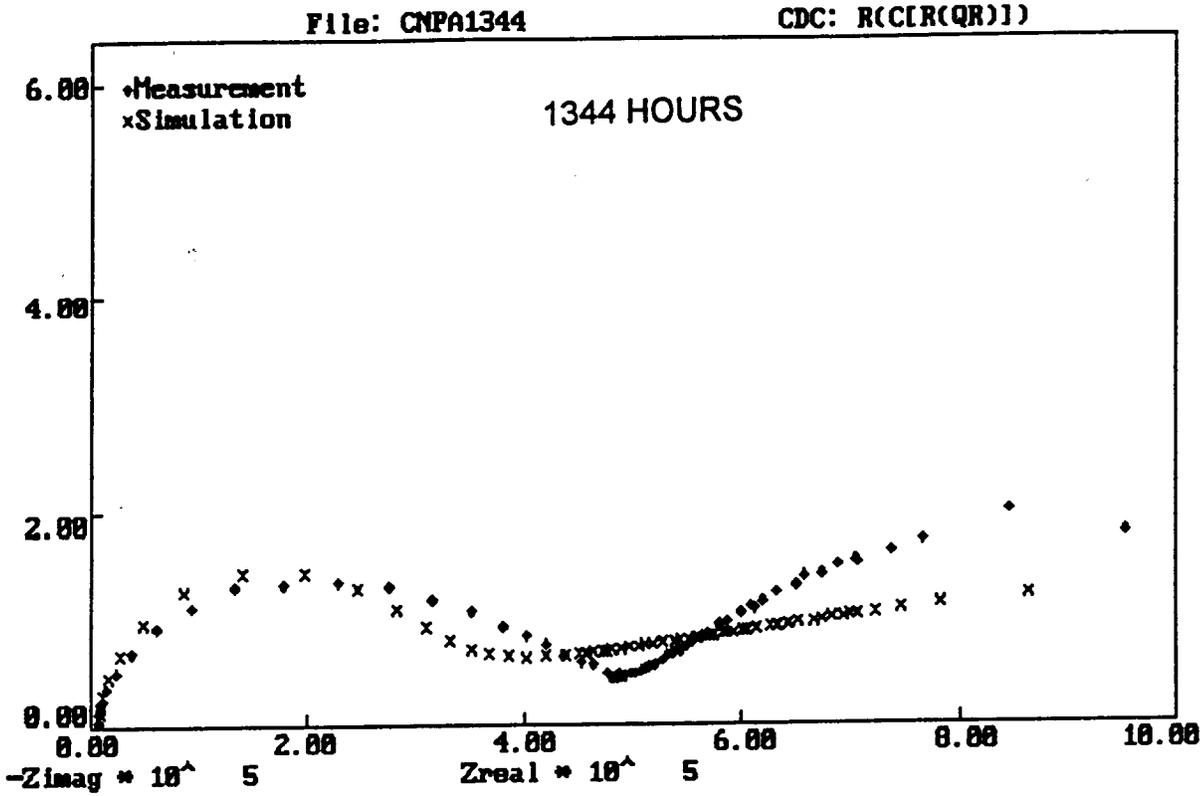


FIGURE B-12. NYQUIST, BODE, AND BODE MAGNITUDE/PHASE ANGLE, PLOTS FOR COATING B AFTER 1344 HOURS IMMERSION IN NACL

95-1M0070

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