ARMY RESEARCH LABORATORY

Joint Test Protocol: Environmentally Friendly Zirconium Oxide Pretreatment Demonstration

by Fred L. Lafferman

ARL-MR-857 **December 2013**

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Army Research Laboratory
Aberdeen Proving Ground, MD 21005

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14. ABSTRACT

There is a need to implement innovative and cost-effective replacement technologies to address the multiple health, safety, and compliance issues associated with the use of zinc phosphate and chromate/chrome containing conversion coatings while maintaining military readiness for national defense. The new technology must be compatible with original equipment manufacturer/depot infrastructure to and with the current coatings and substrates used by the Department of Defense, and have corrosion performance equivalent or better than current pretreatment technology. The objective of this program is to demonstrate a novel zirconium-based pretreatment that will be environmentally acceptable, cost effective, and perform equal to baseline pretreatments for multimetal application. The demonstration will be on a military asset coated and compatible with the existing chemical agent–resistant coating system.

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Preface

This report was prepared under Weapons Project 201318 for the Environmental Security Technology Certification Program (ESTCP). The structure, format, and technical content were determined by ESTCP, government technical representatives, and government contractors in response to the specific needs of the project and eventual users of this technology.

We wish to acknowledge the invaluable contributions by the following organizations involved in the creation of this document:

- U.S. Army Research Laboratory
- U.S. Naval Air Systems Command
- U.S. Army Aviation and Missile Command
- Letterkenny Army Depot
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- U.S. Marine Corps Corrosion Prevention and Corrosion
- PPG Industries
- Elzly Technology Corporation

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1. Introduction

The Environmental Security Technology Certification Program (ESTCP) selected the Environmentally Friendly Zirconium Oxide Pretreatment project, let by the U.S. Army Research Laboratory (ARL), to assist in mitigating the significant environmental, safety, and occupational health risks associated with the use of zinc phosphate and chromate/chrome-containing conversion coatings.

There is a need to implement innovative and cost-effective replacement technologies to address the multiple health, safety, and compliance issues associated with the current systems while maintaining military readiness for national defense. In addition, the new technology must have the following attributes: (1) compatibility with original equipment manufacturer (OEM)/depot infrastructure, (2) corrosion performance equal to (or better than) current phosphate-based pretreatments, (3) broad compatibility with the current suite of military coatings, and (4) compatibility with substrates used by the Department of Defense (DOD). The objective of the proposed program is to demonstrate a novel pretreatment technology in relevant DOD environments. The zirconium-based pretreatment will be shown to be both environmentally acceptable (no hazardous air pollutants or heavy metals such as hexavalent chromium (Cr6+) or nickel) and a cost-effective alternative to existing phosphate-based pretreatments. At the conclusion of the program plan, the zirconium-based pretreatment will have been demonstrated to perform equal to (or better than) existing phosphate-based pretreatments while offering compatibility with the current suite of military coatings and a range of ferrous and nonferrous substrates.

The proposed zirconium-based pretreatment was originally developed by PPG Industries for the automotive industry, where it is providing corrosion protection equivalent to zinc phosphate on a variety of substrates (including cold-rolled steel). The proposed technology provides a high-quality, continuous zirconium-based pretreatment on multiple types of ferrous, zinc, and aluminum substrates by immersing the metal into a dilute solution of fluoro-zirconic acid (FZA) and proprietary additives at ambient temperature for 30–120 s. The dilute, aqueous FZA pretreatment bath is only slightly acidic (pH = 4.5) and does not contain any volatile organic compounds. During the treatment process, the substrate is etched slightly, which results in a pH increase at the substrate-solution interface. This change in pH results in the precipitation and subsequent bonding of zirconium oxide and additives to the surface of the substrate.

The chemical agent–resistant coating (CARC) systems application specification, MIL-DTL-53072 (*1*), requires that metal surfaces on tactical vehicles be treated to improve adhesion and corrosion resistance prior to coating with an epoxy primer and a camouflage topcoat. In OEM processes, the surface treatment is generally performed by a five-stage dip process, e.g., zinc phosphate prescribed in TT-C-490 (*2*).

Zirconium-based metal pretreatment technology is an alternative to conventional technologies such as zinc phosphate, chromate-containing etch primers, and chromate conversion coatings. Compared with these conventional technologies, zirconium-based pretreatments can provide the following advantages:

- Operation under ambient conditions versus greater than 125 °F for metal phosphate systems.
- Reduction in the amount (80% less) and toxicity of waste materials generated from pretreatment application and disposal processes.
- Reduction in water use in the pretreatment process.
- Reduced exposure to toxic and regulated materials during the pretreatment process.
- Reduced deposition of metallic compounds, such as chrome, that could be released during rework and other downstream operations.

ESTCP program requirements include the development of a joint test protocol (JTP), which contains the technical requirements and tests necessary to evaluate nontoxic-metal-containing pretreatments against qualified and approved control conversion coatings containing phosphate, chromate, and chrome. It also includes the technical requirements of the U.S. Marine Corps, which also plans to evaluate the test results to select materials for implementation. Alternative materials are expected to be validated with these technical requirements under this project.

The overarching objective of this project is a comprehensive evaluation of applications and requirements for environmentally friendly conversion coatings, characterization of the performance and maturity of available or proposed alternative conversion coating technology, and recommendations and actions for development, optimization, and demonstration/validation of toxic metal-free conversion coatings.

All stakeholders* maintained a continuous and open dialogue discussing the technology readiness level of the zirconium oxide conversion pretreatment coating. Over the past several years, ARL and PPG Industries have worked closely to optimize the pretreatment formulation to improve corrosion performance on multiple substrates by communicating the concerns and achievements through many face-to-face and telecommunication meetings. The formula and performance optimization was done through Strategic Environmental Research and Development Program WP-1676. We discussed the demonstration requirements and reviewed variances in application

* The stakeholders of this program are ARL, Fred Lafferman (principal investigator); U.S. Army Aviation and Missile Command, Mark Feathers; U.S. Marine Corps Corrosion Prevention and Control, Andrew Sheetz; Letterkenny Army Depot, Dennis Reed; U.S. Marine Corps Logistics Base, Albany, Steve Allen; U.S. Army Tank Automotive Command, Daniel Nymberg; and PPG Industries, Larry Fitzgerald. See the appendix for a complete list of participating organizations and representatives.

between the zirconium pretreatment and their existing conversion coatings with our stakeholders Marine Corps Logistics Base Albany and Letterkenny Army Depot.

For this project, zinc phosphate and hexavalent chromium, as found in immersion conversion coatings, was identified as the target hazardous material (HazMat) to be eliminated or reduced. Table 1 summarizes the target HazMat, process, application, current specifications, affected programs, and candidate parts/substrates.

Table 1. Target HazMat summary.

Target HazMat	Current Process	Applications	Current Specifications	Affected Programs	Candidate Parts and Substrates
Zinc phosphate conversion coating	Immersion application	Used as a pretreatment for ferrous substrates	TT-C-490F (*2*)	All military GSE[a] platforms and their assets	Substrates: steel 1020
Hexavalent chromium conversion coating	Immersion and spray application	Used as a pretreatment for nonferrous substrates	MIL-DTL-5541 (*3*)	All military GSE and aviation platforms and their assets	Substrates: AA2024-T3 AA7075-T6 AA6061-T6

[a]GSE = general support element.

The purpose of this JTP is to maturate and evaluate the application processes of this pretreatment coating, which has great potential to exceed mature technology in corrosion performance or provide similar corrosion performance at a lower application cost. Successful development will lead to the increased maturity of a technology's application process or coating performance, creating the opportunity for additional demonstrations and validations in areas where current products are insufficient.

2. Performance and Testing Requirements

The project's joint technical team identified engineering, performance, and operational impact (supportability) requirements for zinc phosphate and hexavalent chromium found in conversion coatings used on multimetal components. The technical team then reached consensus on tests with procedures, methodologies, and acceptance criteria for evaluating the zirconium oxide pretreatment conversion coating against approved zinc phosphate and chromate conversion coatings. Data developed from these tests is intended to be used as a guide for implementation for each user and not intended to be used for qualifying or excluding any alternative. Users will select alternatives based on their respective business case.

The major requirements for which the tests in this JTP were chosen are the following:

- Corrosion resistance
- Dry tape adhesion
- Adhesion pull-off
- Stress corrosion cracking
- Fluid resistance
- Flexibility/impact
- Field exposure/static
- Field exposure/on vehicle

Tests should be conducted in a manner that will eliminate duplication and maximize use of each test specimen. For example, where possible, more than one test should be performed on each specimen. The number and type of tests that can be run on any one specimen will be determined by the destructiveness of the test.

Tests in this JTP may involve the use of hazardous materials, operations, and equipment. This JTP does not address all safety issues associated with its use. It is the responsibility of each user of this JTP to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

Table 2 lists all engineering and testing requirements identified for validating the zirconium oxide pretreatment conversion coating to zinc phosphate and chromate conversion coatings that are common to all affected defense systems listed in table 1. Table 3 lists all extended engineering and testing requirements identified by specific stakeholders for conversion coating validation, time permitting. Tables 2 and 3 include acceptance criteria and references, if any, used for developing the tests.

Table 2. Common performance and testing requirements.

Phase	Engineering Requirement	Test	JTP Section	Acceptance Criteria	References
I	Corrosion resistance	Neutral salt fog; flat panels	3.1	336/1,000/2,000 h, minimal scribe corrosion; no loss of adhesion; no blisters; ratings of ≥6 for ferrous and ≥8 for aluminum	ASTM B 117 (*4*); ASTM D 1654 (*5*); ASTM D 714 (*6*)
I	Corrosion resistance	Cyclic corrosion; flat panels	3.2	40/60/120 cycles; minimal scribe corrosion; no loss of adhesion; maximum of five scattered blisters; ratings of ≥7 for ferrous and ≥8 for aluminum.	GMW 14872 (*7*); ASTM D 1654 (*5*); ASTM D 714 (*6*); ASTM G 1(*8*)
I	Adhesion	Dry tape adhesion (cross cut)	3.3	Equivalent or improved performance compared with pretreatment and primer controls; 4B rating minimum.	ASTM D 3359, Method B (*9*)
I	Adhesion	Pull-off adhesion	3.4	Pull strength ≥1,200 psi	ASTM B 4541 (*10*)
I	Flexibility	Mandrel bend	3.5	No cracking or delamination from substrate or intercoat compared with baseline system.	ASTM D522, Method B (*11*)
I	Water resistance	Water resistance, wet tape adhesion	3.6	No peel-away; at least 4A per ASTM D 3359; no blistering of unscribed coating area; no delamination from substrate.	ASTM D 3359 (*9*), FED-STD-141, Method 6301 (*12*)
I	Water immersion	Pencil hardness after immersion	3.7	≤2 pencil hardness difference from an unexposed film; no blistering or delamination of unscribed coating area.	ASTM D 3363 (*13*), ASTM D1308 (*14*)
I	Hydrocarbon immersion (JP-8)	Pencil hardness after immersion	3.8	≤2 pencil hardness difference from an unexposed film; no blistering or delamination of unscribed coating area.	ASTM D 3363 (*13*)
I	In-test hydrogen embrittlement	Stress relief cracking	3.9	There shall be no detrimental effect to K1c of substrate. High-hard K1c at 48–51Rc shall maintain K1eac ≥ 19 (ksi√in)	ASTM E 399-97 (*15*) ASTM G 30 (*16*) ASTM G 38 (*17*) ASTM G 39 (*18*) ASTM G 47 (*19*)

Table 3. Extended performance and testing requirements.

Phase	Engineering Requirement	Test	JTP Section	Acceptance Criteria	References
II	Corrosion	Marine environment, outdoor exposure	3.10	Three years of exposure: rated every 3 months for first year and 6 months thereafter. Specimen will be rated as less or more creepage from scribe than current corrosion protection system.	Approved test site standard practice: ASTM G 50 (*20*) ASTM D 1654 (*5*) ASTM D 714 (*6*)
II	Corrosion and adhesion	Field exposure, on-vehicle	3.11	Exposure time of 2 years on vehicle platform or panels attached to platform. Equivalent or improved performance compared to baseline pretreatment controls.	ASTM D 1654 (*5*) ASTM D 714 (*6*)

The tests (described in section 3 of this report) shall also be conducted for nontraditional candidate substrates such as high-hardness alloy (HHA) (greater than Rockwell hardness Rc39) steels and high-strength low-alloy (HSLA) steels.

A material/corrosion design review will be conducted by the invoking authority to determine if hydrogen embrittlement, corrosion fatigue, or stress-corrosion cracking could occur based on the material and potential exposure environment. However, it will be known that HHA has hardness levels well over Rc39 and is susceptible to environmentally assisted cracking (EAC) whenever residual stresses are present. The invoking authority will specify the appropriate mechanical stability testing required, and the vendor will contract with an independent certified lab to perform the required tests.

The criteria for determining a risk candidate for hydrogen embrittlement is as follows: any ferrous-based alloy exhibiting hardness greater than Rc39 (e.g., high-strength steel) requires testing and heat treatment according to Federal Specification TT-C-490 (*2*). Testing is recommended for materials that will be exposed to an electrochemical environment where hydrogen evolution can occur (e.g., electroplating, pickling).

The basic criteria for determining a risk candidate for stress-corrosion cracking are as follows: (1) any material that will be exposed to a corrosive environment known to cause stress-corrosion cracking, such as sodium hydroxide for carbon steel or chloride ions for stainless steels, and tensile stress due to applied load or residual stresses such as those produced by welding (e.g., any material that will experience a stress greater than 50% of the yield stress) shall be tested, and (2) any material that is known to be subject to stress-corrosion cracking (susceptibility determined by conducting a literature search or consulting with a corrosion expert) shall be tested.

Testing is divided into two phases: alternative screening followed by field demonstration and validation. Alternatives must complete the JTP screening phase before entering into the field portion. Details of field demonstration testing (phase II testing) will be defined in the demonstration plan. In the screening process, corrosion, paint adhesion, flexibility, and immersion testing will be completed first. For all testing, surface preparation and treatment will be applied and tested by DOD or contractor personnel and not at vendor sites. All spray primers and topcoats will be applied by DOD or contractor personnel and tested by both DOD and PPG personnel. PPG will apply the zirconium oxide pretreatment to all specimens, as the required application equipment is currently not available at ARL. All test specimens will be evaluated by DOD and PPG personnel. MIL-DTL-53039 (*21*) topcoats will be used on all test specimens that require topcoat for a specific test.

3. Test Descriptions

The tests identified in table 2 are further defined in sections 3.1–3.9, to include test description, rationale, and methodology. Also included, as needed, are any major or unique equipment requirements as well as data reporting and analysis procedures. Test methodology includes the definition of test parameters, test specimens, number of trials per specimen, any experimental control specimens required, and acceptance criteria. The items listed in table 3 are further defined in sections 3.10 and 3.11 along with the rationale for inclusion in the testing protocol. The primary purpose of this JTP is to provide data to the joint user community, which it can use to select alternatives, if any, for field testing. Decision criteria will vary by user, and it is likely that different users will choose different alternatives based on their business cases.

Test coupons (also referred to as flat panels) will be at least 3 in wide × 6 in long × 0.032 in thick. Common performance tests shall be conducted on test panels made from the same material or alloy as the actual components and agreed upon by the stakeholders. The processes to be used in the preparation of the test panels shall be outlined in the joint test report (JTR). All metal coupon surfaces must be water-break-free prior to use. Water-break tests shall be performed in accordance with ASTM F 22-13 (*22*). Performance and special tests shall be conducted on sections of actual manufactured parts or certified test coupons that accurately simulate current production material and manufacturing processes. Mechanical conditions such as bends, welds, fasteners, crevices, etc., shall be incorporated when applicable. The actual processes used in the test specimen preparation shall be outlined in the JTR.

All painted test coupons shall be allowed seven days of unaided drying time, as per specification requirements of the baseline primers and CARC topcoats, prior to testing to ensure adequate polymerization of the coatings. Industry-standard air spraying equipment will be used to deposit organic coatings to specified thickness. To ensure compatibility of the zirconium pretreatment with the designated CARC primers, duplicate panels and parts will be over-coated with the

following CARC primers: MIL-DTL-53022 (*23*), MIL-DTL-53030 (*24*), and MIL-PRF-32348 (*25*). The topcoat that will be used on all coupons and parts for testing will be either MIL-DTL-53039 (*21*) or MIL-DTL-64159 (*26*). The coupons will use MIL-DTL-53039 (*21*) for the common performance testing but the topcoat choice for the extended performance testing will be determined by what is being used at the demonstration depots. Film thickness for the liquid primers will be maintained at a dry film thickness of 1.5 ± 0.2 mil (37.5 ± 5 µm) to obtain consistent and comparable corrosion results. The powder primer will be applied at a dry film thickness of 2.0 ± 0.2 mil (50 ± 5 µm), and the CARC topcoats will be applied at a dry film thickness of 2.0 ± 0.2 mil (50 ± 0.2 µm).

The stakeholders have established the requirements necessary to evaluate corrosion-resistant candidates for use on U.S. military components. These requirements have been used to identify test methods, derive test procedures, and establish acceptance criteria. It is recommended that different examples of substrates using the candidate, if applicable, be tested concurrently to obtain maximum benefit from the testing effort. Questions regarding the different substrate materials shall be directed to the invoking authority. The candidate must pass the common performance and applicable extended tests with at least minimum performance (MP) in order to be considered for military use. Acceptance criteria for improved performance (IP) and best performance (BP) are provided as well, so that improved corrosion resistance with respect to the current corrosion protection system can be quantified.

Users of this JTP should check the project's JTR, if available, for additional test details or minor modifications that may have been necessary in the execution of testing. Any test procedure modifications will have been agreed upon by the technical stakeholders. Unless otherwise specified by the technical stakeholders at the demonstration depots, table 4 lists the specimen code, alloy name, and composition for the substrates selected for testing and referenced throughout the JTP. These substrates represent a cross section of alloys used to fabricate components in the joint community.

Table 4. Substrate descriptions and test specimen codes.

Specimen Code	Alloy Name	Composition
M1	AA2024-T3 (bare)	4.5% copper, 1.5% magnesium 0.6% manganese (Mn)
M2	1000-series steel	0.05%–0.3% carbon 0.35%–0.90% Mn 0.040% phosphorus 0.050% sulphur

3.1 Neutral Salt Fog for Flat Panels (ASTM B 117) (*4*)

3.1.1 Test Description

This test method describes the procedure and conditions required to create and maintain the neutral salt spray (NSS) (fog) test environment and the evaluation of specimens incorporating the candidate with respect to corrosion, blistering associated with corrosion, loss of adhesion at a scribe mark, or other corrosive attack. The fog chamber will be operated in accordance with ASTM B 117 (*4*).

At least three specimens shall be used for common performance testing (CPT), and at least five specimens shall be used for extended performance testing (EPT). CTP shall be conducted with 102- × 152-mm (4- × 6-in) test panels composed of the material that is used in the end application. Actual or simulated frame structures shall be used for ETP. Each test specimen shall contain a clear identification mark. The testing procedure includes the following steps. Using test specimens incorporating the candidate, scribe an "X" incision through the coating, making sure that the scribed line is all the way through to the substrate. Cover the back of the coupon with wax, paint, tape, or any other material that will prevent corrosion products from contaminating the chamber. Place the scribed test specimens in the chambers, leaning at an angle 15°–30° from the vertical with the scribed surface facing upward. Prepare the salt solution as specified in ASTM B 117 (*4*) such that when atomized at 35 °C (95 °F), the collected solution is in the pH range of 6.5–7.2. The coupons may not contact other surfaces in the chamber, and condensate from a coupon may not contact any other coupons. Prepare a salt solution and the fog chamber as specified in test methodology. Adjust the nozzles in the fog chamber so that sprayed salt solution does not directly impinge on the coupon surfaces. Operate the fog chamber continuously for 2000 h. The testing criteria for ferrous substrates shall be 1000 and 2000 h for aluminum. Ferrous substrates shall be inspected at cycles of 336, 500, 750, and 1000 h. Aluminum substrates shall be inspected at every 500 h.

At the conclusion of the exposure period, remove the test specimens and clean them by gently flushing with running tap water and drying them with a stream of clean, dry, compressed air. Allow the test specimens to recover for 24 h. Scrape the test specimens side to side with the putty knife at a 30° contact angle. Evaluate the corrosion resistance and creepage of the test specimens in accordance with the latest version of ASTM D 1654 (*5*). Rate the corrosion or loss of coating extending back from the scribe mark, and evaluate the unscribed areas for corrosion spots, blisters, and any other types of failure. The test methodology for neutral salt fog testing is given in table 5, and the rating system for corrosion is shown in table 6. Use the rating system in ASTM D1654 (*5*) for scribed areas and D 714 (*6*) for unscribed. Photographically document the surface condition of each of the test specimens using the imaging system.

3.1.3 Test Methodology

The methodology for neutral salt fog testing is detailed in table 5, and the standard corrosion rating system appears in table 6.

Table 5. Test methodology for neutral salt fog testing.

Parameters	Test coupons at a 15° angle. Temperature of exposed salt spray zone = 35 °C ± 1.7 °C (95 °F ± 3 °F). Every 80 cm^2 horizontal area, two collectors gather 1.0–2.0 ml fog/h. 5% salt solution (5 ± 1 parts by weight of sodium chloride in 95 parts of water). pH = 6.5–7.2 when atomized at 35 °C (95 °F). 1000 h (ferrous); 2000 h (aluminum).
Number and Type of Specimens Per Candidate Alternative	Three each of M1 and M2. Include primer-only and topcoated panels.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541 (*3*) for aluminum panels
Acceptance Criteria	Scribe rating of ≥6 for ferrous substrates and ≥8 for aluminum. Performance equal to or better than control zinc phosphate or conversion coating over similar substrates and under similar primers.

Table 6. ASTM D 1654-08 (*5*) corrosion rating system.

Ratings of Failure at Scribe (Procedure A)		
Representative Mean Creepage From Scribe		
(mm)	**(in)**	**Rating Number**
0	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0 to 1.0	1/64 to 1/32	8
Over 1.0 to 2.0	1/32 to 1/16	7
Over 2.0 to 3.0	1/16 to 1/8	6
Over 3.0 to 5.0	1/8 to 3/16	5
Over 5.0 to 7.0	3/16 to 1/4	4
Over 7.0 to 10.0	1/4 to 3/8	3
Over 10.0 to 13.0	3/8 to 1/2	2
Over 13.0 to 16.0	1/2 to 5/8	1
Over 16.0 to more	5/8 to more	0

3.1.2 Rationale

The 1000- and 2000-h neutral salt fog test on scribed, painted substrates is a key accelerated test to determine overall corrosion inhibition compared with controls.

3.1.4 Equipment and Instrumentation

- The NSS (fog) chamber shall consist of a heated fog chamber, salt solution reservoir, supply of conditioned (oil- and contaminant-free) compressed air, atomizing nozzles, and specimen supports.
- Imaging system: a means of visually recording corrosion effects on all tested specimens, such as a digital camera or scanner/software system.
- Scribe tool: ANSI B 94.50, style E (*27*), scriber.
- Straight edge: any straight edge of sufficient length to guide the scribing tool in a straight line across the specimen surface.
- Air source: a source of clean, dry, compressed air capable of delivering at least 10 cfm at 80 psi.
- Air gun and guard: an air dusting gun and nozzle combination meeting the specification in ASTM D 1654 (*5*); and a guard to protect the operator, such as a sandblasting cabinet.
- Scale: a ruler with 1-mm (0.04-in) divisions.
- Putty knife: blunt-edged, 38 mm (1.5 in) wide.

3.1.5 Data Analysis

Report all information required in ASTM B 117 (*4*), ASTM D 714 (*6*), and ASTM D 1654 (*5*), and include the images from the imaging system. Report the final corrosion test results and the specified interval visual observations. Ratings are based on the condition of the scribe, amount and size of undercutting, and number and size of face blisters. The rating system used to quantify results is the frequency of blisters measured according to ASTM D 714 (*6*).

3.2 Cyclic Corrosion on Flat Panels

3.2.1 Test Description

This test method describes a field-correlated laboratory corrosion test method for determining cosmetic corrosion performance that provides a combination of cyclic conditions (salt solution immersion, temperature, and humidity) to accelerate the corrosion process.

Operate the fog chamber for this test in accordance with GMW 14872 (*7*). Use the underbody mode of test cabinet operation, using four salt solution applications per 8-h ambient cycle (see figure 1). The typical ramp time from the ambient stage to the humid stage is 1 h and is part of the 8-h humid stage. The typical ramp time from the humid stage to the dry stage is 3 h and is part of the 8-h dry stage. For extended down time, refer to 4.3.6 of the test method.

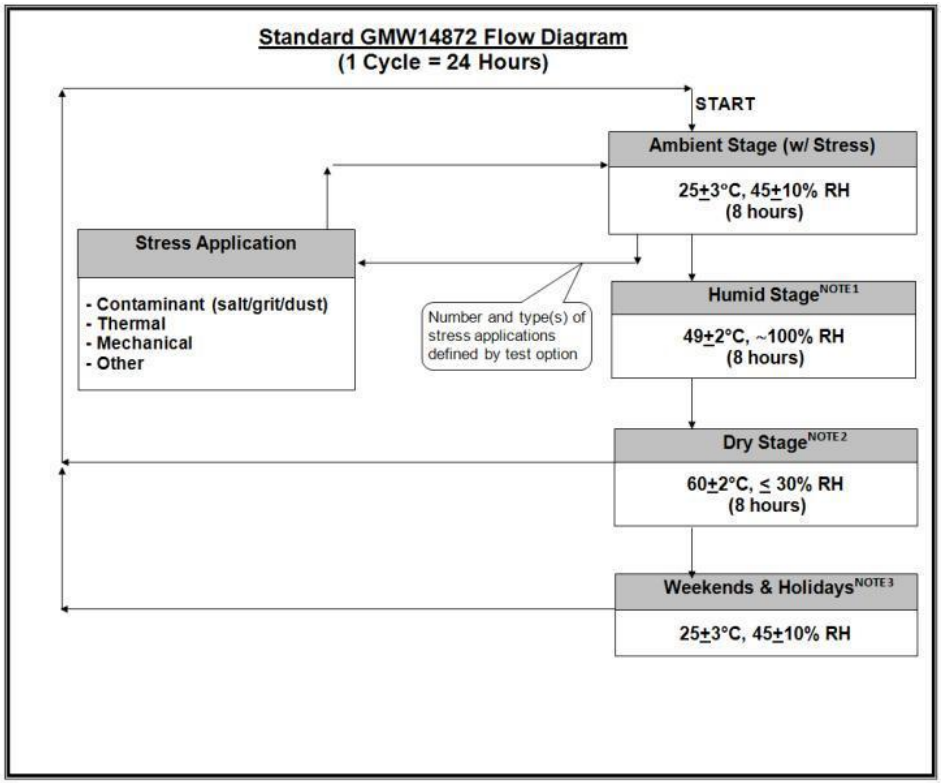

Figure 1. GMW 14872 (*7*) cycle description.

Actual or simulated steel components shall be used for test specimens (see section 3.2 of this report). The number of test specimens depends on the number of cycles selected for the test exposure duration. Use reference coupons consisting of uncoated 25- × 51- × 3-mm (1- × 2- × 1/8-in) pieces of any alloy American Iron and Steel Institute 1006–1010 steel to monitor the average general bare-steel corrosion produced by the test environment. The coupon weight in milligrams shall be recorded and retained for future reference. The number of coupons also depends on the number of cycles selected for the test exposure duration. Each test specimen and reference coupon shall be permanently identified by stamping numbers onto the surface. Using test specimens incorporating the candidate, scribe an X through the coating, making sure that the scribed line is all the way through the coating to the substrate. Place the scribed test specimens and reference coupons in the chamber, leaning at an angle of, at most, 15° from the vertical with the scribed surface facing upward.

Prepare the salt solution per GMW 14872 (*7*) and measure the pH prior to the start of the test and on a weekly basis thereafter. Do not attempt to adjust the pH. Clean the reference coupons (bare steel bars) thoroughly with the cleaning solution prior to placing them in the exposure chamber. For the MP, IP, and BP levels, use test durations of 40, 60, and 120 cycles, respectively. After weighing each reference coupon and test specimen, install them in the exposure chamber. After every 20 cycles, remove two coupons and two test specimens. Weigh each reference coupon (after removal of the rust layers) and determine the average weight loss for that specific number

of cycles. For the test specimens, record the scribe creep-back values with respect to average as specified in ASTM D 1654 (*5*). Conduct the interim creep-back measurements in a rinsed-only condition. At the final number of cycles, two sets of creep-back values will be recorded, one in a rinsed-only condition and one after the scrape-and-tape process.

At the conclusion of the exposure period (or interim period), remove the test specimens and rinse. Scrape the specimens side to side with the putty knife at a 30° contact angle. Evaluate the creepage of the test specimens per the latest version of ASTM D 1654 (*5*) for scribed areas and ASTM D 714 (*6*) for unscribed. Rate the corrosion or loss of coating extending from the scribe mark (using the worst case for the rinsed or scraped methods) and evaluate the unscribed areas for corrosion spots, blisters, and other types of failure. Photograph each of the test specimens and the reference coupons using the imaging system. Clean the reference coupons using a mild sand (or glass bead) blast to remove all corrosion by-products. Once they are clean, wipe the coupons with methanol and weigh them to determine weight loss. Corrosion losses may also be expressed in terms of average corrosion rates from the weight loss, coupon area, test duration, and metal density by use of the calculation described in ASTM G 1 (*8*).

3.2.2 Rationale

The cyclic accelerated corrosion test on scribed, painted substrates is a key accelerated test to determine overall corrosion inhibition compared with controls. Testing at various cycles will provide the information necessary to compare the performance of the zirconium oxide pretreatment to the baseline pretreatments.

3.2.3 Test Methodology

The test methodology for cyclic corrosion testing is given in table 7.

Table 7. Test methodology for cyclic corrosion testing.

Parameters	Test coupons at 15° angle. Up to 120 cycles. One cycle: see figure 1. Fog humidity: per GM 4465 P (*28*) at 49 °C ± 2 °C. Salt solution: 0.9% sodium chloride, 0.1% calcium chloride, 0.25% sodium bicarbonate; pH of 6 to 9. Drying environment: 60 °C ± 2 °C, <30 °C relative humidity (RH). Ambient environment: 25 °C ± 2 °C, 40%–50% RH.
Number and Type of Specimens Per Candidate Alternative	Three each of M1 and M2, including primer-only and topcoated panels.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541 (*3*) (for aluminum panels)
Acceptance Criteria	Scribe rating of ≥7 for ferrous substrates and ≥8 for aluminum, with no more than five scattered blisters in unscribed area. Performance equal to or better than control zinc phosphate or conversion coating over similar substrates and under similar primers.

3.2.4 Equipment and Instrumentation

The following will be required for the testing:

- A test cabinet with the ability to obtain and maintain the required environmental conditions as specified in GMW 14872 (*7*).
- An ANSI B 94.50 style E scriber (*27*).
- A means of visually recording corrosion effects on all test specimens, such as a camera or scanner/software system.
- A source of clean, dry, compressed air capable of delivering at least 10 cfm at 80 psi.
- A ruler with 1-mm (0.04-in) divisions.
- A digital electronic balance capable of weighing up to 10,000 mg with an accuracy of ±1%.
- Any straight edge of sufficient length to guide the scribing tool in a straight line.
- A pH meter to measure the salt solution prior to the start of the test and on a weekly basis thereafter.
- A blunt-edged putty knife 38 mm (1.5 in) wide.

3.2.5 Data Analysis

Report all information required in ASTM D 714 (*6*) and ASTM D 1654 (*5*), including the photographs from the imaging system and the weight loss and/or corrosion rate of the reference coupons.

3.3 Dry Tape Adhesion

3.3.1 Test Description

This test method covers a procedure for establishing adequacy of intercoat and surface adhesion of an organic coating in ambient conditions by applying pressure-sensitive tape over a scribed area of the coating. Perform this test in accordance with ASTM D 3359 (*9*).

Prepare at least three test specimens for performance testing using 102- × 152-mm (4- × 6-in) test panels composed of the material that is used in the end application. Using test specimens incorporating the candidate zirconium oxide pretreatment and the referenced specification epoxy primers, measure the dry film thickness in at least five areas. Make cuts in the coating system per the latest version of ASTM D 3359 (*9*). Remove two laps of tape and discard. Remove an additional length of tape and cut a piece approximately 76 mm (3 in) long. Place the center of the tape over the grid and smooth into place by firmly rubbing a pencil eraser over the area. Within 90 ± 30 s of tape application, remove the tape by holding the free end and rapidly pulling (not jerking) back upon itself at as close as possible to an angle of 180°. Inspect the grid area for

removal of coating from the substrate or from a previous coating. Rate the adhesion in accordance with the latest version of ASTM D 3359, Test Method B (*9*). If ratings differ by more than one rating unit, the results are considered suspect; three additional test specimens shall be prepared and the tests repeated. If applicable, use these latter ratings in the report.

3.3.2 Rationale

This test was identified as a performance requirement for the baseline pretreatment specifications TT-C-490 (*2*) and MIL-DTL-5541 (*3*) when applying specified primers. Loss of paint adhesion is the primary failure mode on ferrous and aluminum substrates.

3.3.3 Test Methodology

The test methodology for dry tape adhesion testing is given in table 8.

Table 8. Test methodology for dry tape adhesion testing.

Parameters	As defined in ASTM D 3359, Test Method B (*9*).
Number and Type of Specimens per Candidate Alternative	Three each of M1 and M2; include primer-only and topcoated panels.
Trials per specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541 (*3*) for aluminum panels.
Acceptance Criteria	No peel away; at least 4B per ASTM D 3359 (*9*); no blistering of unscribed coating area; a rating of 5B provides superior adhesion.

3.3.4 Equipment and Instrumentation

The following items will be used in testing:

- A very sharp razor blade, scalpel, knife, or other cutting device having a cutting edge (tip) angle between 15° and 30°.
- Steel or other hard metal straight edge to ensure straight cuts.
- A steel rule graduated in 0.5-mm (0.02-in) increments for measuring individual cuts.
- 3M Company 250 Flatback masking tape, chosen for its performance over the MIL-DTL-64159 (*26*) and MIL-DTL-53039 (*21*) polymer bead versions of CARC. Care should be taken to use tape only within its reported shelf life; tape beyond the manufacturer's recommended storage date may yield inaccurate results.
- Pencil with eraser to make sure the tape is firmly adhered.
- A light source to determine whether the cuts have been made through the coating into the substrate.
- Dry film thickness gage to measure the thickness of the applied coating.

3.3.5 Data Analysis

Report all information per the latest version of ASTM D 3359, Test Method B (*9*). In addition, report the average of the five dry film thickness measurements (as measured by thickness gauge).

3.4 Pull-Off Adhesion

3.4.1 Test Description

The test method outlined in ASTM D 4541 (*10*) covers a procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating by determining either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached or whether the surface remains intact at a prescribed force (pass/fail). Failure will occur along the weakest plane within the system comprised of the test fixture, adhesive, coating system, and substrate, and will be exposed by the fracture surface. This test method maximizes tensile stress as compared with shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable. Further, pull-off strength measurements depend upon both material and instrumental parameters. Results obtained using different devices or results for the same coatings on substrates having different stiffness may not be comparable.

At least 10 test pulls shall be used for the performance testing and up to 30 test pulls to characterize the comparisons to the baselines. There are a few physical restrictions imposed by the general methods and apparatus. The selected test area must be a flat surface large enough to support the test fixture, have enough perpendicular and radial clearance, and be rigid enough to support the counter force.

Clean the loading fixture and the coating surface to be bonded. Use care to select only those solvents that will not attack the coating and/or leave residues on the fixture. Prepare and apply the adhesive to the fixture or the surface to be bonded in accordance with the adhesive manufacturer’s recommendations, being certain that the entire bonding surface is covered. Based on the manufacturer’s recommendations, allow enough time for the adhesive to cure. Carefully connect the central grip of the detaching assembly to the loading fixture, without bumping, bending, or otherwise prestressing the sample, and connect the detaching assembly to its control mechanism if necessary. After setting the force indicator to zero, increase the load to the fixture in as smooth and continuous manner as possible, at a rate of less than 150 psi/s (1 MPa/s) so that failure occurs or the maximum stress is reached in about 100 s or less.

3.4.2 Rationale

This test method maximizes tensile stress as compared with shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable.

3.4.3 Test Methodology

The test methodology for pull-off adhesion testing is given in table 9.

Table 9. Test methodology for pull-off adhesion testing.

Parameters	As defined in ASTM D 4541 (*10*).
Number and Type of Specimens Per Candidate Alternative	Three each of M1 and M2; include primer-only and topcoated panels.
Trials Per specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541 (*3*) for aluminum panels.
Acceptance Criteria	Minimum average 10 events rating of 1200 psi for pull strength; superior performance for IP would be 1800 psi and 2500 psi for BP. For aluminum, 2000 psi is required.

3.4.4 Equipment and Instrumentation

The following items will be used in testing:

- Commercially available adhesion tester or comparable apparatus as described in Annex A1–Annex A4 of ASTM D 4541 (*10*).
- Loading fixtures: device having a flat surface on one end that can be adhered to the coating and a means of attachment to the tester on the other end.
- Detaching assembly (adhesion tester): a central grip for engaging the fixture.
- Base: part of the detaching assembly, or an annular bearing ring if needed, for uniformly pressing against the coating surface around the fixture either directly or by way of an intermediate bearing ring. A means both of aligning the base is needed so that the resultant force is normal to the surface and moving the grip away from the base in as smooth and continuous manner as possible so that a torsion-free, coaxial (opposing pull of the grip and push of the base along the same axis) force results between them.
- Timer: means of limiting the rate of stress to less than 150 psi/s (1PPa/s) so that the maximum stress is obtained in less than about 100 s. A timer is the minimum equipment when used by the operator along with the force indicator.

3.4.5 Data Analysis

Rate the average results of each set of events.

3.5 Flexibility: Mandrel Bend

3.5.1 Test Description

This test evaluates the resistance to cracking (flexibility) of attached organic coatings on substrates of pretreated sheet metal. Determine flexibility in accordance with ASTM D 522, Test Method B (*11*). Test specimens/coupons shall be prepared with the appropriate pretreatment being tested. These test specimens/coupons shall be wiped and cleaned with solvent prior to applying epoxy primer. Apply epoxy primer conforming to MIL-DTL-53022, type IV (*23*), or MIL-DTL-53030, type II (*24*), to a dry film thickness of 1.5 ± 0.2 mil (37.5 ± 5 µm). Air-dry the specimens/coupons for 7 days. Bend the coated specimens/coupons over a 1/4-in (preferred) or 1-in mandrel, depending upon the type of coupons. Examine the coating for cracks or delamination over the area of the bend.

3.5.2 Rationale

This test method covers the determination of the resistance to cracking (flexibility) of attached organic coatings on metal substrates.

3.5.3 Test Methodology

The test methodology for flexibility of mandrel bend is given in table 10.

Table 10. Test methodology for flexibility of mandrel bend.

Parameters	Cured primer test specimens; 1-in (25.6-mm) mandrel.
Number and Type of Specimens Per Candidate Alternative	M1 and M2; primer only.
Trials Per specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541 (*3*) for aluminum panels.
Acceptance Criteria	Test specimen shall exhibit no cracking or delamination when tested.

3.5.4 Equipment and Instrumentation

The following items will be used in testing:

- 1-in mandrel
- Illuminated magnifier to check for cracks and delamination

3.5.5 Data Analysis

Report cracking or delamination of coating on pretreated substrate of coating system.

3.6 Wet Tape Adhesion and Water Resistance

3.6.1 Test Description

This test method covers a procedure for establishing adequacy of intercoat and surface adhesion of an organic coating immersed in water by applying pressure-sensitive tape over a scribed area of coating. The test also measures the coating's ability to resist penetration by water. Perform this test in accordance with Method 6301 of FED-STD-141 (*12*) and rate according to ASTM D 3359 (*9*). Per MIL-DTL-53022E (*23*), a film of primer, tested as specified in 4.16, shall show no wrinkling or blistering immediately after removal of the panel from the water. The primer shall be no more than slightly affected when examined 2 h after removal. After 24 h air-drying, the portion of the panel which was immersed shall be the same with regard to hardness, adhesion, color, and gloss as compared with the portion not immersed.

Each test panel will be subjected to a cross-hatch tape test. For primer-only panels, a cross-hatch tool making 11 cuts 1 mm apart will be used. All cuts should be through the coating and into the substrate and no closer than 12 mm from any edge. Each line of the cross-hatch should be at least 1.5 in long. Immediately place a piece of tape over the incision parallel to the bottom edge of the panel, ensuring that the tape completely covers the "square" formed by the cross-hatch, and smooth out the tape by rolling a 3-lb roller over it once. Remove the tape rapidly at approximately a 180° angle. Inspect the incision area for peel away. Loss of two or more complete squares shall constitute failure.

3.6.2 Rationale

This test was identified as a performance requirement for the baseline pretreatment specifications TT-C-490 (*2*) and MIL-DTL-5541 (*3*) when applying specified primers. Loss of paint adhesion is the primary failure mode on ferrous and aluminum substrates.

3.6.3 Test Methodology

The test methodology for wet-tape adhesion and water-resistance testing is given in table 11.

Table 11. Test methodology for wet-tape adhesion and water-resistance testing.

Parameters	7-day cured primed panels; 24-h deionized (DI) water immersion at room temperature.
Number and Type of Specimens Per Candidate Alternative	Three each of M1 and M2. Include primer-only and topcoated panels.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541(*3*) for aluminum panels.
Acceptance Criteria	No peel away; at least 4A per ASTM D 3359 (*9*); no blistering of unscribed coating area.

3.6.4 Equipment and Instrumentation

- 1-in masking tape, 3M Company Type 250 only, less than 1 year old.
- Cross-hatch cutter such as PA-2056 and PA-2053 available from Gardco.
- 3-lb rubber covered roller.

3.6.5 Data Analysis

Report all information per the latest version of ASTM D 3359, Method A (*9*). Report also any delamination or blistering in areas away from the scribes.

3.7 Pencil Hardness After Water Immersion

3.7.1 Test Description

This test method covers a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate in terms of drawing leads or pencil leads of known hardness. Perform this test and rate in accordance to ASTM D 3363 (*13*) and MIL-DTL-53022E (*23*).

A coated panel consisting of pretreatment coating and epoxy primer is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (point away from the operator) and pushed away from the operator in a 6.5-mm (1⁄4-in) stroke. The process is started with the hardest pencil and continued down the scale of hardness to either of two end points: (1) the pencil that will not cut into or gouge the film (pencil hardness) or (2) the pencil that will not scratch the film (scratch hardness).

A film of primer, immersed in distilled water per ASTM D 1308 (*14*) for 168 h or 7 days, shall show no wrinkling or blistering immediately after removal of the panel from the water. The primer shall be no more than slightly affected when examined 2 h after removal. After 24 h air-drying, the portion of the panel that was immersed shall be the same with regard to hardness, adhesion, color, and gloss as compared with the portion that was not immersed. Film softening shall not exceed a no. 2 pencil hardness difference (see ASTM D 3363 [*13*]) from an unexposed film with identical cure history prior to water exposure.

3.7.2 Rationale

This test was identified as a performance requirement for designated epoxy primers when applied over the required conversion pretreatment coatings. Loss of paint adhesion is the primary failure mode on aluminum and steel.

3.7.3 Test Methodology

The test methodology for pencil hardness after water-immersion testing is given in table 12.

Table 12. Test methodology for pencil hardness after water immersion testing.

Parameters	7-day cured primed; 168-h DI water immersion at room temperature.
Number and Type of Specimens Per Candidate Alternative	M1, M2; primer only.
Trials Per specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541(*3*) for aluminum panels.
Acceptance Criteria	>2 pencil hardness difference from an unexposed film; no blistering of unscribed coating area.

3.7.4 Equipment and Instrumentation

- Set of calibrated drawing leads meeting the following scale of hardness:

$$\underline{6B\text{-}5B\text{-}4B\text{-}3B\text{-}2B\text{-}B\text{-}HB\text{-}F\text{-}H\text{-}2H\text{-}3H\text{-}4H\text{-}5H\text{-}6H}$$

Softer Harder

- Mechanical lead holder (drawing leads only).
- Mechanical sharpener, draftsman-type (wood pencils only).
- Abrasive paper, grit no. 400.
- Distilled water and container.

3.7.5 Data Analysis

Rate the results according to ASTM D 3363 (*13*). Include with the results discoloration, loss of gloss, delamination, wrinkling, and blistering.

3.8 Pencil Hardness After JP-8 Jet Fuel Immersion

3.8.1 Test Description

This test method covers a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate in terms of drawing leads or pencil leads of known hardness. Perform this test and rate in accordance to ASTM D 3363 (*13*) and MIL-DTL-53022E (*23*).

A coated panel consisting of pretreatment coating and epoxy primer is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (point away from the operator) and pushed away from the operator in a 6.5-mm (1⁄4-in) stroke. The process is started with the hardest pencil and continued down the scale of hardness to either of two end points: (1) the pencil that will not cut into or gouge the film (pencil hardness) or (2) the pencil that will not scratch the film (scratch hardness).

A film of primer, immersed in JP-8 jet fuel for 168 h or 7 days, shall show no blistering or wrinkling and no more than a slight yellow to beige color change on submerged area of panel. Upon removal from the fluid, slight softening is acceptable. After 2 h air-drying, the panel that was immersed shall be almost indistinguishable with regard to hardness, adhesion, color, and gloss from a panel prepared at the same time but not immersed. Film softening shall not exceed a no. 2 pencil hardness difference (see ASTM D 3363) (*13*) from an unexposed film with identical cure history prior to hydrocarbon fluid exposure.

3.8.2 Rationale

This test was identified as a performance requirement for designated epoxy primers when applied over the required conversion pretreatment coatings. Loss of paint adhesion is the primary failure mode on aluminum and steel.

3.8.3 Test Methodology

The test methodology for pencil hardness after JP-8 immersion is given in table 13.

Table 13. Test methodology for pencil hardness after JP-8 immersion.

Parameters	Cured pretreated and primed panels; 168-h JP-8 immersion at room temperature.
Number and Type of Specimens Per Candidate Alternative	M1, M2; primer only.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541(*3*) for aluminum panels.
Acceptance Criteria	>2 pencil hardness difference from an unexposed film.

3.8.4 Equipment and Instrumentation

- Set of calibrated drawing leads meeting the following scale of hardness:

$$\underline{6B\text{-}5B\text{-}4B\text{-}3B\text{-}2B\text{-}B\text{-}HB\text{-}F\text{-}H\text{-}2H\text{-}3H\text{-}4H\text{-}5H\text{-}6H}$$
Softer Harder

- Mechanical lead holder (drawing leads only).
- Mechanical sharpener, draftsman-type (wood pencils only).
- Abrasive paper, grit no. 400.
- JP-8 and container.

3.8.5 Data Analysis

Rate the results according to ASTM D 3363 (*13*). Include with the results discoloration, loss of gloss, delamination, wrinkling, and blistering.

3.9 Stress Corrosion Cracking

3.9.1 Test Description

Hydrogen embrittlement testing shall be performed on any candidate that is considered a risk candidate. Resistance to environmentally assisted cracking shall be assessed using the rising step load method for determination of K_{IEAC}. For this procedure, CV2 Charpy specimens of MIL-A-46100D (*29*) shall be machined in longitudinal-transverse (L-T) and transverse-longitudinal (T-L) orientations in accordance with ASTM E 399-97 (*15*). Unlike the steel test panels, the charpy specimens shall not be abrasive blasted prior to pretreatment.

Specimen fatigue precracking shall be carried out using three stages, each consisting of decreasing loading levels. In the first precracking stage, the load shall be maintained to keep stress intensity values below 80% of the estimated experimental critical stress intensity and the stress ratio ($_{max}/\sigma_{min}$) kept between –1 and +0.1. In the intermediate stage, the cycling load shall be reduced to maintain the stress intensity value as crack growth occurs and the intact cross section reduced. For the final stage of precracking, the load shall be further reduced so the final value of K_{max} will unlikely exceed 60% of the estimated value for K_I during experimentation. Additionally, the final value for K_{max}/E should not exceed 0.0032 $m^{1/2}$, where E is Young's modulus. Precrack length, represented by the dimensionless expression a/W (crack length over specimen width), shall be maintained near 0.5.

Specimens shall be fastened into a double cantilever array test fixture under aqueous conditions with 3.5% NaCl solution at open circuit potential conditions. Specimens shall be loaded by incremental steps in accordance with ASTM F 1624-95 (*30*) using an appropriate load frame apparatus. The specimen load values versus time shall be recorded. The calculation for the onset of environmentally assisted cracking, or K_{IEAC}, is derived as follows for cantilever bending from the four-point bending expression.

$$K_{IEAC} = \left(\frac{6M_{IEAC}\sqrt{\pi a}}{BW^2} \right) \times f(a/W) \qquad (1)$$

3.9.2 Rationale

The calculation of stress corrosion cracking (hydrogen embrittlement) is necessary for assurance that this pretreatment can be applied to ferrous substrates with Rockwell hardness greater than 39.

3.9.3 Test Methodology

The test methodology for stress corrosion cracking testing is given in table 14.

Table 14. Test methodology for stress corrosion cracking testing.

Parameters	CV2 Charpy specimens of MIL-A-46100D (*29*).
Number and Type of Specimens Per Candidate Alternative	Pretreatment only on coupons for stress-cracking testing.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous substrates.
Acceptance Criteria	Results equal to or lower than baseline zinc phosphate.

3.9.4 Equipment and Instrumentation

The equipment shall be determined by the applicable test method.

3.9.5 Data Analysis

Report results of the stress relief cracking as compared to the baseline zinc phosphate pretreatment.

3.10 Marine Environment Outdoor Exposure.

3.10.1 Test Description

This test method evaluates a coating system's (pretreatment/primer/topcoat) ability to prevent substrate corrosion. Outdoor exposure testing will be conducted at the Cape Canaveral Air Force Station (CCAFS) Corrosion Test Site, located 100 yards from and faces the Atlantic Ocean. Test coupons are installed on either wooden or corrosion-resistant metal racks using plastic insulator stand-offs with stainless steel fasteners. The rack angle of the coupons is 30° from horizontal. Figure 2 shows an example of the type of exposure racks that will be used.

Figure 2. CCAFS exposure rack.

Prepare at least five specimens consisting of manufactured parts that accurately simulate current production material and manufacturing processes, incorporating the candidate zirconium oxide pretreatment, and five specimens incorporating the current corrosion protection system. If manufactured parts are not available, panels shall be prepared from the same representative metallic substrates and processed under the same manufacturing process.

Scribe an X incision through the coating and to the substrate so that the smaller angle of the X is 30°–45° as described in ASTM D 3359 (*9*). The scribe should cross the entire length of the panel but ending 1 in from the edges. The edges and back of the panel are properly coated with the same coating system as the one being tested. Test samples are stamped or engraved with an identifying marking under the coating on their backs on the top left corner within a 1/2-in margin from either edge.

Evaluate coupons for surface corrosion (blisters in field) and creep from scribe as per ASTM D 1654 (*5*) at 6-month intervals for 2 years. At each inspection interval, remove and visually examine the coupons using a measuring magnifier device. Corrosive salts or oxides from the scribes running down the surface of the coupon are ignored in these measurements. The inspection is more concerned with perforation of the film than staining. Photos are taken of test coupons at each inspection interval. Upon completion of testing, test coupons are transported to ARL at Aberdeen Proving Ground, MD, for scanning. After being scanned, each panel is scraped as described in TT-C-490 (*2*), rerated using ASTM D1654 (*5*), and scanned a final time.

3.10.2 Rationale

The 24-month outdoor exposure of scribed, painted substrates is a key accelerated test to determine overall corrosion inhibition and paint adhesion compared to controls.

3.10.3 Test Methodology

The test methodology for marine environment outdoor exposure testing is given in table 15.

Table 15. Test methodology for marine environment outdoor exposure testing.

Parameters	Test coupons at a 30° angle. Temperature of exposed coupons varies with outdoor conditions. 24 months (interim ratings at 6, 12, and 18 months).
Number and Type of Specimens Per Candidate Alternative	M1, M2; primed and topcoated.
Trials Per Specimen	1
Experimental Control Specimens	Zinc phosphate pretreatment according to TT-C-490 (*2*) for ferrous panels and conversion coating conforming to MIL-DTL-5541(*3*) for aluminum panels primed with MIL-DTL-53022 (*23*) and topcoated with MIL-DTL-64159 (*26*).
Acceptance Criteria	Equal to or better than experimental control specimens, but having a minimum rating as described in section 3.1 for acceptance criteria.

3.10.4 Equipment and Instrumentation

- Standard racks: see section 5 of ASTM G 50 (*20*).
- Scribe tool: carbide tip scribe as per ASTM D 1654 (*5*).
- Straight edge: any straight edge of sufficient length to guide the scribing tool in a straight line.

3.10.5 Data Analysis

Report the final corrosion test results and 6-month visual observations. Panels are rated according to ASTM D 1654 (*5*).

3.11 Field Exposure, On-Vehicle (ASTM D 1654) (*5*)

3.11.1 Test Description

This test method describes a basic procedure for conducting on-vehicle testing of candidates. This may be performed by selective replacement or refinishing of an appropriate representative substrate/component on a vehicle incorporating the candidate or by the use of test specimens incorporating the candidate attached to the military ground vehicle. This method describes a procedure for monitoring a coating system's (surface treatment/primer/topcoat) performance when applied to specific target areas of an operational platform as compared to the baseline system. The pretreatment and coating system's ability to maintain adhesion to the substrate after exposure to field conditions and to prevent corrosion will be analyzed.

At a minimum, the process shall be conducted to replace or refinish a part or section of the vehicle in accordance with the suggested finishing parameters and the controls established by the CARC applications specification MIL-DTL-53072 (*1*). If using test panels, they shall be prepared in accordance with ASTM G 50 (*20*) for static field testing and evaluated using ASTM D 1654 (*5*). Identify target components of the platform (this will depend on agreements among programs, depot, or logistics center personnel, manufacturers, and the principal investigator) that will observe a significant amount of operational exposure to the environment, representative of the demonstration site. Representative substrates/components will be pretreated in accordance with pretreatment manufacturers recommended specifications finishing parameters and controls established in MIL-DTL-53072 (*1*).

Components substrates will be evaluated during periodic inspections by visual comparison with the base vehicle or control samples attached to the vehicle. Inspection of the demonstration platform or target areas of the demonstration platform shall be at approximate intervals of 3 months, 6 months, 1 year, and 2 years. The Society for Protective Coatings SSPC-VIS-2 (*31*) shall be used for evaluating component substrates and control samples. The success criteria for field testing will be performance greater than or equal to the base vehicle (baseline) or control sample.

3.11.2 Rationale

Pretreatment coatings must perform successfully in the field before they are transitioned to the end users via revisions of technical manuals, military specifications, and qualified products databases.

3.11.3 Test Methodology

The test methodology for field exposure testing is given in table 16.

Table 16. Test methodology for field exposure testing.

Parameters	Ferrous or mixed substrate components.
Number and Type of Specimens Per Candidate Alternative	Number of platforms required is determined by cognizant authority.
Trials Per Specimen	1
Experimental Control Specimens	GSE components coated with standard coating system.
Acceptance Criteria	≥2-year operational service or other user-defined interval, with a minimum of comparable performance with similar platform coated with standard system.

3.11.4 Equipment and Instrumentation

Military ground vehicle: vehicle used for standard deployment.

3.11.5 Data Analysis

After a predetermined exposure agreed upon by the stakeholders, the affected vehicles/parts shall be evaluated for coating adhesion, color, and corrosion resistance in accordance with SSPC-VIS-2 (*31*), MIL-DTL-53072 (*1*), and ASTM D 1654 (*5*).

4. References

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Appendix. Participating Organizations and Representatives

Organization	Representative	Type
Army Research Laboratory	Fred Lafferman (410) 306-1520	Government
Army Aviation and Missile Command	Mark Feathers (256) 842-7355	Government
Army Tank and Automotive Command	Daniel Nymberg (586) 282-7445	Government
Naval Surface Warfare Center, Carderock Division	Andrew Sheetz (301) 227-5037	Government
Letterkenny Army Depot	Dennis Reed (717) 267-8376	Government
Marine Corps Logistics Base Albany	Steve Allen (229) 639-5310	Government
Army Research Laboratory	John Escarsega (410) 306-0693	Government
PPG Industries	Larry Fitzgerald (412) 492-5396	Contractor
PPG Industries	Jonathan Love (412) 492-5206	Contractor

List of Symbols, Abbreviations, and Acronyms

ANSI	American National Standards Institute
ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
BP	best performance
CARC	chemical agent–resistant coating
cfm	cubic feet per minute
CPAC	Corrosion Prevention and Control
$Cr6^{+}$	hexavalent chromium
DOD	Department of Defense
EAC	environmentally assisted cracking
ESTCP	Environmental Security Technology Certification Program
FZA	fluorozirconic acid
GMW	General Motors Worldwide
GSE	ground support equipment
HazMat	hazardous material
HHA	high-hard armor
IP	improved performance
JTP	joint test protocol
JTR	joint test report
MMC	metal-matrix composite
MP	minimum performance
Ni	nickel
NSS	neutral salt spray
OEM	original equipment manufacturer

pH	hydrogen ion concentration
SSPC	The Society of Protective Coatings
TACOM	U.S. Army Tank Automotive Command

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