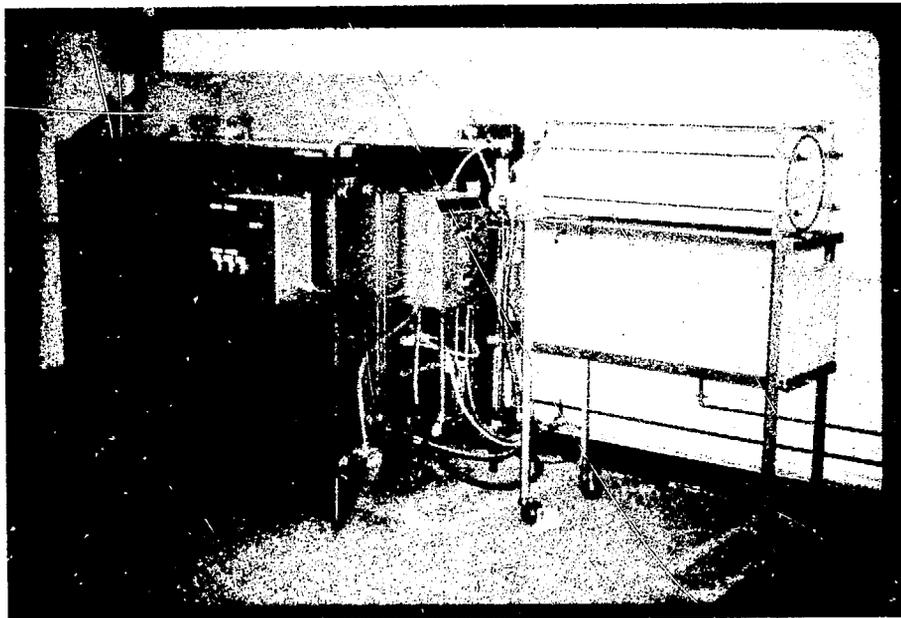


# Comparison of Laboratory Testing Methods for Bridge Coatings

Publication No. FHWA-RD-94-112

June 1995



U.S. Department of Transportation  
**Federal Highway Administration**

Research and Development  
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McLean, Virginia 22010-2296

95-036335

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## FOREWORD

This report presents correlations among various laboratory accelerated testings and natural outdoor marine exposure of low volatile organic compound (VOC) coating systems for steel bridges. This report represents the findings from these testings that compared low VOC coating systems for steel bridges.



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Cover Photo: Prohesion test chamber.

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Technical Report Documentation Page

1. Report No. <b>FHWA-RD-94-112</b>		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle <b>COMPARISON OF LABORATORY TESTING METHODS FOR BRIDGE COATINGS</b>				5. Report Date <b>June 1995</b>	
				6. Performing Organization Code	
7. Author(s) <b>Shuang-Ling Chong, Michael Jacoby, John Boone, and Harry Lum</b>				8. Performing Organization Report No.	
9. Performing Organization Name and Address <b>Office of Engineering and Highway Operations R&amp;D Federal Highway Administration 6300 Georgetown Pike McLean, Virginia 22101-2296</b>				10. Work Unit No. (TRAIS) <b>2E4A0212</b>	
				11. Contract or Grant No.	
12. Sponsoring Agency Name and Address <b>Office of Engineering and Highway Operations R&amp;D Federal Highway Administration 6300 Georgetown Pike McLean, Virginia 22101-2296</b>				13. Type of Report and Period Covered <b>Final Report Sept. 1991-Sept. 1993</b>	
				14. Sponsoring Agency Code	
15. Supplementary Notes <b>Acknowledgment: Technical Discussion given by John Peart</b>					
16. Abstract <p>This study was performed to investigate the effect of combining a freeze cycle, an ultraviolet/condensation cycle (QUV), and a salt-fog plus pollutant/dry cycle (Prohesion) in an accelerated laboratory weathering test on the performance of coating systems for steel bridges. The test results were compared with those obtained from the salt-fog test, the prohesion test, and natural marine exposure. The coating systems selected were water-based systems of acrylic, acrylic epoxy, inorganic zinc alkali silicate, vinyl, and zinc-rich epoxy, and solvent-based systems of calcium sulfonate/alkyd, high-solid epoxy, zinc-rich polyurethanes, epoxy mastic, epoxy urethane mastic, and low-VOC epoxy. All of these coating systems contained volatile organic compound (VOC) content less than 340 g/L. Evaluation parameters for coating performance included coating film thickness, coating gloss, hardness, adhesion strength, blistering, rust, and creepages at scribe. The failure modes of these coatings are discussed and the coating performances are compared.</p> <p>Low-VOC solvent-based zinc-rich polyurethane/polyurethane/polyurethane coating systems outperformed the rest of the coatings tested. The epoxy mastic system and the epoxy urethane mastic system developed serious undercuttings at the scribe. The waterborne acrylic system and the waterborne acrylic epoxy system did not protect steel effectively and they blistered rapidly at the scribe. Zinc-rich primers were highly resistant against corrosion without developing any undercutting on steel, but their water-based topcoats exhibited extensive delamination due to the poor adhesion to the zinc primers. Waterborne vinyl blistered badly in all the laboratory tests, but performed fairly well after 28 months of outdoor exposure. The test results obtained in this study were also analyzed by a statistical variance method to determine the difference among the test methods and coating systems. The cyclic freeze/QUV/Prohesion test results were found to generate a much more promising performance trend when compared to the natural marine exposure results than did the Prohesion test alone. However, salt-fog test results exhibited large deviations from those of the natural marine exposure, indicating that salt-fog testing is not a reliable predictor of field performance of coatings.</p>					
17. Key Words <b>Coatings, testing, performance, inhibitive pigments, waterborne, alkyd resins, acrylics, polyurethane, epoxy, inorganic zinc, organic zinc, epoxy mastic, vinyl resins, corrosion, environmental compatibility, statistical analysis.</b>			18. Distribution Statement <b>No restrictions. This document is available to the public through the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, Virginia 22161.</b>		
19. Security Classif. (of this report) <b>Unclassified</b>		20. Security Classif. (of this page) <b>Unclassified</b>		21. No. of Pages <b>67</b>	22. Price

# SI\* (MODERN METRIC) CONVERSION FACTORS

## APPROXIMATE CONVERSIONS TO SI UNITS

## APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
<b>LENGTH</b>				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
<b>AREA</b>				
in <sup>2</sup>	square inches	645.2	square millimeters	mm <sup>2</sup>
ft <sup>2</sup>	square feet	0.003	square meters	m <sup>2</sup>
yd <sup>2</sup>	square yards	0.836	square meters	m <sup>2</sup>
ac	acres	0.405	hectares	ha
mi <sup>2</sup>	square miles	2.59	square kilometers	km <sup>2</sup>
<b>VOLUME</b>				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft <sup>3</sup>	cubic feet	0.028	cubic meters	m <sup>3</sup>
yd <sup>3</sup>	cubic yards	0.765	cubic meters	m <sup>3</sup>
<b>MASS</b>				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
<b>TEMPERATURE (exact)</b>				
°F	Fahrenheit temperature	5(F-32)/9 or (F-32)/1.8	Celcius temperature	°C
<b>ILLUMINATION</b>				
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m <sup>2</sup>	cd/m <sup>2</sup>
<b>FORCE and PRESSURE or STRESS</b>				
lbf	poundforce	4.45	newtons	N
lbf/in <sup>2</sup>	poundforce per square inch	6.89	kilopascals	kPa

Symbol	When You Know	Multiply By	To Find	Symbol
<b>LENGTH</b>				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
<b>AREA</b>				
mm <sup>2</sup>	square millimeters	0.0016	square inches	in <sup>2</sup>
m <sup>2</sup>	square meters	10.764	square feet	ft <sup>2</sup>
m <sup>2</sup>	square meters	1.195	square yards	yd <sup>2</sup>
ha	hectares	2.47	acres	ac
km <sup>2</sup>	square kilometers	0.386	square miles	mi <sup>2</sup>
<b>VOLUME</b>				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m <sup>3</sup>	cubic meters	35.71	cubic feet	ft <sup>3</sup>
m <sup>3</sup>	cubic meters	1.307	cubic yards	yd <sup>3</sup>
<b>MASS</b>				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
<b>TEMPERATURE (exact)</b>				
°C	Celcius temperature	1.8C + 32	Fahrenheit temperature	°F
<b>ILLUMINATION</b>				
lx	lux	0.0929	foot-candles	fc
cd/m <sup>2</sup>	candela/m <sup>2</sup>	0.2919	foot-Lamberts	fl
<b>FORCE and PRESSURE or STRESS</b>				
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in <sup>2</sup>

\* SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.

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## 1. INTRODUCTION

The U.S. Environmental Protection Agency (EPA) has mandated a strict regulation limiting the amount of volatile organic compounds (VOC's) allowed in the architectural and industrial maintenance coatings. VOC's react with nitrogen oxides through a photochemical process in the presence of sunlight to form lower atmosphere (troposphere) ozone. Generation of ozone is restricted by the 1990 Clean Air Act Amendment. The initial rule proposed by EPA's Architectural and Industrial Coatings Regulatory Negotiation (Reg-Neg) Committee was necessitated by a reduction goal of an initial 25-percent cutback in VOC emissions in 1996, based on 1990 VOC emission levels. A proposed second round of reductions would cut VOC's by 35 percent in the year 2000, and a third phase would bring the total reduction to 45 percent in 2003, also based on 1990 VOC levels. After a few months of discussion in the Reg-Neg process, the rule development for the VOC contents is currently at the final stage and the final VOC limits will be announced in the near future.

A reliable accelerated laboratory test method for predicting field performance and durability of the low-VOC coating systems is imperative in order to ensure cost-effectiveness of newly formulated coatings and to meet a short deadline. The salt-fog testing, as designated in the ASTM (American Society for Testing and Materials) B117 method, does not accurately predict the field performance of many of the new generic low-VOC systems. An inclusion of the dry cycle in the conventional wet salt-fog test (made by Timmins, Sherwood, Lyon and Guest, and Jackson) had avoided unrealistic failures.<sup>(1-4)</sup> When a dry cycle, pollutants, and ultraviolet (UV)/condensation (QUV) exposure were incorporated into the salt-fog cycle, a better correlation with field exposure was obtained by Simpson et al.<sup>(5)</sup> Chong and Peart added a freeze cycle to a salt-fog exposure and this cyclic test, in conjunction with a UV/condensation test, has resulted in a performance trend similar to that obtained by an outdoor weathering of 15 coating systems for steel bridges.<sup>(6)</sup> Freezing is an important part of the weather cycle in cold climates and results in significant mechanical stresses being placed on the coating systems due to the volume expansion of water absorbed by a coating at freezing temperatures. It is of interest to determine the effect of the addition of a freezing cycle to the Prohesion/QUV exposure regimen on its ability to predict field performance. To resolve this question, a combined cycle of freeze, QUV, and salt plus pollutant fog/dry (Prohesion test) was employed to evaluate some high-solids and water-based coating systems for steel bridges. The results were compared with the results obtained by salt-fog and Prohesion exposures alone. Preliminary 28-mo outdoor exposure results of these coating systems at a marine environment site was used for determining which of the accelerated laboratory methods was most reliable for predicting coating performance for steel bridges. Furthermore, a statistical method was employed to compare the test methods.

In addition to the primary objective of the study, valuable data were collected to compare the coating performances of the candidate bridge coating systems. The coating systems evaluated in this study were water-based systems of acrylic, acrylic epoxy, inorganic zinc potassium silicate, vinyl, and zinc-rich epoxy, and solvent-based systems of calcium sulfonate/alkyd, high-solids epoxy, zinc-rich polyurethanes, epoxy mastics, epoxy urethane mastic, and low-VOC epoxy. All of the tested coating systems contain VOC amounts of less than 340 g/L. The coating performance data developed in the study will be used to provide a guideline for the selection of durable low-VOC coatings for protecting steel bridges.

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## 2. EXPERIMENTAL PROCEDURES

### PANEL PREPARATION

All coatings were applied on SSPC SP-5 (blast white), A-36 hot-rolled, and 6.35-mm- (0.25-in-) thick steel panels by the coating manufacturers. Panel sizes of 10.2 cm by 15.2 cm (4 in by 6 in) were used for the salt-fog test, the Prohesion test, and the outdoor exposure test. Panel sizes of 7.6 cm by 15.2 cm (3 in by 6 in) were used for the freeze/QUV/Prohesion test. A 5.1-cm (2-in) diagonal scribe was made on the face of the test panels to study blister and rust creepage from the scribe.

### PHYSICAL TEST

Coating film thickness was measured with a PosiTector 2000 magnetic thickness gauge. Pencil hardness was measured with a GARDCO pencil hardness gauge. Gloss was measured with a Novogloss 20/60 glossmeter according to ASTM method D525. Adhesion strength was determined with a Model 106 Elcometer adhesion tester according to ASTM method D4541. An American Optical Corporation optical microscope with a Polaroid camera was used for taking magnified pictures.

### ACCELERATED TESTING

The 13 coating systems tested are described in table 1. Three accelerated laboratory exposures were used to evaluate the candidate coating systems. These tests are as follows:

1. Salt-Fog:  
American Society for Testing and Materials (ASTM B117).
2. Prohesion - 1-h wet/1-h dry cycle.  
Wet cycle: Harrison mixture of 0.35 percent ammonium sulfate and 0.05 percent sodium chloride. The collected condensate has a pH of 5.0.  
Dry cycle: forced-air purging (6.8 m<sup>3</sup>/h).
3. Cyclic Freeze/QUV/Prohesion - 70-h freeze/215-h QUV/215-h Prohesion cycle.  
Freeze temperature: -23 °C (-10 °F)  
QUV: UV/Condensation test  
Test cycle: 4-h UV/4-h condensation cycle  
UV lamp: UVA-340  
UV temperature: 60 °C  
Condensation temperature: 40 °C  
Prohesion: same as test 2.

Table 1. Coating systems.

<u>Code No.</u>	<u>Description</u>	<u>Dry Film Thickness</u> <u>mm x 100 (mil)</u>	<u>VOC (g/L)<sup>1</sup></u>
1	Solvent-based Calcium Sulfonate/Alkyd, 2 coats	13/10 (5/4)	276/288
2	Solvent-based High-solids Epoxy	20 (8.0)	180
3	Waterborne Acrylic, 3 coats	7.5/7.5/7.5 (3/3/3)	132/109/109
4	Waterborne Acrylic Epoxy, 3 coats	7.5/7.5/7.5 (3/3/3)	134/133/133
5	Solvent-based Zinc-rich Polyurethane/ Polyurethane/Polyurethane	6.3/12.5/20 (2.5/5.0/8.0)	336/336/336
6	Solvent-based Zinc-rich Polyurethane/ Polyurethane/Polyurethane	6.3/12.5/20 (2.5/5.0/8.0)	336/250/250
7	Solvent-based Zinc-rich Polyurethane/ Waterborne Polyurethane/Waterborne Polyurethane	6.3/12.5/20 (2.5/5.0/8.0)	336/24/24
8	Solvent-based Epoxy Mastic/Polyurethane	12.5/5.0 (5.0/2.0)	84/288
9	Solvent-based Epoxy Urethane Mastic/Polyurethane	12.5/5.0 (5.0/2.0)	327/288
10	Water-based Inorganic Zinc Potassium Silicate/ Water-based Acrylic/Water-based Acrylic	6.3/8.8/8.8 (2.5/3.5/3.5)	0/237/241
11	Solvent-based Low-VOC Epoxy/Acrylic Modified Epoxy	13.8/5.0 (5.5/2.0)	308/282
12	Waterborne Vinyl, 3 coats	11.3/11.3/11.3 (4.5/4.5/4.5)	2/2/64
13	Water-based Zinc-rich Epoxy/Acrylic/Acrylic	7.5/7.5/7.5 (3.0/3.0/3.0)	86/230/230

<sup>1</sup> 120 g/L = 1 lb/gal

One set of panels was also exposed at Sea Isle City, New Jersey, a marine exposure site. All the test panels were placed at a 45-degree angle on wooden racks, facing directly south. Each panel was sprayed three times daily with seawater (pH = 7.7, specific gravity at 15.6 °C = 1.021).

All the tests were carried out in duplicate to ensure statistical reliability and the results presented are an average of the two panels.

#### EVALUATION METHODS

The accelerated test panels were examined every 500 h to record their failure modes and to study the rate of deterioration. Evaluation criteria were blistering, rusting, creepages at scribe, and undercuttings at scribe. Creepage is the distance of surface blistering and/or rusting that has progressed from scribe that is visible without disturbing the film. No information on primer performance is available when the creepage is shown. Undercutting is the distance traveled from scribe after loose material was removed by a sharp knife after exposure was completed. Degree of blistering was evaluated by ASTM method D714. Surface failures (unscribed area) and creepages at scribe were rated in accordance with ASTM method D1654. To improve accuracy, a grid of 6.35-mm (1/4-in), instead of 12.7-mm (1/2-in), squares was used for measuring surface failure. Both creepages and undercuttings were measured in millimeters to an accuracy of 0.5 mm and two readings were taken at each scribe, one at the left side and the other at the right side.

Coating film thickness and gloss were measured before exposure and after 1,500 h, as well as after 3,000 h of the Prohesion and the freeze/QUV/Prohesion exposures. Coating film thickness and gloss were determined for the salt-fog test panels and the coating adhesion strengths in all the tests were measured only before exposure and after exposure was terminated.

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### 3. RESULTS AND DISCUSSION

The physical properties of all the tested coating systems, such as coating film thickness, 60° gloss, pencil hardness, and adhesion strength, were determined before exposure as a basis for evaluating coating degradation; they are shown in table 2. The pencil hardness determines the rigidity and mechanical property of a coating system. The adhesion test measures both the degree of cohesive failure and adhesive strength of a coating system. The type of adhesion failure obtained was noted. Cohesive failure is the separation within a coating system and the adhesive failure is the separation at either the interface of the top coat, the intermediate coat, the primer, or the steel substrate. The symbols T/P, P/S, and I/P are used to designate adhesive failure between topcoat and primer, between primer and steel surface, and between intermediate coat and primer, respectively.

The exposure of most of the salt-fog test panels were terminated after 6.35 mm (0.25 in) of creepage at the scribe occurred (a general standard criterion for a pass or fail classification). A few panels were exposed for a longer time, due to their peculiar failure modes, to obtain additional information. The Prohesion tests and the cyclic freeze/QUV/Prohesion tests were conducted for a full period of 3,000 h for all the coating systems; this long exposure time was essential because both of these tests included a dry cycle that resulted in a reduced failure rate when compared to the salt-fog results. The additional sets of data points obtained for the later two tests were highly beneficial in studying the exposure method difference using a statistical method.

#### SALT-FOG EXPOSURE

The pencil hardness and the adhesion strength of the tested coating systems after the salt-fog exposure are shown in table 3. The hardness of most of the coating systems remained the same after the exposures, except for the waterborne acrylic (code no. 3), waterborne acrylic epoxy (code no. 4), and the solvent-based epoxy mastic/polyurethane (code no. 8) systems that had slightly lower hardness than prior to exposure.

Seven coating systems retained strong adhesion strength (>7.0 MPa). The solvent-based high-solids epoxy (code no. 2), water-based inorganic zinc/acrylic/acrylic (code no. 10), and waterborne vinyl (code no. 12) retained their original adhesion strength; whereas the solvent-based epoxy mastic/polyurethane (code no. 8) strength decreased from >7.0 to 4.2 MPa. The increase in the adhesion strength of the solvent-based epoxy urethane mastic/polyurethane (code no. 9) from 4.1 to 5.7 MPa after the salt-fog exposure indicates a further curing of the coating system by moisture.

The changes of film thickness and gloss after the salt-fog test are shown in figures 1 and 2. It should be noted that eight of the coating systems were exposed less than 3,000 h because of their early failures; the total exposure times for each coating system are indicated in table 3. A majority of the coating systems gained film thickness, implying that they swelled due to the moisture that diffused into the coatings under the high humidity in the salt-fog test and was maintained in the coating systems. All the coating systems lost some gloss after exposure, except for the waterborne polyurethane (code no. 7), which acquired more reflectivity.

Table 2. Physical properties of candidate coating systems.

<u>Code No.</u>	<u>Thickness,</u> <u>nm x 100 (mil)</u>	<u>60° Gloss</u>	<u>Pencil</u> <u>Hardness</u>	<u>Adhesion Strength, MPa<sup>1</sup></u> <u>(failure type)</u>
1	30.8 (12.3)	6.8	<1-6B	0 (T/P)
2	28.0 (11.2)	19.0	4-HB	5.0 (P/S)
3	25.8 (10.3)	48.2	3-2B	>7.0
4	20.5 (8.2)	56.7	4-HB	>7.0
5	25.8 (10.3)	74.8	3-2B	>7.0
6	28.0 (11.2)	70.6	3-2B	>7.0
7	18.0 (7.2)	32.1	4-HB	>7.0
8	20.5 (8.2)	79.4	4-HB	>7.0 (T/P)
9	16.3 (6.5)	42.7	3-2B	4.1 (I/P)
10	20.5 (8.2)	41.5	3-2B	4.7 (I/P)
11	14.5 (5.8)	85.1	4-HB	>7.0
12	28.5 (11.4)	54.2	2-4B	3.9 (I/P)
13	25.3 (10.1)	48.6	2-4B	>7.0

<sup>1</sup> 1 MPa = 142.9 lbf/in<sup>2</sup>

P: Primer

I: Intermediate coat

T: Topcoat

S: Steel

Table 3. Pencil hardness and adhesion strength after salt-fog exposure.

<u>Code No.</u>	<u>Exposure Time</u>	<u>Pencil Hardness</u> <u>(Before Exposure)</u>	<u>Adhesion Strength, MPa<sup>1</sup></u> <u>(Before Exposure)</u>
1	500	<1-6B(<1-6B)	0 (0)
2	3,000	4-HB (4-HB)	6.9 (>7.0)
3	1,000	2-4B (3-2B)	>7.0 (>7.0)
4	3,000	3-2B (4-HB)	>7.0 (>7.0)
5	3,000	4-HB (4-HB)	>7.0 (>7.0)
6	3,000	3-2B (3-2B)	>7.0 (>7.0)
7	2,500	4-HB (4-HB)	>7.0 (>7.0)
8	3,000	3-2B (4-HB)	4.2 (>7.0)
9	2,000	3-2B (3-2B)	5.7 (4.1)
10	500	3-2B (3-2B)	4.8 (4.7)
11	2,500	4-HB (4-HB)	>7.0 (>7.0)
12	500	3-2B (3-2B)	4.1 (3.9)
13	1,500	2-4B (2-4B)	>7.0 (>7.0)

<sup>1</sup> 1 MPa = 142.9 lbf/in<sup>2</sup>

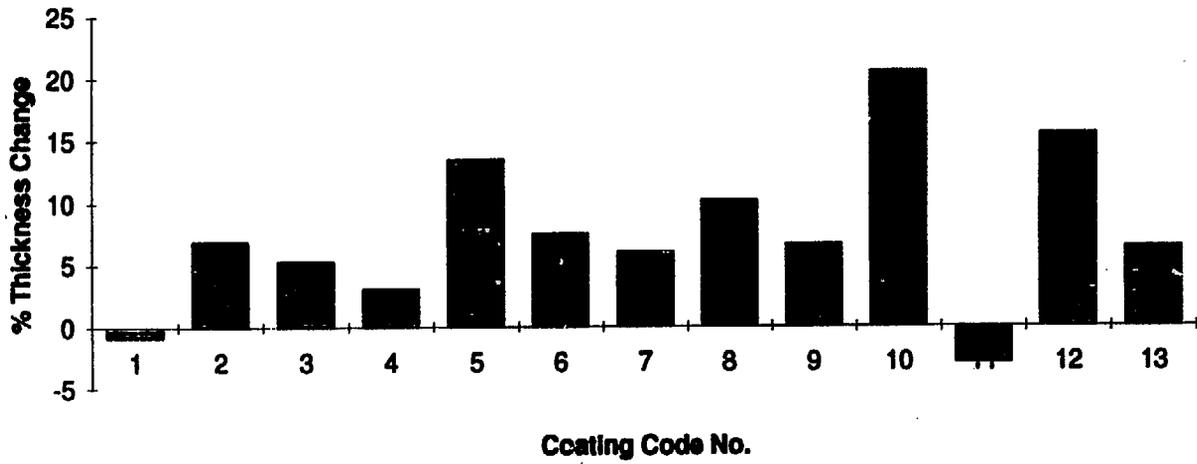


Figure 1. Film thickness change after salt-fog exposure.

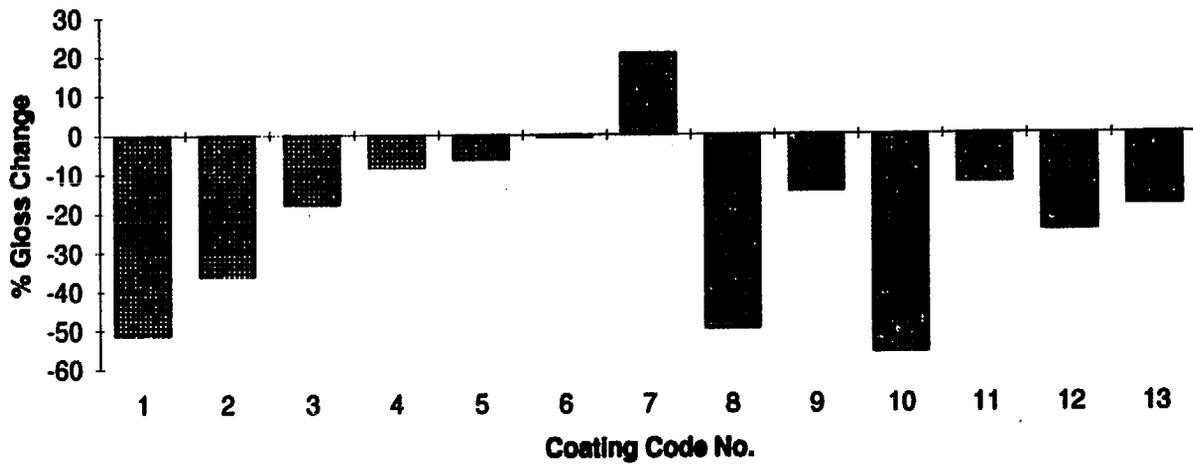


Figure 2. Gloss change after salt-fog exposure.

Two types of coating failures were investigated in this study; they were plane-surface failure and scribe failure. The condensed coating failure results of the salt-fog test are shown in table 4. The characteristic failure modes and relative performance of all the candidate coating systems are described as follows.

### Plane Surface Failure

The salt-fog test, as specified by ASTM method B117, provides an extremely harsh environment for coating due to the high salt concentration (5 percent sodium chloride) and high humidity (97 percent). Under this condition, the diffusion of water through a coating film is a continuous process that provides a strong driving force for blistering. The water diffusion was demonstrated by the increases in film thickness after exposure to salt-fog as previously stated. The result of this water diffusion through a semi-porous coating film is often a rapid formation of blisters. The formation of various patterns of blisters, depending upon the characteristics of a coating, was observed on some of the coating systems in this study.

The water-based inorganic zinc potassium silicate/acrylic/acrylic system (code no. 10) exhibited severe blistering of the topcoats both on the panel surface and around the panel edges after only 500 h of the salt-fog test (figure 3), even though no rusting or undercutting of the inorganic zinc were observed on the surface and at the scribe. A large number of size 6 blisters were distributed over the panel surface and size 1 blisters (larger than size 2 blisters, 9 mm in diameter) developed 2 mm away from the scribe; this blister formation may be a result of relatively weak adhesion of the acrylic topcoat to the inorganic zinc primer (4.7 MPa) as determined by the pull-off adhesion test of the coating on an unexposed panel. The failure mechanism was intercoat adhesive failure between the inorganic zinc primer and the intermediate coat of acrylics. A similar topcoat failure (peeling) was also found for the high-ratio, water-based inorganic zinc potassium silicate system in our previous study, as well as in a similar FHWA study by Kogler and Mott.<sup>(6-7)</sup> Topcoat blistering away from the scribe was also observed on the topcoated inorganic zinc potassium silicate system in a study by Szokolik.<sup>(8)</sup> Topcoat delamination from water-based inorganic zinc has also been experienced on operational bridges. On the contrary, a generically similar, but differently formulated, topcoated, water-based inorganic zinc potassium silicate system performed excellently in the cyclic salt-fog/freeze test in our previous staff study.<sup>(6)</sup> The delamination was possibly caused by the incompatibility of the topcoats with the relatively alkaline zinc primers that alkali carbonate formed together with zinc silicate polymer matrix after curing. In addition, both size and uniformity of the distribution of zinc particles can affect the rheological properties of the coating, which affect the application characteristics and the uniformity of the applied film.<sup>(9)</sup> Extensive topcoat blistering (very fine, smaller than size 8, x40 magnification shown in figure 4) with limited undercutting (3.3 mm) occurred at the scribe of the solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane (code no. 7). The higher-VOC, solvent-based polyurethane topcoats (code nos. 5 and 6) with the same primer, however, did not exhibit any blistering at the scribe. Another incident of blister clusters (6D, x40 magnification shown in figure 5) occurring at the scribe was found on the water-based zinc-rich epoxy/acrylic/acrylic (code no. 13). These very small, but dense, blisters were difficult to see because of the color of the coating; but they were visible with an optical microscope at x40 magnification. These topcoat

Table 4. Results of salt-fog exposure.

Code	Hour	Panel Face			Scribe		Scribe Creepage, mm
		Blisters <sup>1</sup>	Rust	Other	Rust	Blisters <sup>1</sup>	
1	500	- <sup>2</sup>	-	TD <sup>3</sup>	-	-	0
1	500	-	-	TD <sup>3</sup>	-	-	0
2	3000	-	-		SEV	3D	5.0
2	3000	-	-		SEV	2D	6.8
3	1000	-	-		SEV	2D & 4M	9.9 (1,000) <sup>4</sup>
3	1000	-	-		SEV	2D & 4M	10.5 (1,000) <sup>4</sup>
4	3000	-	-		SEV	2VF	7.0
4	3000	-	-		SEV	4F	5.0
5	3000	-	-		SEV	8D	1.5
5	3000	-	-		SEV	8D	3.0
6	3000	-	-		SEV	8F	2.2
6	3000	-	-		SEV	-	2.9
7	2500	8VF	-		SEV	9VD	15.0 <sup>5</sup> (2,000) <sup>4</sup>
7	2500	8VF	-		SEV	9VD	17.3 <sup>6</sup> (2,000) <sup>4</sup>
8	3000	4MD <sup>7</sup>	Edge		SEV	2D	5.3
8	3000	4F <sup>7</sup>	Edge		SEV	2D	5.0
9	2000	9D <sup>7</sup> (Edge)	Edge		SEV	9VD	19.8 (1,000) <sup>4</sup>
9	2000	9D <sup>7</sup> (Edge)	Edge		SEV	9VD	24.0 (1,000) <sup>4</sup>
10	500	2F(Edge)	-		-	1VF <sup>8</sup>	0
		6M					
10	500	2F(Edge)	-		-	1VF <sup>9</sup>	0
		6M					
11	2500	-	Edge		SEV	4F	7.4 (2,500) <sup>4</sup>
11	2500	-	Edge		SEV	4F	7.1 (2,500) <sup>4</sup>
12	500	6M	-		SEV	4VF	5.0
12	500	6M	-		SEV	4F	4.0
13	1500	6D(Corner)	-		SEV	6VD	28.8 <sup>10</sup> (500) <sup>4</sup>
13	1500	6D(Corner)	-		SEV	6VD	24.0 <sup>11</sup> (500) <sup>4</sup>

<sup>1</sup> Method ASTM D714, Evaluation Degree of Blistering of Paints.

<sup>2</sup> None.

**Table 4. Results of salt-fog exposure (Continued).**

3	Topcoat delamination.
4	Hours exposed to develop more than 6.35-mm creepages at the scribe.
5	Undercut is only 3.3 mm.
6	Undercut is only 2.5 mm.
7	Underfilm corrosion.
8	11 mm away from scribe.
9	10 mm away from scribe.
10	Undercut is only 1.3 mm.
11	Undercut is only 1.5 mm.



**Figure 3. Condition of the water-based inorganic zinc potassium silicate/acrylic/acrylic system after 500 h of salt-fog exposure.**



**Figure 4. Condition of the solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane at x40 magnification after 2,500 h of salt-fog exposure.**



**Figure 5. Condition of the water-based zinc-rich epoxy/acrylic/acrylic system at x40 magnification after 1,500 h of salt-fog exposure.**

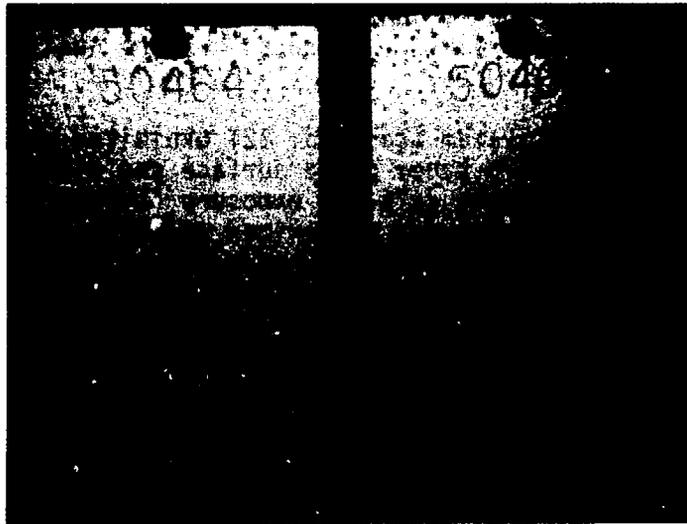
failures demonstrate that the formulation of the water-based topcoats that perform well over water-based zinc-rich primers is difficult, which may also explain why some suppliers resist recommending such topcoats over these zinc primers. More research is needed to better define primer curing requirements and the development of a simple field test to ensure the quality of the primer for topcoating.

The waterborne vinyl coating system (code no. 12) exhibited pronounced blistering (6MD) over the entire panel plane surface and size 4 blisters at the scribe after only 500 h of the salt-fog exposure (figure 6). Again, the relatively weak adhesion found between the primer and the topcoat (3.9 MPa) for the unexposed coating system may be the precursor to the delamination. These blisters were filled with electrolytes (chloride and water) after the salt-fog exposure, but the apparent collapse of the majority of the blisters (figure 7) was noticed 2 d after being exposed to ambient laboratory environment (temperature = 25 °C, relative humidity = 50 percent), implying that drying for a reasonable timeframe has helped to heal the blistering. However, this pronounced blistering phenomenon was not found by Clement for the waterborne vinyl system evaluated after 1,500 h of his salt-fog exposure.<sup>(10)</sup> This discrepancy may be attributed to different coating formulations or variations in panel preparations.

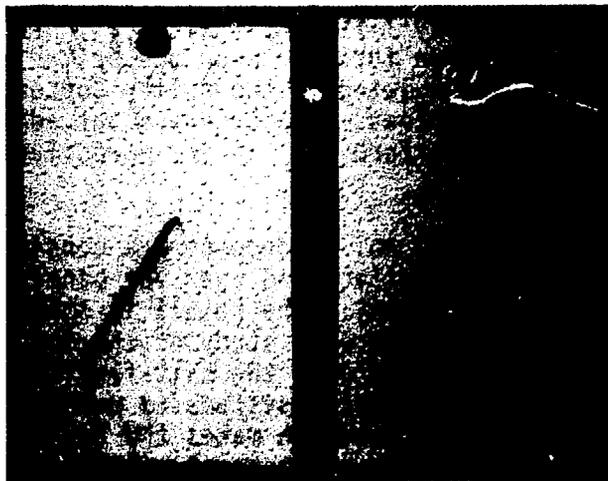
The calcium sulfonate/alkyd coating system is soft and vulnerable to abrasion and impact. Its pencil hardness is less than 1-6B. The topcoat peeled easily at the edges of scribes at the time of scoring, which demonstrates its fragility. Furthermore, in the adhesion test of this coating, the topcoat separated easily when roughed with sand paper prior to the bonding of a dolly, indicating that the adhesion of the topcoat to the primer is very weak. This suggests that the use of the calcium sulfonate/alkyd coating system on structures exposed to vehicle/personnel traffic and dirt conditions would not be advisable. The corrosion protection characteristics are acceptable. After 500 h of salt-fog exposure, the topcoat was easily separable and detachable from the primer coat and was easily lifted by a blade (figure 8). The failure mode demonstrates that the adhesion force between the primer and topcoat is extremely weak so that water accumulated at the primer-topcoat interface when it diffused through the porous topcoat. Improved topcoat adhesion is necessary for the coating system to perform well in an aggressively corrosive environment.

The epoxy mastic/polyurethane (code no. 8) developed a considerable number of rust-filled blisters on the plane surface and near the panel edges after 3,000 h of the salt-fog test (figure 9). The result is consistent with the underfilm corrosion observed in our earlier study of the low-VOC epoxy mastic/polyurethane system after 3,000 h of the salt-fog/freeze test.<sup>(6)</sup> The susceptibility of the low-VOC epoxy mastic systems to underfilm corrosion demonstrated in this and other FHWA research makes the use of these materials to protect bridge steel in a salt-rich environment questionable, particularly since other systems have demonstrated superior performance in such applications.

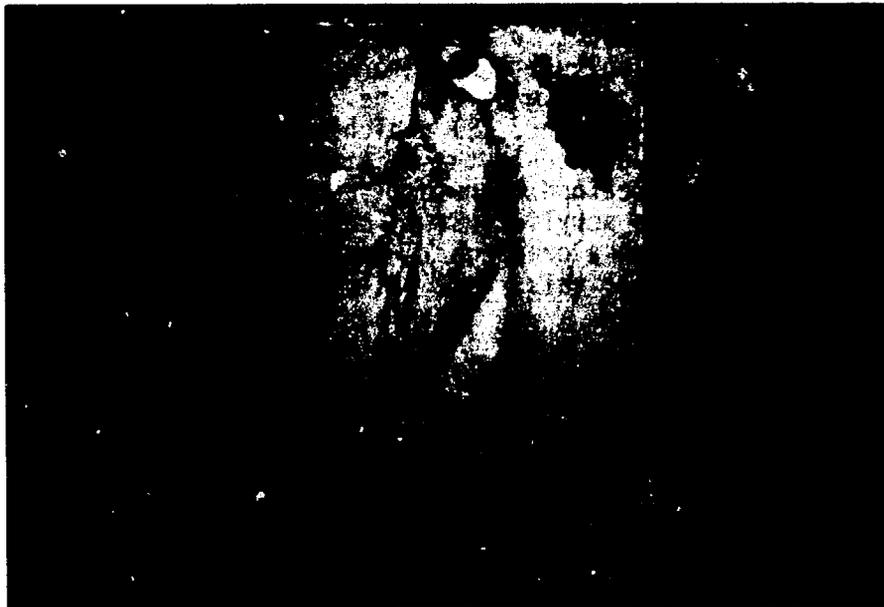
The epoxy urethane mastic/polyurethane (code no. 9) showed 9VD blisters (smaller than size 8 blisters) at the edges after 2,000 h of the salt-fog test (figure 10); these blisters extended about 15 mm from all four edges of the coated panels and underfilm corrosion was observed beneath the blisters.



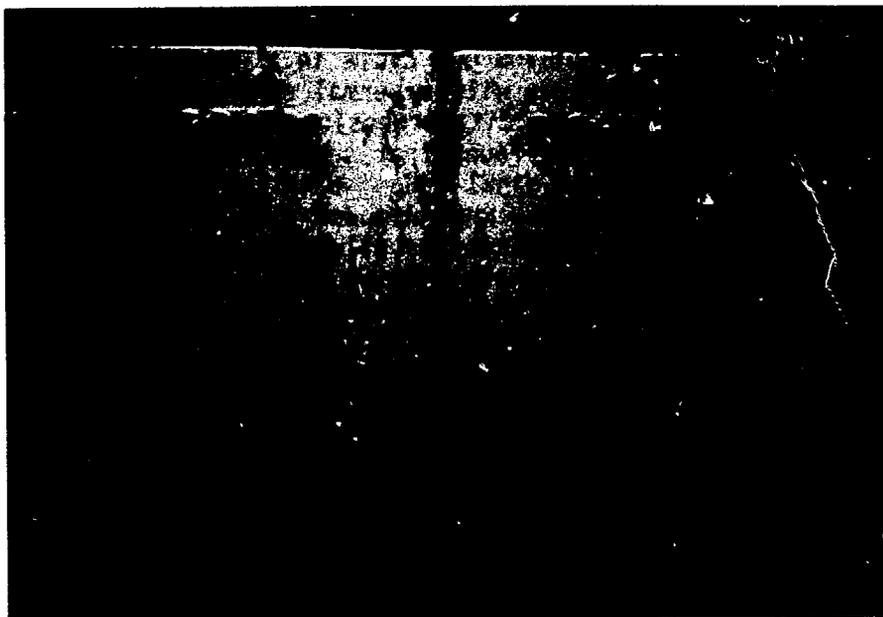
**Figure 6. Condition of the waterborne vinyl system after 500 h of salt-fog exposure.**



**Figure 7. Condition of the waterborne vinyl system after 500 h of salt-fog exposure followed by standing in air for 48 h.**



**Figure 8.** Condition of the calcium sulfonate/alkyd system after 500 h of salt-fog exposure. (The easily lifted topcoat is shown at right.)



**Figure 9.** Condition of the epoxy mastic/polyurethane after 3,000 h of salt-fog exposure.

## Scribe Failure

All of the coating systems developed creepage or cutback at the scribes. A majority of the coatings undercut to the same degrees of creepages, except for the solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane (VOC of topcoat = 24 g/L, code no. 7) and the water-based zinc-rich epoxy/acrylic/acrylic (code no. 13), which had minute undercutting. No underfilm corrosion was found beneath the blisters at the scribe for these materials, illustrating the cathodic protection of steel afforded by zinc-metal primer. The waterborne acrylic system (code no. 3) exhibited severe blistering at the scribe with a creepage larger than 6.35 mm after 1,000 h of the salt-fog test (figure 11); this rapid failure makes its use in a high-salt environment questionable.

To compare coating performance, the failure times yielding more than 6.35 mm (0.25 in) of creepage at the scribes are plotted for all the coatings in figure 12; the arrow symbol (†) denotes time longer than 3,000 h. (Note: code nos. 1 and 10 are not included.) If a creepage of 6.35 mm or greater is used as a criterion for failure, the high-solids epoxy (code no. 2), waterborne acrylic epoxy (code no. 4), two solvent-based zinc-rich polyurethane/polyurethane/polyurethane (code nos. 5 and 6), epoxy mastic/polyurethane (code no. 8), and waterborne vinyl systems (code no. 12) did not fail at the scribe after 3,000 h of the salt-fog test. Other coating systems performed as follows (in decreasing order): low-VOC epoxy/acrylic modified epoxy (code no. 11), solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane (code no. 7), epoxy urethane mastic/polyurethane (code no. 9), waterborne acrylic (code no. 3), and water-based zinc-rich epoxy/acrylic/acrylic (code no. 13).

## PROHESION EXPOSURE

The use of a Harrison mixture of 0.35 percent ammonium sulfate and 0.05 percent sodium chloride followed by a dry cycle in a Prohesion test has been found to produce better correlation with a normal exterior environment than does either salt-fog or the SO<sub>2</sub>/humidity Prohesion testing procedure alone (the Kestenich Test).<sup>(11,12)</sup> Walker found that several coatings tested lost 50 percent to 70 percent of their original adhesion in less than 10 h of immersion, but recovered appreciably when removed from immersion.<sup>(13)</sup> The extent of recovery improves as more time is allowed for drying after immersion, but declines with the length of time that the coatings are originally immersed. This test, with cyclic 1-h salt-fog/1-h dry air, was employed on the coating systems to study their failure modes caused by the exposure.

The pencil hardness and adhesion strength of the coating systems after 3,000 h of the Prohesion test are presented in table 5. The hardness of the high-solids epoxy, waterborne acrylic, waterborne acrylic epoxy, solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane, epoxy mastic/polyurethane, epoxy urethane mastic/polyurethane, and low-VOC epoxy/acrylic modified epoxy decreased after the exposure, whereas those of the other coating systems remained the same. The adhesion strengths of the epoxy urethane mastic/polyurethane and waterborne vinyl lessened. The adhesion strength of the water-based inorganic zinc potassium silicate/acrylic/acrylic system increased with >7.0 and 6.6 MPa at the area above the scribe and that below the scribe, respectively. The area below the



Figure 10. Condition of the epoxy urethane mastic/polyurethane after 2,000 h of salt-fog exposure.



Figure 11. Condition of the waterborne acrylic system after 1,000 h of salt-fog exposure.

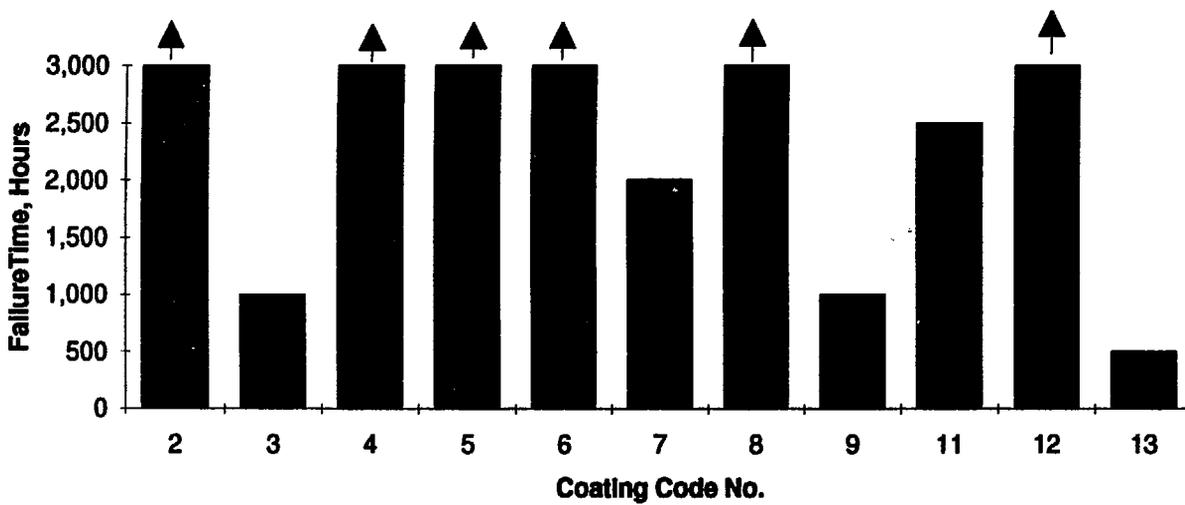


Figure 12. Failure time of the coating systems for developing 6.35 mm of creepage after salt-fog exposure.

Table 5. Pencil hardness and adhesion strength after 3,000 h of prohesion exposure.

<u>Code No.</u>	<u>Pencil Hardness (Before Exposure)</u>	<u>Adhesion Strength, MPa<sup>1</sup> (Before Exposure)</u>
1	<1-6B (<1-6B)	0 (0)
2	3-2B (4-HB)	>7.0 (>7.0)
3	2-4B (3-2B)	>7.0 (>7.0)
4	3-2B (4-HB)	6.4 (>7.0)
5	3-2B (3-2B)	>7.0 (>7.0)
6	3-2B (3-2B)	>7.0 (>7.0)
7	3-2B (4-HB)	>7.0 (>7.0)
8	3-2B (4-HB)	>7.0 (>7.0)
9	2-4B (3-2B)	4.3 (4.1)
10	3-2B (3-2B)	>7.0, <sup>2</sup> 6.6 <sup>3</sup> (4.7)
11	3-2B (4-HB)	>7.0 (>7.0)
12	2-4B (2-4B)	2.2 (3.9)
13	2-4B (2-4B)	>7.0 (>7.0)

- 
- <sup>1</sup> 1 MPa = 142.9 lbf/in<sup>2</sup>  
<sup>2</sup> Area above the scribe.  
<sup>3</sup> Area below the scribe.

scribe was exposed to a more concentrated salt solution due to the drip of salt solution along the panels. The coating with increased adhesive strength after the exposure indicates that moisture may have enhanced the curing of the water-based inorganic zinc coating system.

The changes of coating film thickness and gloss after 1,500 and 3,000 h of the Prohesion test are plotted in figures 13 and 14 respectively. All the coating systems lost some film thickness, except the epoxy urethane mastic/polyurethane (code no. 9) which must have absorbed water during the exposure period. With the exception of a gloss increase for the waterborne polyurethane (code no. 7), all the coating systems suffered gloss reduction after 3,000 h of the Prohesion exposure.

The summarized failure results of the Prohesion exposure are presented in table 6.

### Plane Failure

After 3,000 h of the Prohesion test, the calcium sulfonate/alkyd coating (code no. 1) did not exhibit any blistering or rusting on the plane surface or at the scribe. However, the softness of the topcoat resulted in coating loss on the bottom edge of the panels while transferring the panels in and out of the holding rack during the examination every 500 h; consequently, some areas of the panel bottom and corners became heavily rusted after 3,000 h due to the exposure of the bare steel to the corrosive environment. This failure behavior shows that the 1-h wet/1-h dry cycles did not suppress the delamination of the calcium sulfonate/alkyd topcoat. In the Prohesion test, the waterborne vinyl system (code no. 12) initially developed blistering (8D) on edges after 1,500 h of the exposure, then blisters of 8M were found to be distributed all over the entire coating plane after 2,000 h of exposure. The adhesion strength of this vinyl system decreased from 3.9 to 2.0 MPa after the exposure and the pulled dollies revealed the topcoat/intermediate coat (T/I) intercoat adhesive failure with no underfilm corrosion (figure 15). This failure mode was similar to that found for the same coating system after 500 h of the salt-fog testing. No topcoat failure, i.e. blistering was observed for the water-based inorganic zinc/acrylic/acrylic (code no. 10) after the Prohesion test. This may have been the result of the addition of a dry cycle in the Prohesion regimen. It is not clear if the dry cycle prevented the delamination or the salt concentration (0.05 percent sodium chloride) in the Prohesion test was too low to induce the failure.

### Scribe Failure

All of the coatings exhibited creepage at the scribe, except the solvent-based calcium sulfonate/alkyd, which did not show any rust and creepage at the scribe but developed the topcoat delamination as described in the salt-fog test. The waterborne acrylic epoxy, the solvent-based low-VOC epoxy/acrylic modified epoxy, and the water-based zinc-rich epoxy/acrylic generated the largest amount of creepage (>10 mm). Two epoxy mastic systems and high-solids epoxy coating developed the next largest amounts of creepage (~7.0 and 6.0 mm). The waterborne acrylic system and the waterborne vinyl systems exhibited 5.0 and 4.5 mm of creepage, respectively. The coating systems with the least amount of creepage (2.0 to 3.5 mm) were the water-based inorganic zinc potassium silicate/acrylic/acrylic and the three zinc-rich polyurethane/polyurethane/polyurethane systems (VOC of topcoat = 24 g/L, 250 g/L,

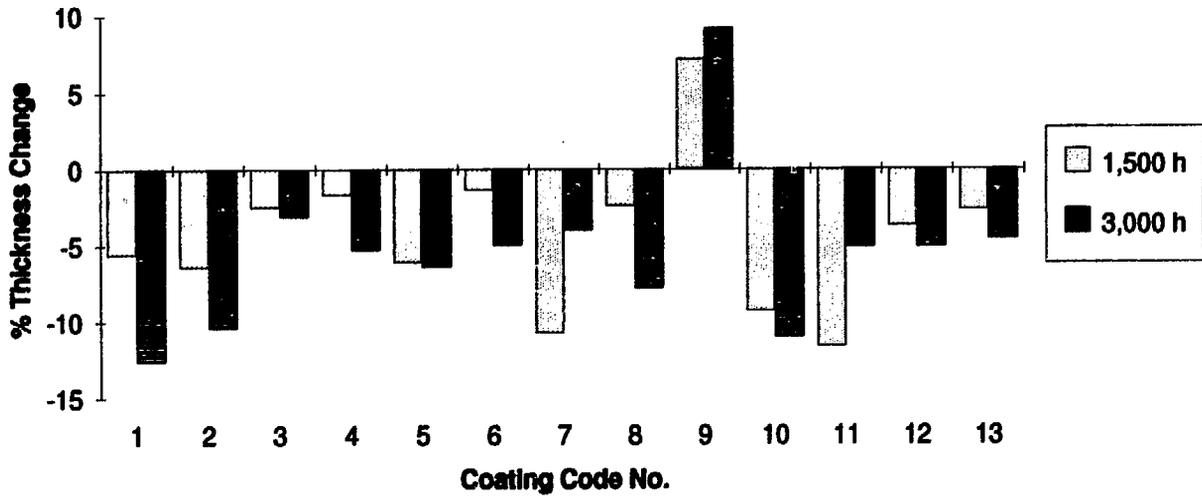


Figure 13. Film thickness change of the coating systems after 1,500 and 3,000 h of Prohesion exposure.

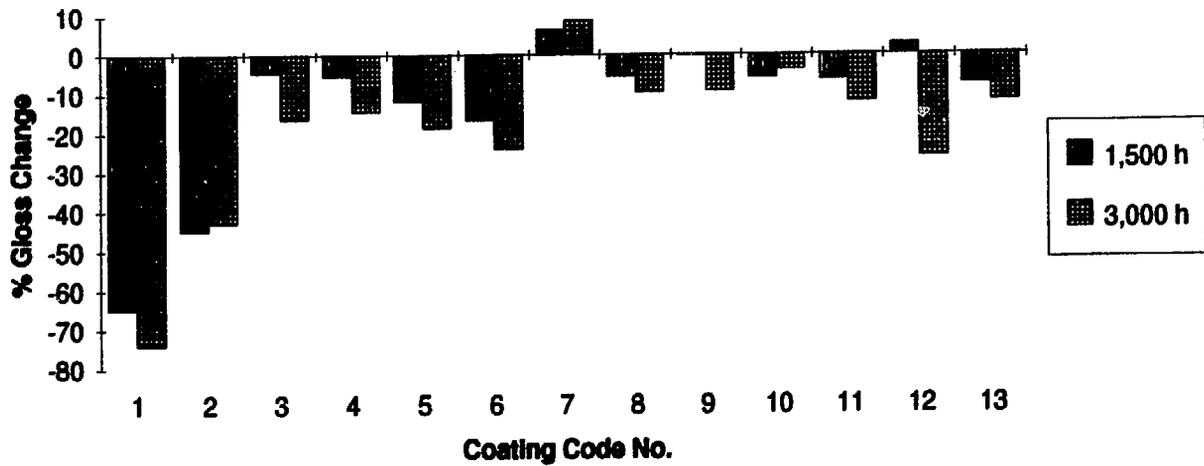


Figure 14. Gloss change of the coating systems after 1,500 and 3,000 h of Prohesion exposure.

Table 6. Results of Prohesion exposure.

Code	Hour	Plane Face		Others	Scribe		Scribe Creepage, mm
		Blisters <sup>1</sup>	Rust		Rust	Blisters <sup>1</sup>	
1	3,000	- <sup>2</sup>	Edge	TD <sup>3</sup>	-	-	0
	3,000	-	Edge	TD <sup>3</sup>	-	-	0
2	3,000	-	-	-	MOD	6MD	7.6
	3,000	-	-	-	MOD	6MD	4.5
3	3,000	-	-	-	SEV	6D	3.5
	3,000	-	-	-	SEV	6D	6.0
4	3,000	-	-	-	SEV	2D	24.5 (1,000) <sup>4</sup>
	3,000	-	-	-	SEV	2D	17.8 (1,000) <sup>4</sup>
5	3,000	-	-	-	MOD	6D	3.8
	3,000	-	-	-	MOD	6D	2.5
6	3,000	-	-	-	MOD	4D	4.3
	3,000	-	5	-	MOD	6VF	2.0
7	3,000	8VF	6	-	SEV	6F	2.0
	3,000	8VF	6	-	SEV	6F	3.0
8	3,000	-	-	-	SEV <sup>7</sup>	2D	7.3
	3,000	-	-	-	SEV <sup>7</sup>	2D	6.5
9	3,000	-	-	-	SEV <sup>7</sup>	2D	6.5
	3,000	-	-	-	SEV <sup>7</sup>	2D	5.5
10	3,000	-	Edge	-	MOD	6D	1.5
	3,000	-	Edge	-	MOD	6MD	2.0
11	3,000	-	-	-	SEV <sup>7</sup>	6VD	9.8 (1,500) <sup>4</sup>
	3,000	-	-	-	SEV <sup>7</sup>	6VD	14.0 (1,500) <sup>4</sup>
12	3,000	8MD	-	-	SEV	4M	4.5
	3,000	8MD	-	-	SEV	4M	3.5
13	3,000	-	-	-	MOD	9D	11.5 <sup>8</sup>
	3,000	-	-	-	MOD	9D	12.0 <sup>9</sup>

<sup>1</sup> Method ASTM D714, Evaluation Degree of Blistering of Paints.

<sup>2</sup> None.

<sup>3</sup> Topcoat delamination.

<sup>4</sup> Hours exposed to develop more than 6.35 mm of creepage.

<sup>5</sup> Three size 6 pits.

<sup>6</sup> Five size 6 pits.

<sup>7</sup> Raised rust.

<sup>8</sup> Undercut is only 2.5 mm.

<sup>9</sup> Undercut is only 2.0 mm.

MOD Moderate

SEV Severe

and 336 g/L, respectively). Among all the coating systems tested, the waterborne acrylic epoxy system failed most rapidly. It developed 8.5 mm of creepage (>6.35 mm) at the scribe after only 1,000 h of the Prohesion test as demonstrated by figure 16; on the contrary, this system showed a creepage of only 6.0 mm after 3,000 h of the salt-fog test. This difference in failure results implies that the waterborne acrylic epoxy may be prone to the attack of the weak acid (ammonium sulfate) used in the Prohesion test.

Virtually all of the coating systems tested in the Prohesion chamber undercut to the same degree of creepage, except the water-based zinc-rich epoxy/acrylic/acrylic coating system (code no. 13), which showed a large area of dense topcoat blisters of size 9 extending 12.0 mm from the scribes, but only undercut 2.2 mm at the scribe.

The failure time for developing 6.35 mm or more of creepage is compared in figure 17. Four coating systems performed poorly; they are waterborne acrylic epoxy (code no. 4), low-VOC epoxy (code no. 11), water-based zinc-rich epoxy (code no. 13), and epoxy mastic (code no. 8). Furthermore, the epoxy mastic, the epoxy urethane mastics, and the low-VOC epoxy/acrylic modified epoxy developed raised rust at the scribes (figures 18, 19, and 20). These extremely severe undercuts strongly suggest that low-VOC epoxy coatings are not good candidates for steel bridges in a salt-rich and sulfate-rich environment.

#### **CYCLIC FREEZE/QUV/PROHESION TEST**

A freeze exposure and a QUV exposure were combined with the Prohesion exposure to better simulate natural weathering. Freezing induces mechanical stress as does temperature in winter, the QUV exposure simulates sunlight and the dew exposure at night. These cyclic stresses should accelerate coating degradation, hopefully in a manner more reproducible in the field.

The pencil hardness and the adhesion strength of the coating systems after 3,000 h of the cyclic freeze/QUV/Prohesion test are presented in table 7. The hardness of eight coating systems (code nos. 2, 4, 6 through 11) were decreased slightly by the cyclic freeze/QUV/Prohesion exposure, while the others remained the same. The increased number of coating systems with lower hardness after 3,000 h of the cyclic freeze/QUV/Prohesion test indicates that this regimen produces somewhat more stress to the coating systems than the other accelerated test exposures, which were the salt-fog and Prohesion exposures.

The changes in the film thickness and gloss after 3,000 h of the cyclic freeze/QUV/Prohesion testing are shown in figures 21 and 22, respectively. The film thickness of all the coating systems were reduced by the exposure; the small increase detected for code no. 5 after 3,000 h may not be significant. The gloss of all the coating systems declined after exposure, except the solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane system (code no. 7), which increased in gloss. A similar increase for the system was noted after the salt-fog exposure and the Prohesion exposure.

The failure results after 3,000 h of the cyclic freeze/QUV/Prohesion test are summarized in table 8.



Figure 15. Topcoat delamination of the waterborne vinyl system after 3,000 h of Prohesion exposure.



Figure 16. Condition of the waterborne acrylic epoxy system after 3,000 h of Prohesion exposure.

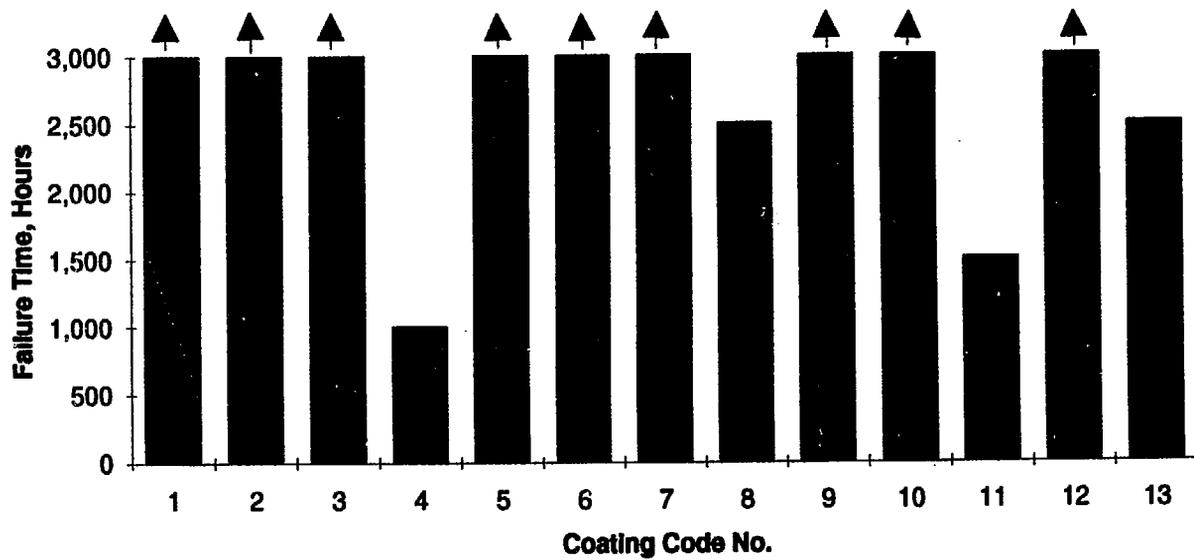


Figure 17. Failure time of the coating systems for developing 6.35 mm of creepage after 3,000 h of Prohesion exposure.

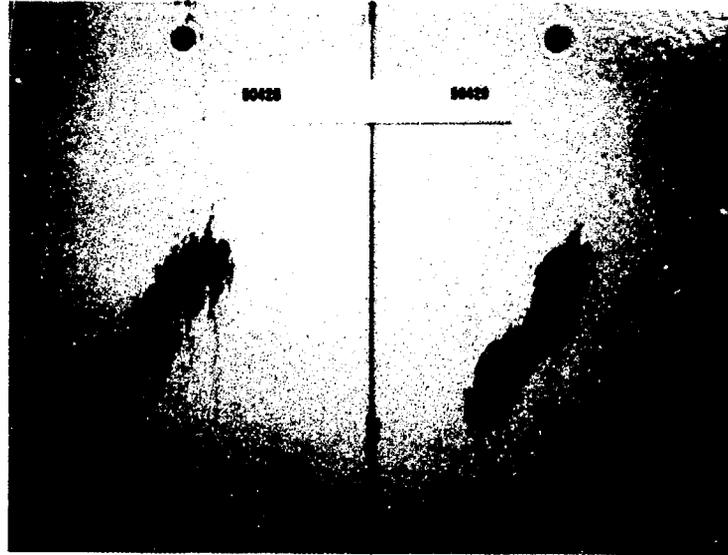


Figure 18. Condition of epoxy mastic/polyurethane system after 3,000 h of Prohesion exposure.

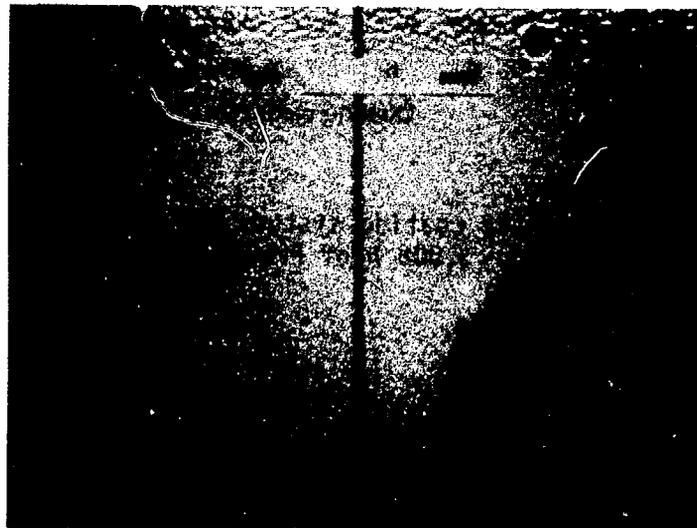
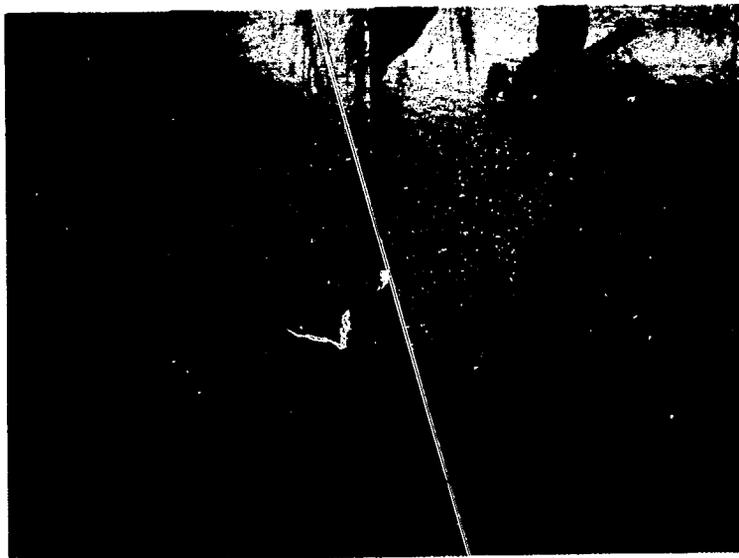


Figure 19. Condition of epoxy urethane mastic/polyurethane system after 3,000 h of Prohesion exposure.



**Figure 20. Condition of low-VOC epoxy/acrylic modified epoxy system after 3,000 h of Prohesion exposure.**

Table 7. Pencil hardness and adhesion strength after 3,000 h of freeze/QUV/Prohesion exposure.

<u>Code No.</u>	<u>Pencil Hardness (Before Exposure)</u>	<u>Adhesion Strength, MPa<sup>1</sup> (Before Exposure)</u>
1	<1-6B (<1-6B)	0 (0)
2	3-2B (4-HB)	5.7 (5.0)
3	3-2B (3-2B)	>7.0 (>7.0)
4	3-2B (4-HB)	5.5 (>7.0)
5	3-2B (3-2B)	>7.0 (>7.0)
6	2-4B (3-2B)	>7.0 (>7.0)
7	2-4B (4-HB)	>7.0 (>7.0)
8	3-2B (4-HB)	6.0 (>7.0)
9	2-4B (3-2B)	5.1 (4.1)
10	2-4B (3-2B)	4.5 (4.7)
11	3-2B (4-HB)	>7.0 (>7.0)
12	2-4B (2-4B)	3.2 (3.9)
13	2-4B (2-4B)	5.9 (>7.0)

<sup>1</sup> 1 MPa = 142.9 lbf/in<sup>2</sup>

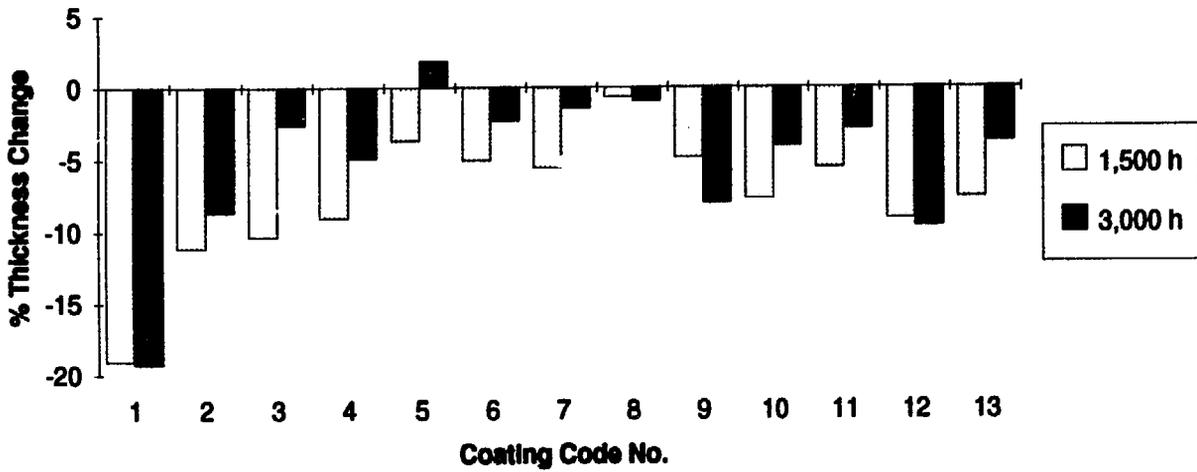


Figure 21. Film thickness change of the coating systems after 1,500 and 3,000 h of cyclic freeze/QUV/Prohesion exposure.

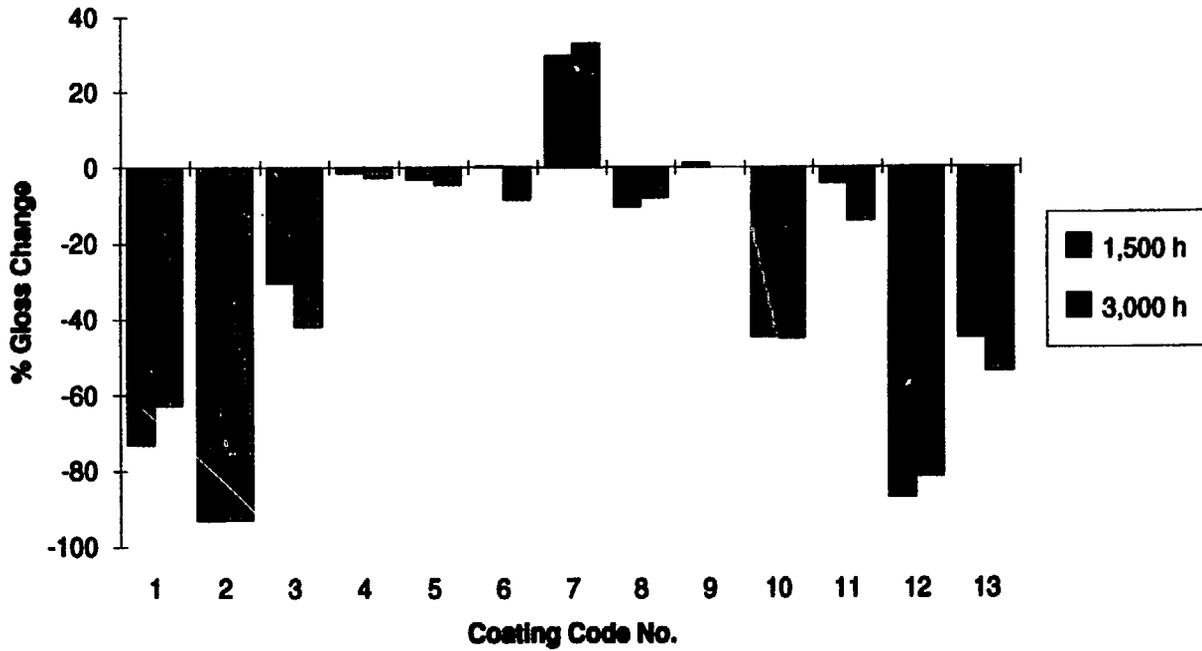


Figure 22. Gloss change of the coating systems after 1,500 and 3,000 h of cyclic freeze/QUV/Prohesion exposure.

Table 8. Results of cyclic freeze/QUV/Prohesion exposure.

Code	Hour	Panel Face		Others	Scribe		Creepage at Scribe, mm
		Blisters	Rust		Blisters	Rust	
1	3000	- <sup>2</sup>	Edge	TD <sup>3</sup>	-	SEV	0
	3000	-	Edge	TD <sup>3</sup>	-	SEV	0
2	3000	-	-	-	4M	MOD	3.3
	3000	-	-	-	4D	MOD	3.3
3	3000	-	-	-	6VD	SEV <sup>4</sup>	7.3
	3000	-	-	-	6VD	SEV <sup>4</sup>	5.0
4	3000	-	-	-	4VD	SEV <sup>4</sup>	6.5
	3000	-	-	-	2VD	SEV <sup>4</sup>	10.8
5	3000	-	-	-	-	MOD	0
	3000	-	-	-	-	MOD	0.4
6	3000	-	-	-	8M	SEV	1.8
	3000	-	-	-	8M	MOD	1.5
7	3000	9VF	Edge	-	8F	MOD	1.5
	3000	9VF	Edge	-	-	SL	0
8	3000	-	-	-	4D	SEV <sup>4</sup>	4.0
	3000	-	-	-	4D	SEV <sup>4</sup>	3.3
9	3000	-	-	-	4D	SEV <sup>4</sup>	3.1
	3000	-	-	-	4VD	SEV <sup>4</sup>	2.5
10	3000	1VF <sup>5</sup>	Edge	-	8F	MOD	1.5
	3000	1VF <sup>5</sup>	Edge	-	8F	MOD	1.8
11	3000	-	- <sup>6</sup>	-	4VD	SEV <sup>4</sup>	8.0
	3000	-	- <sup>7</sup>	-	4VD	SEV <sup>4</sup>	6.3
12	3000	4M	-	-	8MD	SEV	2.0
	3000	4M	-	-	6F	SEV	2.5
13	3000	-	Edge	-	8D	SL	4.0
	3000	-	Edge	-	8D	SL	4.3

<sup>1</sup> Method ASTM D714, Evaluating Degree of Blistering of Paints.

<sup>2</sup> None.

<sup>3</sup> Topcoat delamination.

<sup>4</sup> Raised rust.

<sup>5</sup> On edges.

<sup>6</sup> Three size 9 pits.

<sup>7</sup> Five size 9 pits.

SL Slight

MOD Moderate

SEV Severe Plane Surface Failure

## Plane Surface Failure

Topcoat delamination once again was noted for the solvent-based calcium sulfonate/alkyd system. Three other coating systems developed blistering on the surface. The waterborne vinyl system again exhibited uniform blistering of 4 MD, similar to the results of the salt-fog and the Prohesion tests. The solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane (VOC = 24 g/L) showed a few size 9 blisters on the surface. One large blister of size 2 was found at edge of the water-based inorganic zinc/acrylic/acrylic panel (figure 23). None of these three systems showed any underfilm corrosion. These results again elucidate topcoat delamination as a characteristic for some waterborne systems.

Only one system is strongly degraded by ultraviolet (UV) light. The solvent-based high-solids epoxy faded to a great extent after 3,000 h of the cyclic freeze/QUV/Prohesion exposure, demonstrating that epoxy is highly vulnerable to UV attack and needs to be topcoated.

## Scribe Failure

Among all the coating systems, the waterborne acrylic epoxy (figure 24), the low-VOC epoxy/acrylic modified epoxy (figure 25), and the waterborne acrylic (figure 26) exhibited the largest amount of creepage (6 to 8.6 mm) at the scribe. The solvent-based calcium sulfonate/alkyd and the solvent-based zinc-rich polyurethane/polyurethane/polyurethane (code no. 5, VOC = 336 g/L) showed no creepage, while the other coating systems all developed creepage in the range of 1.0 to 4.0 mm. Rust was developed at the scribe of the calcium sulfonate/alkyd system; it appears that the freeze cycle or QUV cycle has somehow altered the anticorrosive properties of this coating system. It is speculated that the strong affinity of calcium sulfate to steel substrate may have been weakened by the freezing cycle, thereby allowing some rust formation. In addition, the two epoxy mastic systems again formed raised rust at the scribe (figures 27 and 28).

The failure times for the creepage at the scribes for all the coating systems exposed to the cyclic freeze/QUV/Prohesion test are shown in figure 29. The plot shows clearly that the waterborne acrylic epoxy (code no. 4) failed rapidly in the test and the solvent-based low-VOC epoxy/acrylic modified epoxy (code no. 11) failed at the end of 3,000 h of exposure, whereas other coating systems did not fail after 3,000 h of exposure.

## FIELD EXPOSURE

The 28-month outdoor exposure results at the Sea Isle site are summarized in table 9.

## Plane Surface Failure

Only two of the coating systems showed plane surface failures after 28 months of outdoor exposure. The solvent-based calcium sulfonate/alkyd system exhibited topcoat peeling. The water-based inorganic zinc/acrylic/acrylic system developed topcoat delamination on edges and below scribe (figure 30).

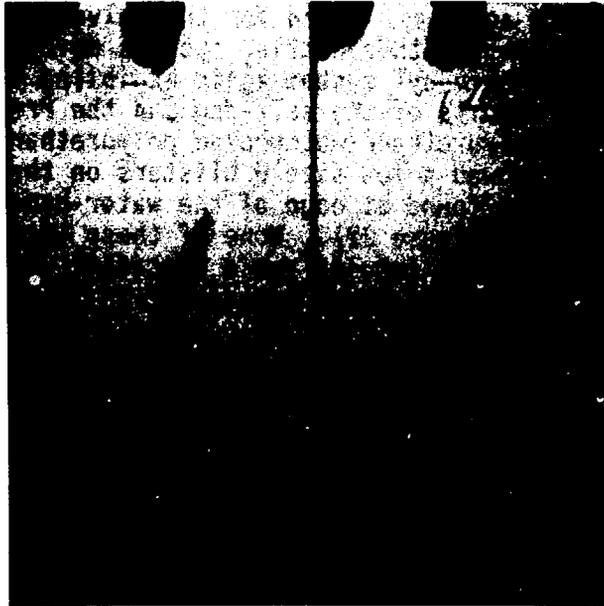


Figure 23. Condition of water-based inorganic zinc potassium silicate/acrylic/acrylic system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.

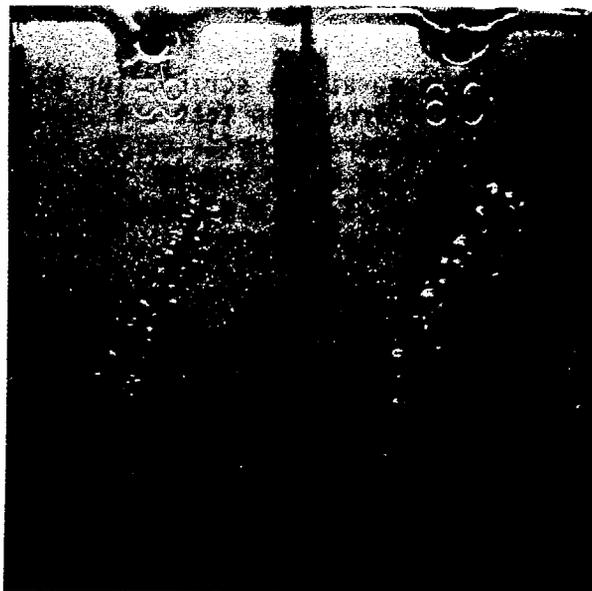
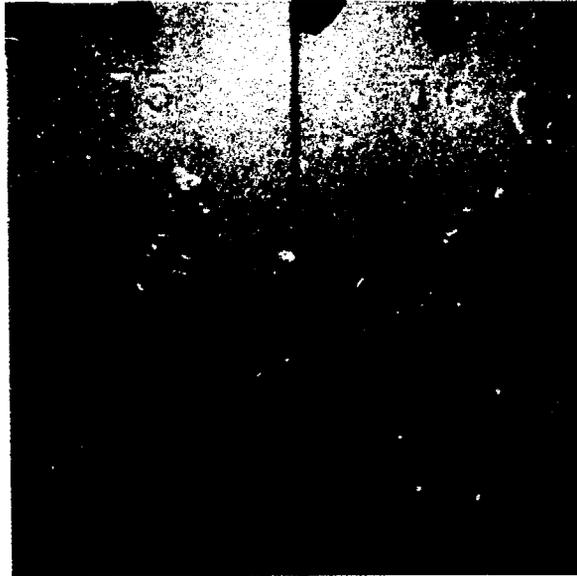
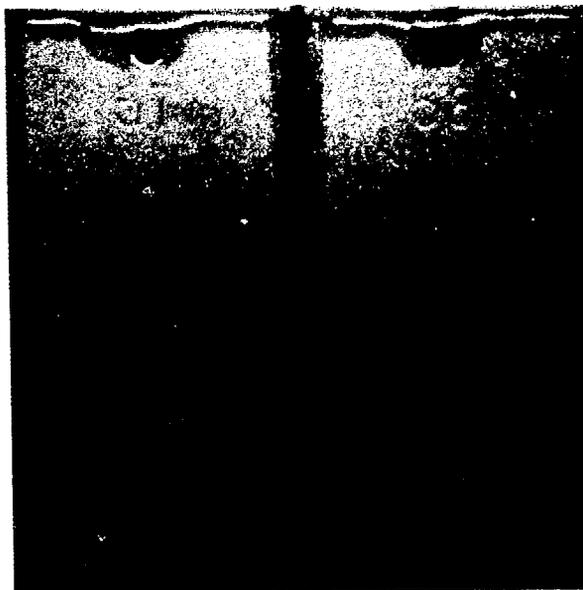


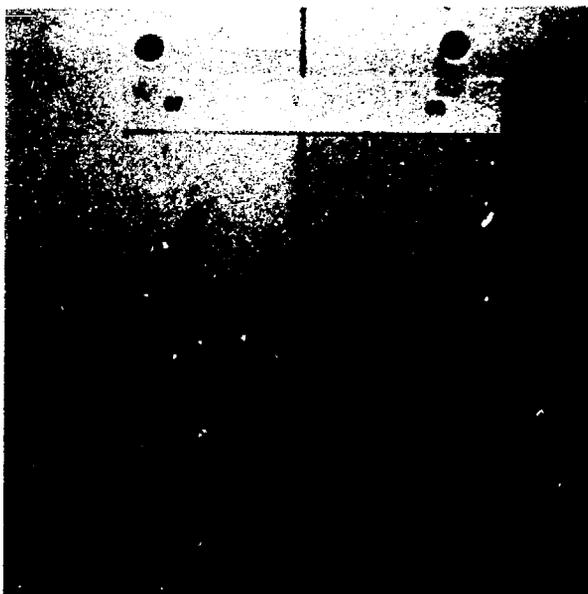
Figure 24. Condition of waterborne acrylic epoxy system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.



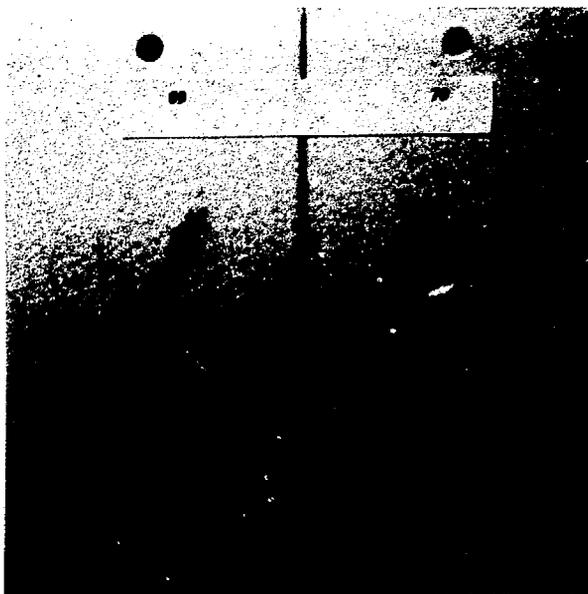
**Figure 25.** Condition of solvent-based low-VOC epoxy/acrylic modified epoxy system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.



**Figure 26.** Condition of waterborne acrylic system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.



**Figure 27. Condition of epoxy mastic/polyurethane system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.**



**Figure 28. Condition of epoxy urethane mastic/polyurethane system after 3,000 h of cyclic freeze/QUV/Prohesion exposure.**

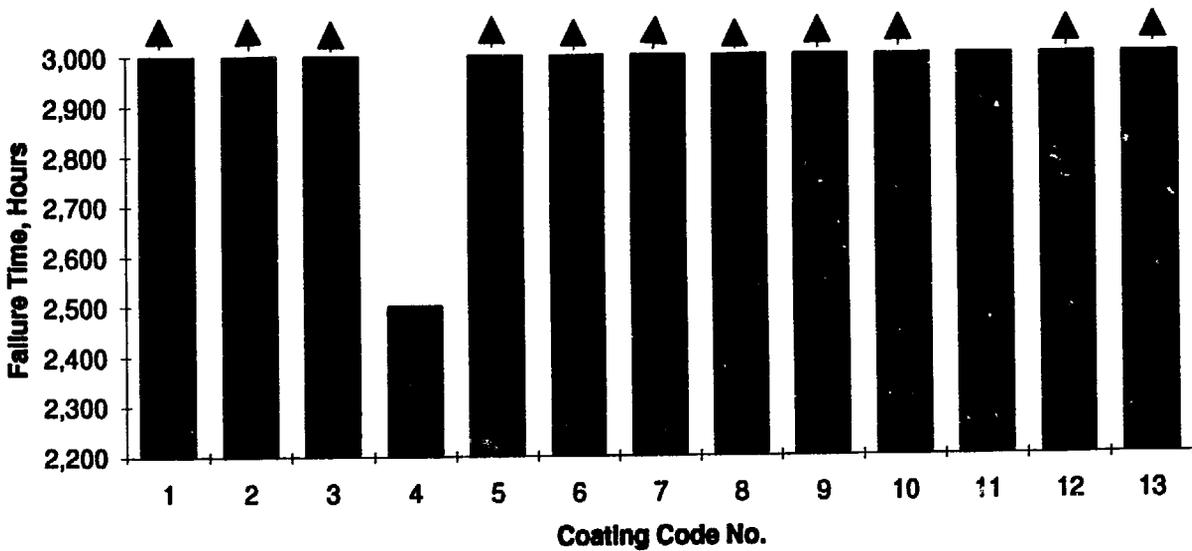


Figure 29. Failure time of the coating systems for developing 6.35 mm of creepage after cyclic freeze/QUV/Prohesion exposure.

Table 9. Results of the 28-month outdoor exposure.

Code No.	Blisters <sup>1</sup>	Plane		Scribe		Creepage at Scribe, mm
		Rust	Other	Blisters <sup>1</sup>	Rust	
1	-	-	TD <sup>2</sup>	-	-	0
1	-	-	TD <sup>2</sup>	-	-	0
2	-	-	-	4F	SEV	2.0
2	-	-	-	4M	SEV	7.5
3	-	-	-	6VD	SEV <sup>3</sup>	7.0
3	-	-	-	6VD	SEV <sup>3</sup>	7.0
4	-	-	-	4D	SEV <sup>3</sup>	9.0
4	-	-	-	4D	SEV <sup>3</sup>	8.0
5	-	-	-	-	SL	0
5	-	-	-	-	SL	0
6	-	-	-	-	SL	0
6	-	-	-	-	SL	0
7	8VF	-	-	-	MOD	0
7	8VF	- <sup>4</sup>	-	-	MOD	0
8	-	-	-	6MD	SEV	4.0
8	-	-	-	6MD	SEV	4.5
9	-	- <sup>5</sup>	-	6VD	SEV <sup>3</sup>	7.0
9	-	- <sup>6</sup>	-	6MD	SEV <sup>3</sup>	6.5
10	1VF	-	-	6MD	SEV	4.0
10	1VF	-	-	6MD	SEV	2.8
11	8VF	-	-	4VD	SEV <sup>3</sup>	11.5
11	-	-	-	4VD	SEV <sup>3</sup>	11.8
12	-	-	-	4VF	SEV	2.3
12	-	-	-	4VF	SEV	1.0
13	-	-	-	8MD	SEV	2.5
13	-	-	-	8MD	SEV	2.5

<sup>1</sup> Method ASTM D714, Evaluating Degree of Blistering of Paints.

<sup>2</sup> Topcoat delamination.

<sup>3</sup> Raised rust.

<sup>4</sup> Five size 8 pits.

<sup>5</sup> Two size 8 pits.

<sup>6</sup> Ten size 8 pits.

<sup>7</sup> Two size 6 pits.

SL Slight.

MOD Moderate.

SEV Severe.



**Figure 30. Condition of water-based inorganic zinc potassium silicate/acrylic/acrylic system after 28-month outdoor exposure.**

## Scribe Failure

The scribe creepage in millimeters for all the coating systems are plotted in figure 31. The low-VOC epoxy/acrylic modified epoxy showed the largest amount of scribe creepage (8.2 mm, figure 32). The waterborne acrylic and acrylic epoxy coating systems developed the next largest amount of scribe creepage (5.2 and 4.9 mm, respectively; figures 33 and 34). The epoxy mastic and epoxy urethane mastic systems exhibited moderate amount of creepage at the scribe (3.5 and 2.6 mm, respectively; figures 35 and 36). The high-solids epoxy (code no. 2), water-based inorganic zinc/acrylic/acrylic, and water-based zinc-rich epoxy/acrylic/acrylic had a very limited amount of creepage at the scribe. However, all three polyurethane systems (code nos. 5, 6, and 7) performed extremely well without any indication of scribe creepage or other failures.

## COMPARISON OF EXPOSURE METHOD AND COATING PERFORMANCE

The most pronounced difference in performance in each laboratory test as compared to an outdoor test are described as follows:

The salt-fog exposure showed extremely early failure for the waterborne acrylic system and the water-based inorganic zinc system, which both failed after 500 h. However, the latter only lost topcoat adhesion. Also, two epoxy mastic systems developed extensive underfilm corrosion in the salt-fog test that was not duplicated in the 28-month marine exposure. The Prohesion test after 1,000 h produced severe scribe failure for the waterborne acrylic epoxy system and the solvent-based low-VOC epoxy/acrylic modified epoxy. Over all, the cyclic freeze/QUV/Prohesion exposure appears to give the closest performance correlation to the outdoor exposure in terms of degree of creepage and the comparison of all the exposure results are shown in figure 37. The total exposure time of less than 3,000 h in the salt-fog test are marked above the data points. In general, the resemblance to the outdoor exposure is in the decreasing order of cyclic freeze/QUV/Prohesion > Prohesion > salt-fog. The salt-fog results showed an extremely different pattern as compared to the cyclic freeze/QUV/Prohesion and the outdoor exposure results.

A rating system for an overall performance was established for the candidate coating systems; it is a summation of ratings for surface failure (unscribed area) and scribe creepage (ASTM D1654) resulting in "20" as the best possible overall rating. In this rating system, "10" indicates perfect performance and "0" indicates total failure in each rating. Using this method, the rating results for all the laboratory tests and the 15-month as well as 28-month outdoor test are presented in tables 10 through 13. A rating for unscribed area evaluates both blistering and rusting on the plane and is only logical method to be used because not much rusting was found on most of these coating systems. Table 14 summarizes the ratings for all the coating systems in all the tests. Most of the laboratory tests were performed for 3,000 h, except some of the salt-fog tests as noted earlier. Undercutting was not included in this rating system since the panels at the outdoor marine site are intended for longer exposure.

An attempt was made to calculate the correlations of performance ratings between the outdoor exposure and the Prohesion test or the cyclic freeze/QUV/Prohesion exposure. The best fit by least squares method produced the following correlation coefficients as shown in table 15. The correlation

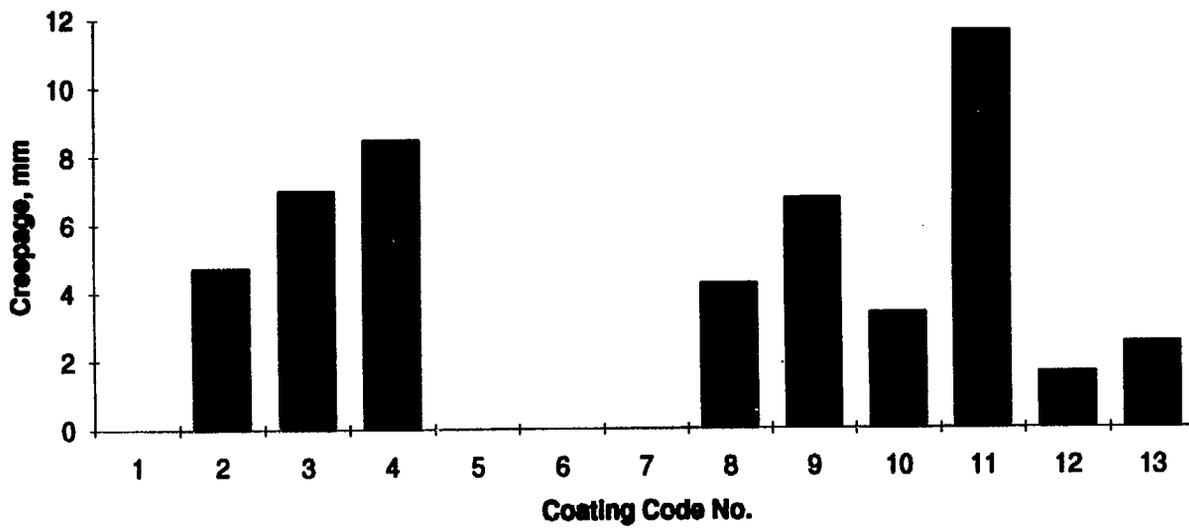
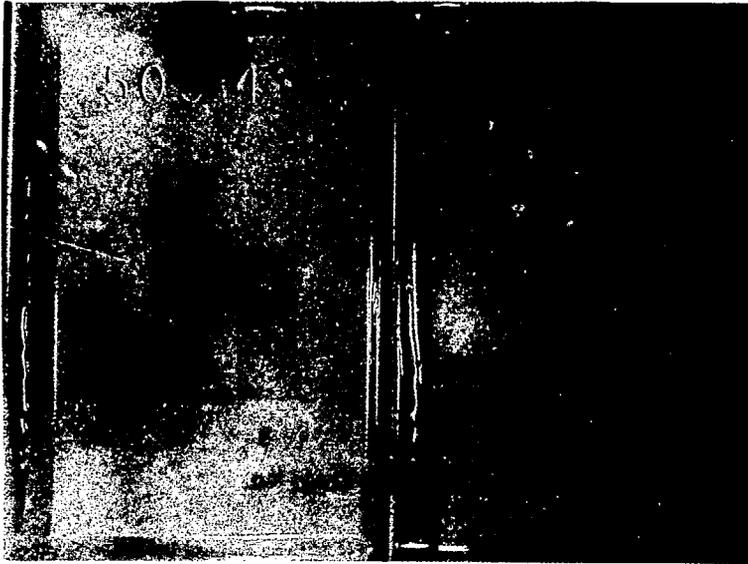


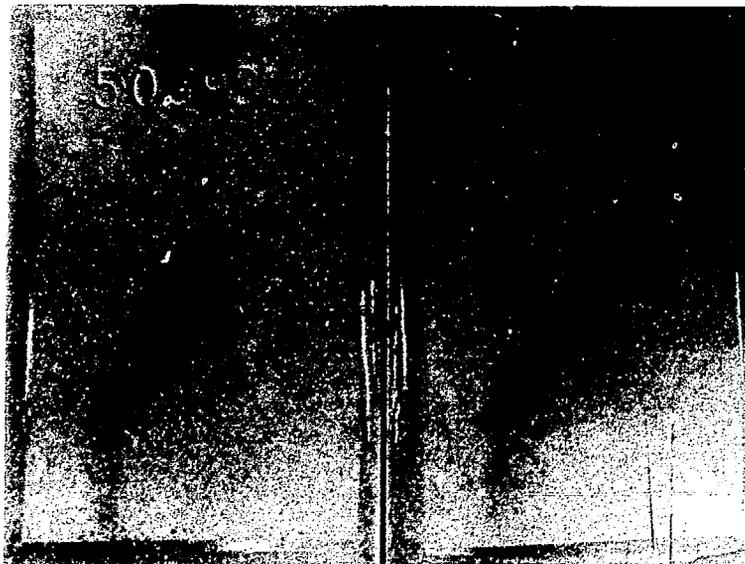
Figure 31. Creepage of the coating systems after 28-month outdoor marine exposure at Sea Isle, New Jersey.



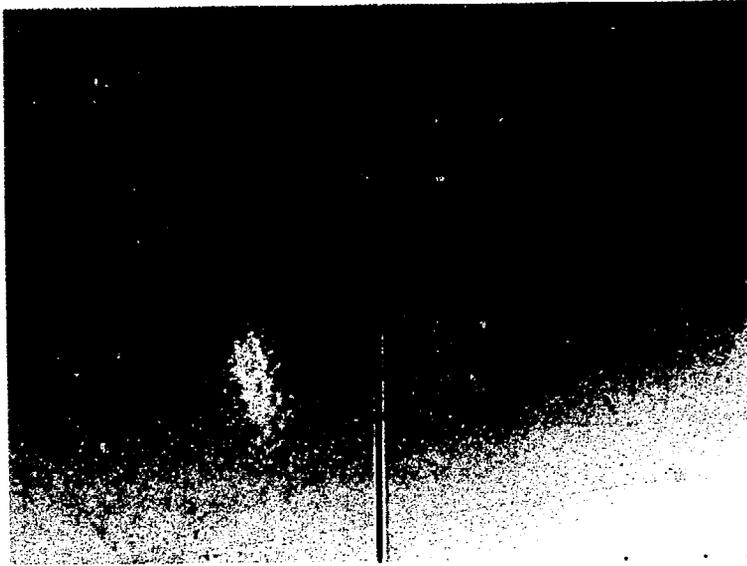
Figure 32. Condition of solvent-based low-VOC epoxy/acrylic modified epoxy system after 28-month outdoor exposure.



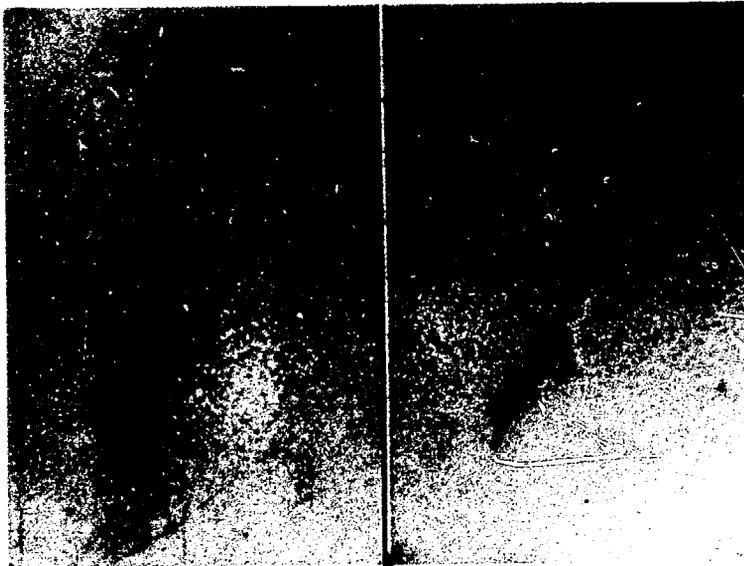
**Figure 33. Condition of waterborne acrylic system after 28-month outdoor exposure.**



**Figure 34. Condition of waterborne acrylic epoxy system after 28-month outdoor exposure.**



**Figure 35. Condition of epoxy mastic/polyurethane system after 28-month outdoor exposure.**



**Figure 36. Condition of epoxy urethane mastic/polyurethane system after 28-month outdoor exposure.**

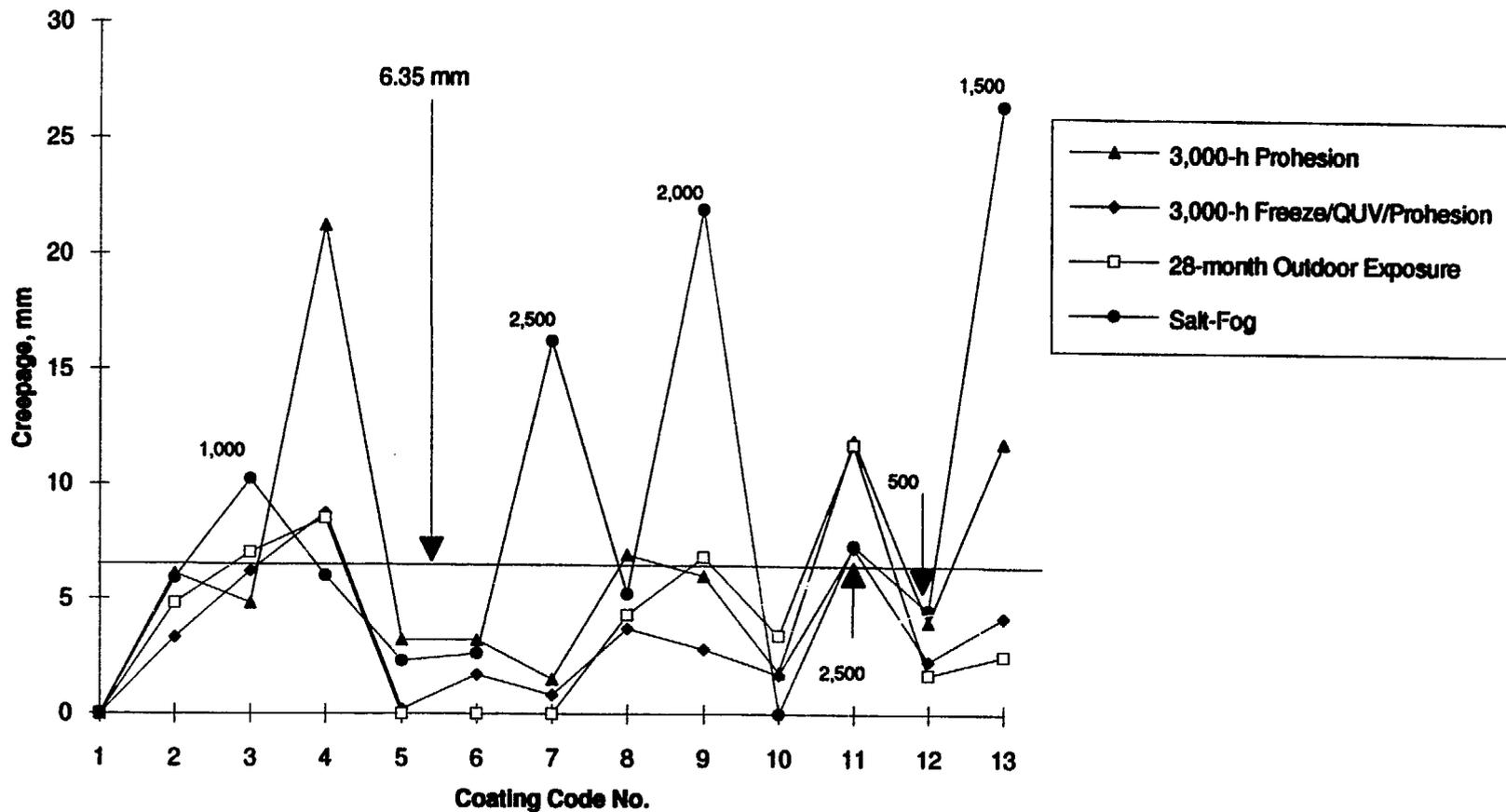


Figure 37. Comparison of creepage for salt-fog exposure, Prohesion exposure, cyclic freeze/QUV/Prohesion exposure, and 28-month outdoor marine exposure.

Table 10. Rating for salt-fog exposure.

<u>Code No.</u>	<u>Unscribed Area</u>	<u>Scribe</u>	<u>Overall</u>
1	0	10	10
2	10	4	14
3	10	2	12
4	10	4	14
5	10	6	16
6	10	6	16
7	9	0	9
8	8	4	12
9	6	0	6
10	6	10	16
11	10	3	13
12	5	5	10
13	9	0	9

Table 11. Rating for Prohesion exposure.

<u>Code No.</u>	<u>Unscribed Area</u>	<u>Scribe</u>	<u>Overall</u>
1	0	10	10
2	10	4	14
3	10	5	15
4	10	0	10
5	10	5	15
6	10	5	15
7	9	6	15
8	10	4	14
9	10	4	14
10	8	7	15
11	10	2	12
12	2	5	7
13	10	2	12

Table 12. Rating for freeze/QUV/Prohesion exposure.

<u>Code No.</u>	<u>Unscribed Area</u>	<u>Scribe</u>	<u>Overall</u>
1	0	10	10
2	10	5	15
3	10	4	14
4	10	3	13
5	10	9	19
6	10	7	17
7	9	8	17
8	10	5	15
9	10	6	16
10	10	7	17
11	10	3	13
12	5	6	11
13	10	5	15

Table 13. Rating for outdoor exposure.

<u>Code No.</u>	<u>Unscribed Area</u>	<u>Scribe</u>	<u>Overall</u>
1	0 <sup>1</sup> , 0 <sup>2</sup>	10, 10	10, 10
2	10, 10	9, 5	19, 15
3	10, 10	4, 4	14, 14
4	10, 10	5, 3	15, 13
5	10, 10	10, 10	20, 20
6	10, 10	10, 10	20, 20
7	9, 9	10, 10	19, 19
8	10, 9	5, 5	15, 14
9	9, 9	6, 4	15, 13
10	9, 8	7, 5	16, 13
11	10, 9	2, 2	12, 11
12	10, 10	10, 7	20, 17
13	10, 10	8, 6	18, 16

<sup>1</sup> 15-month exposure.

<sup>2</sup> 28-month exposure.

Table 14. Comparison of ratings in various exposures.

<u>Code No.</u>	<u>Salt-Fog</u> <u>3,000 h</u>	<u>Prohesion</u> <u>3,000 h</u>	<u>FQP<sup>1</sup></u> <u>3,000 h</u>	<u>Outdoor</u> <u>15 Months</u>	<u>Outdoor</u> <u>28 Months</u>
1	10 <sup>2</sup>	10	10	10	10
2	14	14	15	19	15
3	12 <sup>3</sup>	15	14	14	14
4	10	13	13	15	13
5	16	15	19	20	20
6	16	15	17	20	20
7	9 <sup>4</sup>	15	17	19	19
8	12	14	15	15	14
9	6 <sup>5</sup>	14	16	15	13
10	16 <sup>2</sup>	15	17	16	13
11	13 <sup>4</sup>	12	13	12	11
12	10 <sup>2</sup>	7	11	20	17
13	9 <sup>6</sup>	12	15	18	16

<sup>1</sup> Cyclic freeze/QUV/Prohesion test.

<sup>2</sup> 500 h.

<sup>3</sup> 1,000 h.

<sup>4</sup> 2,500 h.

<sup>5</sup> 2,000 h.

<sup>6</sup> 1,500 h.

Table 15. Correlation coefficients for performance ratings between laboratory test and outdoor exposure.

	<u>Prohesion<sup>1</sup></u>	<u>Cyclic freeze/ QUV/Prohesion<sup>1</sup></u>	<u>Salt-fog<sup>1</sup></u>
a. For 13 coating systems			
15-mo outdoor exposure	0.14	0.55	
28-mo outdoor exposure	0.27	0.62	
b. For 12 coating systems <sup>2</sup>			
15-mo outdoor exposure	0.64	0.81	
28-mo outdoor exposure	0.65	0.80	
c. For 8 coating systems <sup>3</sup>			
28-mo outdoor exposure	0.91	0.88	0.20

<sup>1</sup> Prohesion and cyclic freeze/QUV/Prohesion run for 3,000 h for tests a and b. Prohesion, cyclic freeze/QUV/Prohesion, and salt-fog run for 2,000 h for test c.

<sup>2</sup> Excluding the waterborne vinyl system..

<sup>3</sup> Code nos. 2, 4-9, and 11.

coefficients of 0.55 and 0.62 clearly suggest that there is a closer agreement between the cyclic freeze/QUV/Prohesion test exposure and the 15-mo and the 28-mo outdoor exposure than between the other accelerated test regimens. Another correlation was calculated for all the coating systems except the waterborne vinyl system that exhibited severe blistering in all three laboratory tests, but showed no surface failure after the 28-mo outdoor exposure. The exclusion of the waterborne vinyl system (code no. 12) in the linear regression analysis has tremendously improved the correlations between the laboratory test results and the outdoor exposure results. The improved correlation coefficients, 0.81 and 0.80 (see table 15), obtained for the relationship between the cyclic freeze/QUV/Prohesion test and the 15-mo and the 28-mo outdoor exposures, respectively, suggest that the cyclic freeze/QUV/Prohesion test produced closer failure results to the natural marine exposure results as compared to the Prohesion test. It is understandable that the performance of the three-coat waterborne vinyl system with a minimal solvent content (VOC = 2/2/64 g/L) showed a large discrepancy between laboratory tests and natural marine exposure. This waterborne coating material with high hydrophilic character easily absorbs water and does not allow sufficient time for water to diffuse out under the experimental conditions employed in the accelerated testers as compared to presumably less humid and longer drying cycles in the natural environment.

The correlation between the salt-fog test and the outdoor exposure could not be obtained for all 13 coating systems due to some early terminations of the salt-fog test. However, a correlation was attempted between the 2,000 h of salt-fog test results and the 28-month outdoor exposure results for eight coating systems (code nos. 2, 4 through 9, and 11) that had complete data points; a correlation coefficient was found to be 0.20. This extremely low value strongly suggests that using the salt-fog test result to predict field performance is inappropriate.

A computation of all the test results suggests that the solvent-based polyurethane organic zinc-rich primer with acrylic aliphatic polyurethane as intermediate coat and topcoat (code nos. 5 and 6) performed the best. The water-based inorganic zinc potassium silicate/acrylic/acrylic, solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane, and water-based zinc-rich epoxy/acrylic/acrylic also performed fairly well, but developed topcoat delamination. The high-solids epoxy showed reasonable corrosion resistance against all severe environments used in this study, but was susceptible to UV attack. The low-VOC epoxy/acrylic modified epoxy, epoxy mastic/polyurethane, epoxy urethane mastic/polyurethane, waterborne acrylic, and waterborne acrylic epoxy systems performed poorly in all the cases, with severe raised rust developed at the scribe for two epoxy mastics. The calcium sulfonate/alkyd system exhibited total topcoat delamination in all exposures.

The waterborne vinyl systems blistered very badly in all three accelerated tests, but showed no signs of failure after 28 months of outdoor exposure. One possible explanation for the difference in results is that the dry cycle in the natural outdoor exposure may be much longer than in the Prohesion test (cyclic 1-h wet/1-h dry). As noted in the previous discussion of results, dry time in the exposure cycle seems beneficial for healing the blisters formed on the vinyl system. The correlation analysis of the waterborne vinyl system between the accelerated testing and outdoor exposure results will be performed again as the coating panels are exposed further to the natural marine environment.

## STATISTICAL ANALYSIS

A statistical analysis was carried out to study the variation of results among the three laboratory test methods employed in this work. The creepages at the scribe were used for the analysis because they are more accurate than the percentage of surface failures in terms of measurements. Eight coating systems (code nos. 2, 4-9, and 11) contained a complete set of scribe creepage results from 500 to 2,000 h for all three test methods shown in tables 16 through 18; these data were evaluated by an analysis of variance procedures shown in table 19.<sup>(14)</sup> The experiment was conducted as a 2-way factorial design in which one of the factors is method of testing (3) and the other factor is type of coating (8) with 8 measurements for each of the 24 combinations.

The statistical results of extremely low probabilities (0.09 and 0.06) of obtaining the reported F-ratio values in table 19 reveals that all three tests and coatings have statistically significant differences at the 10-percent level. In other words, different laboratory exposure methods generated different amounts of creepage at the scribe as did different coating systems. In fact, the actual difference is much bigger than that presented here because the extremely severe creepages developed for the waterborne acrylic (code no. 3) and the water-based zinc-rich epoxy/acrylic/acrylic (code no. 13) and the creepage of the waterborne vinyl system were not included in the analysis due to their earlier termination of exposure (see table 4).

The differences in methods and coating systems can also be seen in the plot of averages for scribe creepage using three methods (figure 38). To distinguish the degree of failure by each test method, the mean creepage at the scribes at exposure times of 0, 500, 1,000, 1,500, and 2,000 h is plotted in figure 39. The extent of creepage for the salt-fog test and the Prohesion test are similar up to 1,500 h; above 1,500 h, the salt-fog test caused larger creepage than did the Prohesion test. The cyclic freeze/QUV/Prohesion test produced the least amount of scribe creepage among all three test methods due to its less severe environment.

## EFFECT OF VOC CONTENT

The effect of VOC content on the coating performance can be clearly investigated by comparing the scribe creepage of code nos. 5 through 7, which are the polyurethane systems with topcoats containing different solvent content. In the plot of creepage versus exposure time for the salt-fog test (figure 40(a)), the waterborne topcoat system with a VOC of 24 g/L performed the poorest and failed very rapidly after 1,000 h. However, after 1,000 h of exposure, two solvent-based polyurethane systems (code nos. 5 and 6) still showed similar good performance. The coating system with the highest VOC content (VOC = 336 g/L) had the best performance as compared to two lower VOC systems (VOC = 250 and 24 g/L) in the cyclic freeze/QUV/Prohesion test (figure 40(c)). On the contrary, all three systems showed no visible difference in performance in the Prohesion test (figure 40(b)). In general, the performance of the three-coat polyurethane system decreases with decreasing VOC content of the topcoat.

Table 16. Scribe creepage after salt-fog exposure.

<u>Code No.</u>	<u>Panel No.</u>	<u>Scribe Creepage in mm After Various Exposure Times</u>			
		<u>500 h</u>	<u>1,000 h</u>	<u>1,500 h</u>	<u>2,000 h</u>
2	1	1.8	2.9	3.5	3.6
	2	0.9	3.0	3.0	6.0
4	1	2.0	3.0	3.7	4.9
	2	1.4	2.8	4.0	4.4
5	1	0.4	0.8	0.8	1.0
	2	0.5	1.3	1.8	2.5
6	1	0.5	1.6	1.8	1.9
	2	0.5	1.4	2.0	2.6
7	1	1.0	1.3	3.5	13.8
	2	0.5	1.5	3.5	15.3
8	1	2.0	3.3	4.1	4.4
	2	1.6	2.9	4.6	5.0
9	1	1.5	7.0	10.8	19.8
	2	1.9	8.5	13.0	24.0
11	1	1.6	2.3	4.4	6.5
	2	1.8	2.6	4.5	4.5

Table 17. Scribe creepage after Prohesion exposure.

<u>Code No.</u>	<u>Panel No.</u>	<u>Scribe Creepage in mm after Various Exposure Times</u>			
		<u>500 h</u>	<u>1,000 h</u>	<u>1,500 h</u>	<u>2,000 h</u>
2	1	4.1	4.5	6.0	6.9
	2	1.8	1.8	2.3	2.3
4	1	4.3	8.9	11.5	16.8
	2	4.1	8.1	11.8	12.3
5	1	0	0	1.3	1.8
	2	0	0	1.0	1.8
6	1	0	0	2.8	2.9
	2	0	0	0	1.3
7	1	0	0	0.5	1.3
	2	0	0	1.4	1.8
8	1	2.0	3.0	4.9	5.5
	2	1.3	3.5	3.5	4.1
9	1	1.5	3.0	4.0	4.8
	2	1.8	2.9	3.3	3.9
11	1	3.0	4.9	7.3	9.0
	2	3.8	6.4	7.8	9.8

Table 18. Scribe creepage after cyclic freeze/QUV/Prohesion exposure.

Code No.	Panel No.	Scribe Creepage in mm After Various Exposure Times			
		500 h	1,000 h	1,500 h	2,000 h
2	1	0	0	0	2.0
	2	0	0	2.5	2.5
4	1	3.5	4.8	5.3	6.0
	2	4.0	4.5	6.0	6.3
5	1	0	0	0	0
	2	0	0	0	0
6	1	0	0	0	0.3
	2	0	0	0	0.8
7	1	0	0	0	0
	2	0	0	0	0
8	1	0	1.5	1.5	2.5
	2	0	1.5	1.5	1.8
9	1	1.0	1.8	2.3	2.8
	2	1.0	1.3	1.5	1.6
11	1	1.0	3.0	4.5	5.0
	2	1.0	2.8	3.8	5.0

Table 19. Analysis of variance: salt-fog, Prohesion, and cyclic freeze/QUV/Prohesion exposures for scribe creepage.

Component	Sum of Squares	df	Mean Square	F-ratio	P-value
Test	223.757	2	111.878	2.323	0.09
Coating	666.972	7	95.282	1.978	0.06
Residual	1633.019	182	48.170		
<b>Total</b>	<b>2523.748</b>	<b>191</b>			

df = Degree of freedom.

F-ratio = Fisher F-ratio.

P-value = Probability of obtaining a reported F-ratio value.

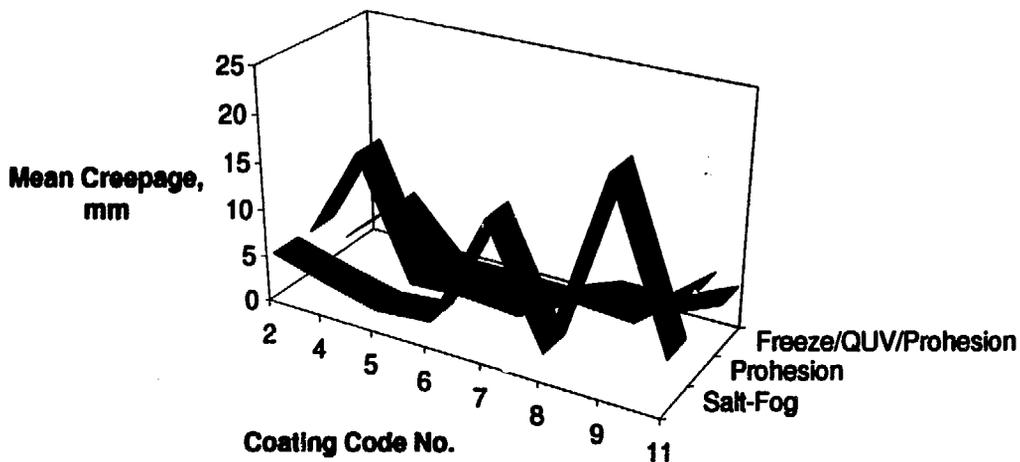


Figure 38. Plot of mean creepage of eight coating systems vs. exposure time for salt-fog test, Prohesion test, and cyclic freeze/QUV/Prohesion test.

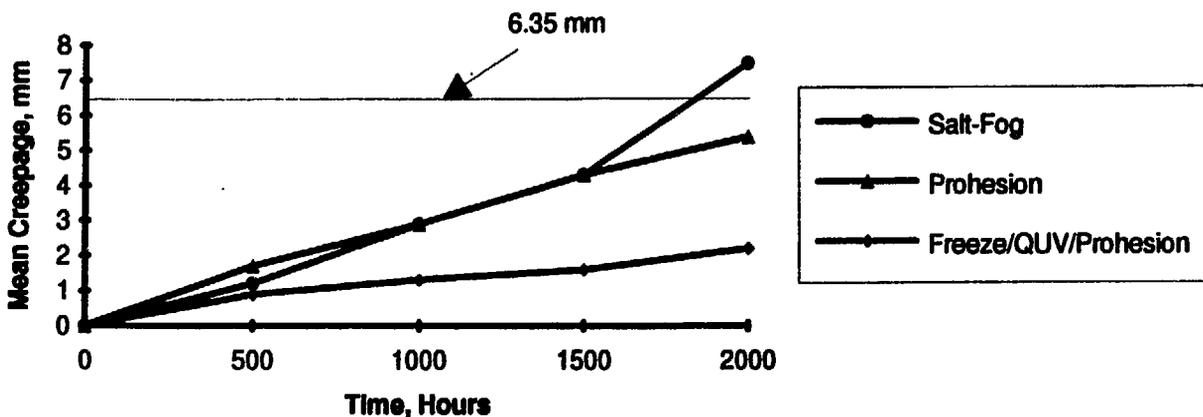


Figure 39. Mean creepage of exposure time of 500, 1,000, 1,500, and 2,000 h for eight coating systems after salt-fog test, Prohesion test, and cyclic freeze/QUV/Prohesion test.

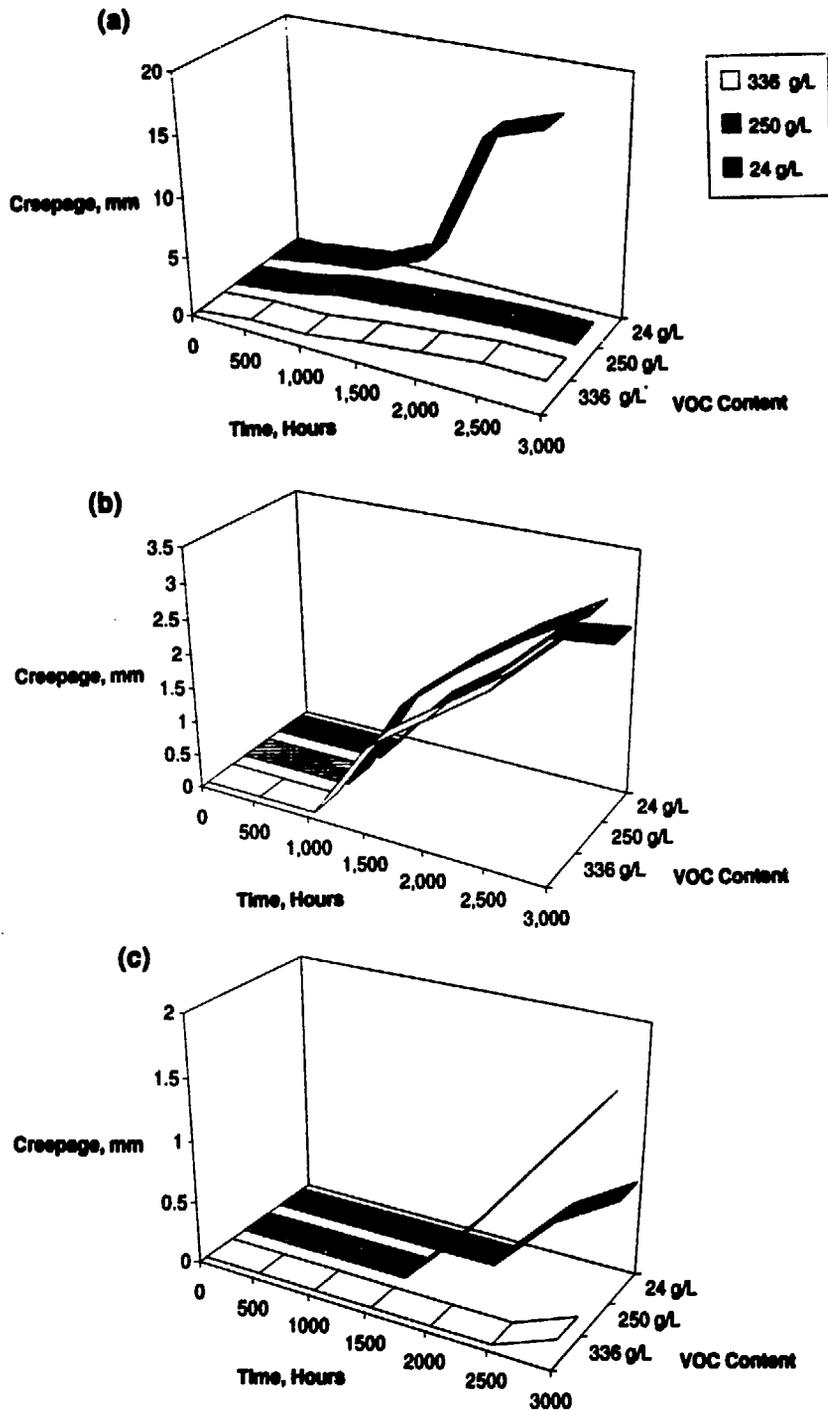


Figure 40. Creepage variation of different VOC content of polyurethane topcoat applied on solvent-based zinc-rich polyurethane: (a) salt-fog exposure, (b) Prohesion exposure, and (c) cyclic freeze/QUV/Prohesion exposure.

## **PANEL UNIFORMITY**

The standard deviations of the film thickness, gloss, and adhesion strength before testing are shown in table 20. These results show that all three sets of panels used in the different tests were very similar. Examinations of panel uniformity were made for each laboratory test using the same statistical variance method. These analysis results are presented in tables 21 through 23. They showed that all the duplicate panels used in the salt-fog test and in the cyclic freeze/QUV/Prohesion test were fairly similar because P-values were 0.660 and 0.550, respectively. However, the duplicate panels in the Prohesion test showed some variations due to the low P-value (0.180).

## **DATA RELIABILITY**

The standard deviations of all the measurements made in this study are shown below (tables 24 and 25) to indicate the significance of any changes. It should be noted here that the values used in the calculation for adhesion strength were limited to those equal to, or below, 7.0 MPa (1,000 lbf/in<sup>2</sup>).

The standard deviations for the film thickness and coating gloss of all the coating systems evaluated are very similar for all three laboratory tests indicating that the experimental variables for these tests were well-controlled. The lack of consistency in standard deviations for the adhesion strength may be due to the testing method and variation on the steel surface, such as profile, cleanness, and the curing degree of coating.

Table 20. Standard deviations of physical properties before exposure.

<u>Test</u>	<u>Film Thickness, mil</u>	<u>60° Gloss</u>
Salt-Fog	0.70	5.10
Prohesion	0.86	4.31
Freeze/QUV/ Prohesion	0.69	4.87

Table 21. Analysis of variance: duplicate panels for salt-fog exposure.

<u>Source</u>	<u>Sum of Squares</u>	<u>df</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>P-value</u>
Panel	2.310	1	2.310	0.168	0.660
Coating	508.438	7	72.634	5.270	<0.001
Residual	758.033	55	13.782		
<b>Total</b>	<b>1268.781</b>	<b>63</b>			

Table 22. Analysis of variance: duplicate panels for Prohesion exposure.

<u>Source</u>	<u>Sum of Squares</u>	<u>df</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>P-value</u>
Panel	7.840	1	7.840	1.842	0.18
Coating	566.155	7	80.879	19.004	<0.001
Residual	234.085	55	4.256		
<b>Total</b>	<b>808.079</b>	<b>63</b>			

Table 23. Analysis of variance: duplicate panels for cyclic freeze/QUV/Prohesion exposure.

<u>Source</u>	<u>Sum of Squares</u>	<u>df</u>	<u>Mean Squares</u>	<u>F-ratio</u>	<u>P-value</u>
Panel	0.006	1	0.006	0.0074	0.550
Coating	180.144	7	25.739	31.737	<0.001
Residual	44.614	55	0.811		
<b>Total</b>	<b>224.764</b>	<b>63</b>			

Table 24. Standard deviations<sup>1</sup> of physical properties after exposure.

<u>Test</u>	<u>Film Thickness</u> <u>mm x 100 (mil)</u>	<u>60° Gloss</u>	<u>Adhesion</u> <u>Strength, MPa</u>
Salt-fog	1.8 (0.7)	4.06	0.47
Prohesion	2.0 (0.8)	3.99	0.12
Cyclic Freeze/ QUV/Prohesion	2.0 (0.8)	5.10	0.61

<sup>1</sup> Duplicate Difference Method.

Table 25. Standard deviations for creepages after exposure.

<u>Test</u>	<u>Standard Deviation</u> <u>mm</u>
Salt-fog	2.20
Prohesion	1.72
Cyclic freeze/QUV/Prohesion	0.72
Outdoor	0.44

#### 4. SUMMARY AND CONCLUSIONS

- The cyclic freeze/QUV/Prohesion accelerated test evaluated in this study generated a failure trend closest to the 28-month outdoor exposure results when compared to the salt-fog test and the Prohesion test.
- The statistical analysis showed large differences between the coating systems and between the testing methods.
- In general, all the coating film thickness decreased after the Prohesion and cyclic freeze/QUV/Prohesion exposure, whereas the film thickness increased after the salt-fog exposure.
- Virtually all coating gloss decreased after all laboratory exposure.
- Among the 13 coating systems, the solvent-based zinc-rich polyurethane/polyurethane/polyurethane (VOC = 336 g/L) performed the best. In general, the performance of three zinc-rich polyurethane systems are fairly similar except that the lowest-VOC coating system with the waterborne topcoat (VOC = 24 g/L) exhibited severe topcoat blistering at the scribe without the undercut.
- The zinc-rich primers with water-based topcoats did not undercut or rust at the scribe, but exhibited topcoat blisters at the panel surface. These systems include the water-based inorganic zinc/acrylic/acrylic, the water-based zinc-rich epoxy/acrylic/acrylic, and the solvent-based zinc-rich polyurethane/waterborne polyurethane/waterborne polyurethane. In conclusion, the majority of the water-based topcoats tested showed a tendency to blister regardless of whether the zinc-rich primer is solvent or water-based. The results verified that the zinc-rich primers protected steel from rusting and undercutting even though topcoat blistering occurred.
- The waterborne vinyl systems failed badly in all three laboratory tests, but have not showed any sign of failure after the 28-month outdoor exposure.
- The epoxy mastic systems performed very poorly with extensive underfilm corrosion after the salt-fog exposure and they developed raised rust at the scribe after all three laboratory tests. The solvent-based high-solid epoxy system was fairly corrosion-resistant, but was prone to UV attack. However, the solvent-based low-VOC epoxy/acrylic modified epoxy system performed the worst and developed severe undercutting.
- Both the waterborne acrylic system and the waterborne acrylic epoxy system did not perform very well and exhibited severe scribe creepage.
- The calcium sulfonate/alkyd system did not develop undercutting, but had extremely low abrasion resistance and experienced extensive topcoat delamination; therefore, it is not suitable for exposed areas.

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