NASA Technology Evaluation for Environmental Risk Mitigation
Kennedy Space Center, FL 32899

Demonstration/Validation of Environmentally-preferable Coatings for Launch Facilities

Test Protocol
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National Aeronautics and Space Administration (NASA)

Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM)

Test Protocol

For Demonstration/Validation of Environmentally-preferable Coatings for Launch Facilities

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PREFACE

This report was prepared by International Trade Bridge, Inc. (ITB) through the National Aeronautics and Space Administration (NASA) Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM). The structure, format, and depth of technical content of the report were determined by NASA TEERM, Government contractors, and other Government technical representatives in response to the specific needs of this project.

We wish to acknowledge the invaluable contributions provided by all the organizations involved in the creation of this document.

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LIST OF ACRONYMS/SYMBOLS

ΔE	One (1) unit of color change
°F	Degree Fahrenheit
A36	ASTM A 36 Carbon Steel
ASTM	American Society for Testing and Materials
CCS	Composite Carbon Steel
DFT	Dry Film Thickness
ft-lb	Foot-pound
GSE	Ground Support Equipment
ITB	International Trade Bridge, Inc.
J	Joules
KSC	John F. Kennedy Space Center
LOX	liquid oxygen
MEK	Methyl Ethyl Ketone
MMH	Monomethylhydrazine
N_2O_4	Nitrogen Tetroxide
N_2H_2	Hydrazine
NACE	National Association of Corrosion Engineers
NASA	National Aeronautics and Space Administration
RH	Relative Humidity
SSPC	The Society for Protective Coatings
TEERM	NASA Technology Evaluation for Environmental Risk Mitigation Principal Center

1. INTRODUCTION

Headquarters National Aeronautics and Space Administration (NASA) chartered the Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM) to coordinate agency activities affecting pollution prevention issues identified during system and component acquisition and sustainment processes. The primary objectives of NASA TEERM are to:

- Reduce or eliminate the use of hazardous materials or hazardous processes at manufacturing, remanufacturing, and sustainment locations.
- Avoid duplication of effort in actions required to reduce or eliminate hazardous materials through joint center cooperation and technology sharing.

Kennedy Space Center (KSC) is responsible for a number of facilities/structures with metallic structural and non-structural components in a highly corrosive environment. Metals require periodic maintenance activity to guard against the insidious effects of corrosion and thus ensure that structures meet or exceed design or performance life. The standard practice for protecting metallic substrates in atmospheric environments is the application of an applied coating system. Applied coating systems work via a variety of methods (barrier, galvanic and/or inhibitor) and adhere to the substrate through a combination of chemical and physical bonds.

Maintenance at KSC and other NASA Centers is governed by NASA-STD-5008B (*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*) which establishes practices for the protective coating of ground support equipment and related facilities used by or for NASA programs and projects. The Standard is for the design of non-flight hardware used to support the operations of receiving, transportation, handling, assembly, inspection, test, checkout, service, and launch of space vehicles and payloads at NASA launch, landing, or retrieval sites. These criteria and practices contained within the Standard may be used for items used at the manufacturing, development, and test sites upstream of the launch, landing, or retrieval sites.

The objective of this effort is to demonstrate and validate environmentally-preferable alternatives in accordance with NASA-STD-5008B and KSC requirements which can then be added to the Approved Products List.

This Test Protocol contains the critical requirements and tests necessary to qualify alternatives for structural steel applications. These tests were derived from engineering, performance, and operational impact (supportability) requirements defined by a consensus of KSC participants. A Test Report will document the results of the testing as well as any test modifications made during the execution of the testing. Users of this Test Protocol should check the project's Test Report for additional test details or minor modifications that may have been necessary in the execution of the testing. The technical stakeholders will have agreed upon test procedures modifications documented in the Test Report.

2. ENGINEERING, PERFORMANCE, AND TESTING REQUIREMENTS

A joint group led by TEERM and consisting of technical representatives from NASA reached technical consensus on engineering, performance, and testing requirements for environmentally-preferable alternative coatings. The joint group defined critical tests with procedures, methodologies, and acceptance criteria to qualify alternatives against these technical requirements.

Once the test criterion is approved, testing will be performed in a manner that will optimize the use of each test panel. For example, where practical, more than one type of test will be performed on the coated test panels. The number and types of tests performed on a given panel will be determined by the destructive nature of the tests in question.

All coating system candidates will be tested using an approved NASA-STD-5008B coating system as the experimental control. Coating technicians will follow all manufacturer application instructions and will document all relevant conditions at the time of application.

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

The objective of this project is to qualify the candidates under the specifications for the standard coating system. This project will compare coating performance of the proposed alternatives to existing coating systems or standards. The tests described in this Test Protocol are divided into two (2) phases. Tables 1 and 2 summarize the test requirements for validating alternative coating candidates against existing approved coating systems.

Table 1 and Table 2 show the Phase 1 and Phase 2 tests, respectively. Phase 1 tests are the preliminary tests to be performed on the candidate coating systems. Candidate coatings that do not meet the requirements of the Phase 1 tests will be eliminated from further testing unless otherwise directed by the testing authority.

These tables include acceptance criteria and the reference specifications, if any, used to conduct the tests. All test method references shall use the most recent version unless otherwise specified. The proposed test and evaluation are based on the aggregate knowledge and experience of the assigned technical project personnel and prior testing where "None" appears under *Test Method References*.

Table 1 Phase 1 Testing Requirements for Environmentally-preferable Coatings				
Test	***Section***	***Test Specimen***	***Acceptance Criteria***	***Test Methodology References***
Pot Life	3.1.1.	Mixed Coating System	Equal to or better than control coating based upon Applicator Evaluation	None
Ease of Application	3.1.2.	Coupon	Based on Applicator Evaluation: Smooth coat, with acceptable appearance, no runs, bubbles or sags; Ability to cover the properly prepared/primed substrate with a single coat (one-coat hiding ability); Measure Dry Film Thickness.	SSPC-PA-2
Surface Appearance	3.1.3.	Coupon	Based on Applicator Evaluation: No streaks, blistering, voids, air bubbles, cratering, lifting, blushing, or other surface defects/irregularities; No micro-cracks observable at 10X magnification	ASTM D 523; ASTM D 2244
Atmospheric Exposure	3.1.4.	Coupon	Gloss/color change and panel condition of candidate coating rated equal to or better than control coatings	ASTM D 610; ASTM D 714; ASTM D 523; NASA-STD-5008B
Heat Adhesion	3.1.5.	Coupon	No loss of adhesion after heating	ASTM D 4541; NASA-STD-5008B

Table 2 Phase 2 Testing Requirements for Environmentally-preferable Coatings				
Test	***Section***	***Test Specimen***	***Acceptance Criteria***	***References***
Hypergol Compatibility	3.2.1.	Coupon	Slight to Moderate Reactivity Observed: When test data based on visual observations with the unaided eye reveal reactivity (but no ignition) and/or any changes in the visual characteristics, bulk characteristics, and/or surface characteristics of the test sample	KSC MTB-175-88; NASA-STD-6001
LOX Compatibility	3.2.2.	Coupon	Twenty samples must not react when impacted at 72 foot-pounds [ft-lbs or 98 Joules (J)]. If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.	ASTM D 2512; NASA-STD-6001
Cure Time (MEK Solvent Rub)	3.2.3.	Coupon	Coating will be tested every two (2) days for a total of 14 days; No effect on surface or coating on the cloth (Resistance Rating 5)	ASTM D 4752
Tensile (Pull-off) Adhesion	3.2.4.	Coupon	Pull-off strength achieved at time of failure equal to or better than control coatings	ASTM D 4541
Removability	3.2.5.	Coupon	Less than one minute to penetrate substrate; Tested during Reparability and Abrasion Resistance Tests; Measure Dry Film Thickness of remaining coating	ASTM G 155; SSPC-PA-2

Table 2 Phase 2 Testing Requirements for Environmentally-preferable Coatings				
Test	***Section***	***Test Specimen***	***Acceptance Criteria***	***References***
Reparability	3.2.6.	Coupon	Ease of removal and replacement of damaged areas of the test coatings, color matching of aged versus new material; No streaks, blistering, voids, air bubbles, over-spray "halo", cratering, lifting, blushing, or other surface irregularities, No peel away of the repaired coating during the dry tape adhesion test	ASTM D 523; ASTM D 2244; ASTM D 3359
Mandrel Bend Flexibility	3.2.7.	Coupon	No peeling or delamination from the substrate and no cracking greater than ¼-inch from the edges.	ASTM D 522

3. TEST DESCRIPTIONS

Test requirements identified in Tables 1 and 2 are further defined in this section to include the test description, rationale, and test methodology. The *Test Methodology* lists the major parameters, test coupon descriptions, number of test coupons, number of coupons per coating system, number of control coupons and acceptance (pass/fail) criteria. Any U*nique Equipment or Instrumentation* requirements and *Data Analysis and Reporting Criteria* are also included.

The coating of coupons will be documented using the Application Record Sheet in NASA-STD-5008B (*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*), or an equivalent form. For each test requiring coupons, a minimum of five (5) coupons shall be prepared; those with the best coating as determined by the technician shall be used in accordance with the number of coupons required as specified in the *Test Methodology*. Unless otherwise required by a specific test, all coupons will be prepared as follows:

Unless otherwise noted, test coupons will be four (4) inches wide by six (6) inches long and of suitable thickness. Metal coupons shall be prepared in accordance with NACE-STD-RP0281 [*Method for Conducting Coating (Paint) Panel Evaluation Testing in Atmospheric Exposures*]. Surface roughness shall be measured in accordance with NACE-STD-RP0287 (*Field Measurements of Surface Profile of Abrasive Blast Cleaned steel Surfaces Using a Replica Tape*) and recorded for informational purposes prior to application of the primer.

The surface conditioning for steel test coupons will be in accordance with the Society of Protective Coating Standards SSPC-SP-1 (*Solvent Cleaning*) and SSPC-SP-5 (*White Blast Cleaning*). Each test will be performed on identical test panels prepared with the candidate alternative coating systems and the NASA standard control coating as the test control.

Test coupons shall be allowed 24 hours of unaided drying time prior to dry film thickness measurements. If powder coatings are being tested, test coupons shall be cured in accordance with manufacturer's recommendations prior to dry film thickness measurements. The dry film thickness measurements shall be made nondestructively in accordance with SSPC-PA-2 (*Measurement of Dry Coating Thickness with Magnetic Gages*).

If liquid coatings are being tested for a destructive test, coupons shall be allowed to cure for an additional 14 days before they undergo any destructive testing to ensure full polymerization of the coating. Coating process parameters, including application method and cure schedule, shall be documented by the facility that prepares the test coupons.

Each coating system will be prepared and applied according to instructions provided by the manufacturer. Coating systems should be applied by spraying, or, in the case of advanced film technology, by hand to the dry film thickness recommended by the coating manufacturer. Application should be conducted at a minimum temperature of 75 ± 5 degrees Fahrenheit (°F) and 50 ± 10% relative humidity (RH), unless otherwise specified. The coating system may be applied in one or two coats if allowed by the manufacturer and provided that the manufacturer's instructions are carefully followed. Unless otherwise specified, test panels with organic topcoats should be held at 75 ± 5 °F and 50 ± 10% RH prior to testing.

If a topcoat is to be applied over the primer, the topcoat should be applied within 24 hours of primer application. In many cases, the topcoat will be applied before the primer is fully cured; however, the topcoat should never be applied sooner than specified by the manufacturer or before the primer is dry to the touch (dry-to-handle). Unless otherwise specified, the topcoat should be applied to the total dry film thickness recommended by the coating manufacturer.

Table 3 contains a listing of substrate types that will be used for testing and their test specimen code.

Table 3 Test Specimen Codes and Substrate Descriptions

Test Coupon Code	***Substrate Description***
A36	Carbon Steel Four (4) inch x six (6) inch x 3/16 inch panels fabricated from ASTM A 36 hot rolled carbon steel; primed, intermediate coated (if required), and topcoated with the candidate coating system. Coatings applied per the coating manufacturer specifications.
CCS	Composite Carbon Steel Four (4) inch x six (6) inch x 3/16 inch panels with a one (1) inch channel welded on front face fabricated from ASTM A 36 hot rolled carbon steel; primed, intermediate coated (if required), and topcoated with the candidate coating system. Coatings applied per the coating manufacturer specifications.

3.1. Phase 1 Tests for Environmentally-preferable Alternative Coatings

Coating systems that meet the requirements of the Phase 1 tests will be subjected to the additional tests listed in this Test Protocol. The initial screening of the coating candidates will compare the test candidates against the control coating as described in each *Test Methodology*.

3.1.1. Pot Life

Test Description

This test will be conducted based on the Applicator Evaluation and will make note of any issues experienced by the applicator during the application process.

Rationale

This test provides data to characterize the pot life envelope. Pot life is a concern for participants.

Test Methodology

Table 4 Test Methodology for Pot Life Test	
Parameters	Applicator Evaluation
Coupons Per Coating System	None
Control Coupons Required For Testing	None
Acceptance Criteria	Equal to or better than the control coating system

Unique Equipment or Instrumentation

- None

Data Analysis and Reporting

- Report notes on the Application Record Sheet in NASA-STD-5008B, or an equivalent form.

3.1.2. Ease of Application

Test Description

This procedure is used to determine how easily a coating system may be applied.

Prepare the test coupons as described in Section 3, noting the appropriate coating application processes and equipment. This evaluation will be conducted while

preparing test coupons for each coating described in this Test Protocol. Accomplish tests at 75 ± 5 °F and 50 ± 10% RH.

The Dry Film Thickness (DFT) shall be measured in accordance with SSPC-PA-2 (*Measurement of Dry Coating Thickness with Magnetic Gages*).

Rationale

This screening test is conducted to identify and eliminate those candidate coating systems that are difficult to properly apply under normal maintenance operation conditions. All participants have agreed that *Ease of Application* is a performance requirement.

Test Methodology

Table 5 Test Methodology for Ease of Application Test	
Parameters	Coating Manufacturer instructions; 75 ± 5 °F and 50 ± 10% RH
Coupons Per Coating System	Not Applicable
Trials Per Test Coupon	One (1)
Control Coupons Required For Testing	Not Applicable
Acceptance Criteria	Smooth coat, with acceptable appearance, no runs, bubbles, or sags. Ability to cover the properly prepared/primed substrate with a single coat (one coat hiding ability). Measure DFT.

Unique Equipment and Instrumentation

- Magnetic Gage per SSPC-PA-2

Data Analysis and Reporting

- Report applicator evaluation of the coating application and DFT in accordance with SSPC-PA-2 on the Application Record Sheet in NASA-STD-5008B, or an equivalent form.

3.1.3. Surface Appearance

Test Description

The purpose of this test is to evaluate and compare the surface appearance of the candidate and control coating systems.

Examine the surface of each coated test coupon for coating defects with unaided eye and with 10X magnification. Micro-cracks extending no more than ¼-inch from the panel edge are acceptable. A slight orange peel appearance is acceptable. Color and gloss measurements shall be conducted on each coated coupon per ASTM D 2244 (*Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates*) and ASTM D 523 (*Standard Test Method for Specular Gloss*), respectively, to document the specular gloss of the original finish of the control test coupons. The surface appearance of the topcoat is required to be evaluated only after the entire primer/topcoat system has been applied.

Rationale

This test is conducted to provide critical detailed evaluation of coating appearance and integrity. All participants agreed the surface appearance evaluation is a performance requirement.

Test Methodology

Table 6 Test Methodology for Surface Appearance Test

Parameters	10X Magnification
Coupons Per Coating System	Not Applicable
Trials Per Test Coupon	One (1)
Control Coupons Required For Testing	One (1)
Acceptance Criteria	No streaks, blistering, voids, air bubbles, cratering, lifting, blushing, or other surface defects/irregularities. No micro-cracks observable at 10X magnification.

Unique Equipment or Instrumentation

- 10X optical magnifier
- Hunter Lab "Miniscan" Spectrophotometer (using CIE L*a*b* Color Measurement System) or equivalent
- Hunter Lab "Progloss" Meter or equivalent

Data Analysis and Reporting

- Measure and report observation on any coating defects, original color readings, and gloss readings on the Application Record Sheet in NASA-STD-5008B, or an equivalent form.

3.1.4. Atmospheric Exposure Test

Test Description

This test evaluates the performance of the test and control coatings after an 18-month outdoor exposure in a marine environment.

The four (4) inch x six (6) inch CCS panels shall be used for this test. Coat all surfaces of the test panels with the prescribed coating. Install the test panels at the Kennedy Space Center (KSC) outdoor exposure rack 150 feet from the ocean high tide line. Follow all KSC test rack procedures for fasteners, exposure angle, and inspection interval.

At the conclusion of the test, rate the test coupon condition per ASTM D 610 (*Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces*). Use the numerical grade scale in ASTM D 610, Table 1, *Scale and Description of Rust Grades*, where 0 indicates 100% surface rusting and 10 indicating less than 0.01% surface rusting.

At the conclusion of the test, rate the test coupon condition per ASTM D 714 (*Standard Test Method for Evaluating Degree of Blistering of Paints*); use the reference standards in section 3. Also at the conclusion of testing, measure gloss changes on each coated coupon per ASTM D 523 (*Standard Test Method for Specular Gloss*).

Rationale

This test documents the actual exposure of the coatings to ultraviolet radiation, as well as different cycles of salt spray exposure, and heat adhesion. NASA requires this test for validation of alternative coating systems.

Test Methodology

Table 7 Test Methodology for Atmospheric Exposure Test	
Parameters	Per NASA-STD-5008B, ASTM D 523, and ASTM D 714; NASA Corrosion Beach Test Site
Coupons Per Coating System	Three (3) CCS
Trials Per Coupon	One
Control Coupons Required For Testing	Three (3) CCS
Acceptance Criteria	Gloss change and panel condition (per ASTM D 610 and ASTM D 714) of candidate coating rated equal to or better than control coatings

Unique Equipment or Instrumentation

- Outdoor test rack located 150 feet from ocean high tide line
- BYK Gardener Micro Tri-Gloss Glossmeter or equivalent

Data Analysis and Reporting

- Report corrosion rating per ASTM D 610, Table 1; blister rating per ASTM D 714, Section 3; and gloss measurements per ASTM D 523.
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

3.1.5. Heat Adhesion

Test Description

This test evaluates the performance of primers after exposure to prolonged heat as required by NASA-STD-5008B.

Primer-only coated panels will be tested for tensile adhesion using ASTM D 4541 (*Standard Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers*). The same primer-only coated panels will then be exposed in a high temperature oven to a temperature of 750°F for 24 hours. The panels will be allowed to cool at room temperature. The coating will then be re-tested for tensile adhesion to check for adhesion loss or film deterioration caused by the heating. Loss of adhesion after heating will constitute a failure due to temperature effects on the film.

Rationale

This test documents the exposure of the primers to heat followed by adhesion testing. NASA requires this test for validation of alternative coating systems.

Test Methodology

Table 8 Test Methodology for Heat Adhesion Test	
Parameters	Per NASA-STD-5008B and ASTM D 4541
Coupons Per Coating System	Three (3) CCS
Trials Per Coupon	One
Control Coupons Required For Testing	Three (3) CCS
Acceptance Criteria	No loss of adhesion after heating

Unique Equipment or Instrumentation

- Outdoor test rack located 150 feet from ocean high tide line
- BYK Gardener Micro Tri-Gloss Glossmeter or equivalent

Data Analysis and Reporting

- Report the pre-heat and post-heat tensile adhesion per ASTM D 4541.
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

3.2. Phase 2 Tests for Environmentally-preferable Alternative Coatings

The engineering, performance, and testing requirements of candidate alternatives are listed in Table 2-2. Phase 1 tests that are intended for use prior to Phase 2 tests are described in Section 3.1.

3.2.1. Hypergol Compatibility

Test Description

This procedure evaluates the effects on coatings from casual exposure to hypergolic fluids [nitrogen tetroxide (N_2O_4), hydrazine (N_2H_4), and monomethylhydrazine (MMH)]. This procedure provides the method to determine if a fluid could react exothermally or spontaneously ignite on contact with a material.

This test will be performed in accordance with NASA KSC MTB-175-88 (*Procedure for Casual Exposure of Materials to Hypergolic Fluids*), Test Method 7.1, *Reactivity Test Method*. The materials to be tested shall be identified on the "Material Identification Form" from KSC Report MTB-175-88 Figure 1, or an equivalent form. The results of the tests shall be recorded on the "Chemical Analysis Laboratory Compatibility Report," KSC Form 3-539NS, or an equivalent form.

The alternative coatings shall be applied in a thickness equivalent to normal use on aluminum foil measuring four (4) inches by four (4) inches and cured, if necessary, in accordance with the manufacturer's instructions.

Rationale

This test is specified in NASA-STD-6001 (*Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion*) and was identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified ground support equipment (GSE) must meet the requirements of this document.

Test Methodology

Table 9 Test Methodology for Hypergol Compatibility Test	
Parameters	Per NASA KSC MTB-175-88: N_2O_4, N_2H_4, and MMH
Coupons Per Coating System	One (1) four (4) inch x four (4) inch aluminum foil coupon
Trials Per Coupon	One (1)
Control Coupons Required For Testing	None
Acceptance Criteria	Slight to Moderate Reactivity Observed: When test data based on visual observations with the unaided eye reveal reactivity (but no ignition) and/or any changes in the visual characteristics, bulk characteristics, and/or surface characteristics of the test sample.

Unique Equipment and Instrumentation

- None

Data Analysis

- Complete the "Material Identification Form", or an equivalent form.
- Complete the "Chemical Analysis Laboratory Compatibility Report", or an equivalent form.

3.2.2. LOX Compatibility

Test Description

The purpose of this test is to determine if materials in liquid oxygen (LOX) environments react when mechanically impacted. A reaction from mechanical impact can be determined by an audible report, an electronically or visually detected flash, or obvious charring of the sample, sample cup, or striker pin.

Perform this test in accordance with NASA-STD-6001 (*Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion*). Specifically reference Test Method 13A, *Mechanical Impact for Materials in Ambient Pressure LOX*. The test system should be identical to that described in ASTM D 2512 [*Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold* and *Pass-Fail Techniques*)].

Rationale

This test is specified in NASA-STD-6001 and was identified as a testing requirement. Materials intended for use in space vehicles, specified test facilities, and specified GSE must meet the requirements of this document.

Test Methodology

Table 10 Test Methodology for LOX Compatibility Test	
Parameters	Per NASA-STD-6001; The thickness of the sample must be the worst-case thickness. Test conditions (pressure and temperature) are the ambient pressure of the test facility and the boiling point of LOX at that pressure.
Coupons Per Coating System	Twenty (20)*
Trials Per Coupon	One (1)
Control Coupons Required For Testing	None
Acceptance Criteria	Twenty samples must not react when impacted at 72 ft-lbs (98 J). If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.

*Minimum required

Unique Equipment and Instrumentation

- ABMA-Type Impact Tester

Data Analysis

- The test report must include sample identification, configuration, test conditions, number of reactions, and observations from the test. Proper reporting of the test observations, especially of unusual behavior, is critical.

3.2.3. Cure Time (MEK Solvent Rub)

Test Description

This test determines how long an applied coating system requires to fully cure at room temperature up to a period of 14 days at 50 ±10% RH. Liquid primer/topcoat coating systems are required to be tested against this requirement.

Every two (2) days, for a period of 14 days, perform 25 double-rubs (back and forth) on the coated panels with clean cheesecloth wetted with methyl ethyl ketone (MEK).

Perform this test in accordance with ASTM D 4752 (*Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub*). Each test should be conducted on a previously untested area of the coating. Visually examine the coating for substrate metal exposure. Pigment on the cheesecloth does not indicate failure.

Rationale

Although MEK use is being phase out, the participants deemed the MEK solvent rub test as the test of choice as it is more stringent than an acetone rub test. This test is a commonly accepted industrial criterion for determining coating cure and only small amounts of MEK is consumed. Inspecting at two-day intervals is required by participants to determine the actual cure time. All participants agreed the MEK rub test is a performance requirement.

Test Methodology

Table 11 Test Methodology for Cure Time (MEK Solvent Rub) Test

Parameters	MEK saturated terry cloth rag, 50 double rubs
Coupons Per Coating System	Three (3) A36
Trials Per Coupon	Three (3)*
Control Coupons Required For Testing	Three (3) A36
Acceptance Criteria	No effect on surface or coating on the cloth (Resistance Rating 5)

*Perform succeeding trials on coupon areas that have not previously been rubbed.

Unique Equipment and Instrumentation

- None

Data Analysis and Reporting

- Report the DFT of the coating, elapsed time between the application of the coating and the running of the tests, and results of MEK rub test on candidate coating using rating system in ASTM D 4752, Table 1 (*Scale for Resistance Rating*).
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

3.2.4. Tensile (Pull-Off) Adhesion

Test Description

This test evaluates the pull-off strength (commonly referred to as adhesion) of a coating. The test determines either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or whether the surface of the material remains intact at a prescribed force.

This test method uses a class of apparatus known as portable pull-off adhesion testers. They are capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible. Measurements are limited by the strength of adhesion bonds between the loading fixture and the specimen surface or the cohesive strengths of the adhesive, coating layers, and substrate.

Perform this test in accordance with ASTM D 4541 (*Standard Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers*).

Rationale

Participants agreed that adhesion is a critical performance requirement.

Test Methodology

Table 12 Test Methodology for Pull-Off Adhesion Test	
Parameters	Per ASTM D 4541
Coupons Per Coating System	One (1) A36
Trials Per Coupon	Three (3)
Control Coupons Required For Testing	One (1) A36
Acceptance Criteria	Pull-off strength of candidate coating achieved at time of failure equal to or better than control coatings

Unique Equipment and Instrumentation

- Patti adhesion tester or equivalent

Data Analysis

- Record the strength at which adhesion fails; there should be three (3) data points for each coupon
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1)

color photograph of each tested coupon and the dolly shall be taken after the test.

3.2.5. Removability

Test Description

This test determines the relative ease of removing the coating on a two (2)-inch diameter area on a test coupon using aluminum oxide blast media after artificial weathering.

The coupons used for this test shall be three (3) inches by six (6) inches to accommodate the weathering chamber. Coated test panels shall be weathered for 500 hours in accordance with ASTM G 155 [*Standard Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials*], G 155 corresponding test cycle for G 26, Method A (continuous light with intermittent water spray), prior to testing for removability.

Only half of the coupon will have the coating removed. The weathered panels shall be placed on a rack and tilted to a 60° angle to the horizontal. Adjust the system air pressure to 90 pounds per square inch (psi). Use only aluminum oxide blast media for this test. Media flow must be set in accordance with the media manufacturer's specifications. Direct the abrasive blast jet at the same area for 1 minute. Record the dry film thickness of the coating remaining in the abrasive blast area. Identical removal procedures shall be used for both the candidate and control coating systems.

Rationale

Coating systems must typically be removed after prescribed periods of use. Evaluation of relative removal ease for candidate alternate coating systems after aging is necessary for predicting the effectiveness of field maintenance operations.

Test Methodology

Table 13 Test Methodology for Removability Test

Parameters	Aluminum oxide blast material; Blast pressure 90 psi
Coupons Per Coating System	Three (3) A36; 3x6 inch panels
Trials Per Coupon	One (1)
Control Coupons Required For Testing	Three (3) A36; 3x6 inch panels
Acceptance Criteria	Less than one minute to penetrate to substrate; Measure DFT of remaining coating

Note: The initial gloss, color, and DFT of each panel will be recorded on the Application Record Sheet in NASA-STD-5008B, or an equivalent form, during application.

Unique Equipment or Instrumentation

- Sand Blast Cabinet
- Magnetic gage per SSPC-PA-2

Data Analysis and Reporting

- Report the coating DFT and the dwell time to substrate.
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

3.2.6. Reparability

Test Description

This test determines the relative ease of replacing and blending-in coatings that have been removed or otherwise damaged. The dry tape adhesion test provides a procedure for establishing acceptability of intercoat and surface adhesion of an organic coating by applying pressure-sensitive adhesive tape over a scribed area of the coating, then removing that tape.

The coupons used for this test shall be three (3) inches by six (6) inches to accommodate the weathering chamber. Three procedures will be required for accomplishing this task; (A) Repair the baseline control coating with a baseline coating, (B) Repair the baseline control coating with each of the alternative coatings, and (C) Repair each alternative coating with the alternative coating. Only one (1) set of coupons with the baseline coating repaired with the baseline coating is required for comparison. Test panels from which coatings have been removed (Section 3.2.1., *Removability*) shall be used for this evaluation.

Replace the removed coating in accordance with the coating manufacturer's repair instructions. Examine the surface of each test panel to evaluate the appearance of the repair. The repaired area must be free of voids, air bubbles or other significant defects. The repaired area shall be inspected for coating quality and match to the original, aged coating on the top half of the test coupon using ASTM D 523.

Conduct a dry tape adhesion test on the repaired areas after the prescribed cure times of test coating on the repaired area to ensure the coating adherence. Perform this test in accordance with Method A of ASTM D 3359 (*Standard Test Methods for Measuring Adhesion by Tape Test*), except use a four and one-half (4.5)-pound roller instead of finger pressure for smoothing down the tape. In performing this test,

scribe two “X” incisions through the coating so that the smaller angle of each “X” is 30-45°, making sure that the coating has been scribed all the way to the substrate. The scribe must have a 45° bevel, and each line of each “X” should be approximately 1.5 inches long. Immediately place a piece of tape over the intersection of each “X” and smooth down by passing a 4.5 lb. roller over it eight (8) times. Remove the tape rapidly at approximately an 180^{o} angle. Inspect the incision area for peel away.

Rationale

This test provides data to evaluate how effectively coatings can be replaced/repaired in field maintenance environments. All participants have agreed that coating reparability is a performance requirement.

Test Methodology

Table 14 Test Methodology for Reparability Test

Parameters	Coating Manufacturer's instructions for coating repair
Coupons Per Coating System	Three (3) A36 with the baseline coating; 3x6 inch panels Three (3) A36 with the alternative coatings; 3x6 inch panels
Trials Per Coupon	One (1)
Control Coupons Required For Testing	Three (3) A36 total for comparison
Acceptance Criteria	Ease of replacement of damaged areas of the test coatings, color matching of aged versus new material; No streaks, blistering, voids, air bubbles, over-spray “halo,” cratering, lifting, blushing, or other surface irregularities; No peel away of the repaired coating during the dry tape adhesion test

Unique Equipment or Instrumentation

- 10X optical magnifier
- Hunter Lab "Miniscan" Spectrophotometer (using CIE L*a*b* Color Measurement System) or equivalent per ASTM D 2244 (*Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates*)
- Hunter Lab "Progloss" Meter or equivalent
- One (1) inch masking tape, 3M Company Type 250 or equivalent
- Four and one-half (4.5) pound roller
- Carbide tip scribe

Data Analysis and Reporting

- Technician evaluation of coating quality and match to the original, aged coating on the top half of the test coupon.
- Report coating color measurements of aged area and repaired area per ASTM D 523 and the adhesion rating as specified in ASTM D 3359, Method A, Section 7.
- One (1) color photograph of a coupon coated with each candidate primer or primer/topcoat system shall be taken after recoating is completed. One (1) color photograph of each tested coupon shall be taken after the tape test is completed.

3.2.7. Mandrel Bend Flexibility

Test Description

This test evaluates coating flexibility and adhesion to substrate limits when the test coupon is bent around a ¼-inch fixed diameter mandrel.

The bend test shall be conducted in accordance with the version of ASTM D 522 (*Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings*), Test Method B for Ambient Temperature.

Rationale

This method will determine whether the coatings will provide the necessary flexibility when compared to other more conventional coatings. All participants have agreed that the mandrel bend test is a performance requirement for the coatings.

Test Methodology

Table 15 Test Methodology for Mandrel Bend Flexibility Test

Parameters	¼-inch diameter mandrel at ambient temperature
Coupons Per Coating System	Three (3) A36
Trials Per Coupon	One (1)
Control Coupons Required For Testing	Three (3) A36
Acceptance Criteria	No peeling or delamination from the substrate and no cracking greater than ¼-inch from the edges.

Unique Equipment or Instrumentation

- Mandrel bend apparatus (¼-inch diameter mandrel)

Data Analysis and Reporting

- Report pass/fail on the ¼ inch mandrel test and the material characteristics of the alternatives after this test is performed.
- One (1) color photograph of a coupon coated with each candidate coating and of a coupon coated with the baseline coating shall be taken before the test. One (1) color photograph of each tested coupon shall be taken after the test.

4. REFERENCE DOCUMENTS

The documents in Table 16 were referenced in the development of this Test Protocol.

Table 16 Summarized Test and Evaluation Reference Listing

Reference Document	Title	Test	Section
ASTM D 522	*Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings*	Mandrel Bend Flexibility	3.2.7.
ASTM D 523	*Standard Test Method for Specular Gloss*	Surface Appearance; Atmospheric Exposure Test; Reparability	3.1.3.; 3.1.4.; 3.2.6.
ASTM D 610	*Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces*	Atmospheric Exposure Test	3.1.4.
ASTM D 714	*Standard Test Method for Evaluating Degree of Blistering of Paints*	Atmospheric Exposure Test	3.1.4.
ASTM D 2244	*Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates*	Surface Appearance; Reparability	3.1.3.; 3.2.6.
ASTM D 2512	*Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold* and *Pass-Fail Techniques*)	LOX Compatibility	3.2.2.
ASTM D 3359	*Standard Test Methods for Measuring Adhesion by Tape Test*	Reparability	3.2.6.
ASTM D 4541	*Standard Test Method for Pull-off Strength of Coatings Using Portable Adhesion Testers*	Heat Adhesion; Tensile (Pull-off) Adhesion	3.1.5.; 3.2.4.
ASTM D 4752	*Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub*	Cure Time (MEK Solvent Rub)	3.2.3.

Table 16 Summarized Test and Evaluation Reference Listing			
Reference Document	**Title**	**Test**	**Section**
ASTM G 155	*Standard Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials*	Removability	3.2.5.
FED-STD-141	*Paint, Varnish, Lacquer and Related Materials*	X-Cut Adhesion	3.2.4.
KSC Report MTB-175-88	*Procedure For Casual Exposure Of Materials To Hypergolic Fluids*	Hypergol Compatibility	3.2.1.
NACE-STD-RP0281	*Method for Conducting Coating (Paint) Panel Evaluation Testing In Atmospheric Exposures*	Test Descriptions	3.
NACE-STD-RP0287	*Field Measurements of Surface Profile of Abrasive Blast Cleaned Steel Surfaces Using a Replica Tape*	Test Descriptions	3.
NASA-STD-5008B	*Protective Coating of Carbon Steel, Stainless Steel, and Aluminum on Launch Structures, Facilities, and Ground Support Equipment*	Introduction; Atmospheric Exposure Test; Heat Adhesion	1.; 3.1.4.; 3.1.5.
NASA-STD-6001	*Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures for Materials in Environments that Support Combustion*	Hypergol Compatibility; LOX Compatibility	3.2.1.; 3.2.2.
SSPC-PA-2	*Measurement of Dry Coating Thickness with Magnetic Gages*	Test Descriptions; Ease of Application; Removability	3.; 3.1.2.; 3.2.5.
SSPC-SP-1	*Solvent Cleaning*	Test Descriptions	3.
SSPC-SP-5	*White Blast Cleaning*	Test Descriptions	3.

n/a = Not Applicable