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High Solids and Zinc-rich Epoxy Coatings for Corps of Engineers Civil Works Structures

by Alfred D. Beitelman and Dennis Huffman
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Prepared for Headquarters, U.S. Army Corps of Engineers

Repair, Evaluation, Maintenance, and Rehabilitation Research Program

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Preface

This study was authorized by Headquarters, U.S. Army Corps of Engineers (HQUSACE), as part of the Electrical and Mechanical problem area of the Repair, Evaluation, Maintenance, and Rehabilitation (REMR) Research Program. The work was performed under the Civil Works Research Unit 32667, "Universal VOC Compliant Coating System for Locks and Dams for which Mr. Alfred D. Beitelman is the principal investigator. Mr. John Gilson (CECW-EE) is the technical monitor for this work.

Mr. David B. Mathis (CERD-C) is the REMR Coordinator of the Directorate of Research and Development, HQUSACE; Mr. Jim Crews and Mr. Tony C. Liu (CECW-EG) serve as the REMR Overview Committee; Mr. William F. McCleese, U.S. Army Engineer Waterways Experiment Station, is the REMR Program Manager; Mr. Alfred D. Beitelman is the Problem Area Leader for the Electrical and Mechanical problem area.

This work was conducted by the U.S. Army Construction Engineering Research Laboratories (USACERL) under the general supervision of Dr. Ellen Segan, Acting Chief, Materials Science and Technology Division (FL-M). The technical editor was Linda L. Wheatley, Technical Information Team.

COL James T. Scott is Commander and Acting Director of USACERL, and Dr. Michael J. O'Connor is Technical Director.

Conversion Factors, Non-SI to SI Units of Measurement

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or Kelvin[1]
gallons (U.S. liquid)	3.785412	liters
inches	25.4	millimeters
mils	0.0254	millimeters
pounds	453.6	grams
centipoise	$1X10^{-3}$	pascal seconds
pounds/square inch	6.894	kilopascals

[1] To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: C = (5/9) (F - 32). To obtain Kelvin (K) readings, use: K = (5/9) (F - 32) + 273.15.

1 Introduction

Background

For many years, the Army Corps of Engineers has relied heavily on the use of vinyl paints for coating hydraulic structures. The systems have performed well in many environments, however their high solvent content has made their use illegal under some local air pollution control legislation. As a result, alternative systems that are environmentally safer were sought. An early study by the Corps of Engineers (Baker and Beitelman 1992) investigated a number of proprietary coatings meeting the more restrictive air pollution regulations. This study coupled with field evaluations concluded that high solids epoxies held the best hope as replacement coatings systems. Coatings currently available in specifications E-303d and MIL-P-24441 provided a level of protection equal or superior to any of the proprietary coatings evaluated. These coating systems are listed in the Civil Works Guide Specification (CWGS) 09940, *Painting: Hydraulic Structures and Appurtenant Works*, as System No. 21 AZ and System No. 21 BZ.

Experience has shown that high performance coatings of this type are often applied to excessive thicknesses, in weather that is either too hot or too cold, over improperly prepared substrates or in adverse weather conditions. It was a concern that these improper application procedures could result in poor adhesion of the coating and would ultimately lead to failures, thus resulting in damage to the underlying structure. Evaluating these epoxy coatings was necessary to respond to field inquiries relating to specific application irregularities and anticipated problems.

Objective

The objective of this investigation was to evaluate the application parameters of the currently used E-303d (and the proposed MIL-P-24441/19 [Formula 159]) zinc-rich primers, and of MIL-P-24441 Type IV Formula 150 primer/Formula 151 top-coat epoxy polyamide coatings.

Approach

Laboratory test methods were developed to simulate the poor application and poor application environment often encountered on hydraulic structures. Heavy emphasis was placed on the coatings' physical properties, including the spraying and drying characteristics. Application variables included the following: high and low temperature application, excessively low and high thickness variation, poor surface preparation, improper thinning, improper spray gun-to-substrate distance, ultraviolet (UV) exposure on primer, and extended cure time between coats. The adhesion was evaluated and all of the coatings were exposed to laboratory environments that simulated actual conditions found on hydraulic structures.

Products Tested

Appendix A shows the formula for the epoxy zinc-rich paint E-303d. The batch of material used in this work was manufactured by Kop-Coat Inc., Pittsburgh, PA for a specific Army Corps of Engineers job. The batch was tested and found to comply with all specification requirements. Appendix B shows the E-303d test report. The other zinc-rich primer, MIL-P-24441/19 (Formula 159), was prepared in the laboratory according to the formula listed in the MIL-P-24441/19 specification (Appendix C). Appendix C also includes the formulas for MIL-P-24441 Type IV Formula 150 primer and Formula 151 topcoat. The products used were manufactured by Ameron Inc., Brea, CA.

2 Preparation and Procedure

Panel Preparation

All the coatings were applied to abrasive blasted, 3 x 9 x 1/16 inch cold-rolled steel panels. These panels were first wiped with a 50/50 mixture of Methyl Isoamyl Ketone (MIAK) and an aromatic naphtha (HI-SOL 10) to remove any deposits of oil or grease. After solvent cleaning, the panels were abrasive blasted to SSPC-SP5 White Metal Blast Cleaning, using a suction-feed abrasive blast cabinet. Arrowblast #24 aluminum oxide abrasive was used. Surface profile measurements were taken according to American Society for Testing and Materials (ASTM) D 4417 Method C (Replica Tape); profile measurements proved to be in the 1 to 2 mil range. All coatings were applied using a DeVilbiss MBC conventional spray gun. This spray gun was equipped with a DeVilbiss MBC-444E needle, a 0.070 inch fluid tip, and a #30 air cap. The air pressure was maintained at 55 psi. A standard gun-to-substrate distance of 8 to 10 inches was maintained except as noted. To aid in the dispersal of the zinc dust, a 1-quart agitator cup was used. Viscosity readings were taken according to ASTM D 1200 using a #4 Ford cup. The E-303d was thinned according to specification requirements. Formula 159 was thinned with the specification thinner (Appendix C). The zinc-rich paints required the viscosity be reduced with thinner to 15 seconds while mixtures of Formula 150 and 151 were thinned with naphtha and butanol to a viscosity of 60 sec. (i.e., thinning of Formula 150 approximately 25 percent by volume and thinning Formula 151 approximately 20 percent by volume). The coatings were sprayed at average laboratory temperatures (approximately 70 °F). A Positector 5000 thickness gage was used to measure the dried film thickness according to ASTM D 1186 Method B. After a cure time of 1 week, a diagonal cut (approximately 3 inches long) was made on the lower half of one side of each exposure panel. This cut extended through the surface of the coating to the substrate.

Testing Procedure

Adhesion tests

The E-303d and the Formula 159 were cured and then subjected to the cross cut adhesion test according to ASTM D 3359 Method B. This test requires scoring the coating and using an adhesive tape to attempt to remove the coating in the scored area. Numerical values are given to the result with "5" being no removal and "0" being the greatest level of failure evaluated by the test. The Formula 150 primer and Formula 151 topcoat were subjected to a pulloff adhesion according to ASTM D 4541. Circular aluminum test fixtures ("dollies") required for the test were affixed to coating surfaces using 3M Scotch Weld 1838 epoxy cement. To promote adhesion of the dolly to the coating surface to be tested, the dollies were roughened on the contact side prior to applying the epoxy cement. The dollies were secured to the painted test panels using C-clamps in order to apply firm pressure on both epoxy-cemented contact surfaces while the adhesive cured for 24 hours to its full strength. Finally the dollies were loaded in tension and pulled from their coated substrates. All adhesion tests were run in triplicate.

Environmental testing

Following application and cure, test panels were exposed to several environments in an effort to observe the effects of the irregular application or cure conditions. All panels were evaluated after 30 days and returned to the given exposure. The panels were given an additional visual examination after 6 months and will remain in test indefinitely to determine long-term performance of the coatings. Exposure conditions included: warm (85 °F) aerated tap water immersion, cold (75 °F) aerated tap water immersion, cold (70 °F) aerated synthetic sea water immersion (ASTM D 1140), and atmospheric exposure (ASTM G 7; 45° south, Champaign, IL). The panels will remain in test until a failure is noted. Evidence of failure is detected through visual inspection. Signs of failures included: color variations, blisters, poor adhesion (e.g., peeling or flaking of the coating), any difference in texture, and presence of "chalking" or any other abnormal films on the surface.

Application temperatures

Various high and low temperature variables were investigated to determine if this system is susceptible to incomplete cure or premature adhesion failures when substrate, primer, and topcoat are subjected to unusually high or low temperatures during application and cure.

The elevated temperature application testing was conducted using a Fisher Iso-Temp 400 Series convection oven. The panels to be coated were put into the oven for 7 hours at the temperatures indicated in Table 1. The paint was mixed and allowed to stand in the indicated temperatures for a period of 2 hours before application. Temperatures were increased in 10 °F increments until a satisfactory application could not be obtained. At each temperature the viscosity of the paint was rechecked to ensure that it was still satisfactory for spray application. If the viscosity had increased, sufficient thinner was added to restore the original viscosity. After application of the first coat of Formula 150, the panels were returned to the oven at the given temperature for a drying time of 1 to 3 days before the application of the Formula 151 topcoat. A dry-film thickness of 3 (± 0.2) mils per coat was obtained on all panels. After spraying, all panels were cured for 1 week at their respective temperatures, and the adhesion was checked preceding exposure.

The low temperature application testing was conducted using a constant temperature cabinet. Both the mixed paint and the panels to be coated were put into the cabinet for 7 hours at the settings indicated in Table 1. Temperatures were decreased in 10 °F increments until satisfactory application or cure could not be obtained. At each temperature, the viscosity of the paint was rechecked to ensure that it was still satisfactory for spray application. If the viscosity had increased, sufficient thinner was added to restore the original viscosity. After application of the zinc-rich coating, the panels were cured at the given temperature for 1 week. The Formula 150 primer was cured at the given temperature for 1 week before the application of the Formula 151 topcoat and for an additional week at 70 °F before performing the adhesion test. A dry-film thickness of 3 (± 0.2) mils per coat was obtained on all panels. The adhesion of all coatings was evaluated and the panels were placed into the indicated tests.

Application thickness

A series of panels was prepared varying thickness of each coat. This test was performed to determine if excessively thin or thick coatings are more susceptible to failure. All coatings were applied at 70 °F. The Formula 151 topcoat was applied no sooner than 24 hours, but no later than 96 hours, after the primer; all topcoats were allowed to cure. After a 1-week cure time, the adhesion of all coatings was evaluated as specified, and the panels were placed into the indicated tests.

Only the zinc-rich coatings were evaluated at reduced film thickness. The paint was thinned to standard viscosity and applied at a thickness of 1 (± 0.2) mil. After application the panels were cured for 1 week at standard laboratory conditions.

Initial work with the zinc-rich coatings indicated no noticeable effect of minor increases in coating thickness, so a set of panels was prepared with a single-coat thickness of 10 (± 0.2) mils. The panels for the Formula 150/151 system were prepared with each coating at 2x, 3x, and 4x the standard 3-mil thickness. All coatings were cured for 1 week at 70 °F.

Incomplete surface preparation

An improperly cleaned testing series was performed to determine the effect improperly cleaned substrates would have on E-303d and Formula 159 only. To simulate the effects of improper blasting, three different techniques were used to prepare the panels. Initially, all panels were SSPC-SP5 White Metal Blasted using the same abrasive and suction-feed abrasive blast cabinet as detailed in the Panel Preparation section. The panels were divided into three groups as follow:

a. The first group of panels was placed outside on an atmospheric testing rack and misted with deionized water for approximately 1 hour until flash rust appeared. After rusting, both coatings were applied (3 mils) and allowed to cure for 1 week at standard laboratory conditions. After curing, the adhesion of the coatings was evaluated, and the coatings were exposed in the indicated environments.

b. The second group of panels was also placed on the atmospheric testing rack after blasting. These panels were misted with deionized water three times daily for 2 weeks until 100 percent rust (no pitting) was present. Both coatings were applied (3 mils) and allowed to cure for 1 week. After curing, the coatings were evaluated for adhesion as noted and were exposed in the respective environments.

c. The third group of panels was placed on the atmospheric testing rack for normal ambient exposure for 6 months (summer and fall). The surface of the panels became 100 percent rusted with pitting. The top half of this group of panels was abrasive blasted (with the same equipment and grit as previously stated) to the requirements of SSPC-SP7, Brush Off Blast. The lower half of each panel was left unprepared. Both paints were then applied (3 mils) and cured for 1 week. After curing, the adhesion was evaluated, and the coatings were exposed in the indicated environments.

Thinning variation of zinc-rich primers

The effect of varying the amount of thinner added to the zinc-rich primers was tested to determine if high levels of thinning would have any ill effect. Both paints were thinned with the specified thinner to progressively lower viscosities until a failure during application was evident. A failure during application was identified when the paint became too thin to hold the zinc onto the vertical panel surface. Multiple coats were applied as necessary to obtain the required 3-mil thickness. After application, the adhesion of all panels was checked using ASTM D 3359 Method B and the panels were placed into their respective exposures.

Overspray resistance of zinc-rich primers

The ability of the zinc-rich primers to resist dry spray failure was evaluated. The paints were thinned to standard spraying viscosity, and applied at a spraying distance of 17 to 20 inches from the panels. After application, the panels were allowed to cure at standard laboratory ambient conditions for 1 week. The adhesion of the coatings was measured according to ASTM D 3359 Method B, and the panels were given a qualitative visual examination during and after application. During application, it was noted if the paint was not sufficiently thinned to provide a wet spray and avoid deposition of particles that were semi-dry when they struck the surface. After application, each panel as examined for a lack of gloss and the presence of a sandy or course texture typically found in overspray/dryspray situations. The panels were then exposed to their respective environments.

Effect of UV radiation on MIL-P-24441

A series of test panels was prepared using Formulas 150 and 151 to determine if coating adhesion is reduced due to UV-induced chalking of the primer (as would occur if the primers were subjected to long periods of sunlight prior to topcoating). Test panels were prime-coated at normal dry-film thicknesses and cured in an Atlas UVCON Model UV-1 Ultraviolet/Condensation Cabinet using the UV test as per ASTM G 53 for periods of 1, 2, 3, and 4 weeks before topcoating. Topcoats were allowed to cure under ambient laboratory conditions for 1 week. The adhesion of the coatings was evaluated according to ASTM D 4541.

Effect of long cure time between coats of MIL-P-24441

A series of panels was prepared to determine if coating adhesion is reduced due to excessive hardening of the Formula 150 primer as a result of a long cure time. Panels were prime-coated and cured in ambient laboratory conditions for 1, 2, 3, and 4 weeks before topcoating. All coatings were applied under normal dry-film thickness conditions. The topcoat was allowed to cure for 1 week under ambient laboratory conditions. The adhesion was evaluated as indicated and the panels were placed into the respective exposures.

3 Results

Application Variations

Elevated temperature application

During application of the elevated temperature series, additional thinner was necessary at increased temperatures for both the primers and the topcoats. Problems such as a buildup of dried paint on the tip of the gun, and large particles of coagulated paint resulting in internal clogging of the gun became apparent at temperatures of 110 °F. As long as the mixture flowed through the spray gun, the clogging was not readily apparent; however, the clogging became evident as soon as the spray gun was set aside for more than 10 sec at the highest temperatures. As spraying temperatures exceeded 120 °F, these application difficulties occurred more often and became more difficult to correct. Additional thinner and a closer gun-to-substrate distance (6 to 8 in.) were required at the two highest temperatures. All four paints dried quickly after application at these higher temperatures (almost as soon as they struck the substrate), making it difficult to get good wet-film thickness readings and leaving a course texture. Results of the elevated temperature application series (Table 1) show no degradation in adhesion values for either of the zinc-rich primers for temperatures up to 110 °F. Adhesion values were significantly reduced at 120 °F and 130 °F, and application failure was found at 140 F. At this temperature, neither the Formula 159 nor the E-303d could be applied due to the extremely rapid evaporation of the solvents.

Table 1 shows the results of the adhesion tests of Formulas 150 and 151 applied and cured at elevated temperatures. The data shows both the adhesion (in psi) at the point of failure as well as the type of failure observed. Failures were observed at three locations: (a) at the dolly or within the epoxy adhesive, (b) within the topcoat, and (c) at the interface between the topcoat and the primer. In two instances, failures were recorded at the primer-substrate interface or within the primer itself. Most of the results show some level of failure of the topcoat. Dollies having this failure usually had a quite thin layer of the topcoat adhering to the recorded area of the dolly. In some instances, the topcoat was in a continuous area; in other instances, the topcoat was seen as dozens of small islands adhered to the dolly. The individual adhesion results varied widely ranging from a high of 550 psi to a low of 150 psi. The majority of the results, as well as the averaged results, fell in the 325 to 400 psi range. The test method does not indicate the precision or bias however, experience indicates this range may be within the reproducibility for the test.

The exposure results of the elevated temperature panels after 12 months in the indicated environments proved excellent. No paint showed any sign of failure, including the systems applied at the highest temperatures. Slight corrosion was noticed at the scored area on all panels subjected to salt water immersion. Slight chalking was also noticed on the systems that were atmospherically exposed.

Low temperature application

For the low temperature applications, additional thinner was not necessary to maintain the workability of the zinc-rich paints. Table 1 shows adhesion values for the coatings. The results for the 70 °F panels were excellent. Only a slight decrease in adhesion was noted for both coatings when applied at 50 °F. At 40 °F, both materials could be spray applied; however, neither of the zinc-rich primers attained a suitable degree of cure. An incomplete cure was identified if the coating felt sticky or tacky to the touch or was soft and deformed easily under light to moderate finger pressure. After 1 week of cure at this temperature, the coatings were still soft enough to be removed with moderate thumbnail pressure. No attempt was made to conduct the adhesion test.

At temperatures of 60 °F and below, the Formula 150 took at least 1 week to cure. A complete cure was not obtained for the system at 30 °F. Table 1 shows the adhesion values for Formula 150/151 systems. Intercoat adhesion failed in only two cases. The majority of the failures were within the topcoat at all temperatures. The adhesion values ranged from a high of 500 psi to a low of 150 psi.

The exposure results after 12 months of the low temperature application were consistent with the elevated temperature application. No system showed any obvious signs of failure. Slight corrosion was again observed at the score on all panels immersed in salt water, and slight chalking was also noticed on panels exposed atmospherically.

Thin and thick applications

During the thin and thick application series, no modification of thinning procedures was needed. In all cases, a multiple-pass application procedure was used to apply the thick coatings without creating runs or sags. The adhesion of the thin applications showed only a minor difference between the two zinc primers. The 4B values recorded for the thick applications represent less than 5 percent coating removal. Close examination of the test areas indicated that most of the coating removed was probably due to the action of the cutter on the brittle coating and not to the pulling action of the tape.

Table 2 shows the results of the thick applications of the Formula 150/151 Systems. The adhesion values for this series ranged from a high of 595 psi to a low of 350 psi. The 595 psi was found in the 2x thickness, and was the highest of all of the recorded adhesion values.

The exposure results of both the thin and thick applications indicated no failures after 12 months' exposure to the indicated environments. Slight undercutting was present at the scored area on the thickest panels, which were salt-water immersed. Heavy chalking was present on the thickest panels (3X and 4X) subjected to atmospheric exposure. Neither of these conditions was deemed detrimental to the protective properties of the paint systems.

Other Variations and Effects

Incomplete Surface Preparation

During the application of the zinc-rich primers, both materials tended to resist flowout on the poorly cleaned panels. The appearance immediately after application was somewhat more dry than when applied to a properly cleaned surface. Extra thinning was deemed to be necessary to increase the wetting capabilities and flow-out of both primers. Table 3 shows the results of the adhesion test. The 4B rating of both the Formula 159 and the E-303d probably reflect the brittleness of the coatings rather than an actual reduced adhesion. The 3B ratings on the 100 percent rust and pitting panels appeared to be a loss of adhesion to the substrate. A slightly course texture was also noted on the unprepared lower half of these panels.

When examined after immersion exposure, the poorly cleaned panels (panel groups B and C) had a noticeable increase in corrosion around the score, and slight to moderate undercutting was also present. An increased level of corrosion and undercutting was also noted on panel groups B and C for the atmospherically exposed panels.

Effects of thinning zinc-rich primers

Table 4 shows the results of the thinning tests. It was found that E-303d would not flow properly when applied with 10 percent thinning. The adhesion data indicates 40 to 60 percent thinning by volume produced greatest adhesion of E-303d. Formula 159 had the greatest adhesion when thinned 20 to 30 percent by volume. As more thinner was added to each paint, more caution was necessary during application because of the greater tendency of the zinc to run and sag. When Formula 159 was sprayed at 60 percent, thinning the sagging was so extreme that the zinc appeared to flow off the panel leaving no appreciable film build. At 100 percent thinning by volume, the E-303d displayed a significant decrease in adhesion.

Exposure results of the thinning series showed no signs of failure. Corrosion was again present on the scored area of the immersed panels. Chalking on the atmospheric panels was not readily observed.

Overspray resistance of zinc-rich primers

Both Formula 159 and E-303d were thinned the normal amount and applied at approximately double the standard spray distance. Using this procedure resulted in similar problems for both materials. Extreme difficulties were noted in providing a good flow-out and the applied coatings had a dry, sandy texture. The adhesion results for both materials was 3B.

Corrosion on all immersed panels was moderate to heavy after exposure. Rust stains were extensive and covered the entire surface of these panels; evidence of generalized rusting of the entire panel. Chalking was not noted on any of the atmospherically exposed panels.

Effect of UV radiation on MIL-P-24441

After exposure in the UV chamber, all Formula 150 primers exhibited some level of chalking. Panels that were exposed to 3 and 4 weeks in the UV chamber were more heavily chalked than those exposed for only 1 or 2 weeks. The Formula 151 topcoats were applied and cured according to the test method and adhesion values were determined. Table 5 shows the adhesion test results. One loss of intercoat adhesion was noted when the primer was exposed for only 1 week; however, failure topcoat adhesion was noted as a mode of increased failure when the length of time in the UV chamber increased.

Exposure results of this series showed no noticeable increase in failure on any of the panels. Corrosion was evident only at the scored area on the immersed panels, and slight chalking was present on the atmospheric panels.

Effect of long cure time between coats of MIL-P-24441

Panels were coated with Formula 150 and cured for 1 to 4 weeks. The Formula 151 topcoats were applied and cured according to the test method, and adhesion values were determined. Table 6 shows the adhesion test results. The results do not indicate that the length of cure had any effect on the intercoat adhesion of the system.

Exposure results were consistent with the results from the UV-radiated panels. This series showed no noticeable increase in failure on any of the panels. Corrosion was evident only at the scored area on the immersed panels, and slight chalking was present on the atmospheric panels.

Table 1
Results of Application and Temperature Cure Variation

	Zinc-Rich Primers		Formula 150/151 System				
Temperature (°F)	E-303d Adhesion	Formula 159 Adhesion	Primer Cure (days)	Adhesion (avg. psi)	% Topcoat	% Intercoat	% Primer
140	Failure	Failure					
130	2B	2B					
120	3B	2B	1	431.3	96.3	0	0
			2	375.0	43.8	0	0
			3	343.8	66.9	0	0
110	5B	5B	1	305.0	27.5	0	0
			2	390.0	58.8	4.2	0
			3	437.5	80.0	0	0
100	5B	5B	1	370.8	51.7	.8	0
			2	462.5	26.3	0	0
			3	425.0	88.8	0	0
90	5B	5B	1	368.8	60.0	0	0
			2	425.0	35.0	0	0
			3	400.0	47.5	0	0
70	5B	5B					
60			7	370.0	76.0	2.2	0
50	4B	4B	7	420.8	19.2	.8	0
40	No Cure	No Cure	7	358.3	40.0	0	0
30			7	No Cure			

Table 2
Effects of Application Thickness

	Zinc Rich Primers		Formula 150/151 System			
Dry Film Thickness (DFT)	E-303d Adhesion	Formula 159 Adhesion	Adhesion (avg. psi)	% Topcoat	% Intercoat	% Primer
<1 mil	4B	5B				
3 mil			450	0	0	0
6 mil			542.5	25.0	0	0
9 mil			482.5	55.0	0	0
>10 mil	4B	4B	425	47.5	0	0

Table 3
Effects of Incomplete Surface Preparation

Substrate Conditions and Preparation	E-303d Adhesion	Formula 159 Adhesion
Flash Rust	4B	4B
100% Rust, No Pitting	4B	4B
100% with Pitting Top ½ SSPC SP-7 Brush Off Blasted Bottom ½ 100% Rust with Pitting	4B 3B	4B 3B

Table 4
Effects of Thinning Zinc-rich Primers

% Thinning	Adhesion of E-303d	Adhesion of Formula 159
0		Dry Spray
10	Dry Spray	3B
20	4B	5B
30		5B
40	5B	4B
50		3B
60	5B	Failure
80	4B	
100	2B	

Table 5
Effect of UV Radiation Exposure on Primecoat (Formula 150/151 System)

Primer Cure (weeks)	Adhesion (avg. psi)	% Topcoat	% Intercoat	% Primer
1	435.7	0	18.0	0
2	268.8	12.5	0	0
3	383.4	19.2	0	0
4	395.8	85.0	0	0

Table 6
Effect of Long Cure Time (Formula 150/151 System)

Primer Cure (weeks)	Adhesion (avg. psi)	% Topcoat	% Intercoat	% Primer
1	383.4	33.4	0	0
2	400.0	34.5	1.60	0
3	329.2	25.8	0	0
4	443.8	8.75	0	0
5	358.4	19.2	0	0

4 Conclusions

Applications and Effects

Elevated temperature application

As application temperatures increased up to 140 °F, severe application problems were encountered. Blockages in the spray gun finally made application impossible at 140 °F. The applicator in the field may find it increasingly difficult to apply the zinc-rich primers as temperatures rise to between 90 and 110 °F. However, if the coating can be applied satisfactorily, intercoat adhesion should not significantly differ. Similarly, no differences were noted in the intercoat adhesion of the MIL-P-24441 coating at temperatures up to 120 °F even when the coating cured up to 3 days before topcoating. The recorded adhesion values show some variation but no specific trend. In evaluating the location of the failure, the greatest was at the adhesion used to secure the dollies to the coated panel. Failure of the adhesive probably contributed significantly to the reported adhesion values. The exposure results of the elevated temperature panels after 12 months in the indicated environments proved excellent, reinforcing the adhesion data. Only slight corrosion was noted at the scored area on all panels subjected to salt water immersion. Slight chalking was also noticed on the systems that were atmospherically exposed.

Low temperature application

As application temperatures decreased to 40 °F, application difficulties increased and adhesion strengths decreased for both zinc-rich primers. Topcoats, when applied and cured at colder temperatures, exhibited relatively high adhesive strengths, although not as high as adhesion strengths for topcoats applied at higher temperatures. Intercoat adhesion failures after 7 days' cure are extremely low (probably not significant to the overall adhesion results). The slightly lower test results for the zinc-rich coatings at 50 °F may indicate the low temperature limit for these coatings. Obviously, the failure of the zinc-rich primers to cure at 40 °F and the MIL-P-24441 at 30 °F shows these temperatures are beyond the limits of the coatings. The 12-month exposure results reinforce these observations, and no failures were observed.

Thin and thick applications

MIL-P-24441/19, E-303d, and MIL-P-24441 Formulas 150 and 151 do not seem susceptible to failure when applied excessively thin or thick. The thick (>10 mils) application had no effect on adhesion for either of the zinc-rich primers or the Formula 150/151 system. The adhesion values for the 6-mil test for the Formula 150/151 system proved the highest of the series.

Incomplete surface preparation

Improperly cleaned substrates obviously affected the performance of both the E-303d and the Formula 159 primers. Both primers exhibited consistent adhesion values, indicating that both could be expected to exhibit poor adhesion in the field if applied to improperly cleaned substrates. All the tests displayed 4B adhesion, indicating a slight decrease in adhesion caused by the unclean substrate. The results for the untouched 100 percent rust with pitting are also significant. Both the primers exhibited 3B adhesion and course textures, indicating their degraded status. The exposure results reinforce this observation. Panel groups B and C had a noticeable increase in corrosion and undercutting of the scored area for all immersed panels, and the atmospheric panels showed an increase in corrosion.

Effects of thinning zinc-rich primers

Neither zinc-rich primer was severely affected by the addition of thinner up to a point just short of sagging. (Anyone using the data from this report should use caution because the actual amount of thinning will be determined by not only the paint but also by the equipment being used and the ambient conditions). The results of the work this test do show that, under equal conditions, E-303d requires a higher amount of thinner than Formula 159. They also show that, up to the point just short of sagging, the high amount of thinner has no adverse effect on the performance of the paint.

Overspray resistance of zinc-rich primers

Both zinc-rich primers exhibited fair adhesion at 3B, but the exposure results clearly indicate that the protection provided by the dry-sprayed coating was severely degraded. All immersed panels exhibited generalized rusting of the entire panel, and considerable corrosion at the scored area. Both primers performed equally under the dry-spray conditions.

Effect of UV radiation on MIL-P-24441

UV radiation causes both Formulas 150 and 151 to chalk, and somewhat degrades the intercoat adhesion. Exposure results indicate that the chalking present on the primer did not severely impede the coatings' protective properties.

Effect of long cure time between coats of MIL-P-24441

Allowing the Formula 150 to cure for up to 5 weeks under laboratory conditions before applying the Formula 151 topcoat did not appear to affect the performance of the Formula 150/151 epoxy-polyamide paint system.

Other Conclusions

In addition to these tests, it should be noted that E-303d was more tolerant of less than ideal application conditions, but when the accepted application limits were not exceeded, Formula 159 performed better. Formula 159 was also found to be easier to mix, thin, and apply than the E-303d because the zinc dust remained in suspension longer. Constant agitation is necessary with the E-303d, but is seldom required for Formula 159.

This work was not performed with the intent of altering the recommended application parameters currently specified in Corps guidance. The results of this work do not indicate a need for revising CWGS-09940 at this time. The work supports the existing guidance in that, if the guidance is followed, satisfactory performance can be expected. Field application is quite different than the applications done under laboratory conditions in this study. Many of the requirements placed in the guide specification were written for purposes other than those evaluated in this work. For example, the field guidance may require two coats of zinc-rich primer in order to obtain satisfactory film build on complex surfaces. This study shows that the high builds obtained on adjacent areas of the structure will also provide satisfactory performance.

Overall, this study has shown that the coatings are tolerant of a wide range of application and curing variables that are frequently encountered under field conditions. The study also indicates that MIL-P-24441 Formula 159 provides application and performance characteristics very similar to E-303d. Exposure panels will continue to be monitored to confirm that the coatings may be used interchangeably on hydraulic structure applications.

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Appendix A
E-303d Epoxy Zinc-rich Paint

Formula for E-303d

INGREDIENTS	PERCENT BY WEIGHT	POUNDS	GALLONS
	Component A		
Epoxy Resin, Type 1	35.9	277.8	28.1
Methyl n-Amyl Ketone	44.2	342.5	50.3
Toluene	6.0	46.3	6.9
Butanol	6.0	46.3	6.9
Suspending Agent M	6.5	50.0	6.9
Phthalocyanine Blue Pigment	1.4	11.0	0.9
	100.0	773.9	100.0
	Component B		
Polyamide Resin	38.1	277.8	34.3
Isopropanol	12.7	92.6	14.2
Toluene	12.7	92.6	12.3
Butanol	35.4	257.6	38.2
Catalyst	1.1	8.1	1.0
	100.0	728.7	100.0
	Component C		
Zinc Dust Pigment	100.0	5,000.0	85.0

a. Components A, B, and C of Formula E-303d shall be packaged separately as kits (unitized packaging permissible). The standard size kit shall be 2.85 gallons (mixed paint volume) consisting of 1 gallon of Component A, 1 gallon of Component B, and 50 pounds of zinc dust (Component C), packaged in a 2-gallon pail. Kits of larger or smaller sizes will be permitted, provided that the quantity relationship of the components shall be the same as the standard kit.

b. In addition to standard labeling requirements, each container of each component shall be properly identified as to component type and each container label of Component A shall carry the following: "MIXING INSTRUCTIONS: To prepare this paint for application, combine Components A and B and, while the mixture is being vigorously stirred with a heavy duty power stirrer, sift in the zinc dust (Component C). Continue the stirring until the zinc dust has been well dispersed and the mixture is smooth. The mixed paint shall at some point be strained through a 30-60 mesh screen to prevent any undispersed zinc dust slugs from reaching the spray gun nozzle. Thin with an appropriate thinner where necessary to obtain satisfactory application results. The pot life of the mixed material, extended from time to time by the addition of small amounts of thinner, will normally be in excess of 24 hours but may be less in very warm weather. Stir the material continuously after mixing and during application."

Ingredients for E-303d

- *Zinc dust pigment* shall conform to ASTM D 520, Type II.

- *Phthalocyanine blue pigment* for epoxy zinc-rich paint shall have properties similar and equal to Peacock Blue 249-1282 manufactured by Sun Chemical Co.

- *Suspending Agent M* shall be of soft translucent paste consisting of a thixotropic agent dispersed in toluene. It shall have a nonvolatile content of approximately 25 percent and a specific gravity of approximately 0.872. It shall be capable of minimizing the tendency of zinc dust to settle hard without increasing the viscosity of the paint significantly. MPA-60 (toluene), produced by Rheox, Inc., has these properties.

- *The catalyst* for Formula E-303d shall be 2, 4, 6 tri(dimethylaminomethyl) phenol. DMP-30, Rohm & Haas Co., is such a chemical.

- *The epoxy resin* for Formula E-303d shall be of the solid type conforming to ASTM D 1763 for a Type I, Grade 1, Class D resin except that it shall have weight per epoxy equivalent (WPE) of 425-550 and the softening point shall be between 65 and 75.

- *The polyamide resin* for Formula E-303d shall be a condensation product of a dimerized fatty acid and polyamines. It shall be a solid resin at room temperature and have the following characteristics:

	Minimum	Maximum
Amine value	85	95
Color (Gardner)	--	12
Specific Gravity, 25/25 °C	0.97	0.99
Viscosity, Poises, 150 °C (Brookfield)	7	9

- *Isopropyl alcohol* shall conform to ASTM D 770.
- *Butanol* (butyl alcohol) shall conform to ASTM D 304.
- *Methyl n-amyl ketone* (MAK) shall conform to ASTM D 4360.
- *Toluene* shall conform to ASTM D 841.
- *Alc-50* shall consist of 50 percent methyl n-amyl ketone and 50 percent butanol by volume.

Appendix B
Report of USACERL Paint Laboratory Testing of E-303d

CERL PAINT LABORATORY TESTING REPORT

Specification: E-303D Contract No.: DACW29-92-B-0054

Manufacturer: KOP-COAT MIPR No.:

Batch No.: 2C2126M

Analysis:	Min	Max	Result	Pass
Component A:				
Nonvolatile matter, %	39.15	43.25	39.50	Yes
Condition in container			OK	Yes
Component B:				
Nonvolatile matter, %	37.25	41.15	40.27	Yes
Condition in container			OK	Yes
Mixture:				
Mixing properties			OK	Yes
Thinning properties			OK	Yes
Spraying properties			OK	Yes
Drying properties			OK	Yes
Appearance			OK	Yes
Knife test			OK	Yes
Water immersion			OK	Yes

☐ Recommendation: Accept _X_ Reject ___

☐ Remarks:

Signature:

Appendix C
Military Specification Sheets for Formulas 159 and 150

MIL-P-24441/19 (SH)
31 August 1987

MILITARY SPECIFICATION SHEET

PAINT, EPOXY-POLYAMIDE, ZINC PRIMER, FORMULA 159, TYPE II

This specification is approved for use within the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

The requirements for acquiring the paint described herein shall consist of this specification and the latest issue of MIL-P-24441 (SH).

FORMULA. This formula covers zinc epoxy-polyamide paint designated by Navy Formula 159, Type II for interior or exterior use. The paint shall consist of the ingredients specified in the quantities specified.

Component A

	Pounds
Polyamide	8
Polyamide adduct	106
2-ethoxy ethanol	24
2-butoxy ethanol	7.6
Paint thinner, TT-T-291, type II, grade A	13.2

Component B

Zinc pigment	1900
Epoxy resin	190
Thixotrope	15
2-ethoxy ethanol	100.6
2-butoxy ethanol	30.2
Paint thinner, TT-T-291, type II, grade A	58.7

Formula 159 Thinner

2-ethoxy ethanol	387
2-butoxy ethanol	151
Paint thinner, TT-T-291, type II, grade A	198

QUANTITATIVE REQUIREMENTS. The paint shall meet the following quantitative requirements and the qualitative requirements of section 3 of the general specification. Components A and B shall be mixed 1:4 by volume for mixed components tests. Tests shall be performed in accordance with the general specification.

Requirements	Component A Min	Component A Max	Component B Min	Component B Max	Mixed Component Min	Mixed Component Max
Pigment content, %	----	----	81.5	85.5	----	----
Volatiles, %	42.8	46.8	8.1	8.5	----	----
Nonvolatile vehicle, %	53.1	57.0	8.0	8.6	----	----
Weight per gallon, lb	7.8	8.2	28.5	28.9	----	----
Water, %	----	0.5	----	0.2	----	----
Flash point, °F	99	----	110	----	99	----
Sag resistance, mils	----	----	----	----	8	----
Dry-hard, hours at 73°F, 3 mils wet film	----	----	----	----	----	8
Set-to-touch time, 3 mils wet film	----	----	----	----	----	2
Pot life, hours at 73°F	----	----	----	----	4	----
Consistency, grams	----	----	250	500	150	300

1) The amount and type of thixotrope shall be selected by the manufacturer to meet all requirements of the general specification and this specification sheet.

2) GENAMID 2000, Henkel Corporation; UNIREZ 2810, Union Camp; AZAMIDE 600, AZ Products; ANCAMINE 507, Pacific Anchor Chemical Corp.; EPOTUF SF7791, Reichhold Chemical; TriChem 9200, Trimont Chemicals.

3) VERSAMID 280B75, Henkel Corporation; UNIREZ 2180B75, Union Camp; AZAMIDE 680B75, AZ Products; ANCAMINE 700B75, Pacific Anchor chemical Corp.; EPOTUF SF 7792, Reichhold Chemical; TriChem 9280-B-75, Trimont Chemicals.

4) 90 percent pure by gas chromatography, density 0.93, refractive index (68 °F) = 1.4.18, IR spectrum characteristic of compound.

5) 99 percent pure by gas chromatography, density 0.90, refractive index (68 °F) = 1.418, IR spectrum characteristic of compound

6) Zinc No. 555, American Smelting and Refining; Zinc No. 422, New Jersey Zinc; or Intermediate No. 32 non-gassing, Pacific Smelting Company.

7) EPON 828, Shell Chemical Co.; ARALDITE 6010, Ciba-Geigy; DER 331, Dow Chemical Co.; AZEPOXY 128, AZ Products; EPO-TUF 37-140, Reichhold Chemical; EPIX-REZ 510, Cleanese; Trichem 727, Trimont Chemicals.

8) Tests on mixed components shall consist of one volume Component A mixed with four volumes Component B then reduced with 10 percent thinner by volume. Thinner used shall be Formula 159 thinner.

Use of alternate ingredients in this formula must have prior approval of the Naval Sea Systems Command. Approval will be based on review of data showing equivalent physical and chemical characteristics to the specified ingredient. It will be necessary to demonstrate that paint made using the ingredient will conform to all requirements of this specification.

MILITARY SPECIFICATION SHEET

PAINT, EPOXY-POLYAMIDE, GREEN PRIMER, FORMULA 150, TYPE IV

This specification is approved for use within the Naval Sea Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

The requirements for acquiring the paint described herein shall consist of this specification sheet and the issue of the following specification listed in that issue of the Department of Defense Index of Specifications and Standards (DODISS) specified in the solicitation: MIL-P-24441.

FORMULA. This formula covers green epoxy-polyamide paint designated Navy Formula 150, Type IV, for interior or exterior use. The paint shall consist of the ingredients specified in the quantities specified.

Component A

	Pounds
Thixotrope	----
Polyamide	36
Polyamide adduct	309
Magnesium Silicate	364
Titanium Dioxide	101
Black iron oxide	15.5
Butyl alcohol	258

Component B

Thixotrope	----
Epoxy resin	515
Magnesium silicate	390
Naphtha	208

QUANTITATIVE REQUIREMENTS. The paint shall meet the following quantitative requirements and the qualitative requirements of section 3 of the general specification. Components A and B shall be mixed 1:1 by volume for mixed components tests. Tests shall be performed in accordance with the general specification.

Requirements	Component A		Component B		Mixed Component	
	Min	Max	Min	Max	Min	Max
Pigment content, %	42.0	47.0	33.0	38.0	----	----
Volatiles, %	23.0	28.0	16.0	21.0	----	----
Nonvolatile Vehicle, %	28.0	33.0	44.0	51.0	----	----
Water, %	----	1.5	----	0.5	----	----
Coarse particles, %	----	0.3	----	0.3	----	0.5
Consistency, g	200	300	300	470	180	245
lb/gallon	10.9	11.4	10.8	11.3	10.9	11.4
Set-to-touch time, hr at 40°F	----	----	----	----	----	3
Set-to-touch time, hr at 73°F	----	----	----	----	----	3
Dry-hard, hr at 40°F	----	----	----	----	----	24
Dry-hard, hr at 73°F	----	----	----	----	----	8
Fineness of grind, NS	4	----	4	----	4	----
Flash point, °F	99	----	100	----	99	----
Titanium dioxide, % of pigment	18.5	----	----	----	----	----
Pot life, hr at 73°F	----	----	----	----	6	----
Sag resistance, mils	----	----	----	----	12	----
Color dry film to approximate the standard color chip	----	----	----	----	----	Conform
Weight per epoxy, (vehicle)	----	----	175	195	----	----
Contrast ratio, 3 mils dry film	----	----	----	----	0.98	----
Epichlorohydrin content (vehicle) ppm	----	----	----	2	----	----
Gloss 60 degree specular,%	----	----	----	----	----	30
VOC, ASTM D 3960, lb/gal	----	----	----	----	----	2.8

1) Thixotrope to be used is the manufacturer's choice. In the development of the Component A, 15 pounds (2.1 gallons) of Dislon NS-30 made by King Industries was used. The manufacturer is responsible for choosing a thixotrope that meets all the requirements herein, including shelf life. Thixotrope is a pigment for calculation purposes.

2) GENAMID 2000, General Mills Chemicals; UNIREZ 2810, Union Camp; AZAMIDE 600, AZ Products; ANCAMINE 507, Pacific Anchor Chemical Corp.; EPOTUF SF7791, Reichhold Chemicals.

3) X-HE-283, Ciba-Geigy Corporation.

4) Titanium dioxide conforming to ASTM D 476, type IV. In the development of component A, Dupont Taper R960 was used.

5) Mistron 400, "Mistron 500" Cyprus Industries.

6) Butyl alcohol conforming to ASTM D 304.

7) Sunfast Blue NCF, Sun Chemical Corp.; Paslomar Blue G B-4810, Mobay Chemical Corp.; Monarch Blue G-FR XX-3374 or

Irgalite Blue LGLD, Ciba-Geigy Corp.; Cyanine Blue B7000 (352751), BASF Wyandotte Corp.

8) Yellow Iron Oxide YO-3587, Charles Pfizer Chemical Corp.

9) Thixotrope to be used is the manufacturer's choice. In the development of the Component B, 15 pounds (1.8 gallons) of Dislon 6500 made by King Industries was used. Manufacturer is responsible for choosing a thixotrope that meets all the requirements herein, including shelf life. Thixotrope is a pigment for calculation purposes.

10) EPON 828, Shell Chemical Co.; ARALDITE 6010, Ciba-Geigy Corp.; DER331, Dow Chemical Co.; AZEPOXY 128, AZ Products; EPO-TUF 37-140, Reichhold Chemical; EPI-REZ 510, Celanese.

11) Conforming to ASTM D 3734, Type I. In the development of Component B, AMSCO Super High Flash Naphtha was used.

12) Huber 70C, J.M. Huber Corp.; Satintone #1, Englehard Mineral and Chemical Co.

13) Use FED-STD-595 color chip no. 24272. Color shall approximately match the color chip.

14) For VOC calculations, Component A makes approximately 101 gallons and Component B makes approximately 102 gallons.

Use of alternate ingredients in this formula must have prior approval of the Naval Sea Systems Command. Approval will be based on review of data demonstrating equivalent physical, chemical, and performance characteristics of paint manufactured with the proposed alternate material. Development of the required data package is the responsibility of the proposing agent. Paint incorporating the proposed alternate ingredient shall conform to all the requirements of this military specification sheet and the general specification.

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The Army Corps of Engineers relies heavily on the use of vinyl paints for coating hydraulic structures. Though the systems have performed well in many environments, their high solvent content has made their use illegal under some local air pollution control legislation. High solids epoxies available in the current guide specification will comply with the regulations and may provide acceptable performance in some applications.

Responding to field inquiries relating to specific application irregularities and anticipated problems, this research investigated the application parameters of the currently used E-303d and MIL-P-24441 Formula 159 zinc-rich primers and MIL-P-24441 Type IV Formula 150 primer/Formula 151 topcoat epoxy polyamide coatings.

Overall, this study showed that the coatings are tolerant of a wide range of applications and curing variables frequently encountered under field conditions. The results of this work do not indicate a need for revising CWGS-09940 at this time. The work supports the existing guidance in that, if the guidance is followed, satisfactory performance can be expected.

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