TECHNICAL BULLETIN LF-8144

Introduction to Cyclic Corrosion Testing

This paper is intended as a general introduction to cyclic corrosion testing. It outlines the rationale for cyclic testing, includes some guidelines for using cyclic tests and explains some common cycles and their applications. This discussion is not intended to be a complete, exhaustive tutorial on cyclic corrosion testing. Consult the referenced technical papers for more detailed information.

Background

Salt spray was first used for corrosion testing around 1914. In 1939, the neutral salt spray test was incorporated as ASTM B117.[1] This traditional salt spray test specifies a continuous exposure to a 5% salt fog at 35 °C. During the course of over a century of use, there have been many modifications and refinements to ASTM B117. In spite of all these refinements, there has long been general agreement that salt spray test results do not correlate well with the corrosion seen in actual atmospheric exposures. Nevertheless, ASTM B117 has been generally accepted as the standard corrosion test method and is still widely specified for testing painted and plated finishes, military components, and electrical components.

As the demand for improved corrosion protection increased, engineers and scientists attempted to develop test procedures to more accurately predict the corrosion of materials.
In England, during the 1960's and 1970's, Harrison and Timmons[2, 3] developed the cyclic Prohesion™ test, which has been found especially useful for industrial maintenance coatings. More recently, the Society of Automotive Engineers (SAE) and The American Iron and Steel Institute (AISI) have been studying cyclic testing for automotive applications. Their progress has been encouraging and is well documented.[4,5,6,7, 8,9,10] Japanese researchers have also developed a number of cyclic corrosion test methods, as have automakers around the world.

What is Cyclic Corrosion Testing?

Cyclic corrosion testing is intended to be a more realistic way to perform salt spray tests than traditional, steady state exposures. Because actual atmospheric exposures usually include both wet and dry conditions, it makes sense to pattern accelerated laboratory tests after these natural cyclic conditions. Research indicates that, with cyclic corrosion tests, the relative corrosion rates, structure, and morphology are more similar to those seen outdoors. Consequently, cyclic tests usually give better correlation to outdoors than conventional salt spray tests. They are effective for evaluating a variety of corrosion mechanisms, including general, galvanic, and crevice corrosion. All Q-Lab testers can perform programmed cyclic corrosion testing, though the capability to perform complex exposures varies by model.

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1. ASTM B117, "Standard Practice for Operating Salt Spray (Fog) Apparatus".
2. Cremer, N.D., "Prohesion Compared to Salt Spray and Outdoors: Cyclic Methods of Accelerated Corrosion Testing", Federation of Societies for Coatings Technology, 1989 Paint Show.
3. Timmins, F.D., "Avoiding Paint Failures by Prohesion," J. Oil & Colour Chemists Assoc., Vol. 62, No. 4, p. 131 (1979).
4. M. L. Stephens, "SAE ACAP Division 3 Project: Evaluation of Corrosion Test Method", Paper No. 892571, Automotive Corrosion and Prevention Conference Proceedings, P-228. Society of Automotive Engineers, Warrendale, PA (1989), pp. 157-164.
5. H. E. Townsend, "Status of a Cooperative Effort by the Automotive and Steel Industries to Develop a Standard Accelerated Corrosion Test", Paper No. 892569, ibid., pp. 133-145.
6. F. Blekkenhorst, "Hoogovens' Contribution to AISI Program "Accelerated Corrosion Testing: A Cooperative Effort by the Automotive and Steel Industries" Paper No. 892570, ibid., p 147-156.
7. M. Petschel, Jr., "SAE ACAP Division 3 Project: Evaluation of Corrosion Test Results and Correlation with Two-Year, On-Vehicle Field Results, Paper No. 912283, Automotive Corrosion and Prevention Conference Proceedings, P-250, Society of Automotive Engineers, Warrendale, PA (1989), pp. 179-203.
8. R. J. Neville, W.A. Schumacher, D.C. McCune, R.D. Granata and H. E. Townsend, "Progress by the Automotive and Steel Industries Toward and Improved Laboratory Cosmetic Corrosion Test", Paper No. 912275, ibid., pp. 73-98.
9. F. Blekkenhorst, "Further Developments Toward a Standard Accelerated Corrosion Test for Automotive Materials, Paper No. 912277, ibid., pp. 99-114.
10. D. D. Davidson and W. A. Schumacher, "An Evaluation an analysis of Commonly Used Accelerated Cosmetic Corrosion Test Using Direct Comparisons with Actual Field Exposure", Paper No. 912284, ibid., pp. 205-220.

Q-LAB

Cyclic corrosion testing is intended to produce failures representative of the type found in outdoor corrosive environments. These tests expose specimens to a series of different environments in a repetitive cycle. Simple exposures like Prohesion may consist of cycling between salt fog and dry conditions. More sophisticated automotive methods call for multi-step cycles that may incorporate immersion, saturating or controlled humidity and condensation, along with salt fog and dry-off. Originally, these automotive test procedures were designed to be performed by hand. Laboratory personnel manually moved samples from salt spray chambers, to humidity chambers, to drying racks. In modern times, microprocessor-controlled chambers typically automate these exposures and reduce variability.

Exposure Environments

Any or all of the following environments may be used for cyclic corrosion testing.

Ambient Environment: As used in cyclic corrosion test procedures, this term means laboratory ambient conditions. Ambient environments are usually used as a way to very slowly change the test sample's condition. For example, the sample is sprayed with salt solution and allowed to dwell at "ambient" for two hours. The sample is actually going through a very slow dry-off cycle while subject to a particular temperature and humidity. Q-FOG CRH chambers can generate conditions that simulate "ambient" conditions for tests that do not specifically require lab ambient.

Typically, "ambient environments" are free of corrosive vapors and fumes. There is little or no air movement. Temperature is usually 23 ± 5 °C. Relative humidity is 50% or less. The ambient conditions should be monitored and recorded for each test.

Chamber Environments: Cycling between non-ambient test conditions is typically performed in an automated test chamber, though in some cases is achieved by physically moving test specimens from one test chamber to another.

Temperature is almost always controlled, while relative humidity can be controlled and monitored in a Q-FOG CRH tester. Whenever possible, automatic control systems should be used. Temperature tolerances should be ±3 °C or better.

Fog (Spray) Environment: Salt fog application can take place in a corrosion-type test chamber or be done by hand in a laboratory ambient environment. The fog nozzle should be such that the solution is atomized into a fog or mist. Commonly, in addition to NaCl, the electrolyte solution contains other chemicals to simulate acid rain or other industrial corrosives. Figure 1 shows a chamber in Fog mode. All Q-FOG testers can generate fog in this manner.

Humid Environment: Cyclic corrosion procedures often call for high-humidity enviroments. Typically they specify 95 to 100% RH. These may be achieved by using a condensation chamber as in ASTM D2247.[11] As an alternative, a salt fog chamber may be used to apply a pure water fog. Figure 2 shows a Q-FOG® cyclic corrosion tester operating in the humidity mode. The Q-FOG CCT uses a vapor generator to achieve this condition in a Humid step, while the Q-FOG CRH controls even very high RH values.

Dry-Off Environment: A dry-off environment may be achieved in an open laboratory or inside a chamber. The area should be maintained with enough air circulation to avoid stratification and to allow drying of the material. The definition of "dry-off" can be problematic. There is disagreement on whether a specimen should be considered dry when the surface is dry, or when the specimen has dried throughout. As corrosion products build up, the time necessary to achieve full dry-off may increase. Figure 3 shows Q-FOG chamber dry-off. All Q-FOG testers are capable of performing drying.

Corrosive Immersion Environment: This environment would normally consist of an aqueous solution with an electrolyte at a specified concentration, typically up to 5%. Typical pH is 4 to 8 and temperature is usually specified. The solution will become contaminated with use, so it should be changed on a regular basis.

Water Immersion Environment: Distilled or deionized water should be used. ASTM D1193[12] provides useful guidance on water purity. The immersion container should be made of plastic or other inert material. Acidity of the bath should be within a pH range of 6 to 8. Temperature should be 24 ± 3 °C.

Q-FOG testers do not perform immersive testing.

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11. ASTM D2247, Practice for Testing Water Resistance of Coatings in 100% Relative Humidity.
12. ASTM D1193, Specification for Reagent Water.

Figure 1

Q-FOG Chamber Fog Function

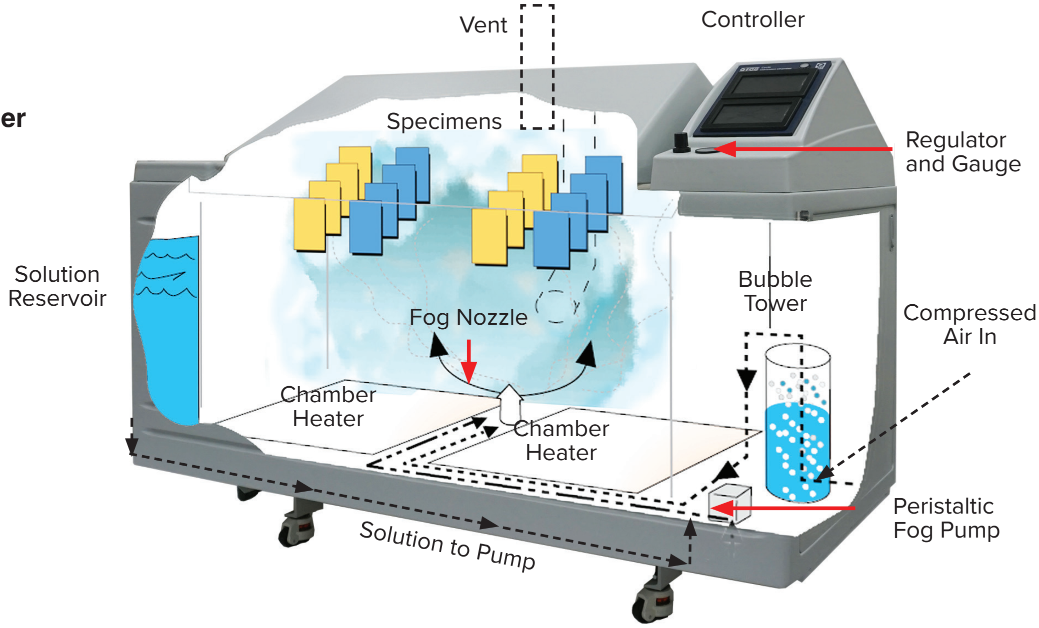


Figure 2

Q-FOG Chamber Humidity Function

Q-FOG CCT model shown

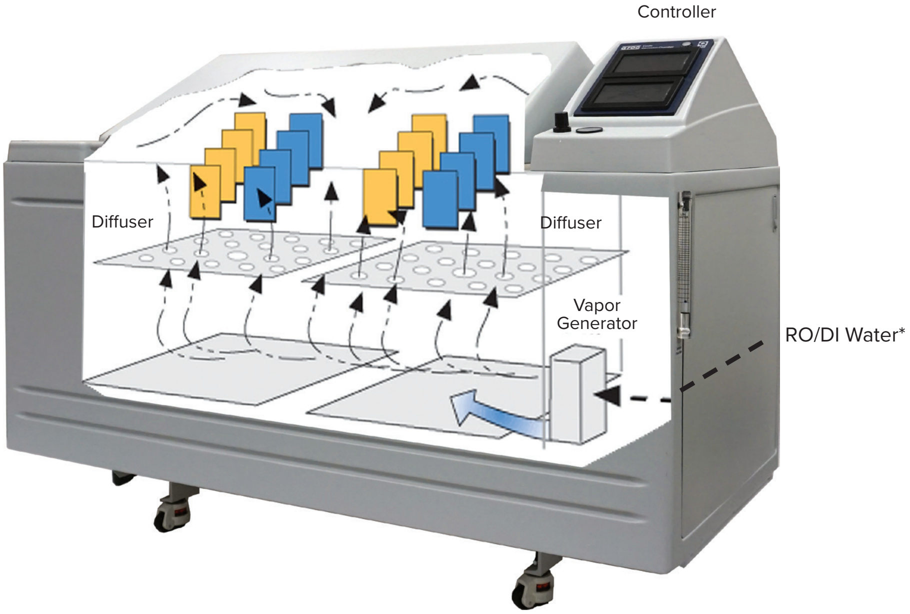
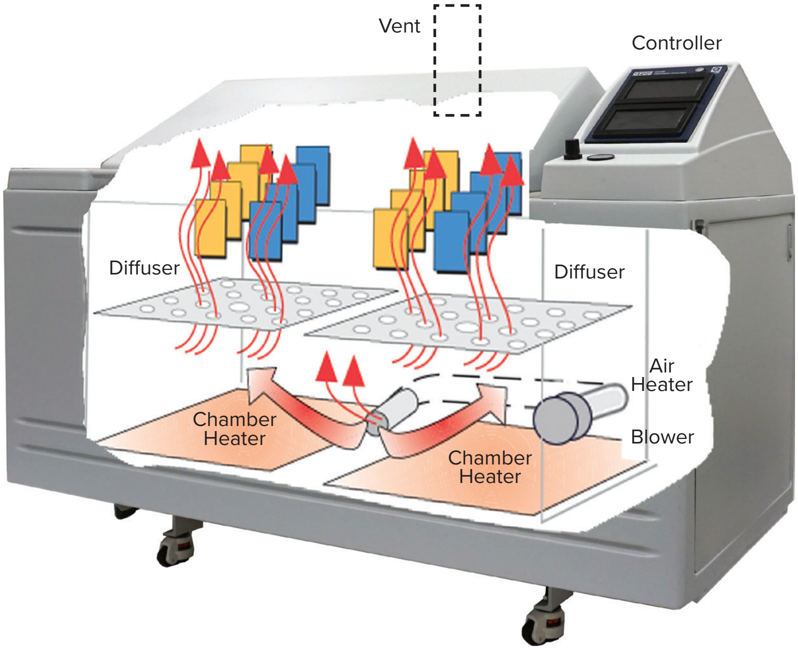


Figure 3

Q-FOG Chamber Dry-Off Environment



Guidelines for Cyclic Corrosion Testing

Because tests are often complicated, multi-step exposures, the procedures themselves can often confound the researcher. The following guidelines are intended to aid the user in understanding the possible sources of variability in cyclic corrosion exposures. The guidelines are also intended to assist in obtaining good inter-laboratory agreement of results.

Use of Reference Specimens

Whenever possible, reference specimens (specimens of known performance in the test being conducted) should be tested concurrently with the actual specimens under test. Preferably, more than one reference specimen will be used and the references chosen will bracket the test specimen's expected performance. The references will allow the normalization of test conditions during repeated running of the test and will also guide comparisons of test results from different repeats of the test.

Preparation of Test Specimens

It is common practice to scribe or chip coated test samples before exposure to the cyclic corrosion test. This provides a break in the coating that accelerates corrosion. When a gravelometer is used, the procedure shown in ASTM D3170[13] is recommended.

There is a growing body of evidence indicating that differences in scribe depth can significantly affect the cyclic corrosion test results. This is particularly important for galvanized substrates. In most cases, the scribe should penetrate into the base metal. It is especially important that the specific scribe tool be reported, since scribe geometry can also affect results. A microscope may be useful for characterizing the scribe damage. A scribing method is described in ASTM D1654.[14]

Exposure Precautions

In addition to the precautions specified in ASTM B117, the multi-functional nature of cyclic corrosion test exposures adds to the potential problems in the area of repeatability and reproducibility of results.

Chamber Loading: Chambers that are loaded to capacity will normally take longer to make transitions between temperatures than will lightly loaded chambers. Chambers should be loaded evenly to maintain good air flow during the test. Validation of test chamber performance should always be conducted with a full chamber.

Transition (Ramp) Time: Transition time can be a factor affecting results in both manual and automated exposures. In manual exposures, transition time is the time that it takes to move the test specimens from one environment or exposure condition to another. In automated chambers, transition time refers to the time it takes the machine to change the exposure conditions inside the chamber. Automated chambers can be expected to give more predictable and reproducible transitions than manual exposures. The effect of transition times on test results still needs to be studied further. Therefore, as much as is practical, transition times should be monitored and reported. Transition time can be expected to vary, depending upon:

- Variability in ambient conditions
- Variability in manual operational procedures
- Type of equipment used
- Cabinet loading

Fog Deposition and Uniformity: In conventional salt spray tests, the uniformity of fog dispersion is typically determined by collecting the fog deposited at various positions around the chamber. Unlike ASTM B117, monitoring of cyclic fog deposition rates cannot be accomplished while the test is operating. This is because most cyclic corrosion exposures specify relatively short fog cycles. Consequently, to determine the fog dispersion uniformity in a cyclic corrosion tester, it is necessary to collect the fog between tests in a special continuous spray run of at least 16 hours.

Test Interruptions: Whenever a test must be interrupted, the test panels should be stored under the least corrosive conditions available. All interruptions and handling of panels should be reported.

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13. ASTM D3170, Standard Test Method for Chip Resistance of Coatings.
14. ASTM D1654, Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments.

Reporting

In addition to all of the usual test conditions that need to be reported in conventional salt spray tests, cyclic corrsion test reports should include:

- Ramp time for all transitions in automated cabinet tests
- Loading (number and distribution of specimens) of all automated cabinets
- Daily range and mean temperature and relative humidity for the laboratory room where “ambient” conditions are maintained in manual tests

Common Cyclic Corrosion Test Cycles

The following cycles are in common use. This list is not comprehensive. The conditions shown below are merely a summary of the full instructions found in the various specifications, test methods and practices. Consult the actual documents for more complete instructions, warnings, etc.

Other cycles may be more appropriate for your application. SAE J1563[15] is particularly useful as a source of guidance for cyclic testing. Visit q-lab.com/webinars and check out our webinar on **Modern Corrosion Testing** for up-to-date information on controlled RH testing pioneered by automotive OEMs.

Advantages of Automated Cyclic Corrosion Testing

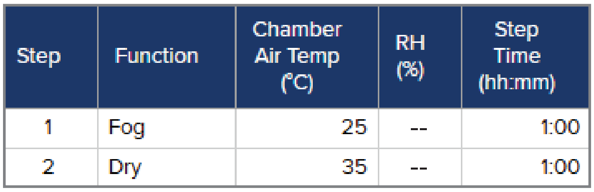
Cyclic corrosion test methods were originally developed as labor-intensive manual procedures. Automated, multi-functional chambers are now the industry standard and can perform cyclic corrosion tests in a single chamber. Some of the advantages of automated systems are that they:

- Eliminate manual moving of test specimens from one chamber to another
- Eliminate laborious spraying of test specimens
- Eliminate variability in results from excessive specimen handling
- Allow more predictable transition times

Prohesion Cycle

The Prohesion test was developed in England for industrial maintenance coatings applications. Prohesion also has a reputation as a good test for filiform corrosion.

The Prohesion electrolyte solution is much more dilute than traditional salt fog. In addition, the spray atomizing air is not saturated with water.



Step	Function	Chamber Air Temp (°C)	RH (%)	Step Time (hh:mm)
1	Fog	25	--	1:00
2	Dry	35	--	1:00

Notes

Electrolyte Solution: 0.05% sodium chloride (NaCl) and 0.35% ammonium sulfate ($(NH_4)_2SO_4$)
Solution pH: 5.0 - 5.4
Solution Collection: 1 -2 mL/hr
Typical Duration: 2,000 hr

(The dry-off is achieved by purging the chamber with fresh air, such that within 0:45-hour all visible droplets are dried off of the specimens.)

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15. SAE J1563, Guidelines for Laboratory Cyclic Corrosion Test Procedures for Painted Automotive Parts.

Corrosion/Weathering Cycle

For industrial maintenance coatings, the addition of UV has been found useful for improving correlation on some formulations.[16, 17] This is because UV damage to a coating can make it more vulnerable to corrosion. The most common Corrosion/Weathering Cycle, ASTM D5894, consists of one week of QUV® weathering tester exposure alternating with one week of Prohesion.

ASTM D5894 consists of two cycles: weathering and corrosion.

Weathering

4 hours	UV exposure, UVA-340 lamps, 60 °C
4 hours	Condensation (pure water), 50 °C

Corrosion (Prohesion as described above)

1 hour	Salt fog application at 25 °C (or ambient)
1 hour	Dry Off at 35 °C

Each test segment (weathering and corrosion) lasts for one week. Specimens are manually transferred betweeen devices at the conclusion of each week of exposure.

Automotive Cyclic Corrosion Test Exposures

The automotive industry has taken the lead in researching cyclic corrosion tests. Consequently, most of the cyclic corrosion test procedures are geared toward automotive applications.

GM 9540P/B[18] According to the research done by the SAE ACAP Committee and the AISI, this is currently considered one of the preferred cyclic corrosion test methods for automotive cosmetic corrosion (painted or precoated metals). GM 9540P/B requires a 16 hour work day or an automatic cycling test chamber. If performed manually, a sprayer is used to mist the samples until all areas are thoroughly wet. Parts should be visibly dry before each mist application. If performed manually, the samples should be left at the ambient conditions over the weekend. There are automated testers available that will perform this exposure in a single chamber.

Step	Function	Chamber Air Temp (˚C)	RH (%)	Step Time (hh:mm)	Ramp Time (hh:mm)
Repeat steps 1-3 4×					
1	RH	25	45	0:27	
2	Shower	25		0:03	
3	RH	25	45	1:30	
4	RH	49	100	7:30	1:00
5	RH	49	95	0:30	
6	RH	60	25	8:00	3:00

Notes	
Electrolyte Solution:	0.9% sodium chloride (NaCl), 0.1% calcium chloride (0.1%), and 0.075% sodium bicarbonate (0.075%)
Solution pH:	not specified
Solution Collection:	not specified

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16. Simpson, C.H., Ray, C.J., and Skerry, B.S., "Accelerated Corrosion Testing of Industrial Maintenance Paints Using a Cyclic Corrosion Weathering Method," Journal of Protective Coatings and Linings, May 1991, Volume 8, No. 5, pp. 28-36.

17. Skerry, B.S., Alavi, A., and Lindren, K.I., "Environmental and Electrochemical Test Methods for the Evaluation of Protective Organic Coatings," Journal of Coatings Technology, October 1988, Volume 60, No. 765, pp 97-106.

18. GM 9540P/B has largely been replaced by GMW 14872 but was still a key early automotive cyclic corrosion test

Japanese Automotive Cyclic Corrosion Tests

Japanese researchers have developed a number of cyclic corrosion tests. Most are primarily for automotive applications.

CCT-1. CCT-1 is specified by some Japanese automotive manufacturers. It is also known as CCT-A.

Step	Function	Chamber Air Temp (˚C)	RH (%)	Step Time (hh:mm)
1	Fog	35	--	4:00
2	RH	60	25	2:00
3	RH	50	100	2:00

Notes

Electrolyte Solution: 5% sodium chloride (NaCl)
Solution pH: Neutral
Solution Collection: 1-2 mL/hr

JASO M609 Japanese Automobile Standards Organization (JASO) test method M609 for automotive corrosion. This test switches the times of the first two steps (making them 2 hours and 4 hours) and specifies minimum transition times between steps to minimize variability.

Step	Function	Chamber Air Temp (˚C)	RH (%)	Step Time (hh:mm)	Ramp	Ramp Time (hh:mm)
1	Fog	35	--	2:00	Less than	0:30
2	RH	60	25	4:00	Less than	0:30
3	RH	50	100	2:00	Less than	0:15

Notes

Electrolyte Solution: 5% sodium chloride (NaCl)
Solution pH: Neutral
Solution Collection: 1-2 mL/hr

CCT-4. CCT-4 is specified by some Japanese automotive manufacturers. In the SAE and AISI research projects, CCT-4 was shown to be one of the exposures that best correlated with actual vehicle corrosion results. There are no special provisions for testing over the weekend.

Step	Function	Chamber Air Temp (˚C)	RH (%)	Step Time (hh:mm)	Ramp	Ramp Time (hh:mm)
1	Fog	50	--	0:15		
2	RH	60	25	2:30	Less than	0:30
3	RH	60	95	1:15	Less than	0:30
Repeat steps 4-5 5×						
4	RH	60	25	2:40	Less than	0:30
5	RH	60	95	1:20	Less than	0:30

Notes

Electrolyte Solution: 5% sodium chloride (NaCl)
Solution pH: Not specified
Typical Duration: 50 cycles (1,200 hours)
Solution Collection: 1-2 mL

Summary

There are a large number of cyclic corrosion procedures to choose from. Each has advantages and limitations. Some researchers prefer fog environments to immersion. Some prefer specialized electrolyte solutions to simulate acid rain. Many prefer the advantages of automated chambers.

The relative advantages of various exposure temperatures, durations, and sequences remain somewhat controversial and researchers will, no doubt, continue to modify cycle times and adjust corrosive solutions. However, there is a strong consensus that, for most materials, cyclic corrosion testing gives more realistic results than traditional salt spray.

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