**Which Accelerated Test Is Best?**

Dr. Nigel Whitehouse  
**Paint Research Association**  
**Teddington, UK:**

The hot salt spray test (ASTM B117, Standard Practice for Operating Salt Spray [Fog] Apparatus) has been an American standard and a benchmark test for more than 50 years. It was developed originally to allow the relative marine corrosion resistance of metals and coated metals to be assessed. The justification for exposure to continuous, neutral (pH 6.5–7.2) salt fog (5% sodium chloride solution) at 35°C (95°F) has always been that a coating system that will resist the test conditions should also perform well in aggressive service environments. However, the assumption was that the mechanisms of corrosion and degradation in service would be the same as those in the test cabinet.

It has now been recognised for many years that little, if any, correlation exists between the results from salt spray tests and in-service performance. Considerable research effort has been directed over the last 20 years towards developing more representative accelerated corrosion test procedures. Progress has been most rapid, however, in the last five years.

It was Harrison and his co-workers who argued that salt spray tests based on sodium chloride spray alone could never simulate corrosion in industrial atmospheres. When Harrison used a salt spray solution consisting of 3.25% (w/w) of ammonium sulphate and 0.25% (w/w) of sodium chloride (“Harrison’s mix”) as the working solution for a salt spray test, he observed improved correlation with coatings exposed for 14 years in an industrial environment.

Timmins developed Harrison’s ideas further. He used a diluted Harrison’s mix (0.35% ammonium sulphate and 0.05% sodium chloride) for his tests and introduced wet and dry cycling (three hours of salt spray followed by one hour of drying at ambient temperature). Timmins called his procedure the Prohesion test.

While the Prohesion test was seen as an important advance, it was also suspected that ultraviolet (UV) radiation plays an important role in natural weathering of coated metals. This led Skerry and his co-workers to investigate test methods based on cyclic salt fog/UV condensation.

The upshot of these and subsequent studies was the publication in 1996 of ASTM D5894, Standard Practice for Cyclic Salt Fog/UV Exposure of Painted Metal, (Alternating Exposures in a Fog/Dry Cabinet and a UV/Condensation Cabinet).

At the present time, ASTM D5894 is the best available published accelerated test method for screening candidate coating systems intended for atmospheric exposure. Used in conjunction with an integrated programme of adhesion and chemical resistance tests (in this case, resistance to refinery products and process chemicals), the maintenance engineer of an oil refinery wishing to upgrade a painting system could generate enough meaningful sets of performance data relatively quickly to make an informed choice with a good measure of confidence.

Brian Skerry  
**Sherwin-Williams Industrial and Marine Coatings**  
**Cleveland, Ohio, USA:**

I would recommend ASTM D5894.1 This test incorporates the most significant factors that will affect paint performance in your application. The test includes wetting and drying cycles in the presence of aggressive chemical contaminants used in the spray electrolyte as well as the concomitant effects of UV/condensation degradation on the paints. In combination, these factors should reasonably mimic the corrosion of the substrate as well as the degradation of the paints that you might expect to observe over time at the refinery site.

Published information is available concerning the background, origins, and development of ASTM D5894.2 Results obtained from this test have also been compared with long-term practical field exposures.2,3,4,5 Paints tested according to ASTM D5894 have been

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shown to exhibit the same types of degradation and modes of failure that occur in natural outdoor environments.\textsuperscript{2,6}

Cyclic tests that do not include weathering factors (e.g., cyclic salt fog testing\textsuperscript{7}) have not been shown to give such good comparisons with long-term field results.\textsuperscript{2,3,4,5}

Although D5894 is highly recommended, it is not yet the panacea of test methods. Much work remains to be done to correlate performance quantifiably and to optimise the test cycle conditions and test period time factors. Nevertheless, it is still probably the best test available at this time. It offers many possibilities to customise the method to suit specific end-user needs.

For example, the standard ammonium sulphate/sodium chloride electrolyte could be modified for composition or concentration according to the prevailing atmospheric conditions if measured at the inland oil refinery. A synthetic acid rain electrolyte could be substituted for the standard sulphate-based electrolyte. This substitution would be advisable when testing zinc-based systems due to the inherent solubility of zinc sulphate corrosion products. Another modification possible is the addition of freeze-thaw cycles if appropriate.

Furthermore, if numerical performance data are required, the test can be instrumented, for example, to measure AC impedance and electrochemical noise under the different paint systems being screened. In summary, the method is probably the best currently available, and it has much potential for further development and enhanced sophistication.

Incidentally, you are on the correct path by avoiding the widely discredited ASTM B117.\textsuperscript{8,9}

**References**


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The traditional method of laboratory testing for corrosion resistance has been ASTM B117. It is now widely known that B117 rarely provides predictive data for outdoor coatings, especially with today’s new formulations. Although many paint laboratories run B117, they may be making unwise decisions about their products.

Will your coating be wet for 24 hours a day, as it will be in the B117 salt spray chamber? No, it will probably be subjected to many environments: corrosive spray, sunlight, heat, rain, and condensation. Therefore, you should test in a way that simulates your coating’s outdoor service environment and then accelerates it. Although there are nearly 100 different cyclic tests, two tests are commonly used for industrial coatings: ASTM D5894 is used to test outdoor industrial coatings, and ASTM G85 Annex A5 is used to test interior industrial coatings.

Typically, the duration of ASTM D5894 is 12 weeks, allowing for six consecutive two-week cycles to be run. During the first week of each cycle, specimens are exposed in a UV/condensation chamber in which the conditions are four hours of UV light at 60 C (140 F) followed by four hours of condensation at 50 C (122 F). In the second week of each cycle, the specimens are moved to a corrosion chamber in which the conditions are one hour of salt spray at 24 C (75 F) followed by one hour of drying at 35 C (95 F). The salt is a dilute mixture of 0.05% sodium chloride.
and 0.35% ammonium sulphate, with a pH between 5.0 and 5.4. This solution simulates a typical industrial environment.

The UV light/condensation exposure is included because corrosion resistance of coatings is affected by sunlight and weathering. The severity of the test may be increased by raising the test temperature, duration, or ultraviolet light intensity during the UV light/condensation exposure. It is always a good idea to expose extra specimens outdoors to confirm the results from the accelerated test.

Marit Bjordal
SINTEF
Trondheim, Norway:
The success of an accelerated test is measured against how well it correlates with real exposure. There are several investigations that compare results from accelerated tests and field tests. Atmospheric exposure involves rain, snow, drying, condensation, UV radiation, temperature changes, and pollution, combined in a random order. The environmental impact can differ considerably from one location to another, and different tests have been developed with different environments in mind. You should seek a test that creates an environment resembling the one you are concerned about.

An accelerated test is normally better at giving correct performance ranking when the systems you investigate are similar. Different coating systems will respond differently to the acceleration. The time dependence of coating degradation may also differ among various coating systems, causing the ranking to change with time. One should consider this when selecting the length of the test period.

We have previously compared the cyclic test, specifically that in the NORSOK M-501 standard (Surface Preparation and Protective Coating), the salt spray test, a cyclic salt fog test, and the Volvo VICT test involving cyclic temperature and humidity. Nineteen systems of modified epoxies with zinc primers and thick polyester coatings were tested. The results were compared to field exposures at a North Sea platform. The cyclic test according to the NORSOK standard was the one found to have the best correlation with 2.5 years of field exposure. The NORSOK test was developed with offshore conditions in mind, using seawater as the electrolyte.

(NORSOK M-501 gives requirements for the materials selection, surface preparation, application procedures, and inspection of protective coatings to be applied during the construction and installation of offshore facilities. Part of the coating pre-qualification in this standard is a cyclic test.)

In another project, polyester and epoxy powder coatings on galvanized steel were exposed in the NORSOK cyclic...
The results were compared to one-year exposure in a coastal atmosphere (corrosivity category 4, as defined in ISO 9223, Corrosion of Metals and Alloys—Corrosivity of Atmospheres—Classification) and an industrial atmosphere (corrosivity category 3). The two cyclic tests correlated well with each other but had low correlation to the one-year field tests.

As noted in the question, cyclic accelerated tests are the ones now considered best able to predict actual degradation. Some claim that the salt spray test is suitable for comparative tests, but our experience is that compared to a marine atmosphere, the salt spray test did not provide a correct ranking.

Dwight G. Weldon
Matco Associates, Inc.
Pittsburgh, Pennsylvania, USA:
It must first be pointed out that there is currently no laboratory test that can accurately predict the performance of a coating as it relates to its actual lifetime. I am frequently asked by clients to test a coating to determine how long it will last and reluctantly have to reply that there is no means of doing so. A properly designed laboratory testing programme can, however, certainly determine which coatings will perform the best in a specific environment.

It is generally recognised that ASTM D5894 is a substantial improvement to the traditional salt spray test in ASTM B117 in terms of reproducing atmospheric corrosion in an accelerated fashion. (ASTM D5894 is frequently referred to as Prohesion testing. This is a slight misnomer, since Prohesion does not have an ultraviolet [UV] component to it, but simply consists of exposure in a cyclic salt fog environment.) Several studies have shown that D5894 testing correlates much better than traditional salt spray testing with “real world” results.

Basically, the D5894 test procedure involves exposing the painted panels to alternating periods (typically one week) in a UV/condensation chamber followed by a similar period in a cyclic salt fog/dry chamber. In contrast to traditional salt fog testing, which exposes the painted panels to a continual fog of 5% sodium chloride solution at 95 F (35 C), the D5894 chamber exposes specimens to a cycle consisting of one hour of fog at ambient temperature followed by one hour of drying at 95 F (35 C). The fog is produced from a relatively dilute solution of 0.05% sodium chloride and 0.35% ammonium sulphate. This electrolyte mixture has been shown to be more typical of what is actually encountered in mild industrial environments. The cyclic nature of the experiment allows not only for some mild thermally induced expansion and contraction but also for the salts to dry out and condense on the face of the panels during the drying step. Both processes are more representative of the real world than is the static environment of a salt fog cabinet. Furthermore, the addition of a UV light component to the test acknowledges the contribution of sunlight to coating deterioration.

Although cyclic salt fog/UV testing is a substantial improvement over traditional salt fog testing, rarely can a single test adequately characterise a coating’s ability to perform in a complicated environment such as an inland oil refinery. The maintenance engineer who asked the question may wish to consider subjecting candidate coating systems to a battery of test procedures specifically selected to reflect the unique environmental conditions encountered at the oil refinery.

The first step in putting together a meaningful testing protocol is to define the environment in which the coating is expected to function. Defining the service environment makes the selection of the test procedures a much simpler task. Some additional tests that might be appropriate would...
include chemical (specifically hydrocarbon) resistance, abrasion resistance, adhesion testing, and impact resistance.

**Shuang-Ling Chong**  
**Turner-Fairbank Highway Research Center**  
**Federal Highway Administration**  
**McLean, Virginia, USA:**

To design an accelerated laboratory test to evaluate durability of coating systems, one must understand the environment to which a coating system will be exposed. The salt fog test (ASTM B117) is an unrealistic test because its constant condition of 95-98% relative humidity and high salt concentration (5% by weight) does not necessarily represent an actual exposure environment for an inland oil refinery. Thus, it generates erroneous coating performance results.

Inland oil refinery areas are exposed to a heavy industrial atmosphere with acid rain, a harsh environment. The basic raw material for refineries is petroleum or crude oil. The chemical composition of crude oils is surprisingly uniform. The elemental composition of crude oil usually falls within the following ranges: 84-87% carbon by weight, 11-14% hydrogen by weight, 0-2% sulphur by weight, and 0.2% nitrogen by weight. During the sulphur and nitrogen removal and recovery processes, sulphur dioxide and ammonia are vented through plant stacks. Sulphur dioxide easily reacts with moisture in the air to form sulphurous acid (H$_2$SO$_3$), which subsequently reacts rapidly with atmospheric oxygen to form sulphuric acid (H$_2$SO$_4$).

Therefore, a sulphur-containing compound such as ammonium sulphate should be incorporated in the accelerated test in addition to general corrosion-initiating elements such as moisture, sodium chloride, and ultraviolet (UV) light.

The solution used in the cyclic salt fog test ASTM G85 (Standard Practice for Modified Salt Spray (Fog) Testing) contains 0.35% ammonium sulphate by weight and 0.05% sodium chloride by weight, and has a pH value of 5.0-5.4. It provides conditions very similar to the oil refinery environment, where sulphuric acid and ammonia react with each other to generate ammonium sulphate, and where a small amount of sodium chloride may come from deicing salts used on roads or platforms in winter. The cyclic salt fog chamber recommended for use in testing the coating systems in question also uses wet and dry cycles that simulate real world weather conditions and generate similar corrosion modes for steel.

For warm climates, ASTM D5894 would be the logical test method to use for coating evaluation. It includes alternating UV and condensation cycles. However, a freeze cycle needs to be added to the test cycles for cold climate areas. The accelerated laboratory test developed by the U.S. Federal Highway Administration would be a better choice for the evaluation. It is essentially a modified ASTM D5894 with an additional freeze cycle.

**Colleen Campbell**  
**Rohm and Haas Company**  
**Spring House, Pennsylvania, USA:**

Salt spray testing is a very severe test run at a constant temperature of 90-95 F (32-35 C) and a salt spray concentration of 5% sodium chloride. Not only are these severe conditions, but they represent only a fraction of the conditions that can lead to weathering of painted metal. Factors that contribute to this outdoor corrosion include UV sunlight, cyclic temperatures, wet and dry cycling, and moisture in the form of rain, high humidity, and dew. Sunlight and moisture can actually have a synergistic effect. Certain materials may be resistant to moisture exposure alone but may fail when exposed to a combination of moisture and sunlight.

The process by which UV light degrades a paint film is called phot-
todegradation. As the polymer absorbs UV radiation, primary bonds of the polymer backbone are cleaved, creating reactive free radicals. The next step that occurs is autoxidation. The free radicals react with oxygen to form peroxyl radicals; these radicals can then remove hydrogen atoms from the polymer backbone to form hydroperoxides and more free radicals. These radicals can easily form additional radicals, all of which can continue to attack the polymer. The end result of the formation of radicals is a loss of the performance properties of the film.

Since coatings are currently expected to provide long-term protection, it has become increasingly more important to attempt to control the degrading effects of UV light. This may be achieved in several ways, but perhaps the easiest way is to manufacture paints containing binders that do not absorb UV light. Binders made solely from acrylic monomers are transparent to UV light and are not directly affected by it. However, this does not mean that acrylic binders are not susceptible to free radical attack by other paint materials, such as additives.

In our corrosion testing, we have been using a modified version of ASTM D5894 to obtain the best prediction of a coating’s performance in outdoor exposure. In this 12-week test, the panels are alternated, one week at a time, between an ultraviolet light/condensation cabinet and a cyclic salt fog cabinet. The UV light/condensation cabinet is set to deliver a cycle of four hours of light and four hours of condensation with temperatures ranging from 35 C to 55 C (95 to 131 F). The cyclic salt fog cabinet is set to deliver one hour of fog at ambient temperature and one hour of drying at 35 C (95 F). The fog delivers a salt solution containing 0.05% sodium chloride and 0.35% ammonium sulphate by weight. We have found this cyclic test to give results more consistent with those found on outdoor exposure.

In addition, we also run salt spray, high humidity, UV and outdoor exposures to obtain a complete picture of how a particular coating will resist these conditions.

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Technical Editor’s Note to this Month’s Question: Because of the presence of ammonium sulphate in the Timmins solution used in ASTM G85 Annex A5 and D5894, the salt-laden mist has a pH of about 5. At this pH, exposed zinc will react at a significantly higher rate than at neutral pH levels. The zinc particles (e.g., in an inorganic zinc-rich

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coating) will not properly form the protective oxides and carbonates necessary for long-term protection. Studies by SSPC: The Society for Protective Coatings in the USA have shown that untopcoated zinc-rich primers degrade relatively rapidly when exposed in accordance with ASTM D5894. Thus, coating evaluation studies that include untopcoated zinc-rich primers have not exhibited good correlation with exterior exposures at marine or industrial sites. It is likely that similar anomalies would be observed for galvanized steel and thermally sprayed metallic zinc coatings.

Got an Answer? Here are some Upcoming Questions

• High-pressure water cleaning and abrasive blasting are recommended to remove mildew from concrete. Do these methods allow residual organisms to reinfect the surface and, if so, what can be done to prevent reinfection?

• What techniques can be used to meet a specification requirement that limits chloride content to 5 micrograms or less per square centimetre inside ships’ ballast tanks prior to recoating? Is the requirement derived from an industry norm? Is it reasonable?

• What coating problems are likely to be encountered in storage tanks that have dissimilar metal sides and floors?

Got a Question?

It may be a candidate for Problem Solving Forum.

Responses should be 1-2 typed, double-spaced pages. Responses and questions should be sent along with your name, address, and telephone and fax numbers to:

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