

R 683



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Technical Report

RELATION BETWEEN CHANGES IN ELECTRICAL
PROPERTIES AND PERFORMANCE OF COATINGS
Experiments With Thirteen Immersed Coating Systems

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Experiments With Thirteen Immersed Coating Systems

Technical Report R-683

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by

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ABSTRACT

In an attempt to find a relatively rapid method of predicting the performance of coatings, changes in electrical properties were compared with the results of long-term field performance of the coatings. Thirteen coating systems on steel panels were immersed in seawater in the laboratory for 400 days, and the AC and DC electrical properties of the coatings were determined. Earlier experiments had indicated a relationship between changes in electrical properties and performance. New results with coatings of comparatively good performance indicate some correlation between changes in electrical properties and coating performance. However, the correlation is not sufficiently high to allow reliable prediction of the comparative performance of good coatings.

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INTRODUCTION

The Naval Civil Engineering Laboratory (NCEL) investigates and evaluates new coatings for the Naval Facilities Engineering Command and conducts research related thereto. The evaluation of the performance of such coatings under conditions similar to those encountered in actual service requires considerable time, especially if the coatings have long service lives. There is thus a strong need for reliable accelerated test methods.

Many accelerated tests have been described in the literature.¹ However, all the tests described appear to be of limited usefulness. Some tests show limited correlation for very specific applications. Others were found suitable for comparing two or three coatings without much being known about their general applicability.

Accelerated tests may be useful in the prediction of performance related to surface effects, such as chalking, fading, or loss of gloss. Thus, accelerated weathering machines are often claimed to give useful information about surface changes caused by aging.² No accelerated tests are known that are useful in reliably assessing the long-term protection or corrosion resistance of coatings. One method that showed some promise of being useful is the assessment of potential performance by electrical measurements on coatings.³

Several authors have suggested that electrical measurements on coatings immersed in an electrolyte can be used to predict coating performance.⁴⁻¹⁰ Organic coatings with good film integrity are good insulators and have electrical properties associated with good insulators, whereas coatings that have lost their film integrity have lost these electrical properties. It has been found that such changes in electrical properties can be detected much sooner than visual changes and that they can therefore be used to predict performance. However, no published papers really show good correlation of these changes in electrical properties with performance for any large number of coatings, and the validity of any such correlation has not really been demonstrated. Further investigations of some of the electrical methods, including measurements of DC resistance and of various AC properties such as capacitance, resistance, and dissipation factor, are needed to determine their validity as accelerated tests.

The results of AC electrical measurements with five coating systems on steel panels immersed in seawater have been reported.¹¹ The systems chosen for the NCEL initial study were of widely different performance in seawater.

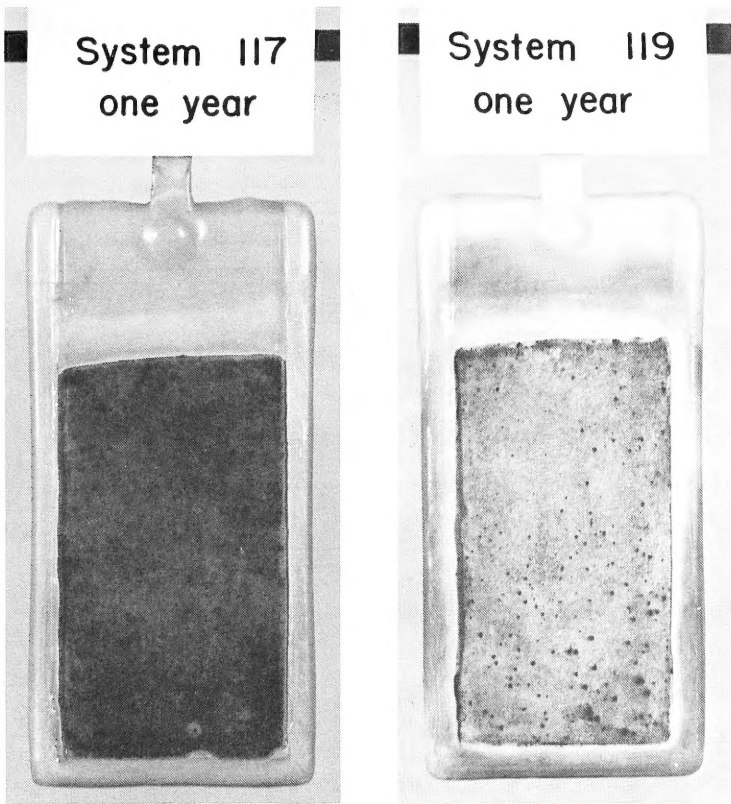
The results showed that there was appreciable correlation between the resistance and capacitance values of the coatings and the deterioration which occurred. Thus, the system with the lowest performance, an oil paint system, was the first to show reduced resistance values. The best performing system, a saran coating, had moderately high and very steady resistance values. The other three coatings were intermediate both in performance and in the stability of the resistance and capacitance values.

The results of both AC electrical measurements and DC electrical measurements of an additional seven coating systems similarly exposed have also been reported.¹² Four of these systems showed good electrical properties and very little deterioration in exposure tests. For two of the systems, the AC and DC resistance dropped considerably during the tests, and these systems also showed some deterioration in the tests. One of the seven systems was highly conductive and its electrical properties therefore could not be used as an indication of performance.

It was apparent from the above results that, in order to determine the validity of the method, it would be necessary to conduct experiments with a larger number of coatings for which the long-term performance was known. Additional experiments were therefore performed with 13 coating systems for which the long-term performance had been or was being determined by NCEL under other work units. The results of these experiments are reported and discussed below.

EXPERIMENTAL METHODS AND RESULTS

The coatings that were employed were applied to steel panels 2-3/4 inches wide and 5-7/8 inches high, which were made from 1/8-inch hot rolled steel plate. The panels were sandblasted and the coating systems were applied at thicknesses of approximately 10 mils (250 microns). Before the panels were coated, handles made of 1/4-inch stainless steel strips were attached to the panels with stainless steel machine screws. The coatings were applied with a spraying machine to insure even coverage. The edges were carefully touched up during the painting operation. After the painted panels had dried, the upper portions of the panels were covered with an epoxy coating which extended down 1-3/8 inches from the top, and the other three edges were dipped 1/4-inch deep into the same epoxy coating to further protect the edges. The top portions of the panels were then brushed with molten ceresin and the edges were dipped 1/4-inch deep into molten ceresin to further insulate the edges electrically from the seawater and to reduce any edge effects in the AC measurements. The area of the coating of the nominal thickness was thus approximately 5.6 cm by 10.6 cm on each side for a total area of 120 cm² on the two faces of the panels. Two of the panels are shown in Figure 1.



(a) System 117 after 400-day exposure.

(b) System 119 after 400-day exposure.

Figure 1. Two exposed panels.

The coating systems that were employed in the experiments discussed below consisted of a variety of organic coatings of different generic types. Included were four vinyl systems, four epoxy systems, two phenolic systems, and three other systems. All these systems had been exposed and evaluated in marine atmospheric environments,¹³ and most of the systems also had been exposed in seawater.¹⁴ The systems are described in Table 1. The coatings were essentially the same thickness as those exposed in the field. One exception is System 120, which was applied approximately twice as thick for the electrical measurements.

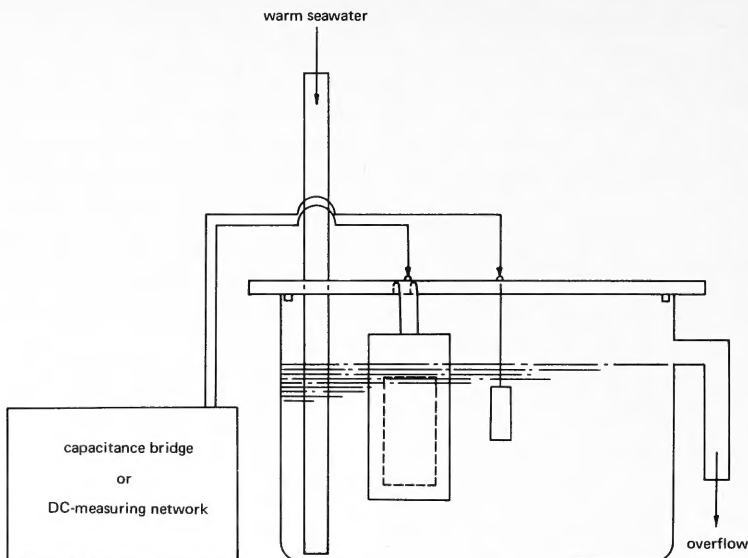


Figure 2. Experimental setup.

Three panels of each system were exposed in flowing seawater. The baths in which the panels were suspended (Figure 2) were rectangular glass jars 8 inches wide, 11-1/2 inches long, and 8 inches high. Aerated seawater was maintained at 25°C in a reservoir and was allowed to flow into the baths through an inlet tube extending to the bottom at one end of the jar. At the other end of the jar, approximately 2-1/4 inches from the top edge, a hole was drilled to accommodate an outlet tube. The panels were suspended from square fiber glass rods which were placed on the top edge of the jars. The length of the hooks and the level of the seawater was such that the 120 cm² of test surface of each panel was always immersed.

The AC electrical measurements were made with a capacitance measuring assembly consisting of a capacitance bridge, an audio oscillator, and a tuned amplifier and null detector (General Radio model 1620). The experimental setup is shown schematically in Figure 2, but for the actual measurements, the glass jar of salt water was removed from the source of warm flowing seawater and was placed into a water bath accurately maintained at 25°C.

Table 1. Coating Systems Investigated

No.	Type	Color	Individual Coatings	Thickness (mils)	Total Thickness (mils)
111	epoxy-phenolic	medium gray	epoxy metal primer epoxy-phenolic finish	4.3 10.2	14.5
112	urethane	gray	polyurethane zinc chromate primer polyurethane finish	1.3 6.3	7.6
113	epoxy	white	epoxy-zinc chromate primer epoxy finish	3.4 9.4	12.8
114	phenolic	gray	modified-phenolic primer modified-phenolic finish	7.9 8.0	15.9
115	epoxy	gray	epoxy primer epoxy intermediate epoxy finish	0.9 1.4 5.3	7.6
116	chlorosulfonated polyethylene	gray	red-lead iron oxide vinyl primer chlorosulfonated polyethylene finish	1.6 6.5	8.1
117	phenolic mastic	gray	mica-filled phenolic mastic primer phenolic mastic finish	13.0 4.8	17.8
118	vinyl	gray	vinyl-phenolic strontium chromate iron oxide primer vinyl finish	1.5 5.0	6.5

continued

Table 1. Continued

No.	Type	Color	Individual Coatings	Thickness (mils)	Total Thickness (mils)
119	epoxy	tan	epoxy primer epoxy intermediate epoxy finish	1.1 2.6 2.5	6.2
120	vinyl mastic	black	vinyl-phenolic strontium chromate iron oxide primer vinyl mastic finish	1.5 35.0	36.5
121	coal-tar urethane	black	coal-tar urethane	8.0	8.0
122	aluminum vinyl	aluminum	pretreatment primer vinyl red-lead primer aluminum-pigmented vinyl finish	0.5 3.4 3.9	7.8
123	saran	white	saran (formula 113/54)	7.0	7.0

The capacitance-measuring assembly provided series capacitance and dissipation factor readings and also parallel capacitance and conductance. The resistance values were calculated from the conductance values. The measurements were made with three-terminal connection to the capacitance bridge. A lead with grounded shield was attached by an insulated alligator clip to the handle of the panel being measured and contact with the seawater was maintained through a cylindrical platinum screen electrode 15 mm in diameter and 50 mm high. The connection from the capacitance bridge to this electrode also had a grounded shield. Measurement errors with this assembly were negligible, even at frequencies up to 10 Hertz.

The capacitance and dissipation factor measurements and the conductance measurements were made as frequently as possible immediately after exposure and during the first day of exposure; they were then made less frequently as the exposure continued. The average values obtained for the three panels are plotted in Figures 3 to 15. The average initial values are indicated at the left-hand ordinate scale of the curves. The first measurement that was made after an initial 24-hour period and further measurements up to 400 days are shown in the curve. Where the measurements for some panels deviated considerably from the average value, these values are indicated at the right-hand ordinate.

Some of the electrical properties are also shown in numerical form in Table 2. The values listed are the initial values, the values after 6 hours, and the values after 10 days, 150 days, and 400 days (or approximately 1 year). The values shown are the averages of the better panels. Values which were considerably inferior to the averages (that is, low resistance values and high capacitance or dissipation factor values) were not included in the averages, as discussed below. In addition to the resistance and capacitance values at the particular exposure times, the ratios representing the changes from the original values, and also the logarithms of these ratios are shown.

The AC resistance, capacitance, and dissipation factor values obtained during the first 6-hour immersion period and during the first 10-day immersion period were also separately plotted for each system. The curves obtained for the four systems having the most significant changes are shown in Figures 16 to 19.

After approximately 3 hours, and again after approximately 2 weeks, the dissipation factors at different frequencies from 200 Hertz to 10 kHz were also determined for each system. The curves for the 14-day values for 12 of the systems are shown in Figures 20 and 21. Also shown is a plot of the 2-hour values for one of the systems.

DC electrical measurements were made by a modification¹² of the methods of Bacon⁴ and of Brown.⁵ Contact to the seawater was maintained with a calomel electrode, and voltage measurements were made for the voltaic cell: panel/coating system/seawater/calomel electrode. An electrometer with an input resistance of 10^{14} ohms was used (Keithley model 610).

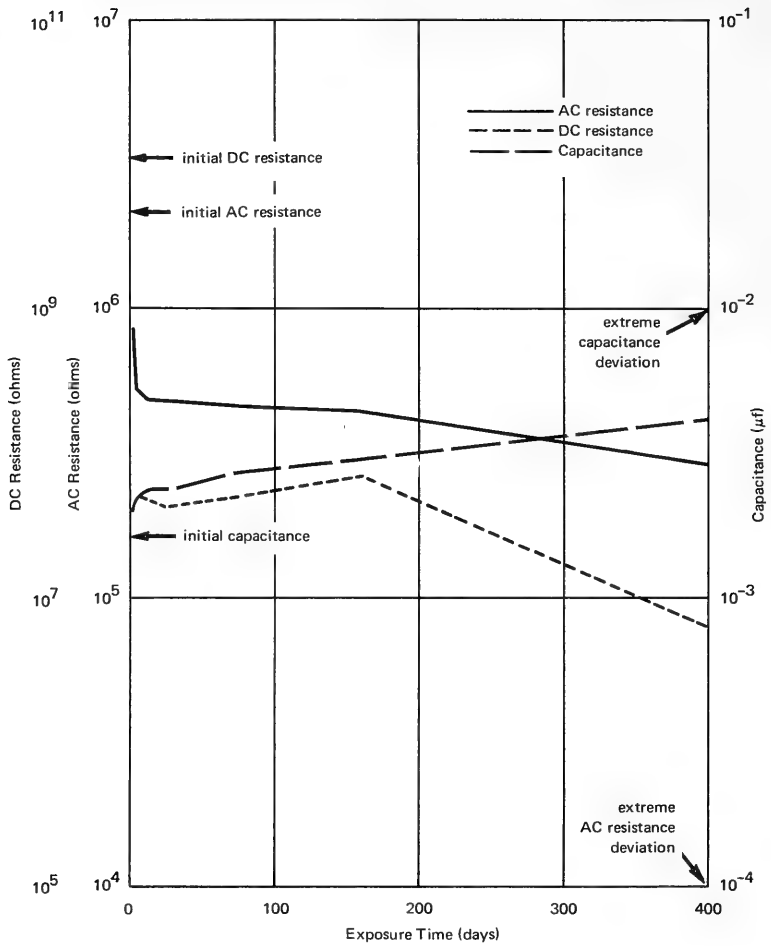


Figure 3. Electrical properties of System 111.

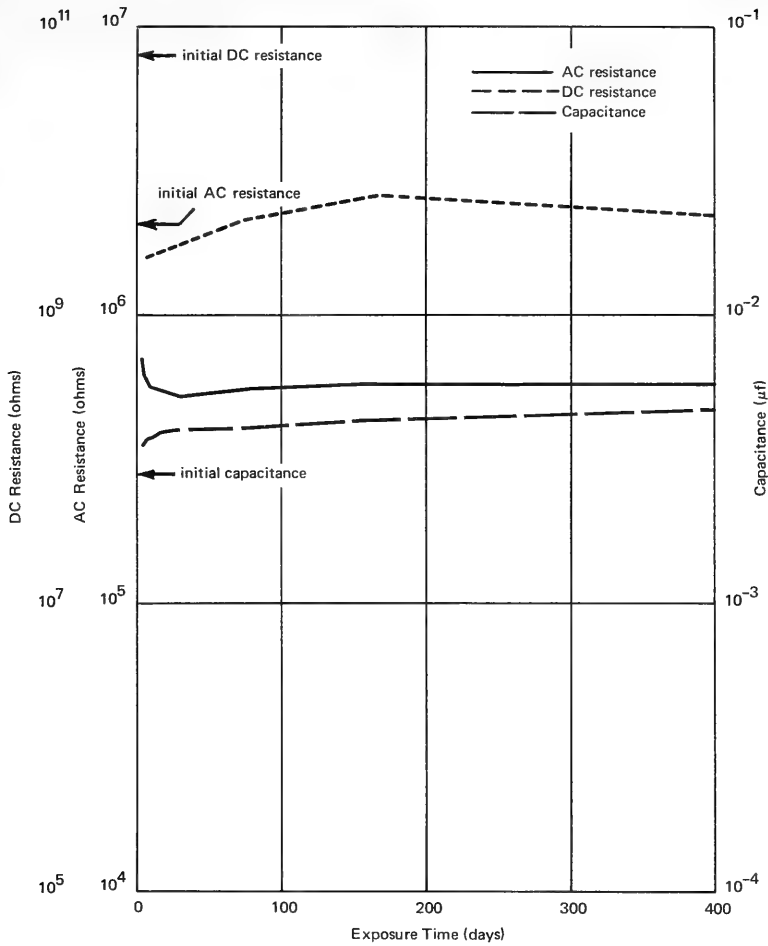


Figure 4. Electrical properties of System 112.

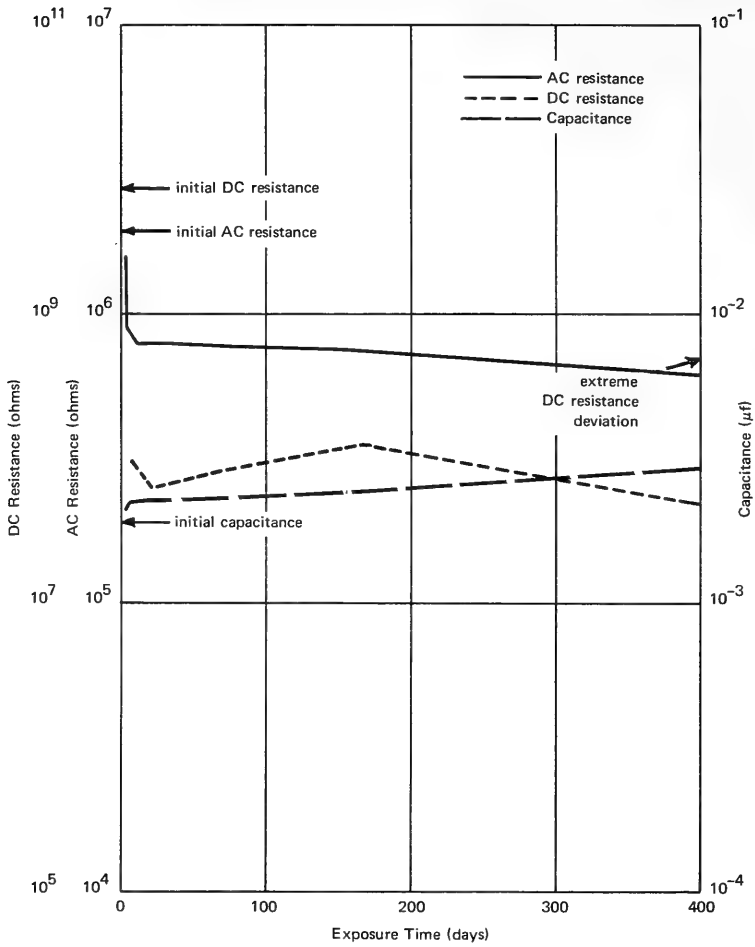


Figure 5. Electrical properties of System 113.

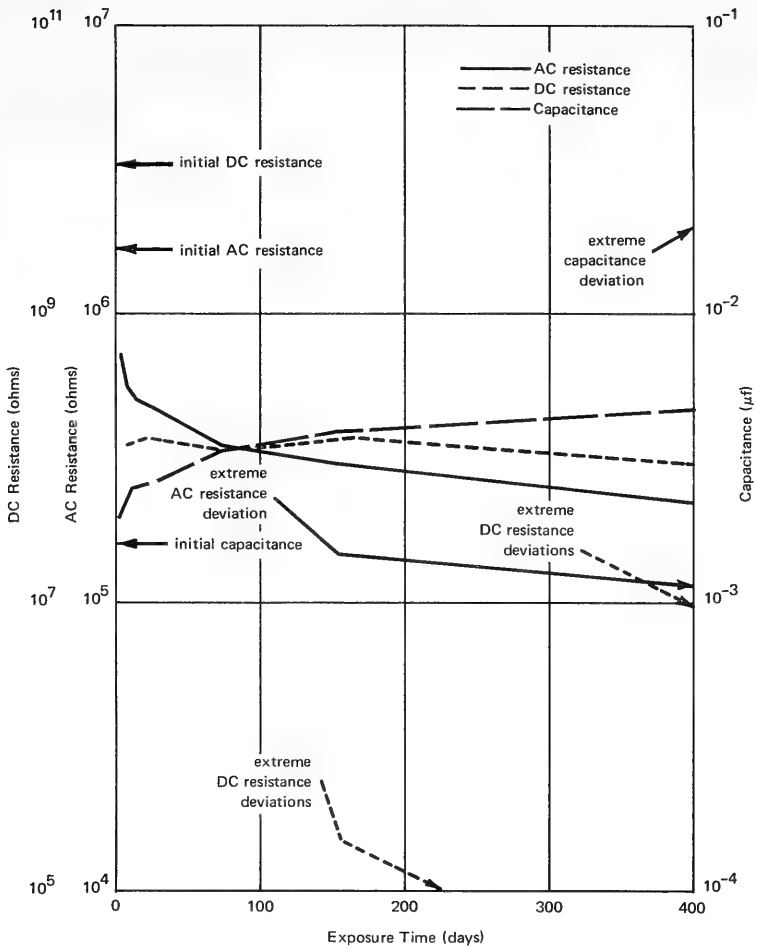


Figure 6. Electrical properties of System 114.

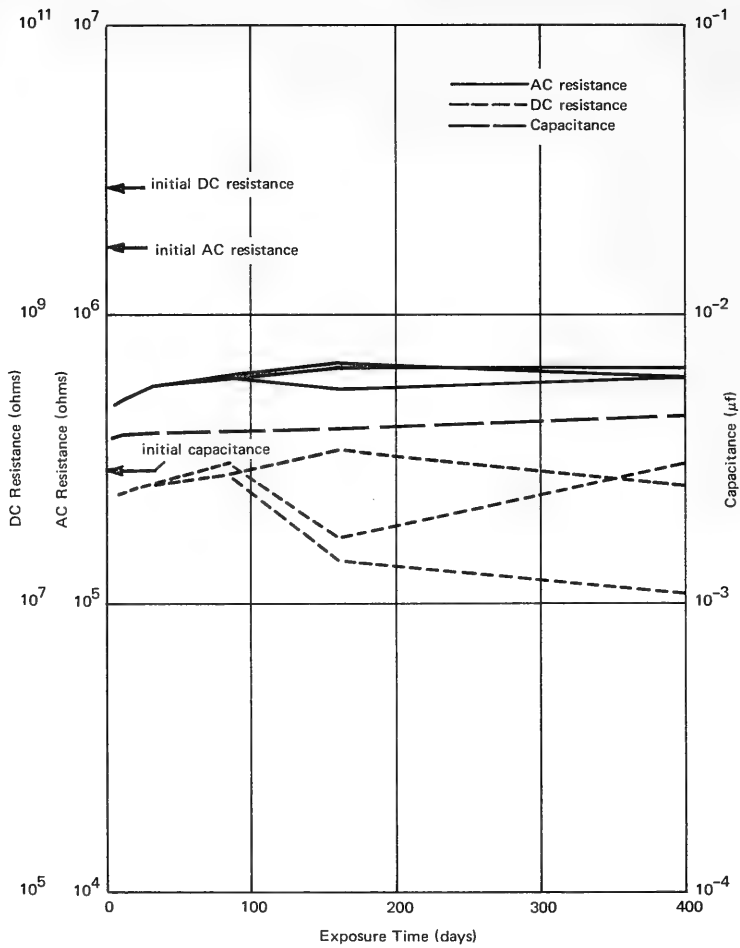


Figure 7. Electrical properties of System 115.

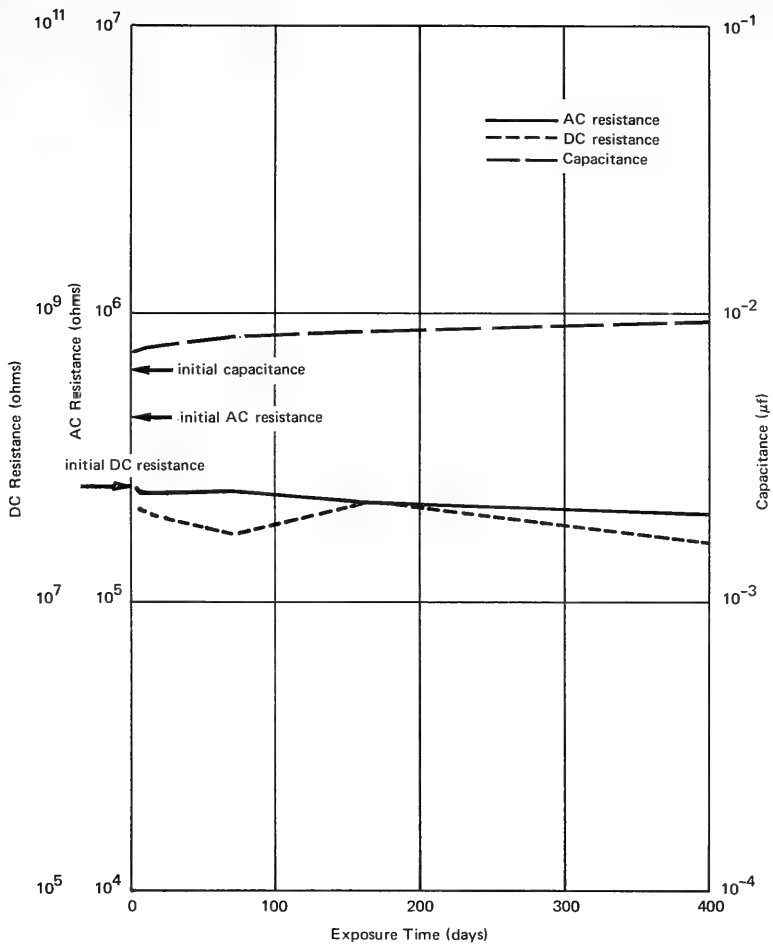


Figure 8. Electrical properties of System 116.

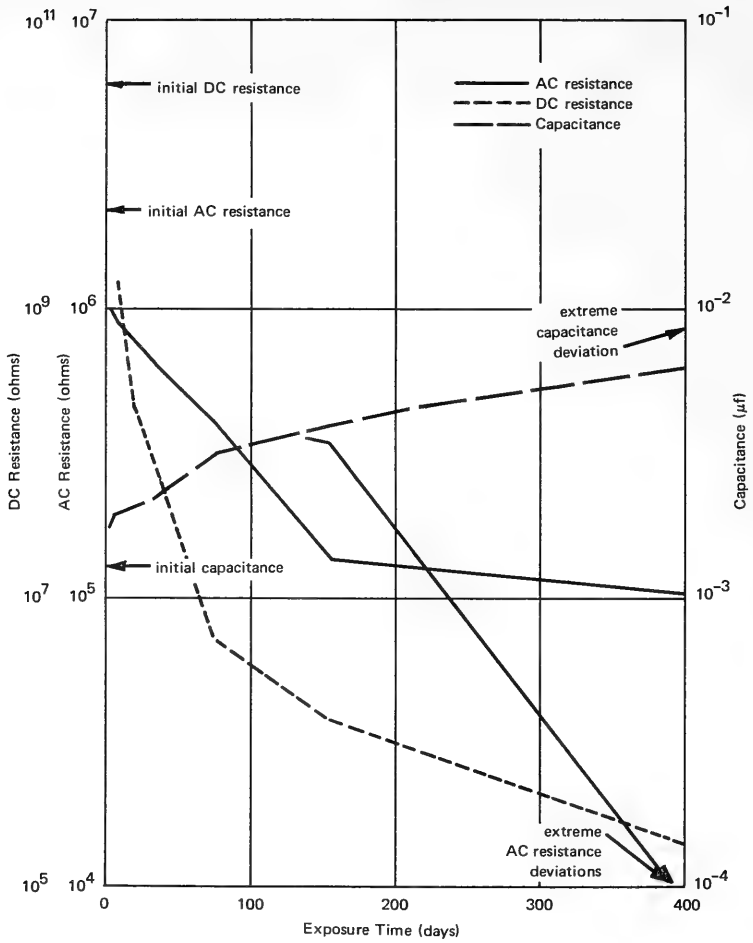


Figure 9. Electrical properties of System 117.

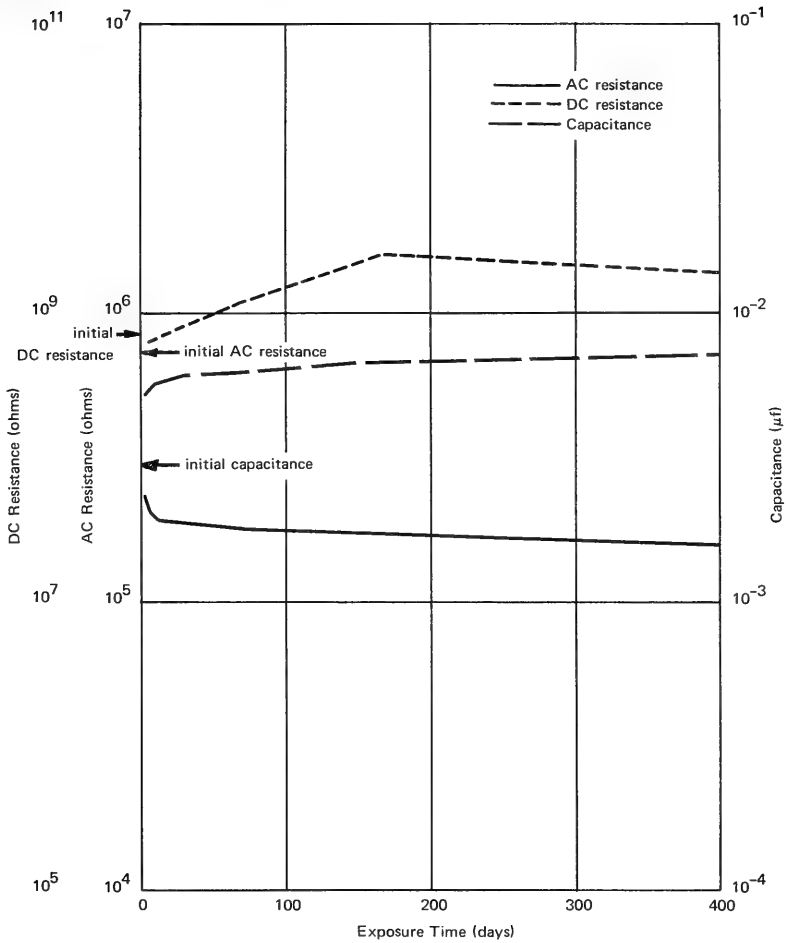


Figure 10. Electrical properties of System 118.

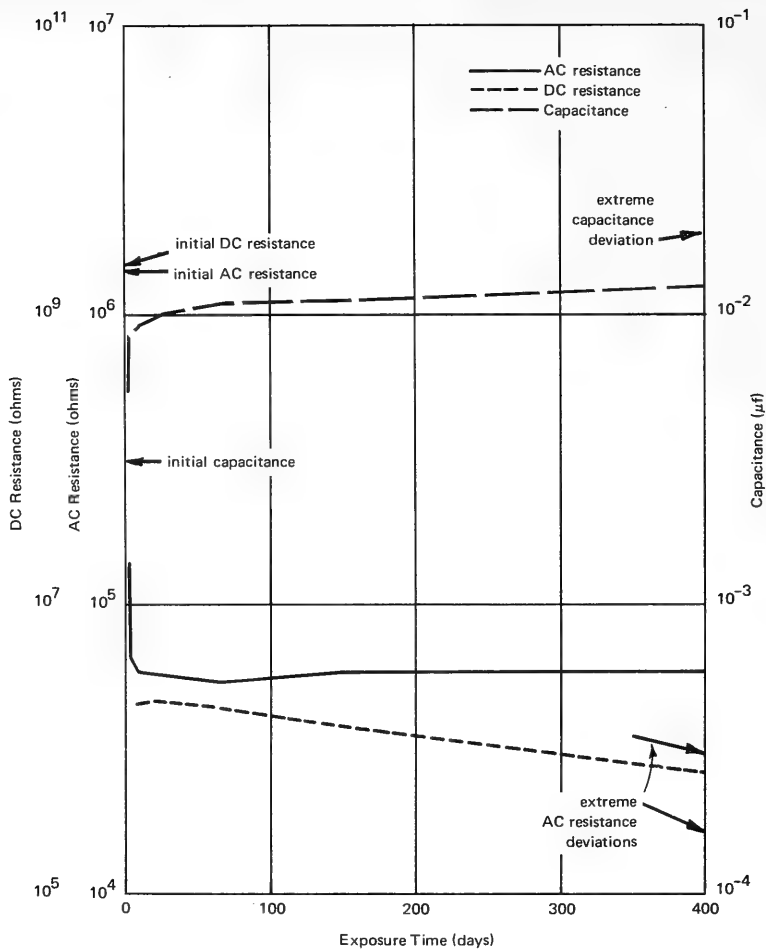


Figure 11. Electrical properties of System 119.

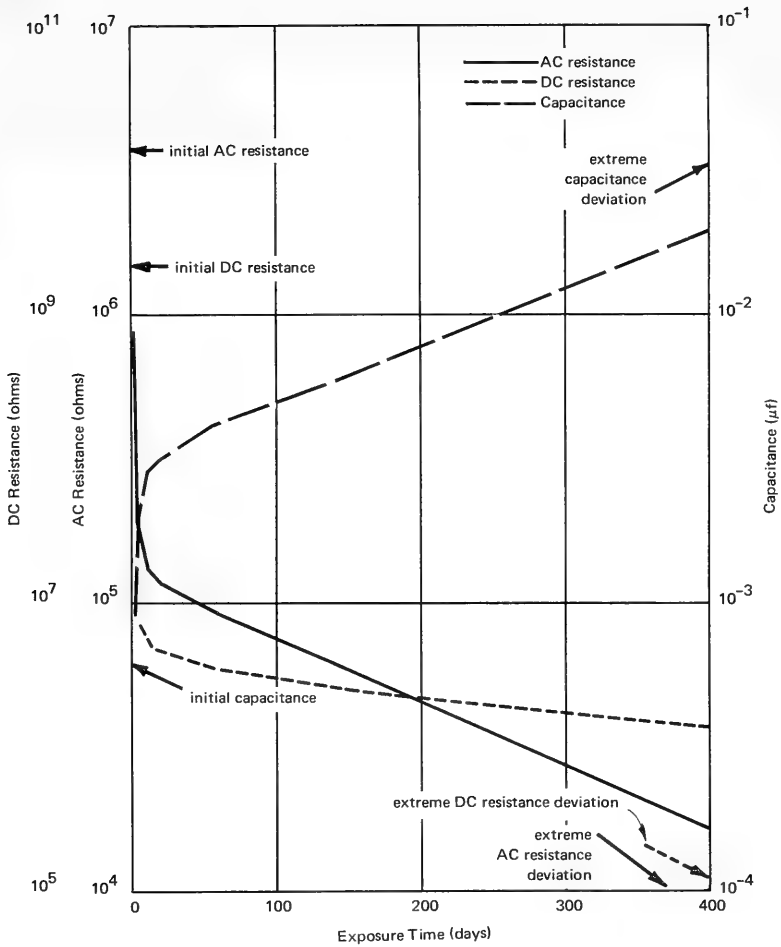


Figure 12. Electrical properties of System 120.

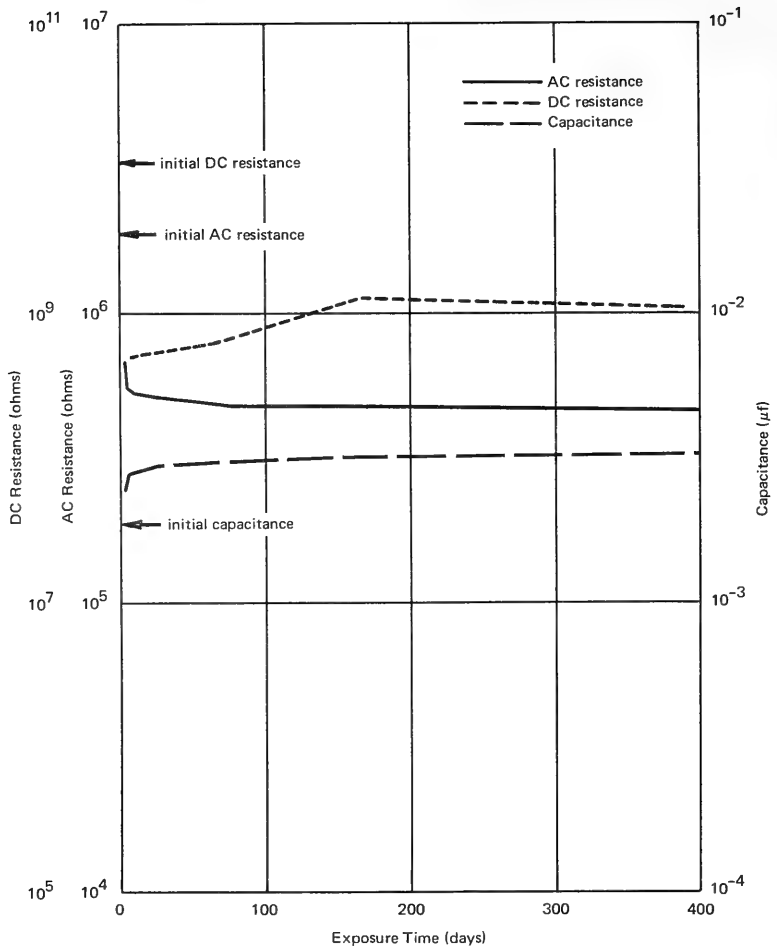


Figure 13. Electrical properties of System 121.

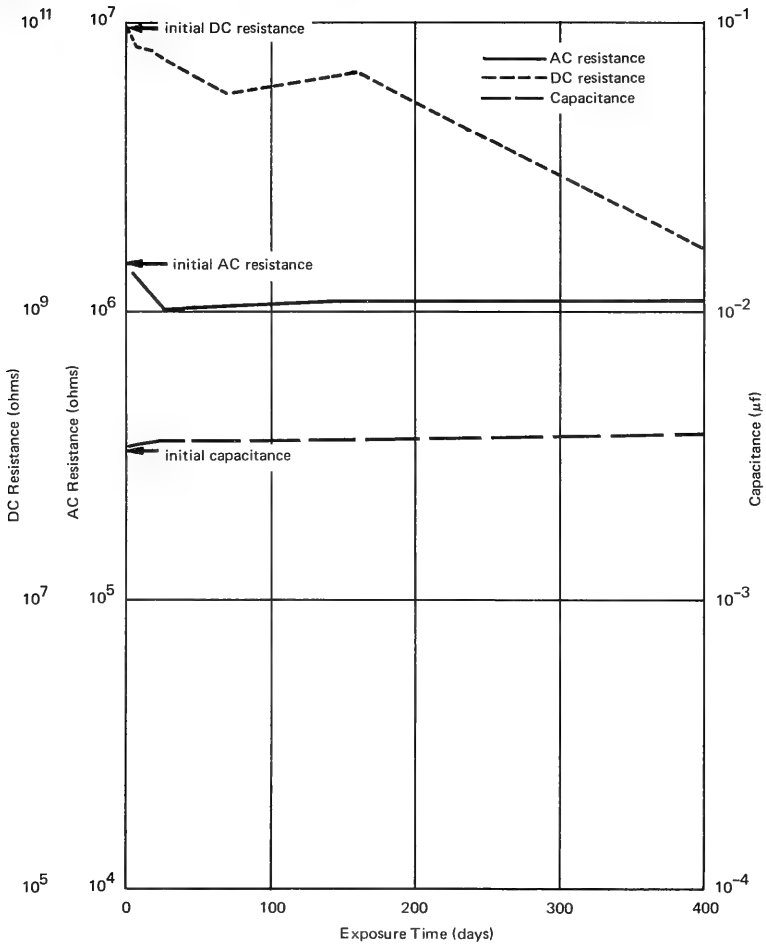


Figure 14. Electrical properties of System 122.

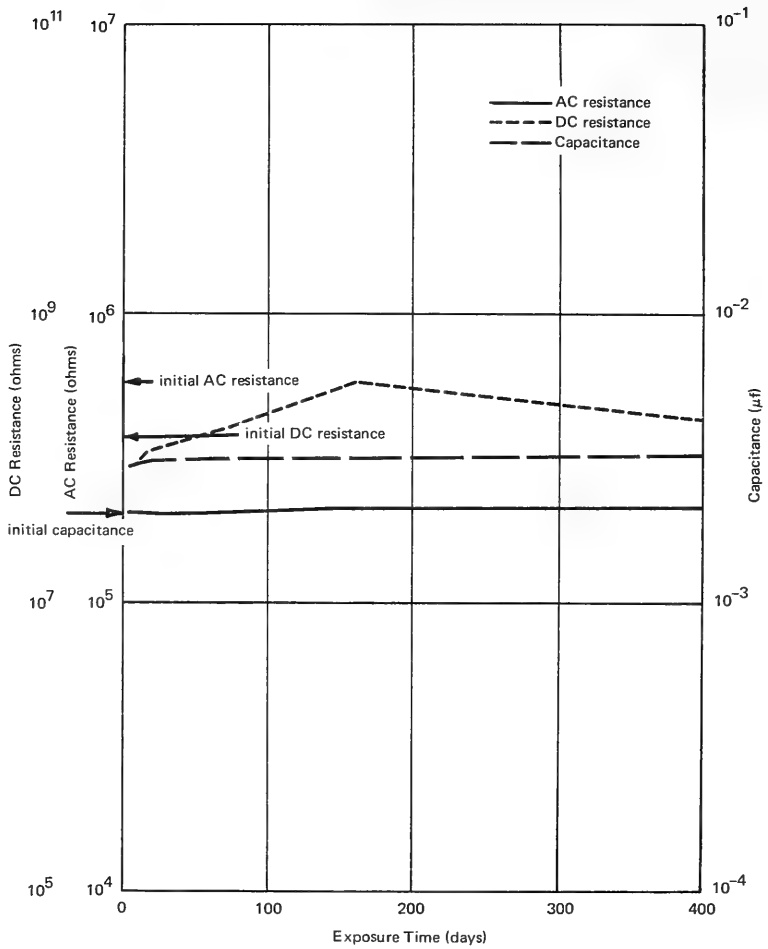


Figure 15. Electrical properties of System 123.

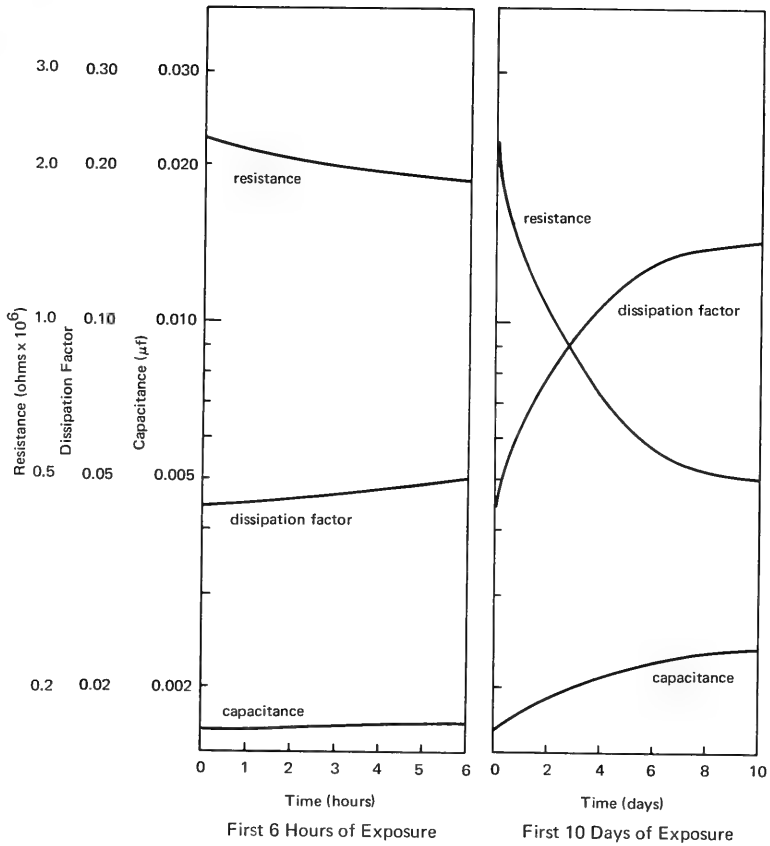


Figure 16. Initial changes in AC electrical properties of System 111.

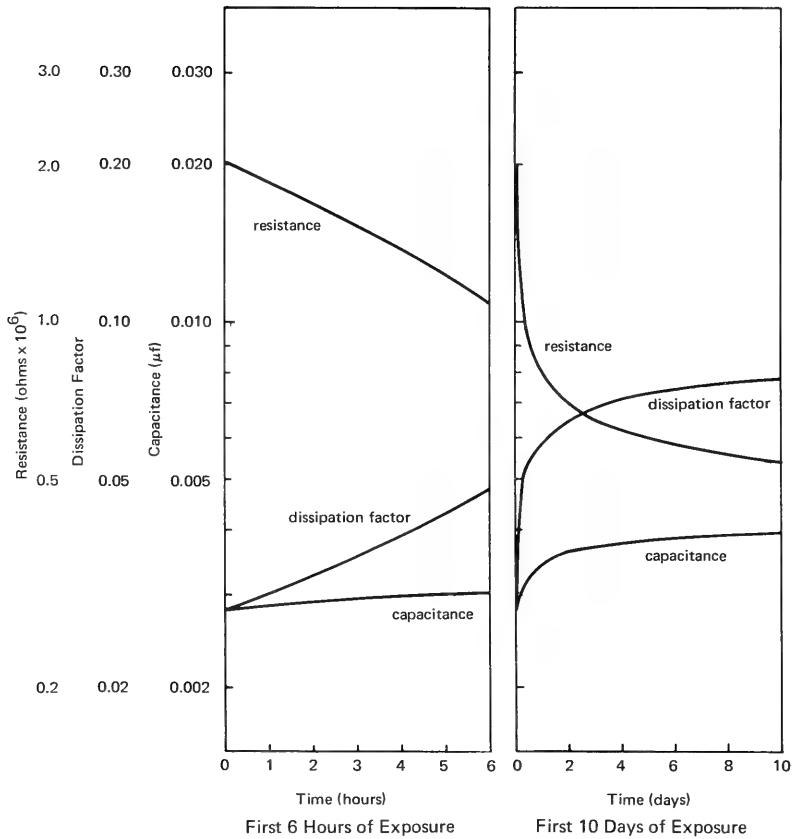


Figure 17. Initial changes in AC electrical properties of System 112.

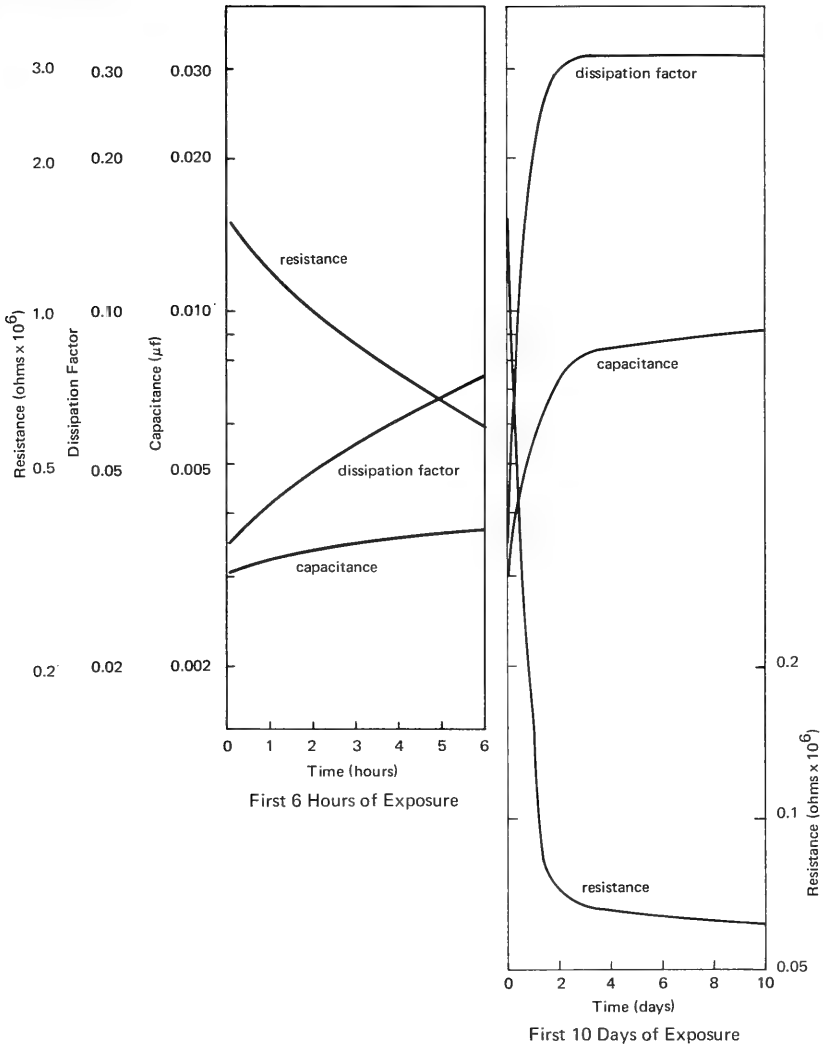


Figure 18. Initial changes in AC electrical properties of System 119.

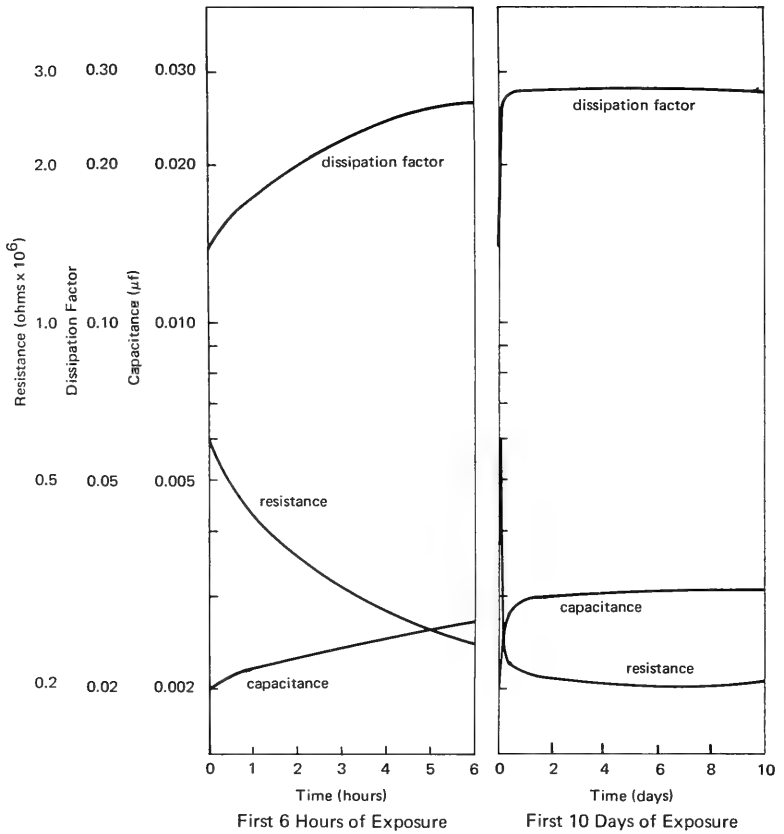


Figure 19. Initial changes in AC electrical properties of System 123.

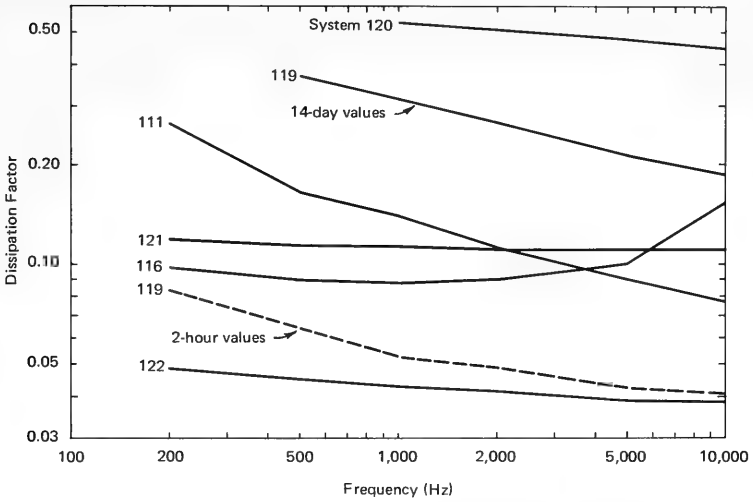


Figure 20. Dissipation factors at different frequencies for Systems 111, 116, 119, 120, 121, and 122. (Dashed System 119 curve is for values after 2 hours, other curves are for 14-day values.)

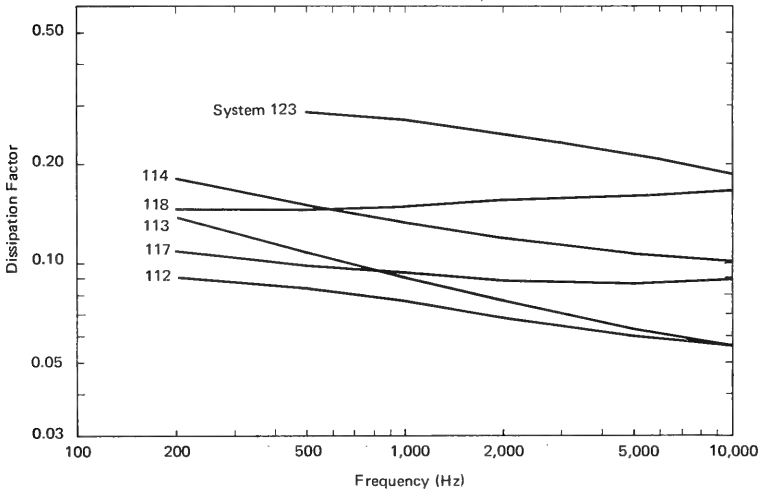


Figure 21. Dissipation factors at different frequencies for Systems 112, 113, 114, 117, 118, and 123. (All curves based on 14-day values.)

Table 2. Electrical Properties of Immersed Coating Systems

System	Exposure Time	DC Measurements ^d		AC Measurements ^e						Dissipation Factor
		Resistance (log ohms)	log R_V/R	Resistance (10^5 ohms)	R_V/R	log R_V/R	Capacitance (μ f)	C/C_i	log C/C_i	
111	Initial	10.1	—	2,200	—	—	0.0017	—	—	0.004
	6 hours	—	—	1,800	1.25	0.1	0.0017	1.03	0.01	0.05
	10 days	7.7	2.4	510	4.4	0.6	0.0023	1.43	0.16	0.14
	150 days	7.8	2.3	450	4.9	0.6	0.0030	1.8	0.25	—
112	400 days	6.8	3.3	290 ^b	7.2	0.9	0.0042 ^b	2.6	0.41	—
	Initial	10.8	—	2,000	—	—	0.0028	—	—	0.028
	6 hours	—	—	1,100	1.8	0.3	0.0030	1.07	0.03	0.046
	10 days	9.5	1.3	540	3.7	0.6	0.0038	1.4	0.15	0.076
113	150 days	9.8	1.0	580	3.4	0.5	0.0044	1.6	0.20	—
	400 days	9.7	1.1	560	3.6	0.6	0.0048	1.8	0.26	—
	Initial	9.9	—	2,000	—	—	0.0018	—	—	0.044
	6 hours	—	—	1,600	1.2	0.1	0.0019	1.03	0.01	0.051
114	10 days	8.0	1.9	790	2.6	0.4	0.0023	1.23	0.09	0.090
	150 days	8.1	1.8	780	2.6	0.4	0.0025	1.4	0.14	—
	400 days	8.0	1.9	620	3.3	0.5	0.0030	1.6	0.20	—
	Initial	10.0	—	1,660	—	—	0.0016	—	—	0.062
115	6 hours	—	—	1,320	1.3	0.1	0.0017	1.06	0.03	0.073
	10 days	8.1	1.9	490	3.4	0.5	0.0025	1.57	0.20	0.134
	150 days	8.1 ^c	1.9	300 ^c	5.5	0.7	0.0039	2.4	0.39	—
	400 days	7.5 ^c	2.5	230 ^c	7.2	0.9	0.0046 ^c	2.9	0.46	—
116	Initial	9.9	—	1,680	—	—	0.0029	—	—	0.032
	6 hours	—	—	900	1.9	0.3	0.0032	1.1	0.04	0.048
	10 days	7.8	2.1	510	3.3	0.5	0.0039	1.3	0.13	0.080
	150 days	7.6	2.3	620	2.7	0.4	0.0042	1.4	0.16	—
117	400 days	7.6	2.3	640	2.6	0.4	0.0044	1.5	0.18	—
	Initial	7.8	—	440	—	—	0.0064	—	—	0.058
	6 hours	—	—	310	1.4	0.2	0.0067	1.1	0.02	0.076
	10 days	7.6	0.2	230	1.9	0.3	0.0079	1.2	0.09	0.090
117	150 days	7.7	0.1	220	2.0	0.3	0.0085	1.3	0.12	—
	400 days	7.4	0.4	210	2.1	0.3	0.0093	1.5	0.16	—
	Initial	10.6	—	2,300	—	—	0.0013	—	—	0.056
	6 hours	—	—	1,760	1.3	0.1	0.0014	1.08	0.03	0.065
117	10 days	8.9	1.7	940	2.5	0.4	0.0020	1.55	0.19	0.089
	150 days	6.1	4.5	190	12.1	1.1	0.0040	3.1	0.49	—
	400 days	5.4	5.2	100 ^b	22.0	1.3	0.0063 ^c	4.8	0.68	—

continued

Table 2. Continued

System	Exposure Time	DC Measurements ^a		AC Measurements ^a					
		Resistance (log ohms)	log R_i/R	Resistance (10 ³ ohms)	R_i/R	log R_i/R	Capacitance (μ f)	C_i/C_i	log C_i/C_i
118	Initial	8.9	—	740	—	0.0031	—	—	0.070
	6 hours	—	—	280	2.6	0.0042	1.3	0.12	0.146
	10 days	8.8	0.1	190	3.8	0.0058	1.9	0.27	0.150
	150 days	9.4	-0.5	170	4.4	0.0066	2.0	0.33	—
119	400 days	9.2	-0.3	160	4.5	0.0072	2.5	0.40	—
	Initial	9.3	—	1,500	—	0.0031	—	—	0.035
	6 hours	—	—	600	2.5	0.0038	1.23	0.09	0.075
	10 days	6.3	3.0	61	25.0	0.0032	3.0	0.48	0.32
120	150 days	6.2	3.1	58	25.8	0.0117	3.8	0.58	—
	400 days	5.9	3.4	58 ^b	26.0	0.012 ^c	4.2	0.62	—
	Initial	9.3	—	3,500	—	0.0063	—	—	0.073
	6 hours	—	—	2,200	1.6	0.0070	1.12	0.05	0.108
121	10 days	6.7	2.6	132	27.0	0.0027	4.2	0.62	0.53
	150 days	6.4	2.9	62	56.5	0.0059	9.4	0.97	—
	1 year	6.1 ^c	3.2	18 ^b	200.0	~0.018 ^b	~30.0	~1.5	—
	Initial	10.0	—	1,920	—	0.0018	—	—	0.047
122	6 hours	—	—	1,140	1.7	0.0021	1.2	0.06	0.068
	10 days	8.7	1.3	510	3.8	0.0028	1.6	0.13	0.114
	150 days	9.1	0.9	480	4.0	0.0031	1.7	0.24	—
	400 days	9.0	1.0	460	4.2	0.0033	1.8	0.26	—
123	Initial	11.0	—	1,480	—	0.0034	1.00	—	0.035
	6 hours	—	—	1,380	1.1	0.0033	0.97	-0.01	0.036
	10 days	10.7	0.3	1,040	1.4	0.0035	1.03	0.01	0.043
	150 days	10.7	0.3	1,100	1.4	0.0036	1.06	0.03	—
123	400 days	9.5	1.5	1,100	1.4	0.0038	1.12	0.05	—
	Initial	8.1	—	590	—	0.0020	—	—	0.138
	6 hours	—	—	250	2.4	0.0027	1.3	0.13	0.26
	10 days	8.0	0.1	205	2.9	0.0031	1.6	0.18	0.28
123	150 days	8.5	-0.4	230	2.6	0.0031	1.6	0.18	—
	400 days	8.3	-0.2	210	2.8	0.0033	1.7	0.23	—

^a R_i and C_i are the initial measurements.^c Values of one panel not included in average.^b Values of two panels not included (value listed is for remaining panel).

To obtain DC resistance values, the open-circuit potential of the above system or cell was first measured. The system was then shunted by switching to the proper amperage scale and thus reducing the input resistance of the electrometer. The new voltage was measured, and the DC resistance was calculated from the following equation:

$$R_i = \left(\frac{E_o - E_s}{E_s} \right) R_s$$

where R_i = internal resistance of the cell (or the resistance of the coating)

E_o = open circuit potential

E_s = shunted potential

R_s = shunt resistance (or input impedance of the electrometer)

When the open circuit potentials were quite low, the total open circuit potentials were increased by the addition of voltage from a potentiometer. When measurements were made on coatings of very high resistance, some time was required to obtain an equilibrium value. For DC coating resistances of 10^{11} ohms, approximately 20 minutes were required to reach reasonable equilibrium values. The time taken for the voltage readings to come to equilibrium was often reduced by imposing, effectively at the electrometer connections, charging potentials very close to the expected equilibrium voltage. These voltages were imposed by the circuit shown in Figure 22. When the time constants were very long, or when there were fluctuations in readings due to external factors, the voltage readings versus time were plotted to arrive at better equilibrium values or average values.

The DC resistance values obtained by the above methods were recorded and plotted, and the results are shown in Figures 3 to 15. The average initial resistance values are indicated at the left-hand ordinate scale, and the average values after about 7 days and continuing to 400 days are shown by the dashed curves. It should be noted that in this semilog presentation, the DC resistance values are plotted on a log scale differing in interval from that used for the AC resistance values. Wherever the resistance values of any of the three panels deviate from the average by more than 0.5 log unit, the averages of the remaining panels are plotted, and the deviations are indicated at the abscissa or right-hand ordinate. For System 115 (Figure 7), the values for each panel are plotted to illustrate the maximum deviations that would be included in the averages.

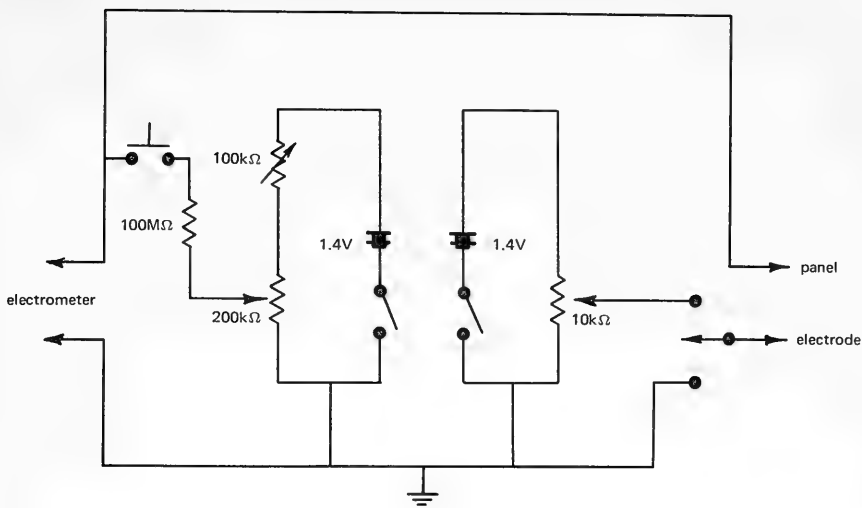


Figure 22. DC source for impressed potentials.

The logarithms of the DC resistance measurements are also shown in Table 2. The initial values were obtained after approximately 1 hour; the 10-day values indicated were obtained after approximately 7 to 10 days. Average values are shown; panels for which $\log R$ would have deviated by more than 0.5 from the averages were not included in the averages. The changes in $\log R$ are also shown.

The panels were rated visually after 2, 5, 13, and 22 months for rusting and blistering.¹⁵ The rusting ratings were assigned according to ASTM method D G10-43, in which a rating of 10 designates no rusting and lower numbers designate increasing rusting. The blistering ratings were assigned according to ASTM method D 714-56, in which a rating of 10 designates no blistering and decreasing numbers indicate increasing blister size, and in which the letters F (few), M (medium), and D (dense) indicate increasing blister density.

Before these ratings, and at other times when the buildup of organic debris or the brown coloration on the panels appeared excessive, the panels were removed from the aerated seawater and were cleaned with a soft brush. They were rinsed with seawater and briefly with demineralized water before being replaced in fresh seawater.

After 1 year of immersion in aerated seawater at 25°C, 10 of the 13 systems showed essentially no rusting or blistering and were given ratings of 10 in both categories. System 117 received a rusting rating of 9 and a blistering rating of 2F. All three panels of the latter system showed some small rusty tubercles. Systems 112 and 119 showed small blisters and were given a rating of 8M. After 2 years of immersion, the blistering ratings of Systems 112 and 119 changed to 6D and Systems 114 and 118 received blistering ratings of 8F. All other ratings remained the same.

The same coating systems used for the electrical measurements had some years earlier been placed on 6 x 12-inch, and in some cases 12 x 14-inch, panels and had been subjected to marine atmospheric environments at Port Hueneme, Calif., Kaneohe, H. I., and Kwajalein, M. I.¹³ One-half of the panels so exposed had been scribed and the others were unscribed. The performance of the 13 systems under atmospheric exposure at Kwajalein is shown in Table 3.

Other coated steel panels 4 inches wide and 10 feet long had been exposed in the harbor at Port Hueneme.¹⁴ These panels were so placed that they were exposed in an atmospheric zone, in an intertidal zone, and in an immersed zone. The performance of 11 of the 13 systems in the immersed zone is shown in Table 4.

DISCUSSION

The majority of organic protective coatings are nonconductive. When such an organic coating on a steel panel is immersed in seawater, the coating provides a comparatively high electrical resistance. This resistance (**R**) is related directly to the specific resistance (ρ) and to the coating thickness (**t**), and it is inversely related to the area of the coating (**A**):

$$R = \frac{\rho t}{A}$$

At the same time, the coating is also a dielectric between two conductors, one of which is the steel plate and the other the salt water. This system is thus a capacitor whose capacitance (**C**) is proportional to the dielectric constant of the coating (ϵ) and to the area of the coating (**A**) and inversely proportional to the coating thickness (**t**), as follows:

$$C = \frac{\epsilon A}{t}$$

Table 3. Performance of Coating Systems Subjected to Atmospheric Exposure at Kwajalein, M. I.

System	Unscribed Panels			Scribed Panels			Protection Ranking ^d	
	Years to Failure ^a	Performance ^b at time of failure (or at last rating) ^c		Years to Failure ^a	Performance ^b at time of failure (or at last rating) ^c			
		Blistering	Undercutting		Blistering	Undercutting		
111	>6.5 ^e	(10)	(10)	~105	3.0	2D	M	28
112	4.0	10	H	40	<2.0 ^f	2D	H	17
113	>6.5	(10)	(10)	~85	1.5	2D	M	14
114	3.5	2F	L	35	1.0	2D	M	9
115	5.5	2M	M	55	4.0	2D	L	40
116	>8.5	(10)	(10)	~125	6.5	2D	H	65
117	8.0	2M	H	80	5.0	2MD	M	49
118	6.0	2M	H	60	<4.0	2D	H	36
119	>4.5	(2F)	(H)	~47	4.5	2F	H	45
120	8.0	2D	H	78	<4.0	2D	10	37
121	3.5	2F	H	34	<2.0	2D	H	18
122	8.0	2D	H	79	4.0	2D	M	39
123	7.0	2F	10	70	<3.0	2D	10	27

^a As described in the text.

^b ASTM rating.

^c For systems which had not yet failed or had failed before the last rating.

^d Protection ranking = approximately 10 times the years to failure (or estimated failure if indicated by ~).

^e > indicates the system had not failed at the time indicated of the last rating.

^f < indicates the system had reached the point of failure before the time of the last rating.

Table 4. Performance of Systems Immersed in Port Hueneme Harbor

System	Time to Comparative Deterioration (yr)			Deterioration ^a After 5 Years of Exposure		Protection Ranking ^b
	Failure	"g" Rusting ^c	"M" Blistering ^d	Rusting	Blistering	
111	>6.5	>6.5	>6.5	10	10	~149
112	>6.5	3.5	>1.5	9	2D	~94
113	>6.5	>6.5	>6.5	10	10	~150
114	>7.5	4	>7.5	9	2F	~105
116	>10	5	10	9	2F	~120
117	>12.5	9.5	9.5	10	2F	~155
119	>9.5	>9.5	>9.5	10	10	~125
120	>12.5	2.5	>12.5	9	6D	~145
121	>8	5	>8	9	2F	~110
122	>5.5	2	4.5	9	6MD	~85
123	9.5	6	>9.5	9+	10	95

^a ASTM rating.

^b Protection ranking = approximately 10 times the years to failure (or estimated failure if indicated by ~).

^c Time to reach an ASTM rusting rating of 9.

^d Time to reach medium blister density according to ASTM rating.

If the coating thickness is reduced by erosion, the resistance decreases and the capacitance increases. If the coating thickness is reduced by distention of the film, caused either by blistering or by rusting, the resistance is also decreased and the capacitance is increased. Other defects or incipient breaks in the coating will make more complicated changes in the effective electrical network of the coating, but they will generally decrease the resistance and increase the capacitance. Water uptake in the immersed film will increase the dielectric constant and decrease the specific resistivity, and therefore, will again have the effect of decreasing the resistance or increasing the capacitance.

When electrical connections are made to the steel panel and to the seawater in which it is immersed, the result is a parallel network consisting of a capacitance and a high resistance. This network is in effect a nonideal capacitor as illustrated in Figure 23. The lower the resistance of this capacitor, the greater is the loss current; and the greater the loss current, the greater is the

dissipation factor or loss tangent. The dissipation factor (**D**) or loss tangent ($\tan \delta$) are related to the loss current (I_l), to the charging current (I_c), and to the resistance and capacitance, as follows:¹⁶

$$D \equiv \tan \delta = \frac{I_l}{I_c} = \frac{1}{\omega R C}$$

As a coating deteriorates it might be expected that the loss current and the dissipation factor increase. This would mean that the resistance must decrease more rapidly than the capacitance increases. For a simple loss of thickness not accompanied by other changes, no change in dissipation factor would be expected.

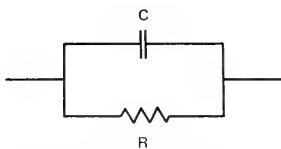
The resistance, capacitance, and dissipation factor at a frequency of 1 kHz can readily be determined with equipment such as that described above. Direct current resistance cannot be determined by a simple resistance measurement because the direct current potential necessary would cause considerable polarization. The method which has been used by others and which was slightly modified for the present work considers the panel, the coating system, the seawater, and the calomel electrode as a cell, or source of electromotive force. The resistance of the coating system is effectively the internal resistance of this cell. From the open cell potential (E_o) and the shunted cell potential (E_s) obtained when the cell is shunted with a resistance (R_s), as shown in Figure 24, the internal resistance (R_i) is calculated:

$$R_i = \left(\frac{E_o - E_s}{E_s} \right) R_s$$

In arriving at the above equation, it is assumed that there is no change in the basic electromotive force of the cell (E) as current is drawn, and that the potential across the internal resistance (E_i) is in fact the difference between the open-cell and closed-cell potentials.

$$E_i = E - E_s = E_o - E_s$$

In practice there will be a drop in the potential of the cell (E) due to polarization, and it will be smaller than E_o . Thus, E_i , and therefore R_i , will be considerably less than the values calculated. This may be the chief reason why reported DC resistances have been much higher than reported AC resistances.



effective network of a capacitor

- I_c = Charging current
- C = Capacitance
- I_l = Loss current
- R = Resistance
- V = Voltage
- δ = Loss angle

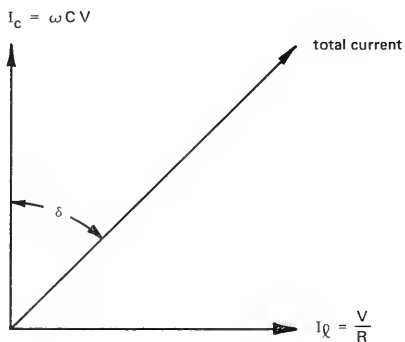
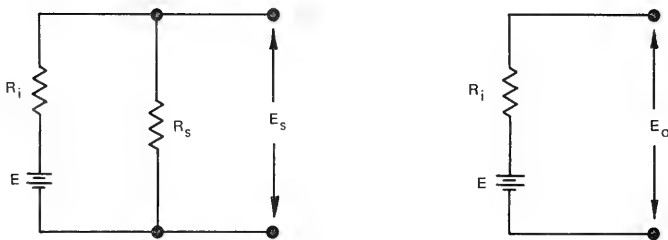


Figure 23. Current in a capacitor.



- E = EMF source (or cell potential)
- E_o = Open circuit potential
- E_s = Shunted potential
- R_i = Internal resistance
- R_s = Shunt resistance

Figure 24. Open and closed cell potentials.

Performance of Coating Systems

The performance of the 13 coating systems under atmospheric exposure at Kwajalein is indicated in Table 3. Each coating system was given a protection ranking for performance on unscribed panels and for performance on scribed panels. This protection ranking is approximately 10 times the number of years of exposure required to produce failure of the coating system. To provide comparative ratings among several coatings that failed at the same time, the protection rankings were weighted on the basis of other performance factors. For coating systems that had not yet failed at the time of the last rating, an approximate protection ranking was assigned, which was obtained by adding to 10 times the years of exposure additional points depending on the condition of the coating at the last rating. These additional points were about 10 for a protection rating of 8, 20 for a protection rating of 9, and 40 for a protection rating of 10, but were varied somewhat depending on other performance factors. Because of the lesser accuracy of these protection rankings they are indicated by ~ in Table 3.

Failure of a system was considered to have occurred when the overall protection rating¹³ decreased to a value of 7. This rating was essentially the same as the ASTM rusting rating, and failure was thus generally the point where 30% of the area had rusted. The degrees of blistering and of undercutting at the time of failure are also shown in Table 3. A "greater than" sign (>) indicates failure after the time specified; a "less than" sign (<) indicates failure before the time specified.

The performance of 11 of the systems immersed in Port Hueneme Harbor is shown in Table 4. The systems had been exposed for varying periods of time before the last rating inspection, as shown by the times during which the systems had not failed. Only one of the systems, System 123, had actually failed. Protection rankings are shown for each system, and these are again approximately 10 times the number of years required for failure. For the systems that had not failed, the protection rankings are based on the degree of protection and blistering at the last rating, and also on the comparative deterioration of the systems after 5 years of exposure. For systems still in perfect condition after 6.5 years of exposure, up to 85 points were added. These quality ratings are thus very approximate, because an extrapolation of the service life to some 12 or 15 years is not valid.

All systems performed well while immersed in seawater at 25°C on the small panels used for the electrical measurements. Only one system, System 117, allowed appreciable rusting, even after 2 years. Three systems had a few blisters after 2 years, and only two systems dense blisters. These Laboratory immersion results are shown in Tables 5 through 8.

Table 5. Coating Performance and Changes in Electrical Properties Arranged According to Coating System Number

System	Performance										Changes in Electrical Resistance			
	Protection Ranking ^d Based on Exposure Tests		Rating ^b After Laboratory Immersion				Strong Initial AC Resistance Changes		Low Final Resistance Values					
	Sea Water Immersion ^c	Marine Atmosphere ^d	Rusting		Blistering		Rapid ^f	Slow ^f	AC	DC				
			1 Year	2 Years	1 Year	2 Years								
111	~149	~100	10	10	10	10	•	•	•	•				
112	~94	40	10	10	10	8M	•	•	•	•				
113	~150	85	10	10	10	10	•	•	•	•				
114	~105	35	10	10	10	10	•	•	•	•				
115	—	55	10	10	10	10	•	•	•	•				
116	~120	~125	10	10	10	10	•	•	•	•				
117	~155	80	9	9	9	2F	•	•	•	•				
118	—	60	10	10	10	10	•	•	•	•				
119	~125	47	10	10	10	8M	•	•	•	•				
120	~145	78	10	10	10	10	•	•	•	•				
121	~110	34	10	10	10	10	•	•	•	•				
122	~85	79	10	10	10	10	•	•	•	•				
123	95	70	10	10	10	10	•	•	•	•				

^d Protection ranking = approximately 10 times the years to failure (or to estimated failure if indicated by ~).

^b ASTM rating.

^c At Port Hueneme Harbor.

^d At Kwajalein, M. I.

^e Within the first 6 hours, as described in the text.

^f Between 6 hours and 10 days, as described in the text.

^g Two of the three panels had low resistance values.

Table 6. Coating Performance and Changes in Electrical Properties Arranged According to Performance of Immersed Coatings

System	Performance										Changes in Electrical Resistance			
	Protection Ranking ^d Based on Exposure Tests			Rating ^b After Laboratory Immersion						Strong Initial AC Resistance Changes		Low Final Resistance Values		
	Sea Water Immersion ^c	Marine Atmosphere ^d		Rusting		Blistering		Rapid ^e	Slow ^f	AC	DC			
		Unscribed Panels	Scrubbed Panels	1 Year	2 Years	1 Year	2 Years							
117	~155	80	49	9	9	2F	2F	•	•	•	•			
113	~150	85	14	10	10	10	10	•	•	•	•			
111	~149	~100	28	10	10	10	10	•	•	•	•			
120	~145	78	37	10	10	10	10	•	•	•	•			
119	~125	117	45	10	10	8M	6D	•	•	•	•			
116	~120	~125	65	10	10	10	10	•	•	•	•			
121	~110	34	18	10	10	10	10	•	•	•	•			
114	~105	35	9	10	10	10	8F	•	•	•	•			
123	95	70	27	10	10	10	10	•	•	•	•			
112	~94	40	17	10	10	8M	6D	•	•	•	•			
122	~85	79	39	10	10	10	10	•	•	•	•			

^d Protection ranking = approximately 10 times the years to failure (or to estimated failure if indicated by ~).

^b ASTM rating.

^c At Port Hueneme Harbor.

^d At Kwajalein, M. I.

^e Within the first 6 hours, as described in the text.

^f Between 6 hours and 10 days, as described in the text.

^g Two of the three panels had low resistance values.

Table 7. Coating Performance and Changes in Electrical Properties Arranged According to Atmospheric Performance of Unscribed Panels

System	Performance						Changes in Electrical Resistance					
	Protection Ranking ^a Based on Exposure Tests		Rating ^b After Laboratory Immersion				Strong Initial AC Resistance Changes		Low Final Resistance Values			
	Sea Water Immersion ^c	Marine Atmosphere ^d	Rusting		Blistering		Rapid ^e	Slow ^f	AC	DC		
			1 Year	2 Years	1 Year	2 Years						
	Unscribed Panels	Scribed Panels	1 Year	2 Years	1 Year	2 Years						
116	~120	65	10	10	10	10						
111	~149	28	10	10	10	10						•
113	~150	14	10	10	10	10						•
117	~155	85	9	9	2F	2F						•
122	~85	80	10	10	10	10						•
120	~145	79	37	10	10	10						•
123	95	70	27	10	10	10						•
118	—	60	36	10	10	10						•
115	—	55	40	10	10	10						•
119	~125	47	45	10	10	8M						•
112	~94	40	17	10	10	8M						•
114	~105	35	9	10	10	10						•
121	~110	34	18	10	10	10						•

^a Protection ranking = approximately 10 times the years to failure (or to estimated failure if indicated by ~).

^b ASTM rating.

^c At Port Hueneme Harbor.

^d At Kwajalein, M. I.

^e Within the first 6 hours, as described in the text.

^f Between 6 hours and 10 days, as described in the text.

^g Two of the three panels had low resistance values.

Table 8. Coating Performance and Changes in Electrical Properties Arranged According to Atmospheric Performance of Scribed Panels

System	Performance										Changes in Electrical Resistance									
	Protection Ranking ^a Based on Exposure Tests		Rating ^b After Laboratory Immersion								Strong Initial AC Resistance Changes		Low Final Resistance Values							
			Marine Atmosphere ^d		Rusting		Blistering		Rapid ^e	Slow ^f					AC	DC				
	Sea Water Immersion ^c	Unscribed Panels	Scribed Panels	1 Year	2 Years	1 Year	2 Years	1 Year			2 Years									
116	~120	~125	65	10	10	10	10	10	10	10	10									
117	~155	80	49	9	9	9	9	2F	2F	10	10									
119	~125	47	45	10	10	10	10	8M	6D	10	10									
115	—	55	40	10	10	10	10	10	10	10	10									
122	~85	79	39	10	10	10	10	10	10	10	10									
120	~145	78	37	10	10	10	10	10	10	10	10									
118	—	60	36	10	10	10	10	10	10	10	10									
123	95	70	27	10	10	10	10	10	10	10	10									
111	~149	~100	28	10	10	10	10	10	10	10	10									
121	~110	34	18	10	10	10	10	10	10	10	10									
112	~94	40	17	10	10	10	10	10	10	8M	6D									
113	~150	85	14	10	10	10	10	10	10	10	10									
114	~105	35	9	10	10	10	10	10	10	10	10									

^a Protection ranking = approximately 10 times the years to failure (or to estimated failure if indicated by ~).

^b ASTM rating.

^c At Port Hueneme Harbor.

^d At Kwajalein, M. I.

^e Within the first 6 hours, as described in the text.

^f Between 6 hours and 10 days, as described in the text.

^g Two of the three panels had low resistance values.

Electrical Properties of Coating Systems

The results of the electrical measurements are presented in Table 2 and in Figures 3 to 20. In the table, average values are shown for the electrical measurements obtained initially and after exposure periods of 6 hours, 10 days, 150 days, and 400 days. The 1/4-, 10-, and 400-day exposure durations vary by a factor of 40. When the values for one of the three panels were considerably below the average, these values were disregarded in computing the averages listed in the table. When two panels had considerably inferior electrical properties (lower resistance or higher capacitance), the value obtained for the remaining panel was used. The values shown for each system therefore tend to show the best properties of each coating system and eliminate possible defects in the panels or in the coating application. The deviations of panels not included in the averages shown in Table 2 are, however, shown in Figures 3 to 15.

The criteria for deciding which values should be omitted from the averages were as follows: (1) for AC resistances, when the deviation of the value from the average was greater than a factor of 1.5; (2) for capacitances, when the deviation was greater than a factor of 1.3; and (3) for the DC resistances, when the deviation from the average was greater than 0.5 log unit.

The changes in electrical properties are probably more important than the absolute values. Therefore, the ratios of these measurements as compared to the initial measurements are also shown in Table 2. Thus, R_i/R is the ratio of the initial resistance to that at the given exposure time, and $\log (R_i/R)$ is the corresponding logarithm. For capacitance measurements the ratio, C/C_i , is given so that, again, increasing changes give increasing ratios.

As shown in Table 2 and in Figures 3 to 15, the initial changes in electrical properties were sometimes quite dramatic. This change presumably is due to water uptake of the coating after it is immersed in seawater.⁹ Such water uptake will change the capacitance of a coating because of changes produced in the dielectric constant of the coating. For a given amount of water, the change in dielectric constant may vary depending on the method of distribution of the water.⁹ No attempt was therefore made to relate the change in capacitance to actual water uptake. The changes in AC resistance were found to be even greater than the changes in capacitance.

For four of the systems (111, 112, 119, and 123) the short-term changes in AC resistance, capacitance, and dissipation factor are shown in graphical form (Figures 16 to 19). The electrical properties of System 111 (Figure 16) change comparatively little during the first 6 hours of exposure, however, there is considerable change during the first 10-day period. System 112 (Figure 17) shows considerable change during the first 6-hour exposure period

and also during the first 10-day period. The greatest of such changes are shown by System 119 (Figure 18). The changes in electrical properties of System 123 (Figure 19) were quite rapid in the initial 6-day period but showed very little change after that time.

The initial water uptake, as reflected in the electrical measurements, could be a factor in the performance of the coating. For this reason strong changes in initial AC resistance, which may be equated with strong water uptake, are shown for each system in Table 5. For some systems strong water uptake occurred rapidly, during the first 6 hours. For others the strong water uptake occurred more slowly, during the period between the 6-hour and 10-day measurements. These changes were considered strong when the resistances dropped to about one half the initial values during the first 6 hours, that is, $\log (R_i/R) \geq 0.3$, or when they dropped to about half the value of the 6-hour measurements at the time of the 10-day measurement. By this criterion five of the 13 systems have fast strong water uptake and eight systems (including two of the five just mentioned) have slow strong water uptake, as shown in Table 5.

Also shown in Table 5 are the low final resistances which are shown by some of the coatings after 1 year. A low final AC resistance was considered to be a panel resistance of less than 10^5 ohms for any of the three panels of a system. This is a value which had been considered a minimum value for good performance on the basis of earlier experiments.¹² A low final DC resistance value was considered to be a value below 10^7 ohms ($\log R = 7.0$) for any panel of a system. This was the minimum value proposed as a qualification requirement.¹⁷ On this basis, four systems showed low final AC resistance after 1 year of exposure, and five systems showed low DC resistance values after exposure for 1 year. It is of interest that all systems which had low AC resistance after 1 year also had low DC resistances at that time. This was true in spite of the fact that the DC resistance values are a combination of low resistance and of the effects of polarization. However, the effect of polarization may be proportionately less at lower resistance values.

For the calculation of the DC resistances, the open-cell potentials were measured. The initial values varied from +100 mv to -195 mv. The final values varied from -75 mv to -850 mv. As in previous experiments, there were considerable variations in the open-cell potentials. Except that in general the potentials became more negative, no specific trends could be established and the individual values are not reported.

Relationships Between Electrical Properties and Performance

It might be expected that the electrical properties of coatings immersed in seawater would correlate most closely with the performance of the coating systems exposed in seawater. Bacon and coworkers⁴ had suggested such a

relationship between the DC resistance changes and the performance of coatings. Anderton and Brown¹⁷ more recently had suggested a performance qualification based on such a relationship, which would require that three coated panels maintain resistances above 10^7 ohms during 1 year of seawater immersion.

In Table 6 the various coating systems for which performance data in Port Hueneme Harbor are available are rearranged in the order of performance of the coatings. As pointed out earlier, all of the systems performed well and the relative protection ranking may not be too meaningful.

The changes in electrical properties do not correlate with these high estimated protection rankings. Four of the five systems which seem to be best in their performance show low AC and DC resistances after 1 year of exposure on small panels immersed in seawater at 25°C.

The above results would appear to cast some doubts on the validity of a qualification requirement for coatings proposed for the Royal Canadian Navy, which requires the maintenance of a minimum DC electrical resistance. The results do not disprove the usefulness of such a test because the coatings that passed the test, by maintaining a resistance above 10^7 ohms for each of three panels, did perform well. But if this test is useful in passing only coatings that perform well, it has the drawback that it would also reject many coatings of superior performance.

A number of claims have been made that the AC electrical properties of coatings, or the changes in these properties, are related to the performance of the coatings under atmospheric exposure. Changes in capacitance have been used as indication of performance,⁸ and it has been claimed that capacitance changes¹⁰ or the values of dissipation factors at various frequencies^{6, 7} could be used to predict performance.

In Table 7 the coating systems are arranged according to the performance of unscribed panels in a marine atmosphere at Kwajalein. The first two systems had not yet failed and their performance is estimated; all other systems had been exposed sufficiently long to produce coating failure. When the coatings are thus arranged, the electrical changes, including strong initial changes in and low final values of the resistances, are somewhat randomly distributed. Coatings of higher protection rankings as well as lower protection rankings show the strong initial changes associated with water uptake and low final resistances.

In Table 8 the coatings are arranged according to the performance of scribed panels in marine atmospheric exposure at Kwajalein. Again, there is limited correlation between changes in electrical properties and the protection rankings of the coatings.

Visual inspection of Tables 7 and 8 thus showed no definite relationships between the protection rankings of the coating systems and major changes in electrical resistance. However, a mathematical comparison of the electrical properties with the protection rankings showed better correlations (see Table 9).

Table 9. Correlation of Electrical Properties of Coatings with Performance

Electrical Property ^a	Correlation Coefficient Between Electrical Properties and Atmospheric Performance ^b					
	Unscribed Panels			Scribed Panels		
	6 Hours	10 Days	400 Days	6 Hours	10 Days	400 Days
Log R_{DC}	–	-0.08	-0.29	–	-0.19	-0.42
Log $(R_i/R)_{DC}$	–	-0.31	+0.01	–	-0.16	+0.14
R_{AC}	+0.01	+0.03	-0.09	-0.27	-0.18	-0.23
Log $(R_i/R)_{AC}$	-0.27	-0.24	-0.05	+0.12	-0.05	+0.09
C	+0.38	+0.16	+0.25	+0.59	+0.59	+0.47
Log C/C_i	-0.27	-0.12	+0.02	+0.01	+0.14	+0.10
All Listed Properties ^c	0.75	0.76	0.76	0.80	0.80	0.88

^a As listed in Table 2.

^b Protection ranking of coating systems on unscribed or scribed panels as determined at Kwajalein, M. I., and listed in Table 5.

^c Absolute value of the square root of R^2 for the best linear equation involving all the independent variables (electrical properties) for which values are listed above.

For each coating system, the electrical properties listed (that is, the DC and AC resistances, the capacitance, and the changes in these values during the immersion periods indicated) were compared to the performance under atmospheric exposure. The correlation coefficients were determined for exposure periods of 1/4 day, 10 days, and 400 days.

For some of the electrical properties, significant correlations were obtained. Thus, for example, the greater the change in AC resistance, the lower is the quality of the coating as based on atmospheric field exposure results with unscribed panels. For these changes in AC resistance after 1/4 day or 10 days of immersion, this correlation is significant, but it becomes less significant after 400 days of exposure.

By treating as independent variables the various electrical properties at the given exposure periods, prediction equations for atmospheric performance were established. The absolute values of the correlation coefficients (the square roots of the "R²" values) of the best linear prediction equations are shown in Table 9. These correlation coefficients are much larger than those obtained for the individual electrical properties. It appears likely that still higher correlation coefficients can be obtained by developing prediction equations which employ as independent variables not only the electrical properties of the coatings but also other properties, such as permeability.

It had been claimed that coatings with the very low dissipation factors, and with dissipation-factor-versus-frequency curves of low slopes, will perform better.^{6,7}

The dissipation factors obtained at different frequencies for six of the systems are shown in Figure 20. These are the systems which had the lowest curve and the highest (Systems 122 and 120), the curve with the least slope and the greatest (Systems 121 and 111), and the systems with the best and the poorest performance in atmospheric exposure of unscrubbed panels (Systems 116 and 121). The curves show values obtained after approximately 2 weeks of exposure in seawater at 25°C. The dissipation factor curves obtained after approximately 2 to 3 hours may be quite similar to or may deviate from those obtained after approximately 2 weeks. The system that showed the greatest variation in this time interval was System 119, and both curves for this system are shown in Figure 20.

When 12 of the coating systems are arranged according to the increasing quality of their dissipation factor curves (which are shown in Figures 20 and 21), the following approximate order results: 122, 116, 121, 117, 112, 118, 113, 114, 111, 123, 119, 120. This order bears little resemblance to the order in Table 7, or to the orders in Tables 6 or 8.

It has been suggested that changes in electrical properties occur much sooner than visual changes in performance, and that electrical measurements may therefore be used to detect early failure of coatings.⁴ In the present series of experiments, only one of the systems allowed noticeable rusting, and five systems had detectable blistering after 2 years of exposure in seawater at 25°C. All these five systems showing blistering had strong water uptake in the first 10 days (as indicated by AC resistance changes), but six of the remaining eight systems also had strong water uptake. Three of the five systems showing blistering developed low AC or DC resistances on one or more panels within 150 days, whereas only one of the remaining eight systems developed low resistances within this time. This difference appears to be meaningful. However, it does not appear that the correlation between such low resistance and the degree of blistering is sufficiently high to make such measurements useful for the prediction of coating performance.

In general, the systems which showed the poorest performance did not necessarily give low final electrical resistances. Conversely, the coatings with the lowest final resistances were not necessarily those that performed the least well.

Prior experiments with coatings that showed appreciable deterioration under laboratory immersion had indicated some correlation between electrical properties and performance.^{11,12} However, no such apparent correlation exists for the present series of coatings, all of which perform well in laboratory exposure.

Changes in electrical properties, as determined in these experiments, cannot in themselves be used to reliably predict performance. Since the changes are related to changes within the coating it is possible that these changes, together with other factors and other changes in properties, could be used to predict performance. Such an investigation is presently being conducted at NCEL.

CONCLUSIONS

1. As demonstrated in earlier experiments with coating systems of widely varying performance, coatings with strong decreases in resistance or strong increases in capacitance generally do not perform as well. Coatings which show little change in electrical properties generally perform well.
2. For coatings of comparatively high performance, the changes in electrical properties (including AC resistance, capacitance, and dissipation factor, as well as DC resistance) show correlation with the relative performance in field exposure. However, this correlation is not sufficiently high to allow reliable prediction of comparative performance of good coatings.
3. Although the changes in electrical properties of coatings immersed in seawater do not in themselves reliably predict performance, there is the possibility that such changes may be useful criteria when considered together with results from other accelerated tests.
4. The qualification requirement proposed for the Royal Canadian Navy (that coated panels, similar to those used in the present experiments, maintain a DC resistance of 10^7 ohms after 1 year of immersion) may be useful in accepting only coatings of high performance, but it would also reject many coatings of high performance.
5. Neither the dissipation factors of coatings immersed for a short time in seawater, nor the curves of the dissipation factors versus frequency, appear useful in predicting the performance of coatings.

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