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It is widely accepted that improved **methods for assessing the corrosion controlling properties of organic coatings on steel are** needed. Difficulties with established testing procedures (primarily ASTM B 117 salt spray) have been well documented[1-3] and need not be restated here. As pointed out in a recent review,[4] the need for reliable short-term testing procedures is greater than ever considering the extensive formulation changes in established coating systems that are expected to result from increasingly stringent volatile organic content (VOC) regulations. More realistic testing procedures will allow meaningful performance comparisons between old and new VOC-compliant compositions without reliance on long-term exterior exposure results.

The most crucial requirements of a "good" or meaningful laboratory test are that it simulates the relative performance rankings of materials observed in practice and that it produces failure modes consistent with field experience. Additionally, a useful test must be reproducible and reasonably rapid. No currently available tests have been shown to meet these requirements.[5]

Although it would not seem practical (or even possible) to simulate, completely, the numerous and complex variables operating in the outdoor environment, a reasonable approach would

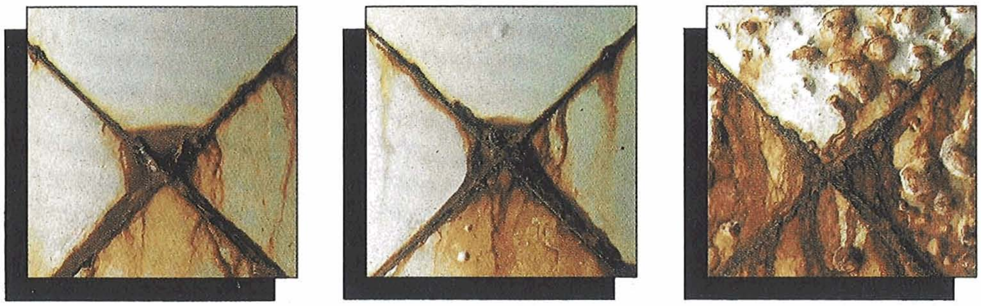

(a) epoxy/epoxy
(b) alkyd/alkyd
(c) latex/latex

Fig. 1 *Scribed regions of panels after 2,000 hours' salt spray testing (1,000 hours for latex)*

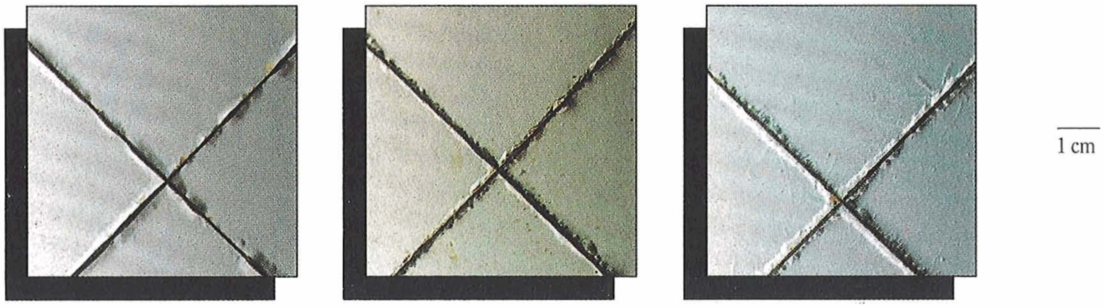

(a) epoxy/epoxy
(b) alkyd/alkyd
(c) latex/latex

Fig. 2 *Scribed regions of panels after cyclic wet/dry corrosion testing (2,000 hours)*

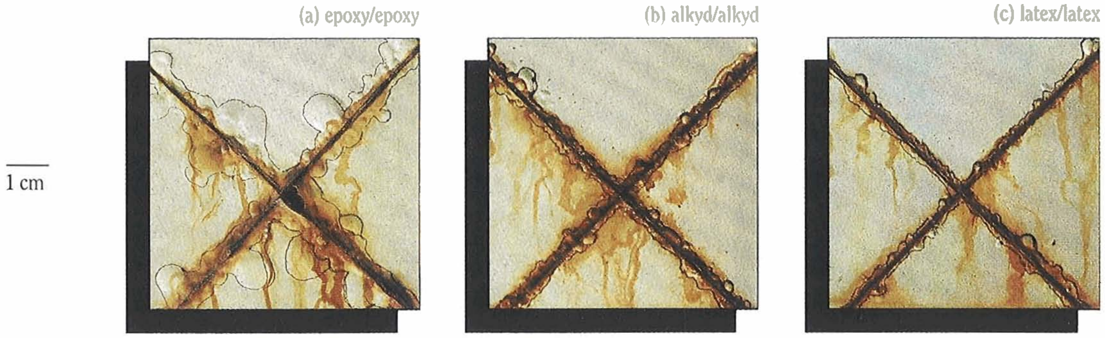
involve identifying the most significant of these variables and incorporating them into an accelerated testing protocol. In recent years, claims of improved correlations with exterior results have been reported based on the use of cyclic wet/dry corrosion chambers[6,7,8] (i.e, chambers producing periods of salt fog alternating with periods when test samples are allowed to dry). This cyclic wetting and drying of electrolyte layers from the panel surface is thought to stress the coating in a more realistic manner than, for example, a continuous ASTM B 117 salt spray test, where panels are placed in a constant, high relative humidity (RH) environment (~97 percent). At the very least, incorporation of wet/dry cycling factors seems intuitively justified, considering that materials exposed to the outdoor environment undergo similar wetting and drying effects on a frequent basis. The importance of electrolyte composition has also been addressed (e.g., the incorporation of ammonium and sulphate species to simulate corrosion processes occurring in industrial environments[1,6]).

Although there has been increased recognition of the importance of wet/dry cycling (or cyclic stress factors in general[9]) and electrolyte composition variables, little attention has been given to the influence of weathering factors (e.g., ultraviolet light exposure, moisture condensation) in the overall paint degradation and corrosion process. Because corrosion at the paint/metal interface and weathering of paint films are processes occurring simultaneously in nature, it does not seem unreasonable to suggest that the 2 processes may be significantly interrelated. For example, weather-induced degradation of a paint's organic binder may result in a more hydrophilic coating surface, which could change the time-of-wetness and subsequent corrosion characteristics of the system.

Other effects may include the following:

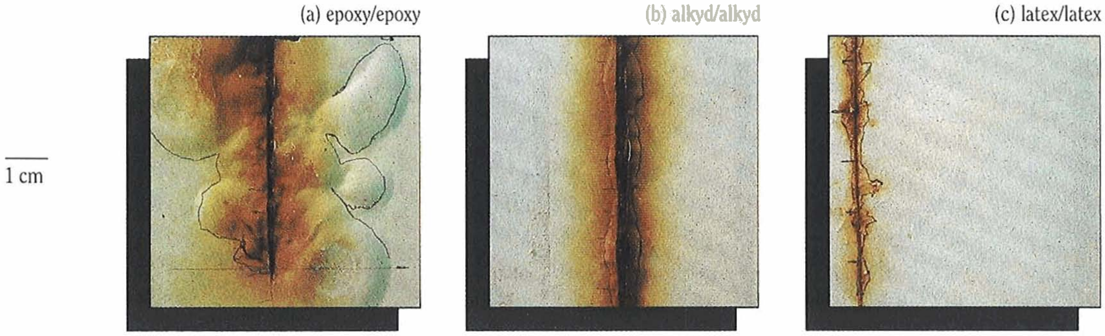
- enhanced retention of surface contamination and transport of detrimental species through the weather-damaged coating;
- changes in physical properties of the paint, such as elasticity, which may have an impact on subsequent performance characteristics; and
- possible dilution of surface species and other effects associated with the deposition of relatively "clean" water on the painted surface during periods of rainfall or condensation.

In an earlier paper[10], the significance of combining weathering and corrosion cycles into a single test method was investigated. The results indicated that the corrosion performance characteristics of organic paint films were markedly affected



(a) epoxy/epoxy (b) alkyd/alkyd (c) latex/latex

Fig. 3 *Scribed regions of panels after combined corrosion/weathering testing (2,000 hours)*



(a) epoxy/epoxy (b) alkyd/alkyd (c) latex/latex

Fig. 4 *Scribed regions of panels after 27 months' marine exposure (21 months for latex)*

by the ultraviolet light-weathering factors in the test. The purpose of this paper has been to compare results obtained from a combined cyclic corrosion/weathering test method with data obtained from outdoor exposures. Further comparisons with data from ASTM B 117 salt spray and wet/dry corrosion cycling (in the absence of the weathering factors) have also been made.

Experimental Design

Coating Systems

Three commercial quality, industrial maintenance coating systems (i.e., primers with appropriate topcoats), representing important generic coating types (catalyzed epoxy-polyamide, acrylic latex, and alkyd), were studied. Further details for these coating systems are shown in Table 1.

All coatings were applied to grit-blasted, 3 in. by 6 in. (8 cm by 16 cm) cold rolled steel test panels (washed and degreased) using an automated air spray technique. Primers were allowed to cure 24 hours before topcoating. Topcoated panels were allowed to cure for 1 full week before testing. The lower half of each coated panel was scribed with an X through the coating down to the metal substrate. (Panels exposed at the marine site were scribed with a single straight line, running vertically along the panels.) In all cases, panels were exposed in duplicate pairs.

Accelerated Testing Procedures

Three accelerated tests were studied:

- standard ASTM B 117 salt spray,
- cyclic wet/dry corrosion testing using a dilute $(NH_4)_2SO_4$/NaCl-based electrolyte, and
- the cyclic wet/dry corrosion test (as above) with weathering factors incorporated (i.e., a combined corrosion/weathering test).

Table 2 outlines the important features of these testing procedures. A more detailed description of each technique is given below. Coating systems were tested for 2,000 hours in each accelerated exposure environment.

Salt Spray (ASTM B 117-85)

Panels were exposed, at a 15- to 30-degree angle

Table 1 Coating Systems Studied

Primer	Topcoat	Substrate	Total DFT mils (microns)
1. Alkyd (zinc chromate inhibitor)	Alkyd	Grit-blasted steel (cold-rolled, 1.5- to 2.0-mil [38- to 50-micron] profile)	6.0 (150)
2. Acrylic latex (barium metaborate inhibitor)	Acrylic latex	Grit-blasted steel (cold-rolled, 1.5- to 2.0-mil [38- to 50-micron] profile)	6.0 (150)
3. Epoxy-polyamide (zinc phosphate inhibitor)	Epoxy-polyamide	Grit-blasted steel (cold-rolled, 1.5- to 2.0-mil [28- to 50-micron] profile	8.0 (200)

Table 2 Salient Features of Test Methods Studied

Test	Type	Features
Salt-spray*	Constant stress	5 (wt) percent NaCl spray, high humidity (~97 percent, non-condensing), elevated temperature (35 C)
Wet/dry test**	Cyclic stress	1-hour 0.35 (wt) percent $(NH_4)_2SO_4$,0.05 (wt) percent NaCl spray at ambient temperature; 1-hour dry at 35 C, purged with air; cycled for total of 2,000 hours
Corrosion/ weathering test***	Cyclic stress	200-hour wet/dry test (as above); 200-hour ultraviolet lig/condensation cycle testing (four-hour ultraviolet light exposure 340 [UVA-bulbs], 60 C alternating with four-hour condensation, 50 C); Cycled for total of 2,000 hours.

**Per ASTM B 117*

***Wet/dry–Prohesion™ (Mebon Paints, Ltd.)*

****Corrosion/Weathering–QUV™ cabinet (Q-Panel Company)*

from the vertical, to continuous deposition of a neutral 5 (wt) percent NaCl solution at elevated temperature (35 C) and high humidity (approximately 97 percent RH).

Wet/Dry (Mixed Salt) Corrosion Test

Panels were exposed to one-hour periods of salt spray (mixed salt) at ambient temperature alternating with one-hour periods with no spray and elevated temperature (35 C). The electrolyte used in this work was a 0.35 (wt) percent $(NH_4)_2SO_4$, 0.05 (wt) percent NaCl solution having a slightly acidic pH (~5.2) upon atomization. Electrolyte was atomized at an approximate rate of 600 ml/hour into a 0.33 m^3 testing chamber. Panels were exposed at a 15- to 30-degree angle from the vertical

Table 3 **Performance Ratings* of Coating Systems After Testing**

Test Method	Conditions Rated	Alkyd System	Latex System	Epoxy System
Salt spray (2,000 hours)	Blistering (size, frequency)	3/7.5	2/0	10/10
	Rust-through	10	5	10
	Undercutting	7	7	8
	Overall	27.5	14	38
Cyclic wet/dry corrosion test (2,000 hours)	Blistering (size, frequency)	10/10	10/10	10/10
	Rust-through	10	10	10
	Undercutting	7	7	7
	Overall	37	37	37
Corrosion/ weathering test (2,000 hours)	Blistering (size, frequency)	10/10	10/10	10/10
	Rust-through	10	10	10
	Undercutting	6	7	4
	Overall	36	37	34
Exterior exposure marine site (27 months)	Blistering (size, frequency)	10/10	10/10	10/10
	Rust-through	10	10	10
	Undercutting	5	7	0
	Overall	35	37	30
Exterior exposure industrial site (12 months)	Blistering (size, frequency)	10/10	10/10	10/10
	Rust-through	10	10	10
	Undercutting	10	10	8
	Overall	40	40	38

** Rating system described in results section*

on shelves attached to the inside walls of the testing chamber. By this arrangement, the panels were positioned in close proximity to resistive heaters that were located in the chamber walls. The resistive heaters were activated during drying periods, thus evaporating the electrolyte layers from the panel surfaces. During these dry periods, the cabinet was also purged with air at a regulated flow rate. This cyclic wet/dry testing procedure is essentially that developed by Timmons in the 1970s.[6] Chambers designed for carrying out this test are commercially available.

Cyclic Corrosion/ Weathering Test

Panels were exposed to 200-hour periods of wet/dry corrosion cycling (as described above) followed by 200-hour periods of ultraviolet light condensation exposure for a total of 2,000 hours (i.e., 5 complete wet/dry/ultraviolet light condensation cycles). For the weathering component of this test, a standard ultraviolet light condensation cabinet, conforming to the ASTM G 53 standard, was employed. Here, a four-hour ultraviolet light exposure period, using UVA-340 bulbs, at 60 C, was cycled with a four-hour condensation period at 50 C.

Outdoor Exposure Sites

Marine Site

Panels were exposed for up to 27 months at a 45-degree angle from the vertical, facing east at a testing site located on the eastern shore of Florida (Ponce Inlet).

Industrial Site

Panels were exposed for 12 months at a 45-degree angle from the vertical, facing south, on the rooftop of our company's technical center in downtown Cleveland, OH. The conditions at this site are corrosive, based on weight-loss measurements made on bare steel samples (e.g., removal rates [short term] greater than 50 micrometers/year for mild steel are typical).

Results

Almost without exception, all failures observed after testing (accelerated and natural) were associated with corrosion and delamination effects along the panel scribes. Figs. 1-4 illustrate the appearance of the scribed regions of the coated panels after salt spray, cyclic wet/dry corrosion, combined corrosion/weathering, and marine site exposure tests, respectively. The epoxy/epoxy system was the only material showing any signs of degradation after 12 months' exposure in an industrial atmosphere. This degradation is illustrated in Fig. 5.

Table 3 shows the average ASTM blister (D 714), rust-through (D 610), and undercutting (D 1654) ratings assigned to the coating systems after 2,000 hours of accelerated testing and after field testing at the marine and industrial sites. In these rating systems, "10" indicates perfect performance, and "0" indicates total failure.

Also reported in Table 3 is an overall performance index, calculated by summing the individual ratings for blister size, blister frequency, undercutting, and rust-through. (The descriptive ratings suggested by ASTM for blister frequency were converted to numeric ratings as follows: none=10, few=7.5, medium=5, medium-dense=2.5, and dense=0.) The best possible overall rating was therefore 40.

The overall performance values were then used to rank the relative performance of the coating systems (on a test-by-test basis), as shown in Table 4.

Discussion

Rank Correlations

From Table 4, several important points may be observed.

Each laboratory test method produced a unique ranking of materials. This suggests that the differences between these test methods (i.e, continuous NaCl salt spray, wet/dry $(NH_4)_2SO_4$/NaCl salt spray, and wet/dry $(NH_4)_2SO_4$/NaCl salt spray with incorporation of weathering factors) cause quite distinct variations in the corrosion protection and degradation characteristics of these organic coatings. The rankings observed at the marine and industrial field sites were similar, in that the epoxy/epoxy panels exhibited the most severe degradation at both sites.

The rankings predicted by the combined corrosion/weathering tests were found to be most consistent with those observed in the field. In contrast, the salt spray test predicted precisely the opposite ranking observed in practice. No clear differentiation of performance was possible using data from the wet/dry corrosion test.

Rankings predicted by the combined corrosion/weathering tests were most consistent with rankings in the field.

Importantly, these rankings, derived from the data in Table 3, were consistent with the overall visual appearance of the coatings after testing, as illustrated in Figs. 1-5.

Modes of Degradation

All samples exposed at the marine site were found to undergo blister formation in areas adjacent to the panel scribes. These blisters were stable (i.e., did not collapse upon storage or upon application of finger pressure), were rust-filled, and were stained orange-brown by the run-off from adjacent corrosion products. In some cases, the scribe line underwent a general lifting due to the accumulation of solid corrosion products visible within. These results are illustrated in Figs. 4(a)-(c). A similar type of scribe line lifting and delamination associated with nearby blisters was also observed on the epoxy/epoxy system after 1 year at the industrial atmospheric test site, as illustrated in Fig. 5. Although a very slight tendency to produce filiform corrosion was observed on the latex/latex sample after marine site exposure (Fig. 4(c)), this was not generally found to be a significant mode of failure in the natural exposure environments.

The modes of degradation observed at the

Table 4 Rank Correlations

Exposure Condition	Ranking Observed (best to worst)
Salt spray	Epoxy>alkyd>latex
Wet/dry corrosion test	Alkyd≈latex≈epoxy
Corrosion/weathering test	Latex>alkyd>epoxy
Exterior (marine)	Latex>alkyd>epoxy
Exterior (industrial)	Latex≈alkyd>epoxy

Table 5 Performance Ratings* of Latex Coatings After 1,200 Hours Corrosion/Weathering Test

Coating	Blistering (size/ frequency)	Rust-Through	Undercutting
A	8/7.5	6	5
B	10/10	6	8
C	10/10	5	6
D	10/10	4.5	7
E	10/10	2	6

Rating system described in results section

marine field sites were generally quite similar to those observed after testing using the combined corrosion/weathering test (Fig. 3). This was apparent, particularly when comparing the marine site exposures with equivalent panels after 2,000 hours of corrosion/weathering testing. For example, with the epoxy/epoxy system, both field and laboratory samples exhibit relatively large (greater than 1 cm in diameter), low-profiled, rust-filled blisters ("scabs") along the scribes. (Compare Fig. 3(a) with 4(a).) Further, a comparison of the performance of the latex/latex system after marine exposure and after corrosion/weathering testing shows that both samples exhibit only very small rust-filled blisters (less than or equal to 2 mm in diameter), negligible loss of adhesion, and only slight rust-staining immediately adjacent to the scribe. (Compare Fig. 3(c) with 4(c).)

Neither the salt spray test nor the cyclic wet/dry corrosion test (Figs. 1 and 2, respectively) was particularly successful in reproducing the types of failures observed after field testing. The wet/dry corrosion test produced some lifting of the coating along the scribe and had a clear tendency to produce filiform corrosion (e.g. Fig. 2(c)). The formation of filiform-type corrosion failures in this cyclic wet/dry corrosion environment is consistent with the findings of other workers.[7,8] Exposure in the salt spray chamber generally resulted in an accumulation of loosely adherent corrosion products covering the scribe and, for the latex/latex system, produced a particularly severe (and unrealistic) breakdown of the coating through the formation of blisters over the entire panel within 1,000 hours of testing. These blisters were of a different nature than those formed along the scribes during exterior exposure and during corrosion/weathering tests. In the salt spray test, the blisters tended to be more hemispherical (i.e., had greater profile) and were not filled with solid corrosion products, as was observed after field testing, but were often filled with liquid electrolyte. The formation of rust-filled blisters around scribed regions of the coatings was not significant in either the salt spray or the wet/dry cycle test.

Based on the relatively good performance of the latex system after field exposure, the salt spray test would appear particularly misleading in the evaluation of these water-borne systems. As a point of interest, the poor salt spray resistance of latex coatings may, to some extent, account for the general reluctance to specify such water-borne paints for service in corrosive atmospheres. The high performance capabilities of these water-borne systems, however, are increasingly being recognized.[3,11] This further demonstrates the need for realistic and meaningful laboratory testing procedures.

Evaluation of Closely Formulated Coatings

Clearly, from the results above, the most meaningful assessments of performance were obtained from the test incorporating both wet/dry cycle corrosion and ultraviolet light condensation weathering fac-

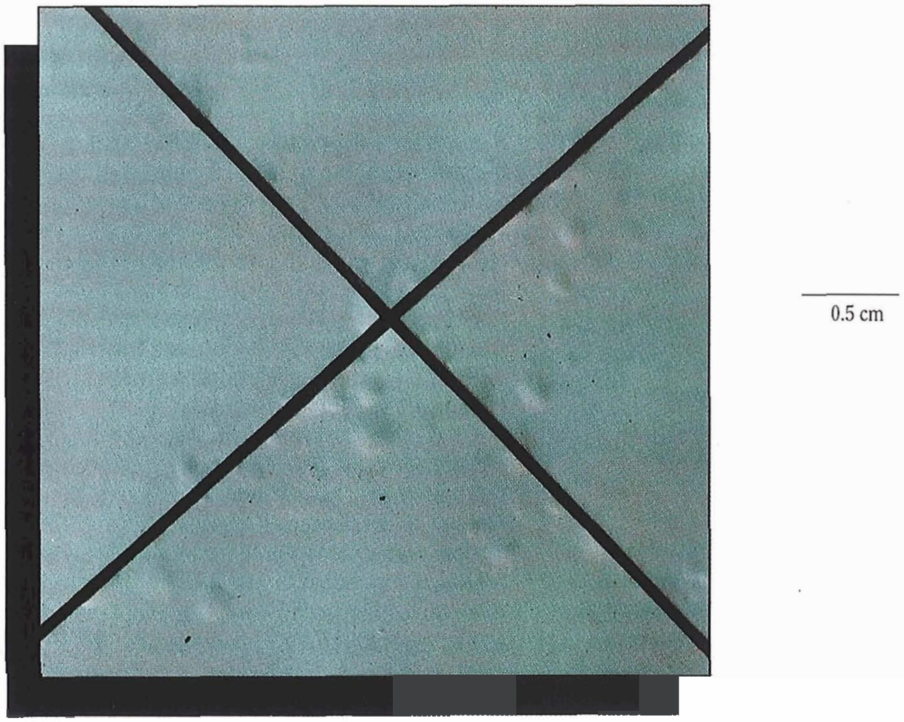


Fig. 5 *Scribed region of epoxy/epoxy system after 12 months' exterior exposure (industrial)*

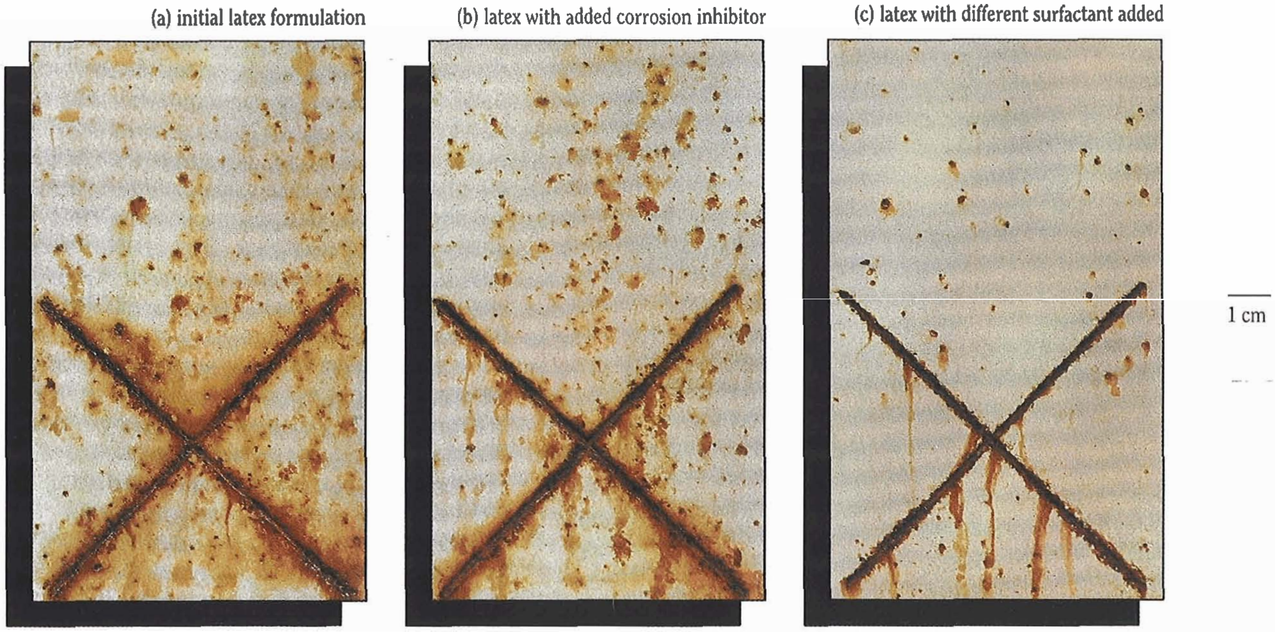


Fig. 6 *Acrylic latex-coated panels (approximately 1.5 mils [3.75 microns]) of blast-cleaned steel after 1,200-hour combined corrosion/weathering test*

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tors. This type of testing approach would thus appear to offer unique advantages over other testing procedures. An important requirement of any useful laboratory accelerated test, however, is that it be able to differentiate the performance capabilities of closely related coatings of the same generic type. A test lacking such sensitivity would be of limited value in product development studies, where it is often necessary to know how small changes in coating composition will affect performance.

To address this issue, a study was conducted in which the cyclic corrosion/weathering test (as described above) was used to evaluate a series of experimental acrylic latex paints applied directly over blast-cleaned steel substrates. These coatings (designated as coating A, B, C, D, and E) differ only slightly in their compositional details. Coated panels were prepared, at equivalent dry film thicknesses of 1.5 (± 0.25 mils [38 ± 6 microns]) using a #75 wirewound drawdown rod. As before, a scribe was cut into the lower half of each equivalently cured panel.

Table 5 lists the ASTM blister, rust-through, and undercutting ratings for the 5 experimental coatings after a 1,200-hour combined corrosion/weathering test. These single-coat samples all exhibited, in varying degrees, formation of rust spots in areas remote from the scribe as well as the formation of rust-filled blisters immediately adjacent to the scribes, as characteristically observed in the combined corrosion/weathering test. This is illustrated for 3 of these coatings in Fig. 6, which demonstrates the effects of incorporating a corrosion inhibitor (Fig. 6(b)) and a different surfactant (Fig. 6(c)) on an initial formulation (Fig. 6(a)). Differentiation of the performance capabilities of these coatings was possible using the combined corrosion/weathering test. Examination of failure modes occurring on generically similar coatings after 12 months' exposure at the marine site further demonstrated the general "realism" of these results, because these exhibited a similar type of scribe line blistering and rust-through in areas remote from the scribe.

Summary

This article demonstrates the importance of incorporating weathering factors in accelerated laboratory tests used to assess the corrosion-controlling properties of an organic coating. The cyclic wet/dry corrosion test, with ultraviolet light condensation weathering factors incorporated, showed significant promise in meeting the requirements of a meaningful accelerated test. Through this testing approach, an improved reproduction of rankings and failure modes observed in practice was possible. The wet/dry corrosion test and particularly the continuous salt spray test were not found to be satisfactory in this respect. Furthermore, the corrosion/weathering testing technique described appeared to have the sensitivity required for applications in product development programs. Based on the results obtained in this work, further investigation using this combined corrrosion/weathering method seems to be warranted. ❑

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