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Multivariate Dynamic Accelerated Corrosion Test Method

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Multivariate Dynamic Accelerated Corrosion Test Method

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Multivariate Dynamic Accelerated Corrosion Test Method

Foreword, Rationale 4
Referenced Standards and Other Consensus Documents 4
Section 1 Scope 4
Section 2 Definitions 4
Section 3 Apparatus 5
Section 4 Test Specimen Construction, Preparation, and Position during Exposure 5
Section 5 Salt Solution 7
Section 6 Facilities Requirements 7
Section 7 Conditions in the Salt Spray Chamber 8
7.1 Temperature 8
7.2 Atomization and Quantity of Fog 8
7.3 Relative Humidity 8
Section 8 Continuity of Exposure 9
Section 9 Period of Exposure 10
Section 10 Reporting on Test Method Compliance 10
Other Referenced Documents 11
Bibliography 11
Appendix A Example Calculations for Solution Collection Rates (Nonmandatory) 12

FIGURES

Figure 1 Top View of Recommended Assembly Properties and Scribe Pattern for Coating Inhibitor Screening 6
Figure 2 Fog and Relative Humidity Cycle Schedule 9

TABLES

Table 1 Salt Solution Information 7
Table 2 Fog Event and Relative Humidity Control Program 9
Table A1 Calculation Sheet for Reporting Solution Collection Rates Following a 6-hour Fog Event 12
Table A2 Example Data and Calculations for a Passing Solution Collection Rate Measurement Following a 6-hour Fog Event 12
Table A3 Example Data and Calculations for a Single Failing Solution Collection Rate Measurement Following a 6-hour Fog Event 13
Table A4 Example Data and Calculations for an Average Failing Solution Collection Rate Measurement Following a 6-hour Fog Event 13

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Foreword

The purpose of this standard is to detail requirements for conducting an accelerated corrosion test with controlled relative humidity (RH). The intended audience of this standard is accelerated corrosion test operators and technicians, as well as materials and process engineers involved with material selection for corrosion-resistant aerospace alloys.

Rationale

This standard is necessary to codify the procedures for a new accelerated corrosion test method with controlled relative humidity.

Referenced Standards and Other Consensus Documents

Unless specifically dated, the latest edition, revision, or amendment of the documents listed in the table below shall apply.

AMPP/NACE/SSPC, www.ampp.org:	
NACE/ASTM G193	Standard Terminology and Acronyms Relating to Corrosion
ASTM International, www.astm.org:	
ASTM D1193	Standard Specification for Reagent Water
ASTM D4285	Standard Test Method for Indicating Oil or Water in Compressed Air

Section 1: Scope

1.1 This document provides instructions for the subject accelerated corrosion test method, detailed chamber performance requirements, recommended monitoring techniques to ensure adherence to performance requirements, and reporting requirements. The subject accelerated corrosion test method was designed to discriminate epoxy primer performance for corrosion mitigation and replicate failure modes observed in the operational environment, namely corrosion within and around fastener holes. Multiple variables within this test method have been modified relative to other test methods (solution chemistry, RH cyclic profile, operating temperature).

1.2 **Limitations:** The parameters that can be controlled in this test method include: relative humidity, temperature, salt solution chemistry (chemical composition and pH), salt solution flow rate during fog events, fog event frequency, and fog event duration. Any other parameters of test execution, such as gaseous contaminant injection (SO_2, O_3, etc.) or ultraviolet radiation, are not covered in this test method in its current version. Details related to the construction and/or geometry of the materials to be tested within this environment are not prescribed in this test method. This test method was primarily developed for aluminum substrates with organic and inorganic coatings with a galvanic fastener. The performance life of the materials tested cannot be estimated by their performance in the accelerated environment alone. The test is meant only to rank performance. Reproducibility estimates are provided based on observed surface area and volumetric damage from round-robin experiments using aluminum substrates and stainless-steel fasteners with a range of inorganic and organic coatings. Reproducibility estimates are highly dependent on the type of specimens tested as well as the evaluation criteria selected. Sufficient replicates shall be included to quantify the variability of the results.

Section 2: Definitions

The definitions of many of the corrosion-related terms used in this standard may be found in NACE/ASTM G193 and/or the SSPC Glossary.[1] There are no other words or phrases in this document that require special definitions.

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Section 3: Apparatus

3.1 The apparatus required for testing consists of a chamber, salt solution reservoir, supply of suitably conditioned compressed air, one or more atomizing nozzles, non-metallic specimen supports, chamber heating elements, and necessary means of control. The size and detailed construction of the cabinet are optional, provided the conditions meet the requirements of this test method.

3.2 Drops of solution that accumulate on the ceiling of the chamber shall not be permitted to fall on the specimens being exposed.

3.3 A solution that has been fogged into the test chamber environment containing samples shall not be returned to the solution reservoir for respraying.

3.4 Material of chamber construction shall be such that it will not affect the corrosiveness of the test environment.

3.5 All water used for solution preparation and relative humidity control in this test method shall conform to Type IV water in ASTM D1193.

Section 4: Test Specimen Construction, Preparation, and Position during Exposure

4.1 The type and number of test specimens to be used, as well as the criteria for evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon by the purchaser and seller. Note, however, that the test was designed to distinguish coating performance for specimens with a galvanic configuration. Paragraphs 4.2 through 4.8 provide a recommendation for sample design.

4.2 Samples should be comprised of two 7.62 cm x 15.2 cm x 0.635 cm panels representing structural alloys fastened together in a lap-joint configuration by six fasteners.

4.3 The center of the fastener holes should be evenly spaced 2.54 cm away from the long edge of the panel. The fastener holes on the top panel should match the profile of the system being replicated. For example, the fasteners may be countersunk if the system to be replicated includes countersunk fasteners.

4.4 The panels should be fastened by nuts and washe and material type of those in the system being replicated.

4.5 The order in which surface treatments, primers, and topcoats are applied should match that of the system being replicated. A reasonable approach would be to apply surface treatment, assemble the panels and fasteners, and then coat the system with a primer and topcoat.

4.6 Special care should be taken regarding the torque of the fasteners. Lower fastener torque values will increase electrolyte wicking into the fastener hole and may increase down-hole damage. Higher fastener torque values will decrease electrolyte wicking into the fastener hole and may decrease down-hole damage. Method development work determined that a reasonable approach would be to install 10-32 x 1 in countersunk steel fasteners at 20 in-lbs.

4.7 Special care should be taken regarding the intactness of the paint coating bridge from the panel to the fastener head. To maximize electrolyte wicking into the fastener hole, remove the fasteners enough to break the paint bridge following the final coating application step. Reinstall the fastener at the torque value of interest.

4.8 To assess the ability of a coating to prevent corrosion in a scribe, scribe the coating around the fastener head in an “X” shape. The lengths of each of the four scribes around the fastener should be 13 mm and should not come in contact with the fastener head. The scribe width should be 1.0 – 1.5 mm.

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4.9 Refer to Figure 1 for a depiction of the recommended test specimen design.

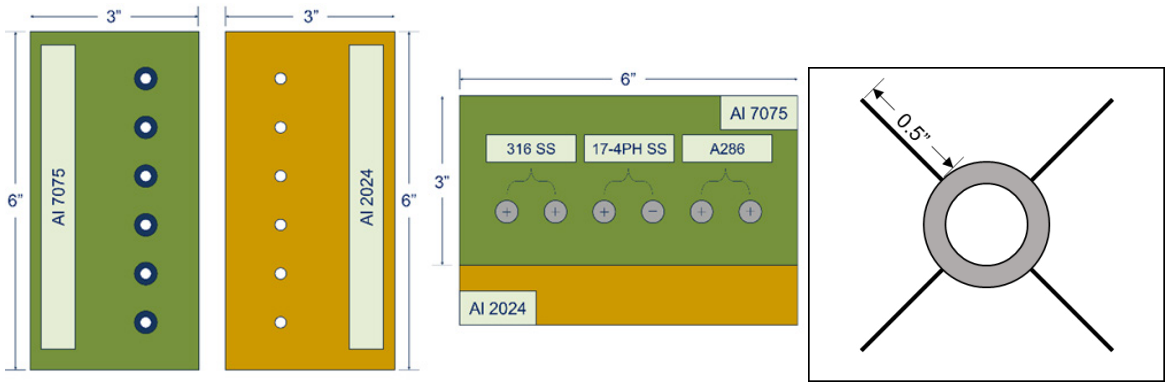

Figure 1: (Left & Center) Top View of Recommended Assembly Properties. (Right) Scribe Pattern for Coating Inhibitor Screening

4.10 The specimens shall be suitably cleaned. The cleaning method shall be dependent on the nature of the surface and the contaminants. Care shall be taken that specimens are not contaminated after cleaning by excessive or careless handling.

4.11 Specimens for the evaluation of paints and other organic coatings shall be prepared in accordance with application specification(s) for the material(s) being exposed, or as agreed upon between the purchaser and the supplier.

4.12 Unless otherwise specified, the uncoated cut edges of specimens and areas containing identification marks or in contact with the racks or supports shall be protected from corrosive environments with a suitable coating (e.g., paint, tape, wax, or other effective media) for the conditions of the test method.

4.13 The position of the specimens in the chamber during the test shall be such that the following conditions are met:

a) Unless otherwise specified, the specimens shall be supported or suspended with the surface of interest at an angle between 15° and 30° from vertical. The vertical orientation of specimens in the cabinet shall be consistent to minimize variability within the chamber.

b) The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

c) Each specimen shall be placed to permit unencumbered exposure to the fog.

d) Salt solution from one specimen shall not drip on any other specimen.

e) Unless otherwise specified, specimens shall be positioned such that they lie on a flat plane of constant vertical height within the chamber.

f) Placement of replicate specimens within the chamber shall be randomized to avoid possible bias caused by variations in fog patterns.

g) Racks and supports shall be made of glass, rubber, plastic, or suitably protected wood.

h) Unless otherwise specified, specimens shall be repositioned each week, or at another agreed-upon interval between the purchaser and seller, to mitigate the effects of environmental condition variation within the chamber.

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Section 5: Salt Solution

5.1 The salt solution shall be prepared in accordance with the simulated sea salt solutions shown in Table 1. Make-up water shall be deionized water conforming to Type IV water in ASTM D1193. All chemicals shall be reagent grade.

Table 1
Salt Solution information

Test Solution #1 – Sea Salt Solution with Nitrate Ions	
NaCl	11.13 g/L
$MgCl_2 \cdot 6H_2O$	5.55 g/L
Na_2SO_4	2.00 g/L
$NaNO_3$	1.635 g/L
HCl (1N)	pH = 3 (acidified with HCl)

5.2 The solution inside the reservoir shall be acidified to pH = 3 ± 0.2 using hydrochloric acid. If pH needs to be increased, 0.1M sodium hydroxide shall be used.

5.3 Unless otherwise specified, the solution pH in the reservoir should be verified weekly by measuring the pH of the solution in the reservoir. For convenience, this can be completed at the same time as the sample rotation specified in Paragraph 4.13 h).

5.4 The salt solution shall be stored in a non-metallic container that will not interact with the salt solution.

5.5 The salt solution shall be stored in a container with a lid to minimize interaction between the salt solution and the laboratory environment. For continuous operation, replace the solution as necessary. For new test startup, drain the old solution, rinse the storage container with deionized water, and fill with new solution.

5.6 Halides (Bromide, Fluoride, and Iodide) other than chloride shall constitute less than 0.1% by mass of the salt content. Copper content shall be less than 0.3 ppm by mass. Sodium chloride that has had anti-caking agents added shall not be used because such agents may act as corrosion inhibitors. Upon agreement between the purchaser and the seller, analysis may be required and limits established for elements or compounds not specific in the chemical composition given above.

Section 6: Facilities Requirements

6.1 The compressed air supply shall be free of grease, oil, water, and dirt. This incoming air pressure shall be maintained per manufacturer specifications and shall meet solution collection rate requirements. ASTM D4285 should be used to verify the air is contaminant-free.

6.2 The compressed air supply to the chamber must be filtered and dried such that the chamber can precisely control relative humidity in order to meet the relative humidity control requirements.

6.3 All water supplied to the system shall be deionized water conforming to Type IV water in ASTM D1193.

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Section 7: Conditions in the Salt Spray Chamber

7.1 Temperature

a) The exposure zone of the chamber shall be maintained at a constant temperature of 40 °C ± 3 °C.

7.2 Atomization and Quantity of Fog

a) Fog events shall occur twice per week and shall last 6 hours. The alignment of the fog event with respect to the relative humidity cycle is shown in Table 2 and Figure 2.

b) Place 6 clean fog collection funnels within the exposure zone so that no drops of solution will be collected from the test specimens or any other source. Position the collectors in a uniformly distributed manner throughout the chamber environment. The fog shall be such that for each 80 cm² of collection area, there will be collected 3.0 – 4.0 mL per hour per 80 cm² collection area across at least 5 collection vessels with no one vessel recording collection rates below 2.6 or above 4.3 mL per hour over 6 hours of continuous fog. Verify solution collection rates when the chamber is opened to rotate the witness coupons.

7.3 Relative Humidity

7.3.1 The relative humidity cycle involves high humidity, moderate humidity, and low humidity steps. The relative humidity steps are cycled and repeated at a 6-hour interval.

7.3.1.1 High Humidity:
a) Set point: 90%
b) Duration: 2 hours

7.3.1.2 Moderate Humidity:
a) Set point: 60%
b) Duration: 3 hours

7.3.1.3 Low Humidity:
a) Set point: 20%
b) Duration: 1 hour

7.3.2 The RH cycle and fog event schedule are summarized in Table 2 and Figure 2. The smallest repeating unit of the test is 168 hours, with the fog events taking place on day 1 (0 – 6 hours) and day 4 (72 – 78 hours). The test wasat a test starting on Monday at 9:00 AM local time will fog on Mondays and Thursdays from 9:00 AM – 3:00 PM local time.

7.3.3 Transitions from step to step should be programmed to occur as quickly as possible, reaching ± 5% of the set point within 10 minutes of transition.

7.3.4 Every 3,000 hours of chamber operation, verify chamber performance using an independent temperature and relative humidity probe. If the probe indicates the chamber is not performing in accordance with the method, recalibrate the chamber's relative humidity sensing elements per chamber manufacturer's instructions. The relative humidity sensor shall be recalibrated, regardless of chamber condition, six months after the most recent recalibration.

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Table 2
Fog Event and Relative Humidity Control Program

Step Number	Action	Duration (h:mm)
1	Fog	6:00
2	Loop steps 3 – 45	Repeat 11x
3	90% RH Hold	2:00
4	60% RH Hold	3:00
5	20% RH Hold	1:00
6	Fog	6:00
7	Subcycle 8 – 10	Repeat 15x
8	90% RH Hold	2:00
9	60% RH Hold	3:00
10	20% RH Hold	1:00
11	Final Step	Return to Step 1

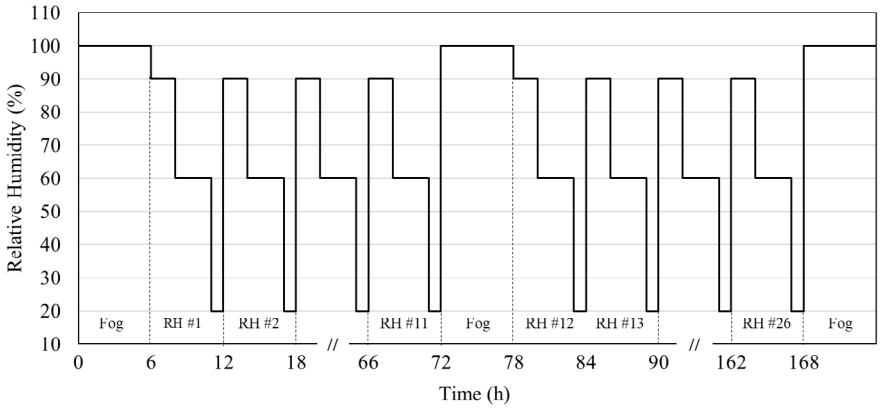

Figure 2: Fog and Relative Humidity Cycle Schedule

Section 8: Continuity of Exposure

8.1 Unless otherwise specified in the specifications covering the material or product being tested, the test shall run continuously for the duration of the entire test period. Continuous operation implies that the chamber be closed except for routine short daily interruptions necessary to inspect, rearrange, or remove test specimens, and to check and replenish the solution in the reservoir. Operations shall be scheduled so that the maximum time for these interruptions is held to 60 min or less per day. It is recommended to have only one interruption per day if possible. Operators shall minimize test downtime and document the stop and start times for all interruptions.

8.2 The chamber lid shall be closed throughout the entirety of the exposure except for routine short periodic interruptions.

8.3 Test interruptions shall take place only during either the 60% or 20% relative humidity step. The test shall never be interrupted during the fog event or the 90% relative humidity step.

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Section 9: Period of Exposure

9.1 The period of exposure shall be as designated by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the seller.

Section 10: Reporting on Test Method Compliance

10.1 The pH of the reservoir solution shall be recorded weekly. If the pH requires adjustment, the pH of the solution before and after the adjustment shall be recorded.

a) If pH adjustment is required, test operators shall document the amount of HCl and/or NaOH used to adjust the pH.

10.2 Collection vessels shall be evenly distributed at 6 locations within the chamber. Collection rates shall be documented weekly.

a) Each collection vessel should be at least 6 in (15.24 cm) away from the chamber walls.

b) The average collection rate of the six collection vessels shall range between 3.0-4.0 mL/hour. A single collection vessel is permitted to collect between 2.6-4.3 mL/hour.

10.3 An independent temperature and relative humidity sensor placed in the center of the chamber shall be used every six months to confirm the chamber is executing the method as described in Section 7.

a) It is recommended that a temperature and relative humidity sensor be employed every 3,000 test hours to validate chamber performance. Method performance should also be validated if the chamber was not in operation for more than three months.

b) If during steps 2-4 and 8-10 described in Table 2, the difference in relative humidity between the independent sensor and chamber varies by more than 5%, the chamber shall be recalibrated. Re-calibration should be executed according to chamber-specific procedures. The recalibration event and magnitude of deviation should be recorded, and the customer shall be informed of the recalibration event. The customer shall determine if the test is to continue or be terminated.

c) If the chamber temperature according to the independent sensor is less than 37 °C or greater than 43 °C for more than 30 minutes during any portion of the test method, the chamber shall be recalibrated. Re-calibration should be executed according to chamber-specific procedures.

d) Chamber recalibration records shall be maintained by the chamber operators. Records shall include temperature and relative humidity values before and after recalibration.

10.4 It is recommended that witness coupons analyzed via mass loss be used to ensure consistent chamber performance.

a) Coupons should be made of AA7075-T6 with dimensions of 7.62 cm by 7.62 cm by 0.32 cm thick.

b) Post-exposure, the coupons shall be cleaned via immersion in nitric acid with ultrasonic cleaning for 15 minutes. After rinsing with copious amounts of water and drying, the clean coupon mass shall be recorded to five decimal places.

c) The data should be reported in units of mg/m^3.

d) Witness coupons shall be deployed in groups of at least three per sampling period.

e) The number of sampling periods will vary depending on the test interval. For 1,000 hours of operation, it is

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recommended that witness coupon recoveries occur at approximately 333 hours and 667 hours of testing time, with an additional set collected at the end of the exposure.

f) Mass loss information can be used to track chamber performance across multiple test periods and, where necessary, determine if a recalibrated chamber impacted test results.

Other Referenced Documents

1. "SSPC Protective Coatings Glossary," (Pittsburgh, PA: SSPC, 2011).

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Appendix A
Example Calculations for Solution Collection Rates
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

A1. This section provides examples of data and calculations for verifying solution collection rates. Table A1 provides an empty worksheet for recording collection volumes, calculating collection rates, and determining pass/fail results. Table A2 provides an example of passing solution collection rates. Table A3 provides an example of failing solution collection rates since vessel 3 had a collection rate of 4.5 mL/hr, which exceeds the upper threshold defined in Paragraph 7.2 b). Table A4 provides an example of failing solution collection rates since the average collection rate of 2.8 mL/hr fails to meet the minimum collection requirement of 3.0 – 4.0 mL/hr defined in Paragraph 7.2 b).

Table A1
Calculation Sheet for Reporting Solution Collection Rates Following a 6-hour Fog Event

Vessel Index	Position	Collection (mL)	Collection Rate (mL/hr)	Performance Criteria (P/F)
1	Back-left		=C2/# of fogging hours since last evaluation	Fail if D2 > 4.3 or D2 < 2.6, otherwise Pass
2	Front-left		=C3/# of fogging hours since last evaluation	Fail if D3 > 4.3 or D3 < 2.6, otherwise Pass
3	Front-center		=C4/# of fogging hours since last evaluation	Fail if D4 > 4.3 or D4 < 2.6, otherwise Pass
4	Front-right		=C5/# of fogging hours since last evaluation	Fail if D5 > 4.3 or D5 < 2.6, otherwise Pass
5	Back-right		=C6/# of fogging hours since last evaluation	Fail if D6 > 4.3 or D6 < 2.6, otherwise Pass
Average	-	**=AVERAGE(C2:C6)**	**=C7/#** of fogging hours since last evaluation	**Fail if D7 < 3.0 or D7 > 4.0 or if individual collection rate Fail, otherwise Pass**

Table A2
Example Data and Calculations for a Passing Solution Collection Rate Measurement Following a 6-hour Fog Event

Vessel Index	Position	Collection (mL)	Collection Rate (mL/hr)	Performance Criteria (P/F)
1	Back-left	17	2.8	Pass
2	Front-left	19	3.2	Pass
3	Front-center	24	4.0	Pass
4	Front-right	21	3.5	Pass
5	Back-right	18	3.0	Pass
Average	-	**19.8**	**3.3**	**Pass**

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Table A3
Example Data and Calculations for a Single Failing Solution Collection Rate Measurement Following a 6-hour Fog Event

Vessel Index	Position	Collection (mL)	Collection Rate (mL/hr)	Performance Criteria (P/F)
1	Back-left	17	2.8	Pass
2	Front-left	19	3.2	Pass
3	Front-center	26	4.5	Fail
4	Front-right	21	3.4	Pass
5	Back-right	18	3.0	Pass
Average	-	20.2	3.4	Fail

Table A4
Example Data and Calculations for an Average Failing Solution Collection Rate Measurement Following a 6-hour Fog Event

Vessel Index	Position	Collection (mL)	Collection Rate (mL/hr)	Performance Criteria (P/F)
1	Back-left	16	2.7	Pass
2	Front-left	16	2.7	Pass
3	Front-center	17	2.8	Pass
4	Front-right	18	3.0	Pass
5	Back-right	16	2.7	Pass
Average	-	16.6	2.8	Fail

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