Adhesion Criteria Between Water-Based Inorganic Zinc Coatings and Their Topcoats for Steel

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FOREWORD

This report presents the findings from the results of an FHWA pooled-fund study investigating the failures of water-based inorganic coatings and their possible causes. This report presents the effects of environmental conditions, substrate salt contamination, improper zinc content in the mixed coating, and the effects of rinsing the primer before topcoating.

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Research, Development, and Technology

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16. Abstract

This report presents the results of a test program designed to investigate, in the laboratory, possible causes for failure of water-based inorganic zinc (WBIOZ) coatings. Failures observed in the field have consisted of either topcoat delamination or the appearance of "freckle rusting" in the cured WBIOZ primer. There are many hypotheses regarding the cause of these failures, but no sufficient explanation exists. Furthermore, there are no proven means to ensure that failures do not occur.

The general consensus of the literature is that the failures relate to improper cure of the WBIOZ primer. This improper curing is further hypothesized to relate to environmental conditions. The project initially focused on identifying what relationship, if any, existed between environmental conditions and the observed failure modes. Since a definitive relationship did not exist, further testing was conducted to investigate the effects of substrate salt contamination, improper zinc content in the mixed coating, and the effects of rinsing the primer before topcoating.

This report presents the more practical results of the study. It demonstrates that environmental conditions may be a contributing cause of the failures but are not the primary cause. Substrate salt contamination and improper mixing of the coating were shown to cause freckle rusting. Acrylic topcoat delamination was related to residual alkaline compounds on the primer surface. The surface pH was reduced by applying a dilute acid solution or by weathering for 2 weeks in a northeastern marine atmosphere. Additional ways of reducing the surface pH (weathering or otherwise) should be investigated.

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APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
	LENGTH			
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
	AREA			
in^2	square inches	645.2	square millimeters	mm^2
ft^2	square feet	0.093	square meters	m^2
yd^2	square yards	0.836	square meters	m^2
ac	acres	0.405	hectares	ha
mi^2	square miles	2.59	square kilometers	km^2
	VOLUME			
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
NOTE: Volumes greater than 1000 l shall be shown in m^3.				
	MASS			
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")
	TEMPERATURE (exact)			
°F	Fahrenheit temperature	5(F-32)/9 or (F-32)/1.8	Celcius temperature	°C
	ILLUMINATION			
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
	FORCE and PRESSURE or STRESS			
lbf	poundforce	4.45	newtons	N
lbf/in^2	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

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

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

(Revised September 1993)

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BACKGROUND

Three significant factors have severely affected the maintenance procedures used for steel bridge structures:

- Compliance with environmental and worker health regulations.
- Continual aging of the Nation's highway infrastructure.
- Resulting increases in the cost of maintaining this infrastructure.

Over the past several years, these factors have combined to make the use of water-based inorganic zinc (WBIOZ) coatings for steel bridges a very attractive option.

In theory, WBIOZ coatings provide excellent long-term corrosion protection in both mild environments and highly corrosive marine environments. This result has been demonstrated by several investigators and in many field applications.[1] In the 7-year low volatile-organic-compounds (VOC) coating study conducted for FHWA, a high-ratio WBIOZ single coat system performed among the best of all coatings tested over a Steel Structures Painting Council (SSPC) SP-10 near white metal blasted surface.(1)

In addition, WBIOZ coatings provide the benefit of containing zero VOCs. This has provided an environmentally compliant option for many high volume fabrication shops that have already been affected by VOC discharge regulations. They are also a potential solution for field painting operations that are subject to stringent limitations of VOC content.

In spite of the rather attractive benefits of the use of WBIOZ coatings, these materials have decreased in popularity within the past few years. This decrease has been caused by the concern over several field failures which have occurred shortly after application of the systems in both shop and field applications. In these failures, two distinct scenarios have been observed—early pinpoint rusting (freckle rusting) and severe topcoat disbondment. Figure 1 shows early pinpoint rusting of underlying steel shortly after application of single coat WBIOZ. Figure 2 shows severe disbondment of water-based topcoats applied over WBIOZ primers.

[1]See the bibliography at the end of this report.

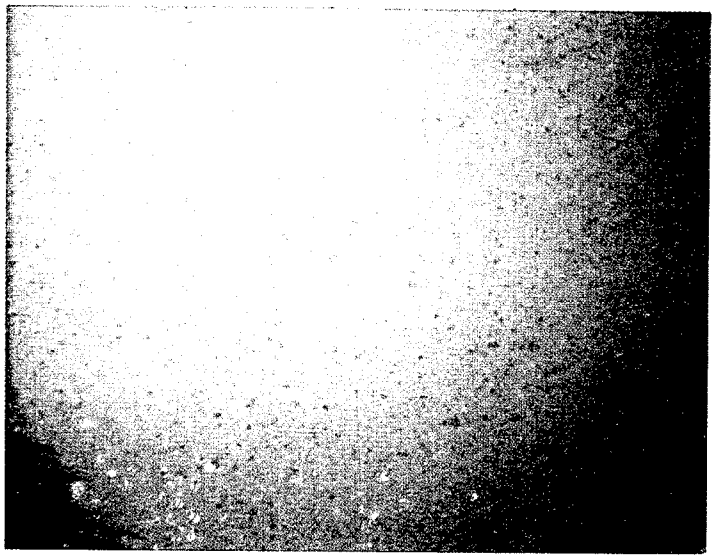

Figure 1. Typical freckle rusting.

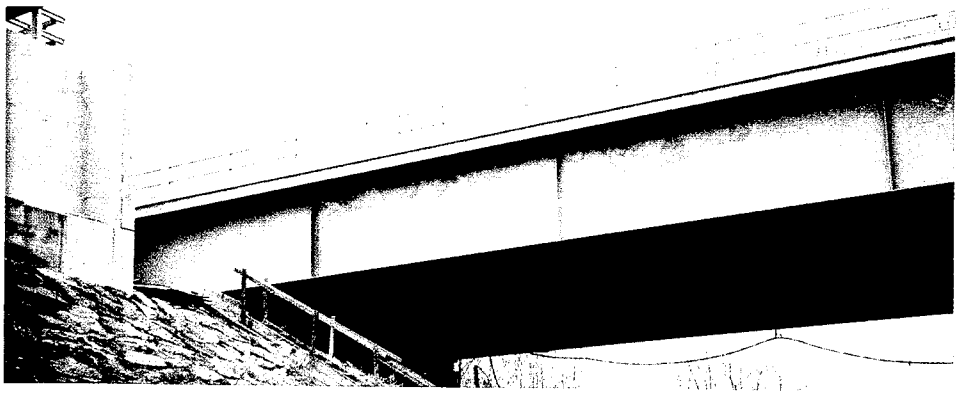

Figure 2. Severe topcoat disbondment.

To help address these concerns, the Federal Highway Administration with the aide of 12 participating state agencies (District of Columbia, Michigan, Wisconsin, Indiana, California, Kansas, Oklahoma, Rhode Island, Missouri, Massachusetts, Mississippi, and Washington) sponsored the subject research program. This research was to be conducted in seven distinct-related tasks. Each of these is listed below:

- Task A - Literature Search. Applicable literature related to the formulation, curing properties, and field performance of WBIOZ coatings was reviewed.

- Task B - Preparation of Coating Test Panels. Candidate coating materials were selected and test panels prepared under strictly controlled and monitored environmental conditions.

- Task C - Accelerated Testing. A battery of accelerated laboratory and natural marine exposure tests were performed to determine differences in the performance properties of the coatings applied under various environmental conditions.

- Task D - Coating Evaluation. Test panels were inspected in detail and the data collected throughout the program was thoroughly analyzed.

- Task E - Critical Parameters for a Proper Cure. The effect of environmental parameters on inorganic zinc cure were discussed using data from test panel inspections.

- Task F - Quality Control. This included the development of test and analysis techniques to conveniently determine proper cure of zinc primers in the field.

- Task G - Presentation and Final Report. The key research results and quality control techniques were presented in a symposium for State and FHWA representatives and documented in detail throughout this report. This task also included the development of the technical summary for FHWA distribution.

The report is divided into sections describing various aspects of the research conducted. Where appropriate, the above referenced tasks are described in further detail. The following describes some of the major sections of the report.

- Literature Review presents the results of task A as well as further review of the literature during the course of the project.

- Technical Approach describes the test procedures, materials, and evaluation techniques used for tasks B through F.

- Results of Testing presents the data analysis from the testing phases of tasks C through F. This section includes technical discussion of the results and the analysis leading to critical parameters and quality assurance guidance. Selected raw data is provided in the appendixes.

- Guidance for Use of Water-based Inorganic Zinc presents the practical results of the research in a clear, concise question-and-answer format. The section includes guidance on critical parameters for proper cure and quality control as required by tasks E and F of the contract. The reader is referred to the results section for the backup data and analysis.

OBJECTIVES

The present research had two primary objectives:

1. Identify a fundamental cause of failure for both topcoated and untopcoated WBIOZ coatings. If it is the result of improper application environment, identify changes in the application and cure environment that will eliminate the failures. If the topcoat adhesion condition is a function of topcoat selection or primer surface condition, identify and provide guidelines to assure satisfactory field performance of the applied coating systems.

2. Develop and/or seek for a quality control field testing procedure to identify if a WBIOZ primer is suitable for exterior exposure or topcoating.

SUMMARY AND CONCLUSIONS

The testing conducted during this study was focused on three areas of WBIOZ performance—two failure modes and coating behavior during cure. The conclusions are presented in three sections, one for each of these issues. The first conclusions presented relate to two failure modes—topcoat blistering and freckle rusting. These conclusions are certainly the more pragmatic of those generated in the study. The final section presents some conclusions related to WBIOZ behavior during cure.

Topcoat Blistering

1. The pH value of the WBIOZ surface before topcoating was found to be the best indicator of the tendency for acrylic topcoat blistering. Other parameters studied (e.g., environmental conditions, time-to-topcoat, salt contamination, and zinc loading) did not consistently predict blistering.

2. A self-cure WBIOZ coating has residual alkaline compounds present on the recently cured film surfaces prepared in this study. Removal of these alkaline compounds appears to minimize or eliminate blistering problems observed with acrylic topcoats.

3. The waterborne acrylic topcoat was susceptible to blistering whereas the epoxy topcoat was not. The research did not find an adequate explanation for this phenomenon, however it may be due to differences in mechanical properties of the films (i.e., how internal stresses are resolved). The possibility of osmotic effects should also be investigated further.

4. The blisters were more readily observed in humid conditions (early morning or rainy days). They typically appeared quickly (within a few days) and shrunk over time. Some blisters were liquid filled while others were gas filled.

5. A few delaminated areas were observed. Delaminated areas were observed where large blisters ruptured.

Freckle Rusting

6. Freckle rusting correlated well with zinc content of the inorganic zinc primer. Less zinc dust in the paint (or dry film) resulted in more freckle rusting. Interestingly, the critical zinc loading to minimize freckle rusting appears to be very near the design loading. It is likely that freckle rusting occurs at coatings areas with locally low zinc content.

7. Freckle rusting correlated well with the level of salt concentration on freshly blasted steel surface. Panels with chloride levels as low as 10 $\mu g/cm^3$ [4.4 grains/ft^3] developed freckle rusting.

8. Freckle rusting was observed relatively soon after coating exposure (within 3 days) and did not tend to increase in severity over the 1-year exposure test period.

9. Higher humidity (80 percent RH) during WBIOZ coating cure did not, in itself, cause freckle rusting. However, high humidity during cure did seem to increase the severity of freckle rusting in the presence of other contributing factors (e.g., low zinc content, surface contamination).

WBIOZ Curing

10. The tendency for the water-based inorganic zinc coatings to exhibit physical defects (e.g., cracking) may be related to the ability of the two curing reaction mechanisms to proceed under the curing conditions. Ideal curing may occur when water evaporates at a rate proportional to the rate at which the inorganic zinc silicate matrix polymerizes. Further work would be necessary to validate this hypothesis.

11. Evaporative cooling must be considered when measuring surface temperature. A 1.7 to 2.8 °C [3 to 5 °F] reduction in the film surface temperature was observed in relation to the steel substrate temperature. This observation quantifies the effects of evaporative cooling during cure of WBIOZ coatings.

12. Initial data suggests that the physical properties of the film can be compromised because of very dry curing conditions. A suitable window for the application of WBIOZ coatings needs to be defined by manufacturers for each product.

13. Alternative measures of the environmental conditions during cure of WBIOZ coatings are being investigated. Since the speed of evaporation of water is the key parameter in coating cure, the use of partial pressures from psychometric charts should prove to be more appropriate than the traditional dew-point and surface temperature calculations.

LITERATURE REVIEW

An information review was conducted regarding the formulation, performance, and field failures of WBIOZ in both the untopcoated and topcoated condition. This search included the following key actions:

- A detailed compilation of the formulation properties of WBIOZ coatings which may have had a significant influence on failure mechanisms.

- Contact with selected State DOT and FHWA representatives to obtain information regarding recent failures of inorganic zinc coatings applied in shop and field environments.

This effort resulted in a summary of the chemistry involved in the curing process for these coatings. The information was obtained in terms of curing mechanisms and their relationship with controllable environmental parameters (i.e., humidity, temperature, and time). This review in conjunction with some preliminary cure testing (discussed later in this report) was used to define the environmental test parameters used in the application of coatings to test panels.

The early failure of water-based inorganic coatings by freckle rusting and topcoat delamination were addressed by this research program. As an initial step, the state of the art in water-based inorganic coatings formulation and curing variables was reviewed. The information obtained covered various aspects of self-cured WBIOZ coating technology. Emphasis was placed on items related to the ability of WBIOZ coatings to be topcoated and considerations for selecting topcoat materials. The following three areas were identified as probable technology sources for further understanding of WBIOZ topcoating:

1. The formulation variables with inorganic water-based coatings.

2. The curing conditions for these coatings, both alone and with topcoats as a coating system.

3. The topcoat materials and the compatibility of these materials with the surface of water-based inorganic coatings.

The publications and articles forming the basis of this review are listed in the bibliography at the end of this report.

WBIOZ Background

Inorganic zinc coatings contain metallic zinc dust and an inorganic alkali silicate binder. The WBIOZ coatings are divided into three types: heat-cured, post-cured, and self-cured. As the names imply, the primary difference in the materials is catalyst for the cure reaction. Heat-cure of

WBIOZ coatings obviously requires the addition of heat for proper cure. Post-cure of WBIOZ coatings requires the application of a curing solution over the uncured coating. This solution, usually a dilute acid, is then rinsed off with water after an appropriate cure time. The primary coating type discussed in this review is the self-cured WBIOZ coating. As its name implies, no external action is required to cure a self-cure coating.

Self-cured WBIOZ coatings are two component products: a powder component containing the metallic zinc dust (with or without other pigments) and a liquid component containing alkali silicate. These coatings have the chemical reactions required for coating cure engineered into the product. The curing of WBIOZ coatings basically occurs in two reactions:

- After application, water evaporation concentrates of the alkali silicate binder.
- Concurrently, a water insoluble zinc silicate coating forms by reaction of the silicate with carbon dioxide in the air and the zinc dust in the coating.

Curing Characteristics

The binder holding the zinc dust particles together within a WBIOZ coating forms a coherent film by chemical reaction after coating application. As stated above, this process can be carried out either by heating, post-curing, or self-curing. Both temperature and humidity (moisture or water) influence the self-curing chemical reactions. After applying self-cured WBIOZ coating, evaporation of the water with precipitation of the alkali silicate is the first step in forming the coating. During this drying phase, high humidity and low temperature significantly extend the dry time for the WBIOZ coatings. Table 1 shows the influence of environmental conditions on the speed and degree of cure for self-cure WBIOZ coatings.

Table 1. Influence of the environment on self-cure WBIOZ film formation.

Process Step	Temperature		Relative Humidity	
	High (100 °F)	Low (32 °F)	High (>80% RH)	Low (<40% RH)
Application	---	---	---	---
Drying	Fast	Slow	Very Slow	Fast
Cure - become water insoluble	Fast	Slow	Very Slow	Fast
Cure - become water resistant	Fast	Slow	Very Slow	Fast
Cure - become abrasion resistant	Fast	Slow	Slow	Fast

In comparison to most other types of chemically cured coatings, inorganic zinc coatings dry quite fast in minutes to such conditions as dry-to-touch or dry-through as compared to hours for typical chemical cured organic resin coatings. After drying, the WBIOZ coating cure process can begin either by heating to a high temperature to convert the alkali metal silicate to a water insoluble binder (heat-cure), treating with an inhibited acidic cure solution (post-cure), or exposure to the atmosphere (self-cure). The speed of cure for self-cure WBIOZ coatings is dependent on the coating formulation and environmental conditions. Tables 2 and 3 show cure rates for various inorganic zinc coatings. As can be seen in the tables, binder formulation variables play an important role in the speed of cure for inorganic zinc coatings. Also note in table 2 that increasing temperature does not necessarily increase time to become water insoluble. For potassium silicate binder, increasing temperature above 30 °C [86 °F] with humidity decreasing below 60 percent RH actually increases time to become water insoluble.

Table 2. Water insoluble time for potassium silicate vs potassium + lithium silicate inorganic zinc coatings after application.

Time to becoming Water Insoluble (minutes)		Temperature (°F/°C)	Relative Humidity (percent)
Potassium Silicate	390	59/15	90
	240	68/20	80
	150	77/25	70
	120	86/30	60
	150	95/35	50
	390	104/40	40
Potassium/ Lithium Silicate	100	77/25	70
	17	122/50	30
	5	140/60	20

Table 3. Dew condensation test (24 hours water contact)* characteristics of potassium silicate vs potassium + lithium silicate inorganic zinc coatings.

Drying Time (hours) 21 °C [70 °F], 58%RH	Potassium Silicate High SiO_2	Potassium/Lithium Silicate High SiO_2
4	Softened/water soluble[1]	Softened/water soluble[1]
8	Softened/water soluble[1]	Softened/water soluble[1]
24	Softened/water soluble[1]	Softened/water resistance[2] Recuperates immediately
48	Slightly soft/water resistant[3] Zinc salts	Hard/water resistant[4] Zinc salts
72	Harder than 48 hours Water resistantZinc salts	Hard/water resistant Zinc salts

* Prepared panels are wrapped with dampened cloth and kept moist for at least 24 hours. The coating is checked for softening and chalking. If softening occurs, the coated panel is allowed to dry to check the coating's recuperation time.

[1] Softened/water soluble—Coating is soft and can be removed by rubbing

[2] Softened/water resistant—Coating is soft and cannot be removed by rubbing

[3] Slightly soft/water resistant—Water insoluble. Same as 2 but harder

[4] Hard/water resistant—Maximum hardness and water resistance

Two stages of cure are usually described for self-cure WBIOZ coatings. These are time to become water insoluble and time for immersion service. The times for these curing stages can be measured with simple laboratory tests. Typical times for these levels of cure are shown in table 4.

Table 4. Typical times for indicated degree of cure of self-cure WBIOZ (3 to 9 mils DFT).

	32 °C [90 °F]	21 °C [70 °F]	10 °C [50 °F]	4.4 °C [40 °F]
Time to Water Insoluble @ 70% RH (minutes)	15-45 min.	30-120 min.	45-180 min.	60-270 min.
Time to Water Immersion @ 70% RH (hours)	12-36 h	24-72 h	48-120 h	72-180 h

Based on this data, the most favorable environmental conditions for proper curing of most self-cure WBIOZ coatings include relative humidities of less than 70 percent and temperatures of at least 21 °C [70 °F]. Conditions to avoid (higher probability for improper cure) include *relative humidity higher than 70 percent, temperatures lower than 4.4 °C [40 °F], and stagnant air flow conditions*. Any combination of these three conditions would be even more likely to cause improper cure of self-cure WBIOZ coatings.

Cure Before Topcoating

The curing time or degree of inorganic zinc coating cure before topcoating with organic coatings has been a subject of much discussion. Some researchers have endorsed short topcoating times, and others have promoted extended topcoating times to achieve more cure of the inorganic zinc coating by weathering before applying the topcoat. There are usually benefits and consequences associated with compromise. In the case of topcoating an inorganic zinc coating, these would be dependent on the type of inorganic zinc coating and the type of topcoat.

Manufacturers' recommendations vary greatly for cure time required before topcoating (i.e., the time at which a topcoat can be applied and the inorganic zinc is not disturbed while achieving satisfactory topcoat film formation). This time can range from as little as 2 hours to a more typical 16-24 hours at normal temperatures and humidities. Many self-cured WBIOZ coatings can be topcoated 24 hours after application at moderate temperatures and good drying conditions. Others may require days or weeks of curing before topcoating because of environmental conditions affecting the cure reactions of the inorganic zinc coating. Early because of topcoating before achieving a higher degree of cure may result in the self-destruction of some WBIOZ coatings or severe blistering of some topcoats.

The temperature, relative humidity, and air movement conditions during application and initial drying of the self-curing WBIOZ will influence the coating film formation. At low temperatures, high humidity, and restricted ventilation or air movement, some self-cure WBIOZ coatings can tend to glaze over (a slight separation of liquid binder migrating to the coating surface), especially at high film thickness, which may reduce topcoat adhesion. When this glaze condition exists, roughening the surface would need to be done to achieve satisfactory adhesion.

Since curing of the inorganic zinc to ultimate film properties takes considerably longer than 24 hours at normal temperatures, the coating system (inorganic zinc plus topcoat) will initially be more sensitive to mechanical stress. This sensitivity is in comparison to either: curing the inorganic zinc longer before topcoating or the properties of the coating system weeks, months, or years after application. When using typical 16-24 hours topcoating time at normal temperatures and humidities, the properties of the coating system need to be taken into consideration. Most coating systems will be sensitive to mechanical damage ranging from moderate to high. Some topcoat materials, such as vinyl, water-based acrylic, and vinyl acrylic, can be "peel-sensitive" initially and gradually adhere normally as solvent or water evaporate and the topcoat film becomes harder.

Topcoating in as little as 2 hours after application of some self-cured WBIOZ coatings is possible. Many topcoats such as the epoxy types can be applied and achieve satisfactory film and adhesion at this short topcoat time. However, the long-term performance needs to be investigated for these coating systems, especially with water-based topcoats.

Topcoat Material Selection

Inorganic zinc coatings, when first developed, were primarily used without topcoats. As the number and types of inorganic zinc coatings expanded, topcoating became more prevalent. All types of topcoats have been applied over inorganic zinc coatings. Many of them have been successful, and some have been disasters. The following generic coating types have been found to develop satisfactory adhesion over inorganic zinc coatings, however, some of these coating types may require a tie coat:

Epoxy - all types
Polyamide Coal Tar Epoxy
Acrylic - Solvent and Water-based
Vinyl and vinyl acrylic
Silicone
Chlorinated rubber
Phenolic - alkali resistant
Aliphatic Polyurethane - alkali resistant reactive types, 2 components or moisture cure
Inorganic Coatings (Polysiloxane, Siloxane, Siloxirane)

The compatibility of a potential topcoat over an inorganic zinc coating needs to be evaluated by taking into account the following considerations:

Resistance to alkalinity. An inorganic zinc coating topcoat material must be resistant to the alkaline conditions which result from the cathodic protection reaction of metallic zinc on steel and the curing of zinc silicate. Topcoats having poor resistance to an alkaline environment should not be directly used on the inorganic zinc coating. Most alkyd-based coatings (which are susceptible to alkaline hydrolysis) need to be avoided or at least have an appropriate intermediate coat used between the inorganic zinc and alkyd. Some highly modified alkyd coatings specifically developed for resistance to water immersion or highly humid environments having some alkali resistance can be compatible over inorganic zinc coatings.

Compatibility with metallic substrates. Inorganic zinc coatings contain metallic (zinc) pigments. As a rule, coatings that do not have good adhesion characteristics to prepared steel, other metals, or other inorganic materials should not be applied directly to inorganic zinc coatings. Vinyl formulated for direct application to inorganic zinc coatings has been the workhorse for highway bridge coating specifications for a long time. Current trends are to replace the vinyl with higher solids coatings because of VOC regulation pressures.

Compatibility with porous substrates. Inorganic zinc coatings, by nature, have a range of porosity in the dry coating film. Most self-cured inorganic zinc coatings have high porosity. This porosity will influence the type of topcoat that can successfully be applied with the popular mist-coat/full- coat application technique.[2] The bubbling or blistering of topcoats over self-cured inorganic zinc coatings and the use of the mist-coat/full-coat application technique for

topcoats with slow evaporating solvents, was described in 1963 by Gatewood Norman.[2] The following types of coatings can present difficulties or be impossible to apply directly to the inorganic zinc without pinholes or intact blisters in the coating:

High Gloss Coatings—All generic types. Coating compatibility should always be confirmed.

Semigloss Coatings—Most generic types. Some can be successful with special variations of the mist coat-full coat application techniques.

Low Build Coatings—Vinyl acrylic, vinyl (below 2 mils DFT/coat)

Coatings that have been successfully applied with the mist coat-full coat technique with a minimum or no pinholes or intact blisters include semi-gloss coatings, flat high build (5 mils DFT/coat or more) coatings, and others specifically designed for application over the porous self-cured inorganic zinc coatings. Typical generic types include:

Epoxy Primers
Epoxy Coatings*
High Build Vinyl*
High Build Chlorinated Rubber
* Those specifically formulated and recommended may require special thinning depending on environmental temperatures.

Post-cured inorganic zinc coatings have presented less difficulty in topcoat application. Many coatings that cannot be applied over self-cured inorganic zinc, because of the bubbling and pin-holing, can be applied over a post-cured inorganic zinc. This does not mean that post-cured inorganic zinc can be topcoated with any coating and not have pinholes or intact blisters result. The post-cured inorganic zinc is also porous and this porosity can result in bubbling when top-coating. The post-curing step may assist in reducing the effects of porosity on topcoating as achieved by allowing self-cured inorganic zinc coatings to cure and weather for months before topcoating. This reduces the amount of bubbling and resultant pinholes or intact blistering in the topcoat.

To minimize topcoat application bubbling, the inorganic zinc coating should be applied to achieve the smoothest possible coating by:

(a) Application in full wet coat, DO NOT DRY SPRAY.
(b) Control DFT—avoid high thickness.
(c) Remove dry over spray before topcoating.

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[2] This technique involves spraying a thin mist of the topcoat material over the WBIOZ primer, allowing it to become tacky so that the high end solvents flash off, and then applying the topcoat to the desired thickness. This technique has been shown to minimize bubbling or blistering of the topcoat.

The topcoat should be applied:

(a) Using thinners appropriate for the temperature conditions.
(b) Using mist-coat/full-coat application technique.
(c) To immediately correct visible pinholes or blistered spots with an additional spray during application.

Susceptibility to mechanical damage. Self-cured inorganic zinc coatings have varying cure rates and most do not develop full physical properties by the time topcoats are applied—typically after 16 to 24 hours. The consequence of slow curing is that the coating system will be sensitive to mechanical stress during handling for shop-applied coatings. The severity of stress is dependent on the specific type of inorganic zinc coating and the number and type of topcoats.

Moisture sensitivity. Moisture may be present in the void spaces of inorganic zinc coating resulting from overnight condensation. If a topcoat is applied before moisture completely evaporates and the inorganic zinc coating is not dry, an amine cured epoxy or coal tar epoxy may show bubbling and severe blistering as a result of gassing. The gassing is a reaction of the zinc with water in the highly alkaline condition on the inorganic zinc surface. It is known that many organic coatings use highly alkaline curing agents. However, polyamide curing agents and many other amides used for curing epoxy coatings have not normally produced this reaction.

Topcoat Delamination

"Auto-delamination" is one term used to describe early catastrophic failure of the adhesion between a primer and the topcoat. For the inorganic zinc coatings, this phenomenon is related to both the cure of the inorganic zinc and more importantly, the application of the inorganic zinc and topcoat as a system. This includes the extended curing conditions for the system along with the characteristics of the topcoat. Inorganic zinc coating that has been dry sprayed will remain powdery and never harden. Powdery overspray not removed from the properly applied inorganic zinc film will act like a mold release. This will prevent proper adhesion of a topcoat to the inorganic coating film. Excessive topcoat thickness in a single coat can result in shrinkage stress such that the topcoat cracks and separation in the inorganic zinc coating follows. Obviously, the failure modes found in the research do not adequately explain all topcoat disbondment phenomena. The present research will investigate failure modes not adequately explained in the literature.

Corrosion Protection

The literature investigates the effect of zinc loading and zinc particle size on the corrosion potential of the zinc primer in a non-aerated 3-percent NaCl solution.[3] This solution had shown the best correlation between corrosion potential on lab test samples and long-term marine atmosphere exposure panels (after 33 months). Lab tests were run on static samples in the solution and on samples with an anodic current of 0.3 mA.

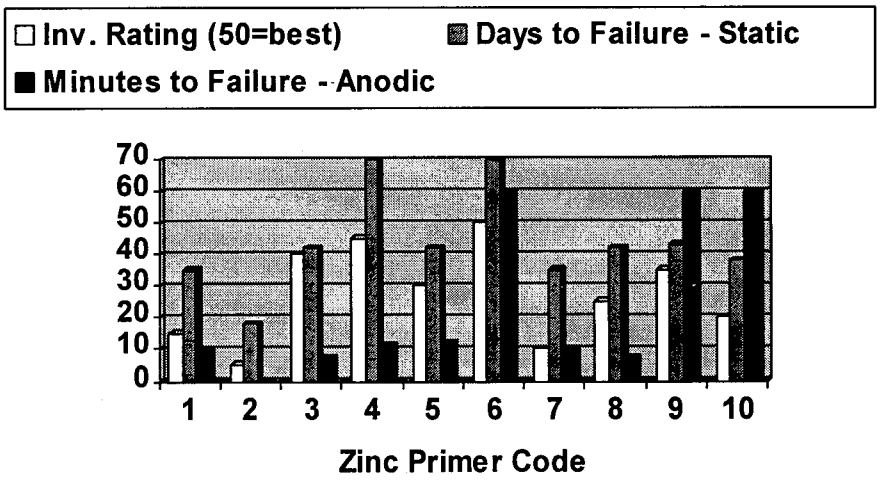


Figure 3. Corrosion test performance of zinc primers.

Test samples were 25 x 12 x 0.2 cm. The surface area (including both sides) was nominally 600 cm^2. Coatings were applied at 2 mils. For the static tests, 15 percent of the steel surface was exposed. For the anodic current tests, the coating was left intact.

The test results are shown in figure 3. There is a general correlation that those materials performing the best show little anodic polarization under the exposure conditions. Those performing the worst generally rapidly polarize in an anodic direction. The anodic current density was about 0.5 $\mu A/cm^2$. One key conclusion is that performance is related to zinc loading and particle size. Figure 4 shows that performance is improved by a higher zinc loading and, over the particle size range investigated, those inorganic zinc primers with a larger average particle size performed better than those with a smaller particle size. There is no discussion as to the reason for this performance.

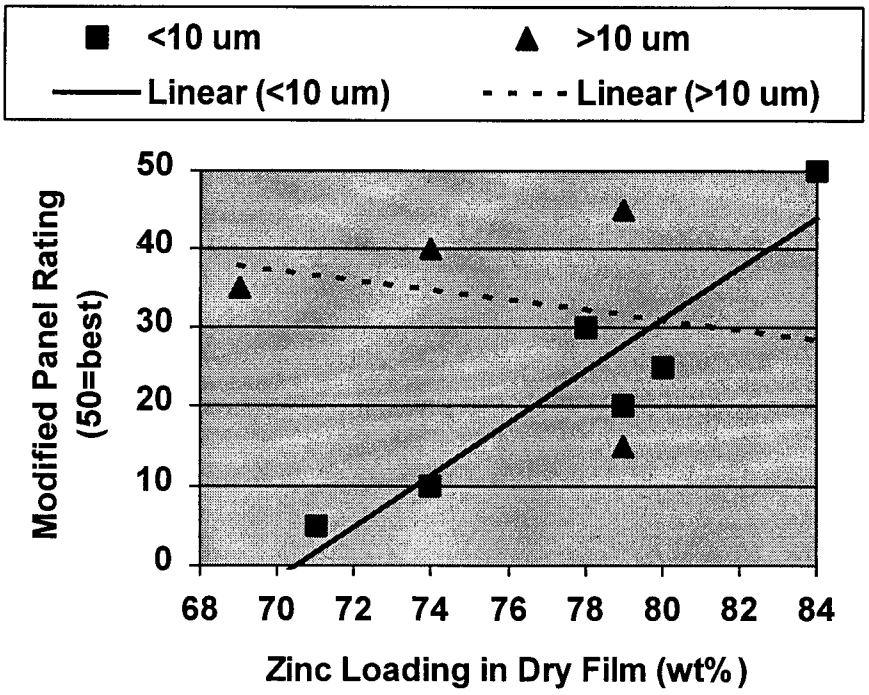


Figure 4. Corrosion performance versus zinc content.

Effect of Surface Salts

Appleman, et. al. bases his safe level of 50 μg/cm² for zinc ethyl silicate (solvent reducible)/epoxy or /epoxy/urethane on the pressurized immersion rather than salt-fog tests.[4] In the salt-fog tests, complete intercoat (IOZ/epoxy) failure was observed on all panels, so the safe level is based on the more aggressive pressure immersion testing where no failures were noted. Conversely, for the zinc alkali silicate (water-based)/acrylic system blistering was observed for all systems in the pressure immersion test, but a failure level of 30 μg/cm² was observed in the cyclic salt-fog test (versus no failures observed on the epoxy systems). The safe level for WBIOZ in the salt-fog test was 16 μg/cm².

Soltz found that as little as 0.25 μg/cm² chloride could effect underfilm rusting of clear epoxy coatings in pressurized seawater immersion.[5] Contamination levels from 8-16 μg/cm² chloride could cause osmotic blistering. In unpublished work by Ocean City Research Corp., it was found that even the lowest chloride level measurable with field equipment (2 μg/cm²) can cause increased blistering of the standard Navy epoxy system in hot deionized water testing.

EXPERIMENTAL APPROACH

The testing conducted included a core test matrix evaluating the effect of environmental variables on WBIOZ cure and performance. As results were obtained from this core matrix, three side issues were examined in preliminary testing. Each of these side issues gave rise to an individual test matrix. Figure 5 illustrates the tests performed. The environmental variables investigated during this research formed the core of the testing. The three additional issues included the effects of surface salt contamination, the effects of varied zinc content, and the effects of various primer washing techniques.

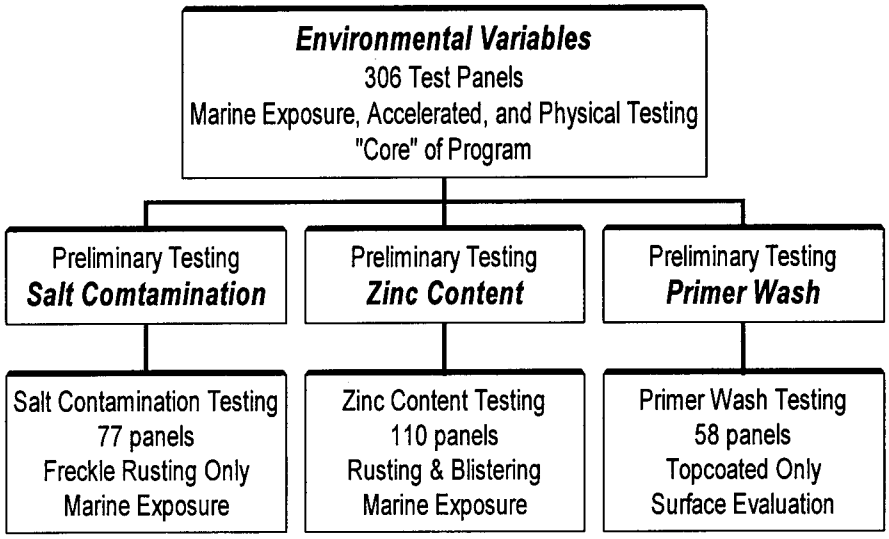


Figure 5. Outline of testing conducted.

This section of the report contains five subsections. The first subsection describes the coating materials used in the testing. Subsequent subsections describe the experimental procedures used for each of the four test matrices in detail.

Coating Selection

A variety of coating materials were used for the various test matrices. To avoid duplication, a description of the coating materials is included here. Note that not all materials were used in all tests. Refer to the subsection on each individual test matrix to see which materials were used for testing.

Self-cure WBIOZ primers

Two self-cure WBIOZ coatings were used in the testing. These will be refered to as Product A and Product B throughout this report. They were selected to represent the WBIOZ class of coatings. Our testing was not intended to distinguish the performance of individual coating formulations but to analyze the characteristics of self-cure as a coating type. Since the rationale was to represent the performance of a coating type, two prevalent materials were chosen for

testing. Both products employ a high silica ratio which is common among accepted self-cure WBIOZ coatings. Both products include a liquid binder and a zinc powder. The powder and liquid are mixed before spraying. Table 5 shows some of the more pertinent characteristics of the two coatings.

Post-cure WBIOZ primer (control)

A post-cure WBIOZ primer was used as a control in most of the experiments. Since the objectives of this study keyed on variables associated with self-cure WBIOZ coating cure, the post-cured material was chosen as a control to best represent the ideal and fully-cured state of a WBIOZ coating. The post-cure coating consists of a liquid and powder that are mixed and sprayed to the surface. The coating is allowed to dry. After drying, a post-cure solution (dilute phosphoric acid) is sprayed over the coating. After the required cure period, the post-cure residue is rinsed from the coating surface with water.

Water-based acrylic topcoat

One of the topcoat materials tested was a water-based acrylic coating. The rationale for this material selection assumes that a high solids/low VOC topcoat would be chosen in conjunction with a WBIOZ primer. This entire coating system would have minimal if any VOC content. The water-based acrylic tested was a water-based acrylic coating that has often met Caltrans specifications as a topcoat for Product A. The single component coating was sprayed on to achieve a dry film thickness of 76μm [3 mils].

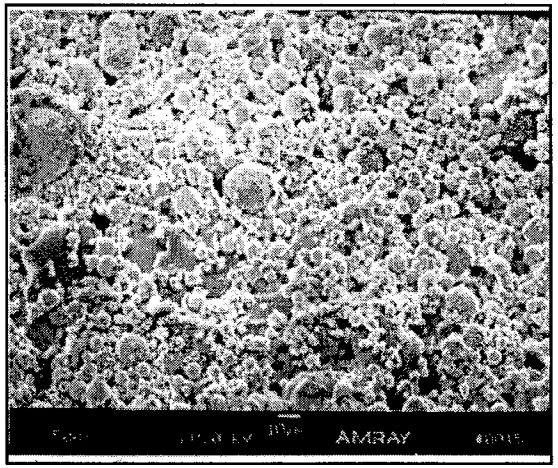
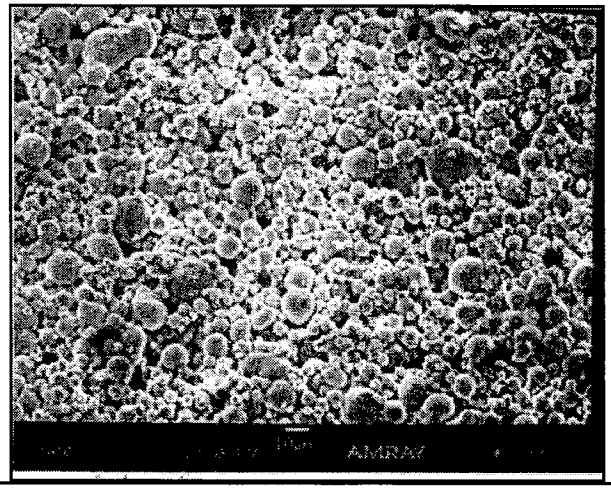
Solvent-based epoxy topcoat

An epoxy topcoat was also chosen for testing. These coatings have a long and successful performance history of application over inorganic zinc coatings yet are generally solvent-based. The material was used as a control coating for compatibility over inorganic zinc primers. The epoxy tested meets MIL-P-24441, F151, a Federal specification for polyamide epoxy. The two component epoxy coating was sprayed on to achieve a dry film thickness of 76 to 127μm [3 to 5 mils].

Ethyl silicate inorganic zinc primer (control)

In one of the experiments, an ethyl silicate inorganic zinc (solvent-based) was used as a control. The chemistries of a cured solvent-based inorganic zinc coating and a cured WBIOZ coating are quite similar. Both contain high percentages of metallic zinc held together by an inorganic binder. Because the vehicles of the wet material and the dynamics associated with coating cure are quite different the solvent-based material was chosen as a control for some experiments evaluating cure factors. The ethyl silicate inorganic zinc consists of a liquid binder and zinc powder which are mixed and spray applied.

Table 5. Selected characteristics of the WBIOZ coatings tested.

Characteristic	Product A	Product B
Mixing Ratio	16.8 lbs Powder/0.7 gal Liquid	18 lbs Powder/6.87 lbs Liquid
Silicate Type	Potassium Silicate	Potassium Silicate
Typical SEM Micrograph		
Minimum Appliaction Temp	4.4 °C [40 °F]	4.4 °C [40 °F]
Surface Temperature	2.8 °C [5 °F] Above Dewpoint	4.4 °C [8 °F] Above Dewpoint
Maximum RH	85%	N/A
Zinc in Dry Film	N/A	90%
Dry to Touch	3 minutes	15-30 minutes
Dry to Topcoat	24 hours	2 hours

N/A - Not Available 1 lb = 0.454 kg

1 gal = 3.785 L

Environmental Variables

The environmental variables testing formed the core of the research. It included two phases—Dry Rate Testing and Accelerated Exposure Testing. The Dry Rate Testing was designed to obtain a rather basic understanding of how the self-cure coatings dry in various environments. This fundamental knowledge then served as the basis for selecting specific conditions to apply coatings for the accelerated testing. The experimental approach to the two phases is described below.

Dry Rate Testing

Preliminary experiments on coating dry rates were conducted to investigate the environmental conditions for the test matrix. The dry rate testing was used to determine which combinations of environmental conditions would be appropriate for conducting coating performance testing. Table 6 shows the rationale for selecting various parameters for the drying conditions.

Table 6. Environmental conditions rationale.

Condition	Level	Intent / Rationale
Environment Temperature	20 ± 1 °C [68 ± 2 °F]	safe level
	8 ± 1 °C [47 ± 2 °F]	just below "critical" level of 10 °C [50 °F]
Relative Humidity	40 ± 2%	safe level
	65 ± 2%	safe level
	80 ± 2%	at "critical" level of 80%
	90 ± 2%	expected detrimental condition
Surface Temperature (relative to the dew point temperature)	>11 °C [20 °F] above	safe level
	3-5.5 °C [6-10 °F] above	just above "critical" value of 3 °C [5 °F]
	0-2 °C [0-4 °F] above	below "critical" value of 3 °C [5°F]
	about 3 °C [5 °F] below	expected detrimental condition

The objective of the dry testing was to better understand the behavior of the WBIOZ primers in the test environmental conditions. The change in weight of coated samples was monitored during the initial drying phase under each of the environmental conditions. Data gathered during this phase also lends insight into the effects of environmental conditions on the curing process of WBIOZ. Note that this initial drying phase may not result in a full cure. The degree of cure that exists when the coating weight has stabilized (resulting in a "dry-to-touch" condition) is what is being evaluated.

Two self-curing WBIOZ coatings and one post-cure coating were tested. The laboratory work of this phase involved closely examining the curing of the zinc coatings under five of the environmental conditions (conditions involving cure near or below the dewpoint were not amenable to the analysis presented).

Applied coatings were dried for up to 4 hours in a controlled atmosphere chamber with a 0.028 m^3 [13 ft^3] enclosed working space and controls which are capable of maintaining relative humidities from 5 to 90 percent and temperatures from 0 °C to 43 °C [32 °F to 110 °F]. The humidity values are varied by using either a humidifier or by circulating air through an anhydrous calcium sulfate desiccant. Circulating air across an electrical heating element or a copper coil containing sub-zero ethylene glycol controlled the temperature. Initial experiments found that evaporative cooling from liquid carbon dioxide or liquid nitrogen interfered with the inorganic zinc curing process. Relative humidity and temperature were measured roughly every 30 minutes through the cure cycle to ensure that they remained within the target range.

Before spraying, each coupon was first weighed, resulting in an initial unpainted coupon weight. If required, the samples were cooled before painting on the thermoelectric cooling device. After painting, the coupon was placed on the scale for drying. The scale was coupled to a computer for automated data acquisition each minute during curing. For conditions requiring the use of the thermoelectric cooling platform, the coupons were removed from the platform and weighed at 10-minute intervals. The normalized paint weight was obtained by subtracting the uncoated substrate weight from the weights recorded through the curing process. Percent weight loss of coating was then plotted as a function of time.

After drying, dry film thickness measurements were taken on at least 10 points scattered over the sample and the average was recorded. Surface characteristics examined both with the naked eye and at a 30X magnification were noted.

Accelerated Exposure Testing

On the basis of dry time results, eight curing conditions were selected to apply the zinc primers. Table 7 shows the actual environmental condition combinations tested.

Test Matrix Design. Eight different environmental curing conditions were selected as variables for this testing. The coating systems tested included two self-curing WBIOZ coatings, one post-cured WBIOZ coating, one water-based acrylic topcoat, and one solvent-based epoxy topcoat. There were two performance tests conducted on each of the individual coating system/curing condition combinations. A series of Quality Assurance (Q/A) and cure evaluation tests were also be performed on the untopcoated self-cure inorganic zinc coatings. The total number of panels prepared for the Environmental Conditions Test Matrix included:

8 cure conditions X 2 self-cure zinc primers X 3 topcoats (acrylic, epoxy, none) =48
1 post cured zinc primer X 3 topcoats (acrylic, epoxy, none) =3
Total number of panels = (48+3) X 6 replicates = 306 panels total

Table 7. Environmental conditions for testing.

Condition #	Dry to Topcoat	RH (%)	Ambient Temp (°C [°F])	Surface Temp (°C [°F])
1	50 minutes	40 ± 2%	20 ± 1 °C [68 ± 2 °F]	20 ± 1 °C [68 ± 2 °F] (7 °C [13 °F] above the dew point)
2	125 minutes	65 ± 2%	20 ± 1 °C [68 ± 2 °F]	Reduced to 14 ± 1 °C [58 ± 2 °F] (0-2 °C [0–4 °F] above the dew point)
3	100 minutes	80 ± 2%	20 ± 1 °C [68 ± 2 °F]	21 ± 1 °C [70 ± 2 °F] (3-5.5 °C [6–10 °F] above the dew point)
4	Condition 3 with a cyclic wetting. This was achieved by lowering the surface temperature below the dew point for a 20-minute duration after the "dry to topcoat" time. The surface was then allowed 1 hour to reach stabilization before topcoating.			
5	50 minutes	40 ± 2%	8 ± 1 °C [47 ± 2 °F]	8 ± 1 °C [47 ± 2 °F] (13 °C [23 °F] above the dew point)
6	125 minutes	65 ± 2%	8 ± 1 °C [47 ± 2 °F]	Reduced to 3 ± 1 °C [38 ± 2 °F] (0-2 °C [0–4 °F] above the dew point)
7	100 minutes	80 ± 2%	8 ± 1 °C [47 ± 2 °F]	8 ± 1 °C [47 ± 2 °F] (3-5.5 °C [6–10 °F] above the dew point)
8	Condition 7 with a cyclic wetting. This was achieved by lowering the surface temperature below the dew point for a 20-minute duration after the "dry to topcoat" time. The surface was then allowed 1 hour to reach stabilization before topcoating.			

Cure and Adhesion Tests. Two panels of each individual scenario were used for cure evaluation and adhesion testing. The cure tests were performed on the zinc primers only and included:

- Pencil hardness per ASTM D 3363-92a "Test Method for Film Hardness by Pencil Test."
- Nickel rub evaluation (pass/fail test).
- Physical examination for microcracking using low power (30X) microscope.

Following the complete cure cycle for each coating scenario, the panels were measured for final dry film thickness and adhesion testing was performed. Two adhesion tests were performed on the topcoated panels only:

- Adhesion per ASTM D 3359-93 "Test Methods for Measuring Adhesion by Tape Test."
- Adhesion per ASTM D 4541-93 "Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers."

Cyclic Accelerated Test. A cyclic accelerated test was used to evaluate coating system performance under conditions of cyclic salt-fog, ultraviolet light exposure, and freeze/thaw temperature exposure. Each accelerated test cycle was approximately 15 days long and was completed two times for a total exposure of approximately 30 days. The specific parameters of this testing follow:

1. 7 days of cyclic salt-fog exposure—This test cycles between 2 hours of ASTM B 117 salt-fog testing at ambient (21±1°C) temperature followed by a dry cycle of 1 hour at 35±1°C.

2. 1 day of exposure in a laboratory freezer maintained at -23±1°C.

3. 7 days of exposure in an Atlas model CXW Sunshine Carbon Arc Weather-Ometer. This testing consisted of a 12-hour cyclic exposure to simulated sunlight and condensing humidity. The panels were exposed for 8 hours to the light source and then 4 hours to the humidity source.

Accelerated Marine Atmosphere Exposure. One set of duplicate panels for each coating system/cure combination were exposed at the Ocean City Research Marine Exposure Site. The total time between coating application and marine atmosphere exposure did not exceed 3 days. Spraying the panels daily with natural seawater accelerated the marine atmosphere exposure. Visual panel evaluations were initially made daily to determine the time to produce pinpoint rusting or topcoat blistering. Testing continued for 1 year.

Coating Evaluation. All panels subjected to the cyclic accelerated test and marine atmosphere exposure were evaluated by ASTM D610 for surface rusting, ASTM D661 for cracking, ASTM D714 for blistering, and ASTM D1654 for scribe cutback. Evaluations were performed following each cycle of the cyclic accelerated test and included panel photographs. Evaluations of the marine atmosphere exposure panels were conducted frequently during the first month of exposure and approximately monthly thereafter up to 1 year. At 60 days of marine atmosphere exposure, a topcoat adhesion survey was conducted.

An evaluation of the data generated in the testing described above was performed to determine the critical environmental parameters for achieving proper curing of WBIOZ primers. The initial Q/A and cured film data were compared with the environmental conditions. The panel performance data following accelerated testing were also evaluated versus the environmental curing conditions to identify any possible relationships between these factors.

Salt Contamination Testing

An additional test matrix was designed to investigate the factors influencing freckle rusting of WBIOZ coatings that occurs when left untopcoated or in the short time before topcoating. The

matrix was based on the contractor's previous experience with WBIOZ coatings and observations from the marine atmosphere exposure panels. We conducted testing according to the following matrix:

1. Untopcoated panels only. Topcoat adhesion was not addressed with this matrix.

2. Product A, Product B, and a solvent-based inorganic zinc (IOZ) control. We applied both WBIOZ coatings used in the remainder of the testing as well as a solvent-based control. This will help to determine if the salts are detrimental during curing or after curing. One would hypothesize that the salts initiate corrosion in the water-based coating because of a corrosive electrolyte. This likely does not exist with solvent-based systems since they have a higher electrolyte resistance in the initial curing stage.

3. Four chloride levels (<2 $\mu g/cm^2$, 10 $\mu g/cm^2$, 20 $\mu g/cm^2$, 30 $\mu g/cm^2$). It seems prudent to select a high level that should fail in the prescribed test program. The 30 $\mu g/cm^2$ was selected based on the failures observed in our preliminary testing. Control panels of <2 $\mu g/cm^2$ will be specified since this is the lowest that can be measured using field techniques. Finally, intermediate salt levels of 10 $\mu g/cm^2$ and 20 $\mu g/cm^2$ were tested. Test panels were doped with a sodium chloride solution and immediately force-dried to minimize flash rusting.

4. Curing at one temperature (21°C) and two humidities (40 and 85 percent RH). The effect of humidity was evaluated by curing the test panels in two environments. Our preliminary dry time evaluation showed that the dry time roughly doubles from 40 to 85 percent RH. This may effect the occurrence of freckle rust since the corrosive electrolyte would be in contact with the surface for twice as long.

5. Three replicates were exposed marine exposure site for 6 months. Preliminary testing showed that panels developed freckle rust after roughly 1 week of exposure. Additionally, field failures are reported to occur rather quickly. Thus, a 6-month marine exposure should be adequate to determine if freckle rusting will occur. Triplicates of 10.2-cm (4-in) by 15.2-cm (6-in) samples were exposed.

In total, 72 panels were prepared and tested. This includes untopcoated panels only, three inorganic zinc coatings applied over four chloride levels, cured under two environmental conditions, with triplicate panels exposed in one environment (1 x 3 x 4 x 2 x 3 = 72). Admittedly, this testing only begins to address the issue of surface contamination. Other concerns such as topcoat application over the freckle rusted surface, effect of other surface contaminants, effect of other environmental conditions (including cyclic), and effect on other inorganic zinc products are but a few of the issues that also might be addressed. However, this limited testing addressed surface contamination adequately and economically.

Mix Ratio Testing

Additional panel testing confirmed the effects of off-ratio (i.e., zinc-to-binder mixing ratios) mixing of WBIOZ. Two WBIOZ coatings were each applied to abrasive-blasted steel panels (SSPC SP-10). Five mix ratio scenarios were simulated for each primer as follows:

1. Mixed with 66 percent of the specified zinc added.
2. Mixed with 90 percent of the specified zinc added.
3. Correct mix ratio with better than 97-percent accuracy.
4. Mixed with 110 percent of the specified zinc added.
5. Mixed with 133 percent of the specified zinc added.

For each of these mixing scenarios, we prepared untopcoated test panels and acrylic topcoated panels. Topcoated and untopcoated panels were exposed at the marine exposure site. They were inspected for topcoat blistering and freckle rusting during exposure. Pull-off adhesion strength of the acrylic topcoat was determined on unexposed and exposed test panels.

Primer Wash Testing

The objective of this testing was to investigate potential surface cleaning methods to eliminate blistering of acrylic topcoats. Experimental variables included different surface treatments after primer application but before topcoat application. Primer surface pH was measured before topcoating. The metric to quantify performance differences among the WBIOZ surface treatments was topcoat blistering. The following variables were used:

1. Two self-cure WBIOZ formulations.
2. One post-cure WBIOZ control coating (did not blister in previous testing).
3. Five surface treatment variables for self-cure WBIOZ:
   - None —topcoated after 24 hours.
   - Post cure solution application/potable water rinse—topcoated after 24 hours.
   - Potable water wash (20.7 MPa [3,000 psi])—topcoated after 24 hours.
   - Phosphoric acid rinse/potable water rinse—topcoated after 24 hours.
   - 14-day weathering at our marine exposure site, potable water rinse, then topcoated.

Samples were exposed at the Ocean City Research marine exposure site and in a condensing humidity chamber. Surface pH measurements were obtained after initial cure and after wash treatment. The panels were inspected frequently during the initial exposure period to quantify the development of topcoat blisters.

RESULTS OF TESTING

The research conducted throughout this program was completed in five phases.

- Dry Rate Testing.
- Environmental Variables Testing.
- Salt Contamination Testing.
- Zinc Content Testing.
- Primer Wash Testing.

Details on how these tests were conducted are presented in the experimental approach section of the report. The following subsections of the report will present the pertinent results of all testing.

Dry Rate Testing

The results of the weight loss testing include the evaluation of five curing conditions for WBIOZ. Table 8 lists the parameters of the test conditions analyzed. The table also includes visual observations of the cured coatings.

Table 8. Test conditions and visual observations.

Condition	Target Values					Panels Exhibiting Cracks
	RH	Ambient Temp.	Surface Temp	Dew Point Spread*	Differential water partial pressure	
1	40%	20 °C [68 °F]	20 °C [68 °F]	8 °C [25 °F]	0.2 in Hg	0 out of 6
3	80%	20 °C [68 °F]	21 °C [70 °F]	4 °C [8 °F]	0.1 in Hg	1 out of 6
5	40%	8 °C [47 °F]	8 °C [47 °F]	13 °C [23 °F]	0.1 in Hg	5 out of 6
6	65%	8 °C [47 °F]	3 °C [38 °F]	1 °C [2 °F]	0.01 in Hg	0 out of 6
7	80%	8 °C [47 °F]	8 °C [47 °F]	4 °C [8 °F]	0.05 in Hg	3 out of 6

*Difference between dewpoint and surface temperature

Figure 6 shows a typical drying curve. The plot shows percent weight loss as a function of drying time. From these curves, drying characteristic values were extracted for dry time and percent weight loss. These are the time and percent weight loss at which the curve levels off. Note that this does not necessarily relate to degree of cure or a completely dry state, but rather the time at which there is no further water loss from the coating (evidenced by weight change) under the environmental conditions tested.

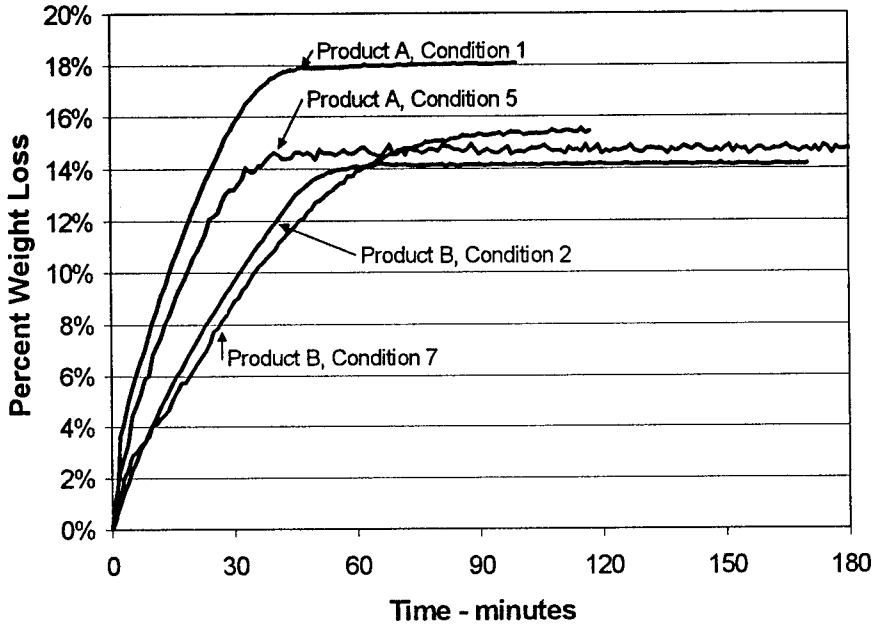


Figure 6. Typical drying curves for WBIOZ primer.

Figure 7 shows the dry time and percent weight loss data grouped by temperature and plotted as a function of humidity. The straight lines are least square linear regressions of the data. The graphs seem to indicate that the percent weight loss (top data) tends to be more a function of temperature and less dependent on humidity. Conversely, the dry time (lower data) tends to be more a function of humidity and less a function of temperature.

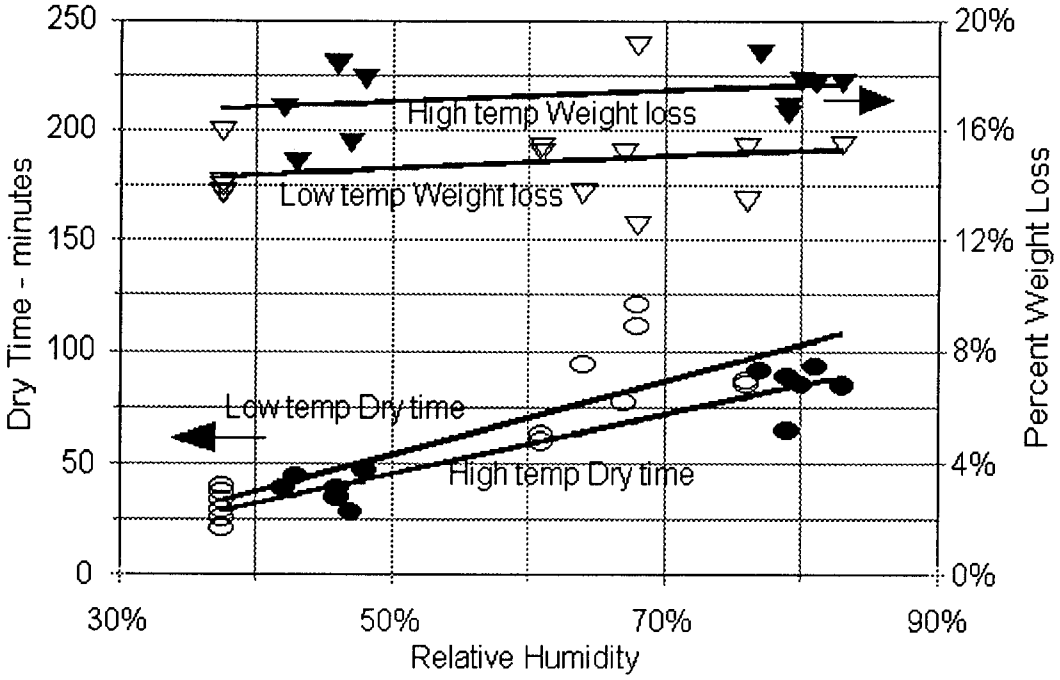


Figure 7. Drying characteristics as a function of target temperature and relative humidity.

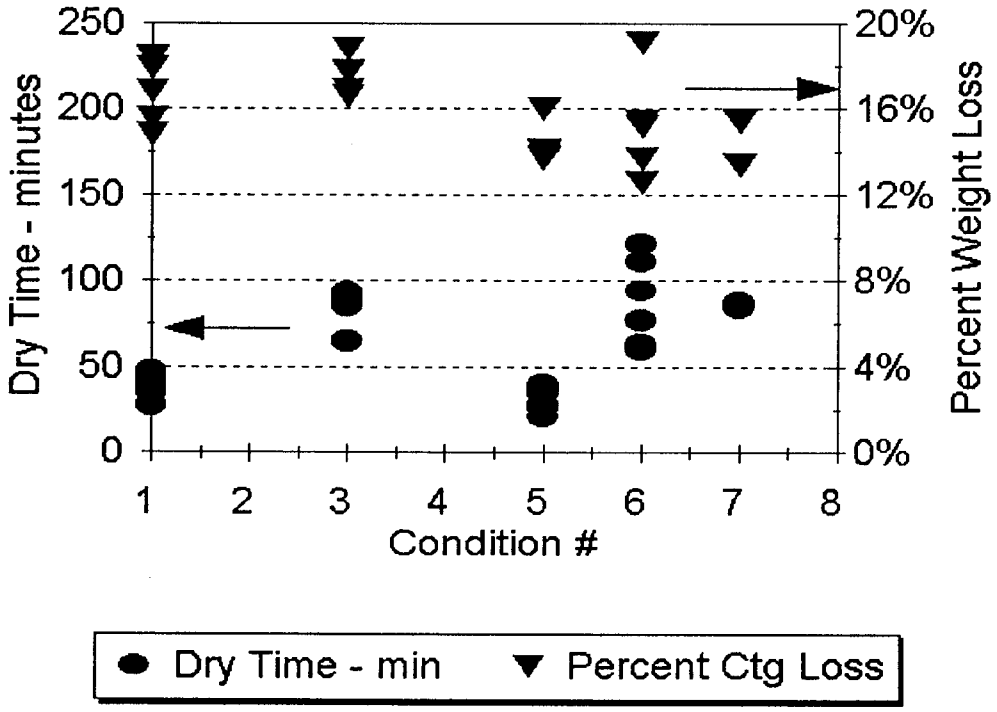


Figure 8. Drying data for the various conditions studied.

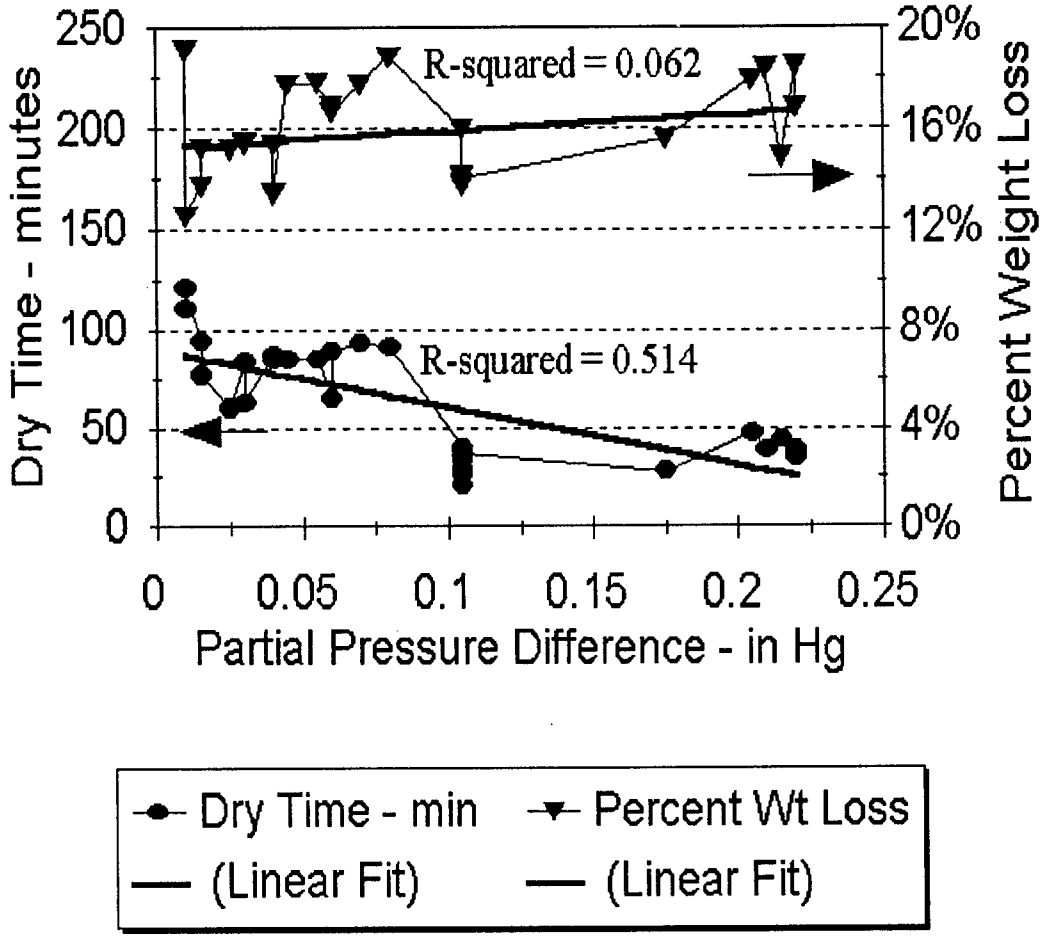


Figure 9. Drying characteristics as a function of actual partial pressure difference.

Figure 8 shows the dry time and temperature of each condition. The higher temperature conditions (condition number 1 and 3) show a higher percent weight loss (triangle points) than the lower temperature data (condition number 5 and 7). The higher humidity conditions (3 and 7) show a longer dry time than the lower humidity (condition number 1 and 5). The intermediate humidity and low temperature condition (condition number 6) shows a significant amount of scatter. This is likely because of the fact that control of the environmental conditions was insufficient to maintain a constant drying condition. At the low drying rate, small changes in temperature will make a large relative change in drying rate.

Table 8 shows the differential partial pressure between water vapor at the specified ambient conditions and water at the panel surface temperature. This parameter should relate to the water evaporation rate phase of the coating cure. Figure 9 shows that differential partial pressure does correlate somewhat with the drying time (the linear regression has an R-squared value of 0.5).

Table 8 also shows that conditions 5 and 7 tend to exhibit cracking where conditions 1 and 3 do not exhibit cracking. These are also the conditions that tend to have a lower percent weight loss. If one looks at the propensity to crack, it is clearly more common at the lower cure temperatures. Yet the partial pressure (and thus evaporation rate) are equal to or less than those at the higher temperature. However, the tendency for cracking at the lower temperature decreases with decreased differential partial pressure (evaporation rate). This suggests that the water evaporation rate must be reduced to maintain sufficient water in the coating for the reversible chemical reaction to proceed at the lower temperature. Thus, at the lower temperatures, a lower differential partial pressure is optimum (condition number 6); whereas at higher temperatures, a higher differential partial pressure is optimum (condition number 1).

Environmental Variables Testing

Application and testing of these panels made up the core of this research effort. A total of 306 panels were prepared in accordance with the coating systems and environmental conditions outlined in the technical approach.

Quality Assurance Testing

The initial quality assurance data collected from unexposed panels is presented in appendix I. Additional pull off adhesion data was collected from untested panels during this reporting period to better compare the effects of the curing conditions and topcoat types. The data demonstrated that the adhesion of acrylic was poorer than the adhesion of epoxy over the WBIOZ. The adhesion of all topcoats was best under curing condition number one (20 °C [68 °F] and 40% RH). Small differences were noticed among the other curing conditions.

Accelerated Testing

Accelerated Marine Exposure. Visual inspection data is presented in tables 9 through 11. Each table cell shows individual panel data for duplicate samples.

Table 9 shows selected inspection results for the epoxy topcoated test panels. None of the epoxy topcoated panels have exhibited rusting or blistering. The adhesion data presented in the table was obtained using ASTM D3359 (method B). The shaded cells show unacceptable adhesion ratings of less than 4B. Note that these ratings are associated with the high humidity conditions.

Table 9. 60-day data summary of accelerated marine atmosphere exposure panels – epoxy topcoated only.

Condition	Temperature	Relative Humidity	Dew Point Spread	Epoxy Adhesion (ASTM D3359)	
				Product B	Product A
1	20 °C [68 °F]	40%	8 °C [25 °F]	4B, 4B	5B, 5B
2	20 °C [68 °F]	65%	1 °C [2 °F]	5B, 5B	4B, 5B
3	20 °C [68 °F]	80%	4 °C [8 °F]	0B, 0B	3B, 3B
4	Condition 3 with a cyclic wetting			4B, 5B	2B, 0B
5	8 °C [47 °F]	40%	8 °C [25 °F]	5B, 4B	4B, 4B
6	8 °C [47 °F]	65%	1 °C [2 °F]	5B, 5B	4B, 4B
7	8 °C [47 °F]	80%	4 °C [8 °F]	0B, 0B	5B, 5B
8	Condition 7 with a cyclic wetting			3B, 3B	3B, 3B

Table 10 shows selected inspection results for the acrylic topcoated test panels. Blistering was the only significant failure mode observed on these panels. The shaded cells indicate that blistering was observed after 60 days. The only clear trend with the data is that no blistering was ever observed when the primer underwent a cyclic wetting before topcoat application. This suggests the importance of a wet/dry cycle after drying, the additional 120 minutes of cure time, or both before topcoat application. A wet/dry cycle might be experienced from overnight condensation or a thorough primer rinsing. Extending the cure time was not explicitly separated out in this test, though it was in later testing. All other conditions, even panels prepared under ideal conditions until a dry state was observed, showed some degree of blistering. Figure 8 shows product B topcoated with acrylic in environmental condition 5 (8 °C [47 °F], 40%RH) after the 60-day accelerated marine exposure.

Table 11 shows the freckle rusting results for the untopcoated test panels. The shaded boxes represent conditions where any rusting was evident at the 60-day inspection. Notice that only a few, random panels showed very minimal freckle rusting. There certainly do not appear to be any trends with environmental conditions.

Table 10. 60-day data summary of accelerated marine atmosphere exposure panels–acrylic topcoated only.

Condition	Temperature	Relative Humidity	Dew Point Spread	Acrylic Blistering (ASTM D714)	
				Product B	Product A
1	20 °C [68 °F]	40%	8 °C [25 °F]	10, 10	2F, 2F
2	20 °C [68 °F]	65%	1 °C [2 °F]	10, 10	2M, 2F
3	20 °C [68 °F]	80%	4 °C [8 °F]	2F, 4F	2F, 10
4	Condition 3 with a cyclic wetting			10, 10	10, 10
5	8 °C [47 °F]	40%	8 °C [25 °F]	2F, 2F	2F, 2F
6	8 °C [47 °F]	65%	1 °C [2 °F]	2F, 10	2F, 2F
7	8 °C [47 °F]	80%	4 °C [8 °F]	2F, 10	6F, 4F
8	Condition 7 with a cyclic wetting			10, 10	10, 10

Table 11. 60-day data summary of accelerated marine atmosphere exposure panels–untopcoated panels.

Condition	Temperature	Relative Humidity	Dew Point Spread	Rusting (ASTM D610)	
				Product B	Product A
1	20 °C [68 °F]	40%	8 °C [25 °F]	9, 10	10, 10
2	20 °C [68 °F]	65%	1 °C [2 °F]	10, 10	10, 10
3	20 °C [68 °F]	80%	4 °C [8 °F]	9, 9	10, 10
4	Condition 3 with a cyclic wetting			10, 10	10, 10
5	8 °C [47 °F]	40%	8 °C [25 °F]	10, 10	10, 10
6	8 °C [47 °F]	65%	1 °C [2 °F]	10, 10	9, 10
7	8 °C [47 °F]	80%	4 °C [8 °F]	9, 9	10, 10
8	Condition 7 with a cyclic wetting			10, 10	10, 10

Figure 10. Product B topcoated with acrylic in environmental condition 5 (8 °C [47 °F], 40% RH) after the 60-day accelerated marine exposure.

Cyclic Accelerated Testing. Table 12 summarizes the test results after two cycles (30 days) of testing. The majority of the failures were evident after the first 24 hours of testing (first test of the first cycle). The results observed were consistent with the accelerated marine atmosphere exposure testing.

Note that the epoxy topcoated panels did not show significant blistering. This is consistent with observations from the marine atmosphere exposure panels. The acrylic topcoated panels have shown substantial blistering. However, it was not consistent with any given environmental conditions.

Freckle rusting was observed on a few of the test panels. As in the marine exposure testing, freckle rusting did not seem to correlate with any of the environmental conditions.

Table 12. Cyclic accelerated testing data summary following two cycles.

Curing Condition	Untopcoated Rusting (ASTM D610)		Topcoat Blistering (ASTM D714)			
			Acrylic		Epoxy	
	Product B	Product A	Product B	Product A	Product B	Product A
1	9, 10	10, 10	2F, 10	1F, 1.5F	10, 10	10, 10
2	10, 10	10, 10	10, 10	1.5F, 1.5M	10, 10	1F, 1F
3	10, 10	9, 10	10, 10	1.5F, 1.5D	10, 10	10, 10
4	10, 10	10, 10	3F, 4F	2M, 10	10, 10	10, 10
5	10, 10	10, 10	1.5F, 1F	1.5M, 1MD	10, 10	10, 10
6	9, 9	6, 7	1M, 6M	3MD, 10	10, 10	10, 10
7	10, 10	10, 7	2F, 1F	2M, 2.5F	10, 10	10, 10
8	10, 10	10, 10	2.5F, 10	4F, 10	10, 10	10, 10

Salt Contamination Testing Results

Preliminary testing showed that Product B coated test panels with approximately 20-25 $\mu g/cm^2$ chloride contamination showed freckle rust after roughly only 2 weeks of marine atmosphere exposure. This prompted testing under a range of salt contamination levels.

Immediate rusting (within 1 day) was noticed on several panels from the two WBIOZ formulations when applied to panels with chloride contamination as low as $8\mu g/cm^2$. Figure 11 shows freckle rust ratings at the end of the 58-day exposure period as a function of contamination level, humidity, and primer material. Key points include:

- Increasing chloride contamination from 0 to 8 $\mu g\ Cl^-/cm^2$ increases the propensity for freckle rusting under marine atmosphere exposure.
- Increasing the relative humidity (RH) level during cure from 40 to 80 percent appears to exacerbate this effect.
- The ethyl silicate control coating showed significantly less freckle rusting than either of the WBIOZ coatings tested.

Zinc Content Testing Results

Figure 12 shows the acrylic topcoat adhesion measured over zinc films with varied levels of zinc. The data do not demonstrate a clear correlation between topcoat adhesion and zinc content. Blister ratings on panels exposed in the marine atmosphere (not shown here) did not demonstrate a clear trend either.

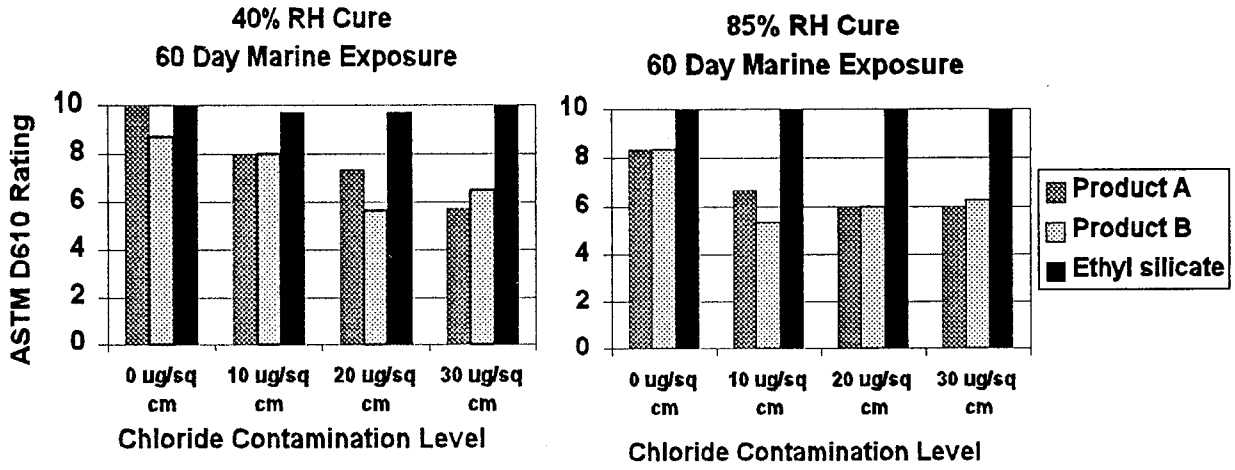


Figure 11. Freckle rusting as a function of surface chloride contamination and relative humidity.

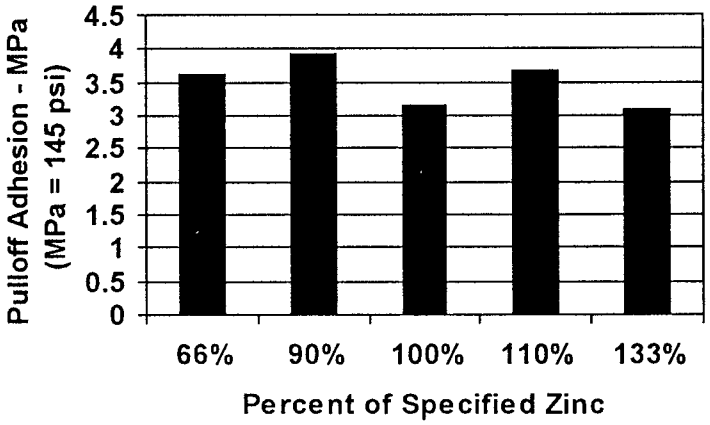


Figure 12. Acrylic topcoat adhesion over films with varying zinc content.

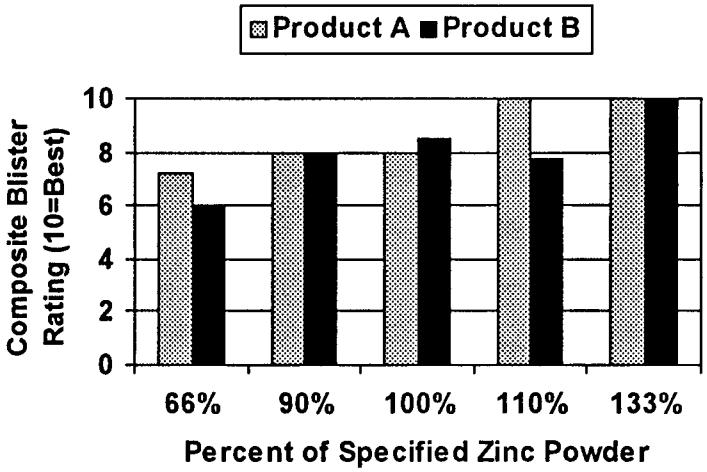


Figure 13. Freckle rusting observed on films with varying zinc content.

Figure 13 shows the freckle rusting observed as a function of the amount of zinc mixed with the binder. There is a very clear trend between zinc content and degree of freckle rusting. It is particularly interesting to note that slightly more than the specified amount of zinc was required to eliminate freckle rusting in these tests. This suggests that coating manufacturers should re-visit the coating formulation with the intent of being able to tolerate lower than specified zinc loading.

Primer Wash Testing Results

Figure 14 shows the surface pH values measured for each of the conditions tested. The data show that neither pressure washing nor a 24-hour cure under laboratory ambient conditions resulted in a lower surface pH. On the other hand, both a 24-hour marine exposure and rinsing with 14 percent phosphoric acid reduced the surface pH below 6.0. The effect of marine exposure may be due to the acidic pH of surface moisture (typically between 4 and 5 at the Ocean City Research marine exposure site) but this must be explored further. When the post-cure solution was applied to the self cure coatings, the pH of Product B was reduced below 6.0 while the pH of Product A remained slightly alkaline. The surface pH of the post-cure was slightly below neutral.

Figure 15 shows the composite blister ratings (average of three panels) after 2 days of marine atmosphere exposure for each of the conditions tested. The panels with alkaline pH showed significant blistering (control and 300 psi [2.1 MPa] water rinse). Product A with post cure solution applied had an average composite blister rating near 8. It also had a slightly alkaline measured surface pH. This seems to suggest that a WBIOZ surface pH below neutral is necessary to minimize topcoat blistering.

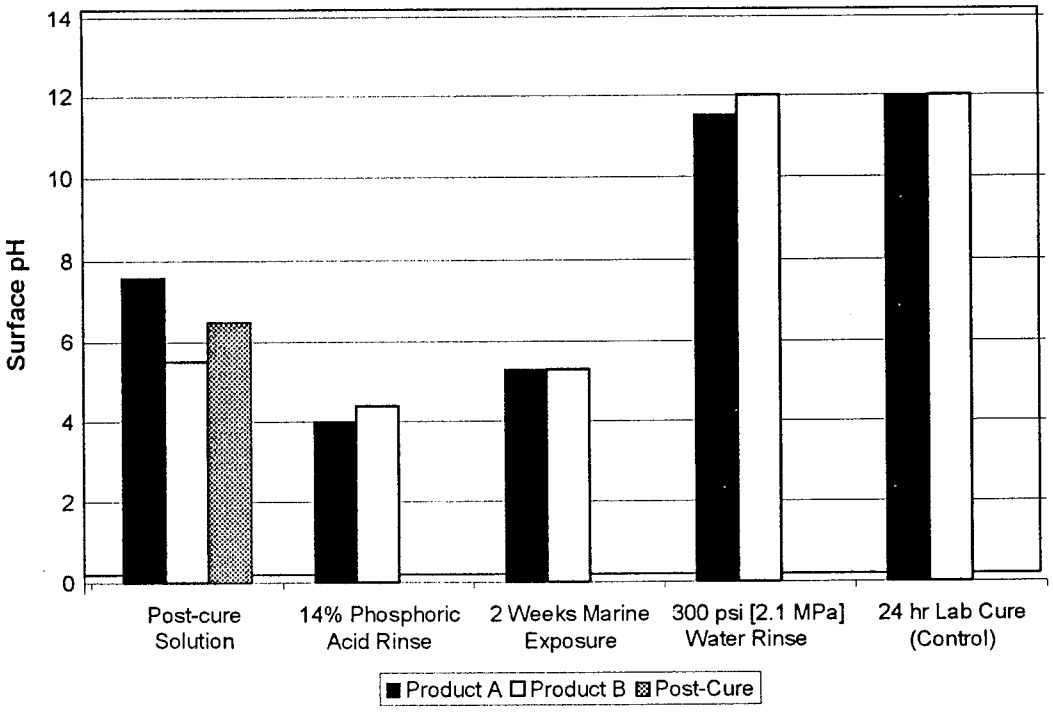


Figure 14. Surface pH measured after various primer treatments.

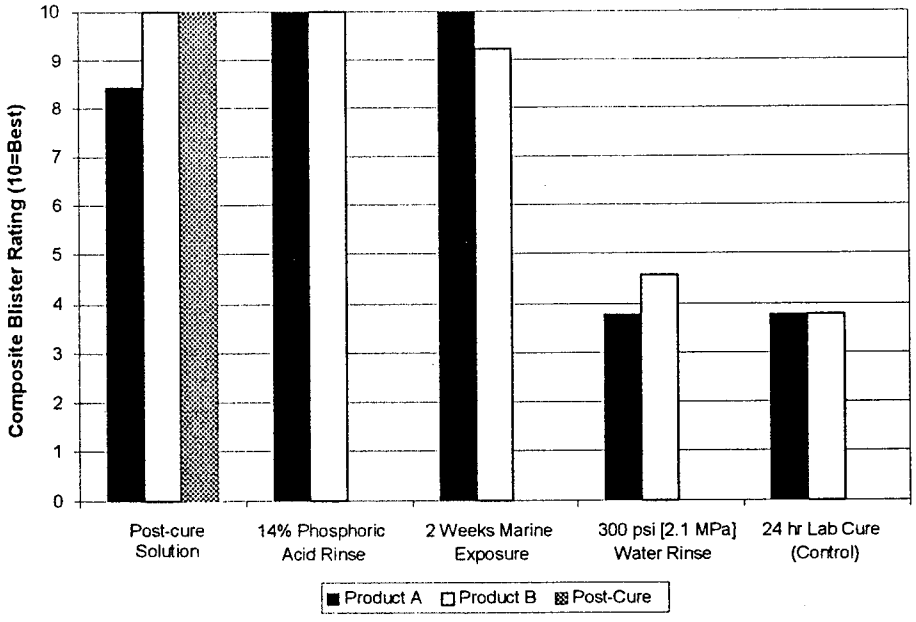


Figure 15. Composite blister ratings after 2-day marine atmosphere exposure.

Figure 16 shows the condensation blister ratings after 8 days of humidity exposure for each of the systems. Again a strong relationship between those conditions resulting in an alkaline pH and blistering is observed. Figure 17 shows the test panels after 12 days of condensing humidity exposure.

Figure 18 shows all blister ratings as a function of surface pH. There is a very clear relationship between blistering and alkaline pH. Acidic pH doesn't seem to correlate with an increase in blistering propensity. Figure 19 is a graph of pH observed as a function of time on triplicate test panels exposed at our accelerated marine atmosphere exposure test facility described in the experimental approach section. The data clearly show a trend to reduced surface pH as a function of exposure time. This shows that extended atmospheric exposure may be an acceptable way to weather WBIOZ before topcoating. Further testing would be necessary to determine what duration and environmental conditions are necessary to achieve acceptable conditions for topcoating.

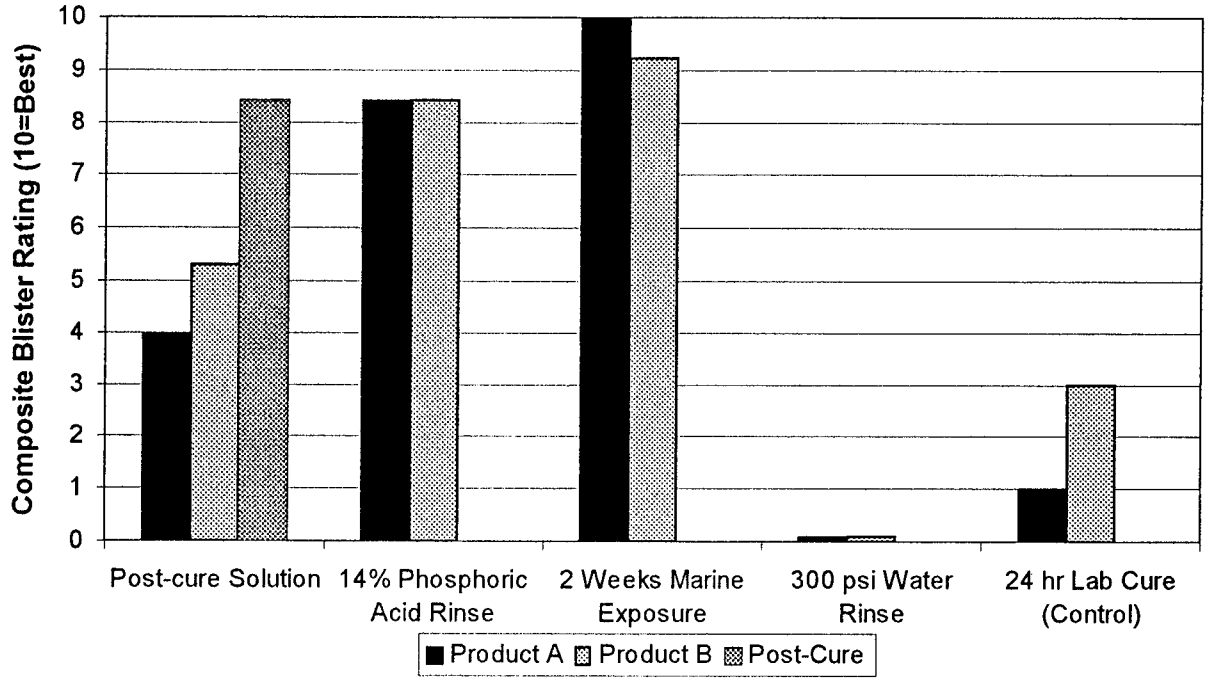
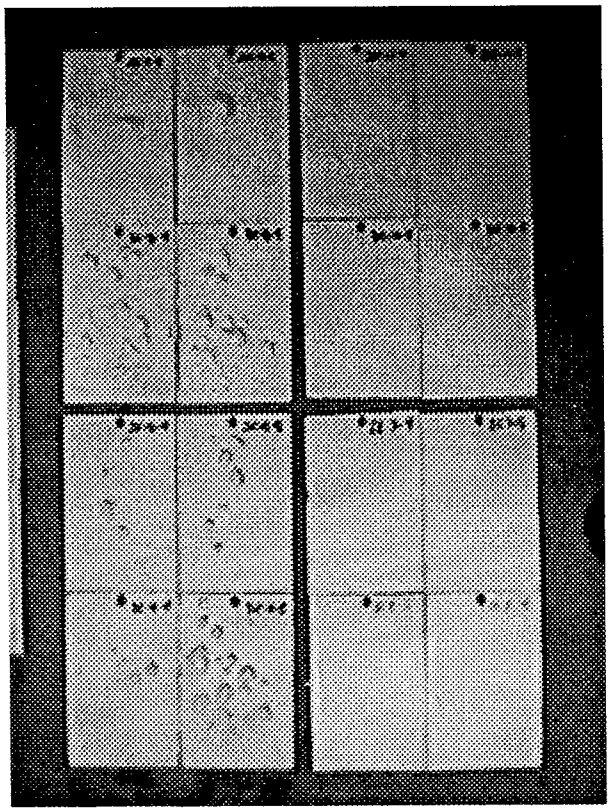


Figure 16. Composite blister ratings after 8-day condensing humidity exposure.



Clockwise from top left:

- Pressure washed resulting in a surface pH of 11.5 to 12.0
- 14 percent phosphoric acid rinse resulting in a surface pH of 4.0 to 4.4
- 2-week marine exposure resulting in a surface pH of 5.3
- 24-hour ambient laboratory exposure resulting in a surface pH of 12

Figure 17. Test panels after 12-day condensing humidity exposure.

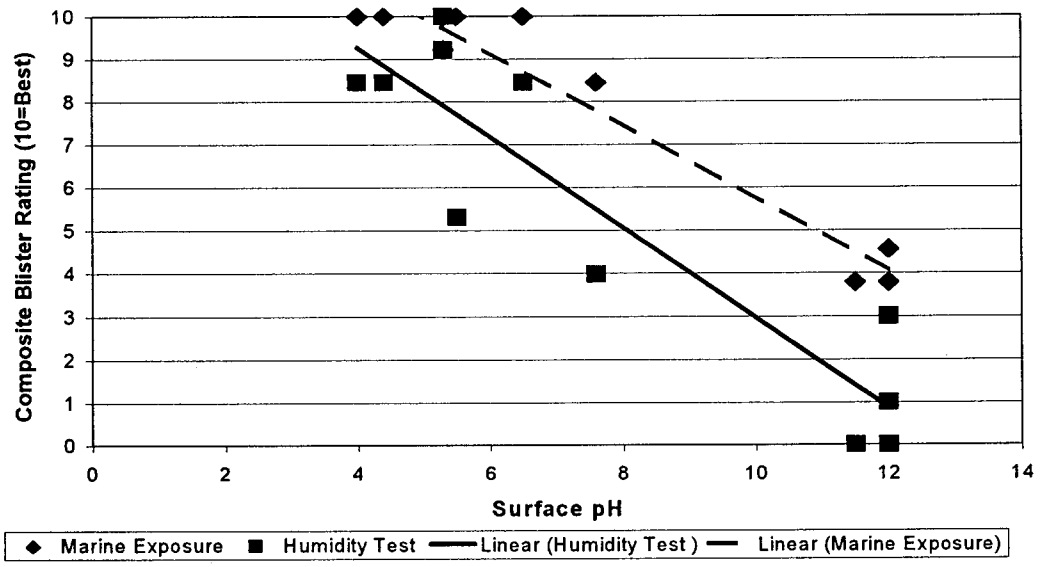


Figure 18. Relationship between surface pH and observed blistering.

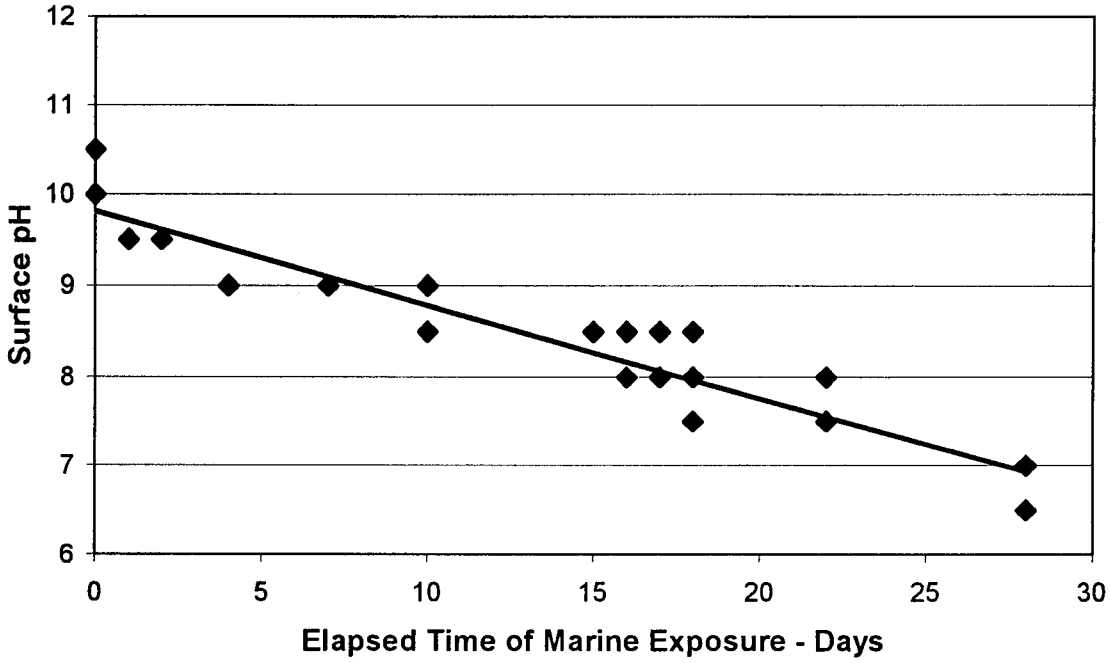


Figure 19. Surface pH versus time in a marine atmosphere.

GUIDELINES FOR THE USE OF WATER-BASED INORGANIC ZINC COATINGS

This section of the report addresses practical questions associated with the use of water-based inorganic zinc (WBIOZ) coatings. The information is presented in an easy to use question-and-answer format. There are several areas of discussion in this section including critical parameters for proper cure of WBIOZ, critical parameters for successful topcoat application, quality assurance, and corrective actions.

Critical Parameters for a Proper Cure

What are acceptable environmental conditions for curing WBIOZ coatings?

The *environmental constraints* that have been put forward by the coatings industry in and of themselves appear sufficient to obtain proper cure. However, other issues (i.e., time before topcoating, acceptable surface contamination, and tolerances for proper mixing) do not appear to be well addressed. Additionally, the extremes of these otherwise acceptable environmental ranges may aggravate other factors that contribute to WBIOZ failure. Thus, interactions between various application and cure parameters do not appear to be well addressed either.

The cure of WBIOZ includes two reactions. The first reaction is simply evaporation of water from the coating surface. The second reaction is the reversible condensation of silanol groups. It is logical that this reaction depends on both temperature and availability of water. Thus temperature and humidity would seem to be the important environmental factors to control for proper curing. However, differential partial pressure is a better metric to determine the rate of the first reaction (i.e., water evaporation).

The differential partial pressure can be calculated quite simply from a psychometric chart. For a given condition of temperature and humidity, there is a unique partial pressure of water, typically expressed in psi or inches Hg. If we assume that the coating contains water (saturated) at the surface temperature, we can obtain the partial pressure of water in the coating. The partial pressure of water in air is obtained knowing the ambient temperature and RH. The rate of drying is predicted based on the difference between these partial pressures. This is the driving factor that is forcing evaporation.

The present research suggests that the evaporation rate needs to be consistent with the coating temperature. That is, a lower temperature requires a lower evaporation rate. In the range of typical application temperatures, the data in this report indicates that you can have too high of an evaporation rate at low temperatures. Evaporation rates obtained at the recommended humidities and ambient (22 °C or 72 °F) temperatures appear acceptable. It is necessary that the coatings manufacturers conduct product specific research in this area to make further specific recommendations.

What causes the appearance of freckle rust after WBIOZ cure?

Freckle rust can result from a number of factors. This research and previous work showed the importance of proper zinc loading to obtaining satisfactory corrosion protection. Unfortunately the critical zinc level appears to be at or near the formulation level. This means that improper mixing or poor agitation may result in failure on an otherwise properly applied coating.

This research also showed that surface chloride levels as low as 5μg/cm^2 contribute to freckle rusting. While other contaminants (e.g., sulfates) were not studied, it is certainly possible that they would also contribute to the freckle rusting phenomena.

Finally, while improper environmental conditions alone did not contribute to freckle rusting, they did certainly increase the appearance of freckle rusting. For example, it is reasonable to expect that freckle rusting will increase under higher humidities if another incubating factor (low zinc in the film or surface salt contamination) is present.

Critical Parameters for Successful Topcoat Application

Are some topcoats less susceptible to blistering problems?

It would appear that water-based coatings (specifically acrylics) are more susceptible to blistering over WBIOZ than other topcoat materials. In the present research, no blistering of the solvent-based epoxy topcoat was observed. Furthermore, topcoat blistering problems reported in the literature and through industry contacts appear to be mostly acrylic topcoats. Unfortunately, the motive for using WBIOZ (reduced VOC emissions) is also a motive for the use of water-based acrylics.

What is the nature of acrylic topcoat blisters over WBIOZ?

The acrylic topcoat blisters observed in this program tended to appear early and occur under high wetness (i.e., morning dew, foggy days, or condensing humidity chamber). The blisters also tended to disappear with time. When popped early in their life, the blisters were water filled. As time went on, the blisters tended to be gas-filled. The research seems to corroborate that the blisters form because of alkali salts on the WBIOZ surface (i.e., blister formation correlated with observed surface pH). However, an exact blistering mechanism has not been definitively demonstrated by this or other research.

How can I reduce the likelihood of acrylic topcoat blistering?

The present research suggests that reducing the alkaline salts on the WBIOZ surface will decrease the likelihood of acrylic topcoat blistering. There is limited information on the methods to reduce the surface pH. This research showed that a dilute acid rinse or

prolonged weathering (on the order of weeks in a marine environment) acceptably reduces the surface pH. Neither high-pressure water washing nor 24-hour ambient cure were, by themselves, able to significantly reduce the surface pH.

In essence, reduction of the likelihood of acrylic blistering seems to relate to an increased level of WBIOZ cure. Additional research needs to be done to define methods that will accelerate thorough curing of the WBIOZ primer.

Quality Assurance

What are critical QA checkpoints for steel surface preparation before applying WBIOZ?

The prepared steel surface should be grit blasted to produce a sharp angular profile with a depth of 38 to 76μm [1.5 to 3 mils]. If shot is used to clean the steel (e.g., in a wheelabrator), it is desirable to follow with a grit blast to produce a sharper profile. The steel surface should be free of visible contaminants, conforming to SSPC SP-10, *Near White Metal Blast Cleaning* or SP-5, *White Metal Blast Cleaning.*

It is also important that the steel surface be free of soluble salts and other invisible contaminants. These may be deposited from the atmosphere, from impure blast media, or from other sources. The detection of invisible surface contaminants is relatively new. Most methods focus on determining surface chlorides. It is important to note that chlorides are not the only salts that may be present. It may therefore be preferable to use an inspection method based on resistivity or conductivity. These methods are not able to distinguish among different ionic species (e.g., chlorides), but indicate what level of total ionic species might be present.

What QA methods need to be conducted during WBIOZ coating application?

During WBIOZ application, the temperature, humidity, wet film thicknesses, zinc content of dry film, and degree of mixing should be closely monitored. Manufacturer data appear adequate to define temperatures and humidity ranges, however other parameters are left undefined (e.g., cure time and interrelationship of temperature and humidity–see above discussion). The limits typically require temperatures above 10 °C (50 °F), relative humidity below 85 percent, and a surface temperature 2.8 °C (37 °F) above the dew point.

There do not appear to be any quality assurance procedures to determine if the proper quantity of zinc is in the wet or dry film. The present study used energy dispersive x-ray spectrometer to analyze the zinc content in the dry film. Unfortunately, these methods are not yet practical for use in the field. However, differential scanning calorimetry and titration techniques can also be used to quantify zinc content. As this stage, it appears

that monitoring mixing is the only practical way of ensuring the zinc content is correct. This would include:

- Requiring that zinc and liquid be accurately measured before mixing.
- Specifying tolerances for zinc and liquid portions to be mixed.
- Requiring that zinc additions be made to compensate for any zinc removed during straining.
- Requiring that agitated pots be used whenever WBIOZ is sprayed.
- Monitoring disposed paint to ensure that zinc settling did not occur.
- Closely inspecting surface for freckle rusting before applying topcoat or putting the coating into service.

What QA steps are appropriate before topcoating WBIOZ?

Before applying a topcoat, the WBIOZ primer should be closely inspected for visual defects. In particular, areas with mud-cracking, freckle rusting, or micro-cracking (visible with 10X magnification) should be identified. Dry film thickness measurements should also be made to determine that the coating thickness is in the specified range. Significant visual defects and areas of inadequate thickness should be repaired before topcoating (see **Corrective Actions**, below).

Data in this report suggest that a surface pH between 4 and 7 minimizes the occurrence of blistering. Standards do not exist for measurement of the surface pH of WBIOZ coatings. In this study, pH was measured using a procedure based on ASTM D 4262-83, *Standard Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces*. The technique involves pressing a strip of pH paper against the surface and wetting the area with deionized water. Color change of the strip is compared with a standard to determine pH. Paper with accuracy of 0.2 pH is commercially available. This research did not conclusively demonstrate an acceptable pH value.

What QA steps are necessary before exposing WBIOZ to service environment?

After curing, the WBIOZ primer should be closely inspected for visual defects. In particular, areas with mud-cracking, freckle rusting, or microcracking (visible with 10X magnification) should be identified. Dry film thickness measurements should also be made to determine that the coating thickness is in the specified range. Significant visual defects and areas of inadequate thickness should be repaired before exposure in the service environment (see **Corrective Actions**, below).

Measurement of zinc content in the dry film may also be an appropriate QA step if field-friendly methods can be developed. Current methods, while possible to use in the field, are likely to be too complex for use as QA tools by coating inspectors on a regular basis.

What nuances as associated with spraying WBIOZ coatings would be helpful to a painter inexperienced with the material?

There are several important aspects to applying WBIOZ coatings that do not apply or are not as important during application of traditional organic coatings. The three most vital of these include *mixing, material agitation/settling, and a wet application.*

Proper mixing of the liquid binder and the zinc powder is critical. The zinc powder is typically passed through a paint strainer to remove any clumps or large particles of zinc. Patience must be used to strain the zinc. It is easy to toss the strainer away before much of the zinc has passed through. The proper level of zinc in the binder is critical to overall coating performance.

Inorganic zinc requires agitation after mixing to maintain uniformity in the mixed material. The density of the zinc pigment causes it to settle and locally collect within the application equipment. This may be in the paint pot or in the coating material lines. Keeping the mixed material moving is the best way to avoid these effects. Keep breaks or downtime to a minimum when painting. The zinc will settle both in the paint pot and in the material lines. If breaks occur, ensure the lines are cleared and pot agitation has occured before continued painting.

Typically the application instructions for inorganic zinc coatings instruct the applicator to "apply a wet coat in even parallel passes." This is especially critical with WBIOZ coatings that dry very fast (seconds). Much of the water evaporation can happen as the material is being propelled towards the surface. To avoid dry spraying WBIOZ coatings, adjust the application equipment to apply enough material in a single 50-percent overlap pass. This means that a single pass over the substrate applies enough material to reach half of the required dry film thickness. The overlapping of successive spray passes will build the application to the proper film thickness.

Corrective Actions

What is the recommended course of action if freckle rust occurs?

Depending on the extent of freckle rusting, the criticality of the application, and the contract requirements, repair of freckle rusting may or may not be warranted. It is recommended that the extent of deterioration requiring repair be well defined in any contract for work. Requirements might be stated as "Any area in excess of 0.1 m^2

exhibiting freckle rusting over 0.1 percent of the area shall be repaired."

If repair of freckle rusting is warranted, the recommended repair procedure would be to re-blast areas requiring repair and repaint with WBIOZ. Of course, all QA requirements should be enforced during the repair.

What is the recommended course of action if topcoat blistering occurs?

Topcoat blisters tended to disappear with time. The aesthetic impact of a shrunken blister varies. Some blisters pop, revealing WBIOZ primer. Some blisters shrink back, leaving a barely distinguishable wrinkle where the blister once was. Still other blisters shrink back and leave no visible trace of their presence. Of course, all blistered coating surfaces have a reduced adhesion to the WBIOZ primer.

Keeping in mind that topcoats are applied for aesthetic and barrier purposes (WBIOZ coatings themselves provide good corrosion protection), one must determine if the blistered topcoat is truly a problem. If necessary, repair should include removal of the topcoat and recoating. If the topcoat can be removed without damaging the primer (perhaps with brush blasting or a chemical stripper), a new topcoat can probably be applied over the original primer. If topcoat removal requires removal or damage to the primer, a new primer and topcoat should be applied. Of course, all repair procedures should include the QA requirements of the originally applied coating.

APPENDIX I—INITIAL PANEL QUALITY CONTROL DATA

Table 13. Untopcoated panel quality control data.

Coating	Condition	Nickel Rub	Pencil Hardness (ASTM D3363 - scratch)	Avg. DFT (mils)
post-cure	ambient control	Pass	6H	3.3
Product B	1	Fail	2H	4.3
Product B	2	Fail	6H	2.7
Product B	3	Fail	6H	4.2
Product B	4	Fail	6H	3.1
Product B	5	Fail	2H	4.7
Product B	6	Fail	6H	3.5
Product B	7	Fail	4H	2.4
Product B	8	Fail	6H	2.3
Product A	1	Fail	4H	4.4
Product A	2	Pass	6H	3.8
Product A	3	Fail	2H	4.8
Product A	4	Fail	6H	4.2
Product A	5	Fail	4H	3.5
Product A	6	Pass	6H	3.5
Product A	7	Pass	2H	4.1
Product A	8	Fail	6H	7.1

Table 14. Acrylic-topcoated panel quality control data.

Coating	Condition	Cross Hatch Adhesion (ASTM D3359)	Avg. DFT (mils)
post-cure/acrylic	Ambient control	4B	6.4
Product B/acrylic	1	3B	5.0
Product B/acrylic	2	4B	6.6
Product B/acrylic	3	4.5B	6.9
Product B/acrylic	4	2B	6.0
Product B/acrylic	5	3B	4.3
Product B/acrylic	6	0B	6.6
Product B/acrylic	7	4B	4.9
Product B/acrylic	8	2B	5.5
Product A/acrylic	1	5B	6.1
Product A/acrylic	2	2.5B	8.8
Product A/acrylic	3	4.5B	6.1
Product A/acrylic	4	0B	6.4
Product A/acrylic	5	2.5B	5.1
Product A/acrylic	6	3.5B	6.7
Product A/acrylic	7	3.5B	4.7
Product A/acrylic	8	0B	7.9

Table 15. Epoxy-topcoated panel quality control data.

Coating	Condition	Cross Hatch Adhesion (ASTM D3359)	Avg. DFT (mils)
post-cure/epoxy	Ambient control	2.5B	6.6
Product B/epoxy	1	5B	9.3
Product B/epoxy	2	4B	9.0
Product B/epoxy	3	4B	6.2
Product B/epoxy	4	4B	6.5
Product B/epoxy	5	5B	6.6
Product B/epoxy	6	4.5B	11.1
Product B/epoxy	7	3B	6.8
Product B/epoxy	8	3B	6.9
Product A/epoxy	1	4.5B	8.5
Product A/epoxy	2	3.5B	8.1
Product A/epoxy	3	5B	7.7
Product A/epoxy	4	1B	7.2
Product A/epoxy	5	5B	8.0
Product A/epoxy	6	4B	6.5
Product A/epoxy	7	4.5B	6.8
Product A/epoxy	8	3B	5.7

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