

## **School of Engineering and Technology**

# **Assessment of Wax Coatings Using a Salt Spray Cyclic Corrosion Test**

**For**

**Bilt Hamber Ltd**

Report Number PG-BH13/001

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Revision **A**



## 1.0 Introduction

Bilt Hamber Ltd specialise in a range products associated with corrosion protection, wax coatings are among these products. The company has supplied 6 different wax coating products, including one of which are their own products – Dynax S50, for an accelerated corrosion test based on ASTM B117-03 using a salt spray chamber. The aim of the test is to qualify the performance of each product by way of a documented visual examination using digital photography at regular stages throughout the test.

## 2.0 Procedure

### 2.1 Sample Preparation

Most of the products have the wax dispersed in a solvent to assist in the coating process, in order that a fair comparison is made the wet film thickness was calculated based on the % solid content and an appropriate bar coater used such that the dry film thicknesses were as closely matched as possible (around 50µm). The table (2.1) below shows the products, the solid content, the bar coater used and the resulting theoretical dry film thickness. For the Mike Saunders product the recommended coating thickness was 500 µm so this was tested at both 50 µm and 500 µm.

**Table 2.1**

<b>PRODUCT</b>	<b>RECOMMENDED APPLICATION</b>	<b>% SOLIDS</b>	<b>BAR COATER</b>	<b>DRY FILM THICKNESS (µm)</b>
Hammerite <b>Wax-Oyl</b>	Spray, Brush	30	200 µm	60
Auson <b>Noxudol 700</b>	Spray, Brush, Dip	100	50 µm	50
<b>Rustbuster</b> Mil-Spec ASTM Rust Preventive	Dip, Spray	55	90 µm	49.5
EFTEC <b>Dinitrol 3125HS</b>	Spray	45	128 µm	57.6
Bilt Hamber <b>DYNAX S50</b>	Spray	45	128 µm	57.6
<b>MIKE SANDERS</b> Korrosions- Schutzfett	Hot dispense @ 120°C	100	50 µm	50
<b>MIKE SANDERS</b> Korrosions- Schutzfett	Hot dispense @ 120°C	100	500 µm	500

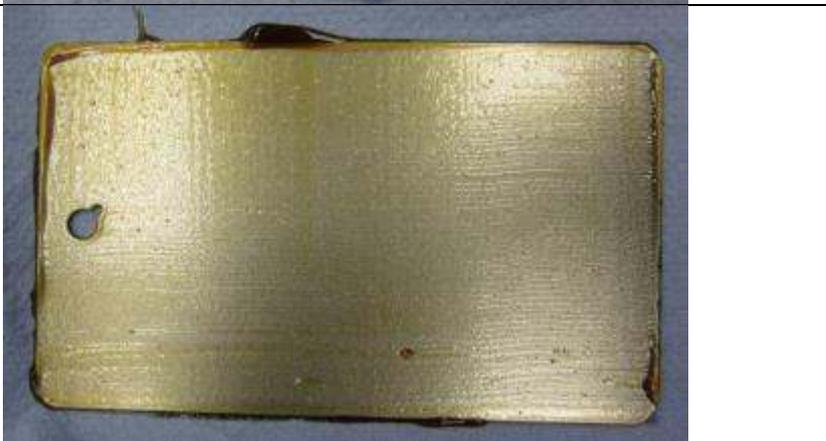
Each of the above products was applied to the steel test panels (Q-Panels) supplied with the specified bar coater, all samples were prepared at room temperature with the exception of the Mike Sanders product which is solid at room temperature so was heated to the recommended 120°C along with the Q-panel to ensure that the product stayed in the liquid state during coating the panel. Each panel was thoroughly degreased with Isopropyl Alcohol (IPA) before the application of the coating. A sample of each of the waxes was dosed onto the surface of the prepared Q-panel, the dose being more than sufficient to completely coat the surface, then the bar coater was drawn across the surface of the panel to distribute the wax evenly over the whole surface, figure 2.1 shows this process.



**Figure 2.1** – Applying Wax with the bar coater.

Three samples of each product type was prepared as above and left in a horizontal position for 48hrs to thoroughly dry. Table 2.2 below illustrates the finish of each sample.

**Table 2.2**

PRODUCT	COATED SAMPLE APPEARANCE	
<p><b>Hammerite Wax-Oyl</b></p>		
<p><b>Auson Noxudol 700</b></p>		
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>		
<p><b>EFTEC Dinitrol 3125HS</b></p>		

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS</b> Korrosions- Schutzfett 50µm</p>	
<p><b>MIKE SANDERS</b> Korrosions- Schutzfett 500µm</p>	

The samples of each coated panel were then loaded onto a rack which orientates the samples at 30° to the vertical, the 3 samples of each product were arranged on the racks so that they were located at different positions within the chamber ie. one sample at mid position between the front and back of the chamber, one located near the front the third located near the back to average out any variances there may be within the chamber. One degreased and uncoated Q-panel was also tested and this was located in a central position.

## 2.2 Test Procedures

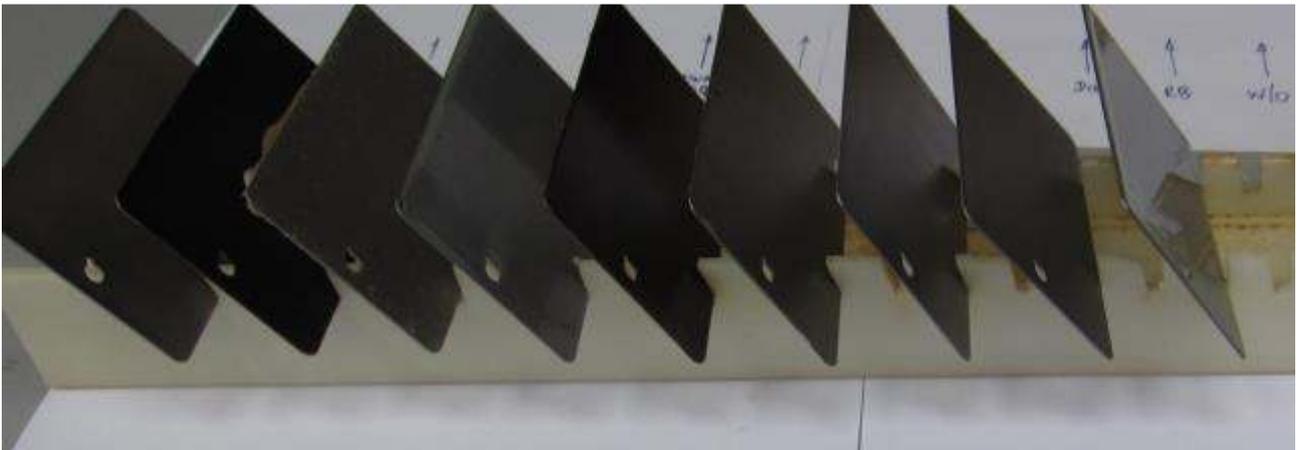
The salt spray test machine was prepared with 1250g sodium chloride per 25litres of deionised water to give a 5% salt solution as per the ASTM B117-03 specification.

Although the ASTM B117-03 states continuous salt spray it also provides freedom to do whatever is appropriate and it has been established that Cyclic Corrosion Testing (CCT - see Appendix 1)) provides a better indication of the performance of a material or coating with respect to real world exposure and there are numerous fog/dry cycles which can be adopted. The exact test adopted in this study has used a 10minute fog cycle + 50 minute dry cycle @35°C (similar to the CCT4

automotive adaptation of the B117 specification). This has been adopted as a standard cycle at the University of Hertfordshire after years of historical testing has shown this gives the best balance between accelerated corrosion and real life performance.

The test was run for 2000hrs and a total of 11 sets of photographs and mass measurements were made throughout the test at regular intervals. Figures 2.2.1 and 2.2.2 show the racked test samples and salt spray test chamber respectively.

After the nominal 2000hrs, those samples which still had the protective wax coating had the bottom half of each Q-panel cleaned with acetone and a soft nylon brush to expose the condition of the underlying steel, again a visual comparison was made.



**Figure 2.2.1 – Racked Q-panels**



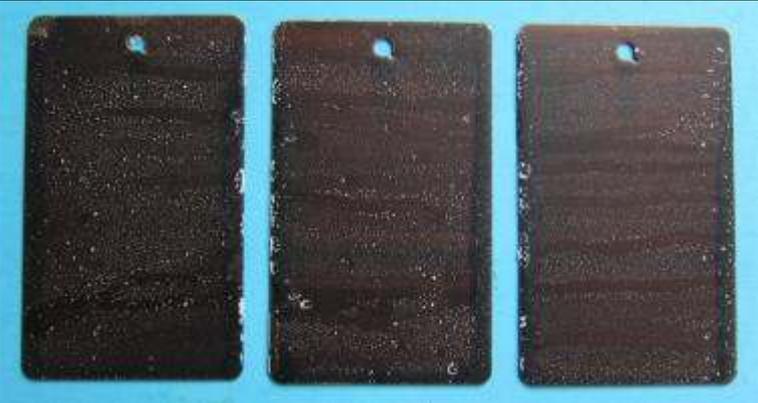
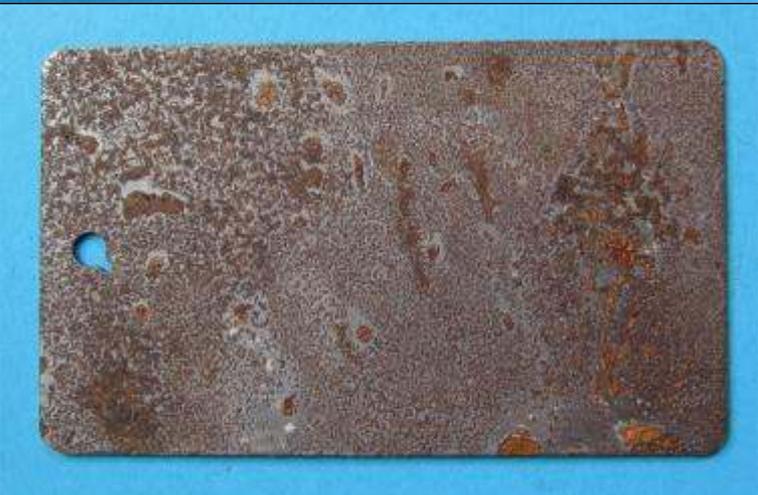
**Figure 2.2.2 – Salt Spray test Chamber**

### 3.0 Results

The following tables (3.1 to 3.11) provide the images at each interval throughout the 2000hr test.

**Table 3.1 – After 47 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 47hrs		
<b>Hammerite Wax-Oyl</b>			
<b>Auson Noxudol 700</b>			
<b>Rustbuster Mil-Spec ASTM Rust Preventive</b>			
<b>EFTEC Dinitrol 3125HS</b>			

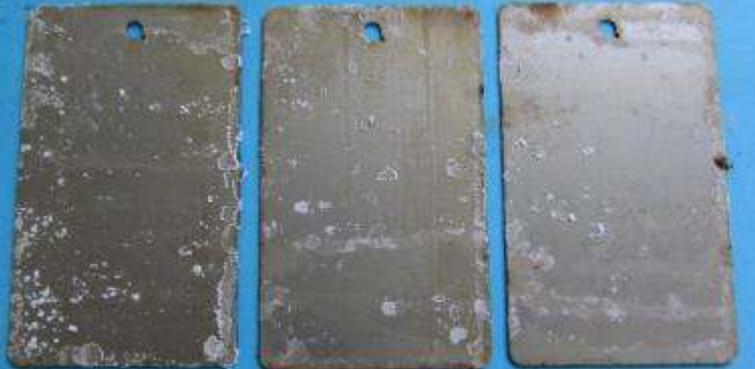
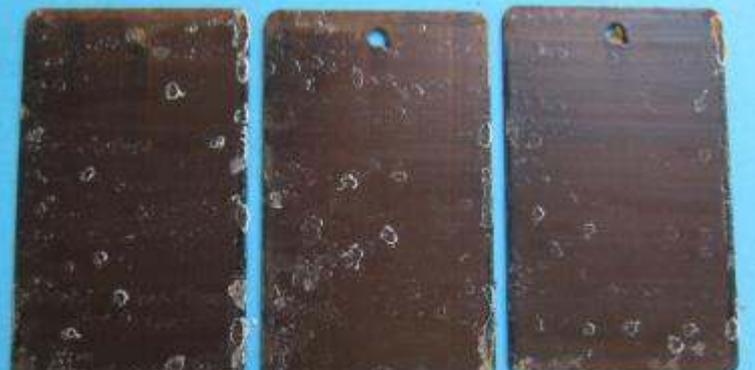
<p>Bilt Hamber DYNAX S50 45%</p>	
<p>MIKE SANDERs Korrosions- Schutzfett 50µm</p>	
<p>MIKE SANDERs Korrosions- Schutzfett 500µm</p>	
<p>UNCOATED</p>	

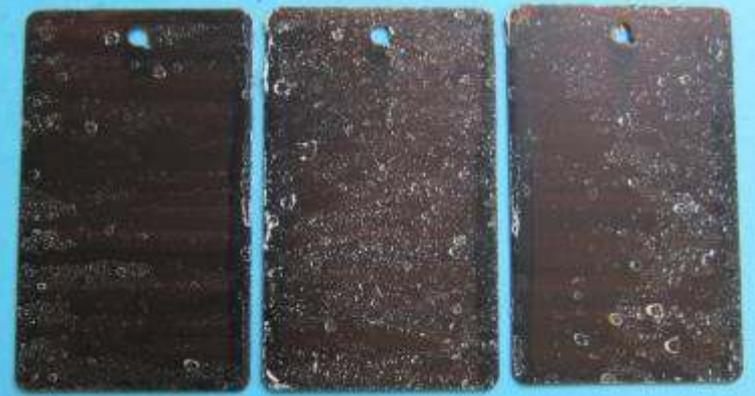
**Table 3.2 – After 143 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 143hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.3 – After 192 hrs**

<b>PRODUCT</b>	<b>COATED SAMPLE APPEARANCE @ 192hrs</b>		
<b>Hammerite Wax-Oyl</b>			
<b>Auson Noxudol 700</b>			
<b>Rustbuster Mil-Spec ASTM Rust Preventive</b>			
<b>EFTEC Dinitrol 3125HS</b>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.4 – After 358 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 358hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

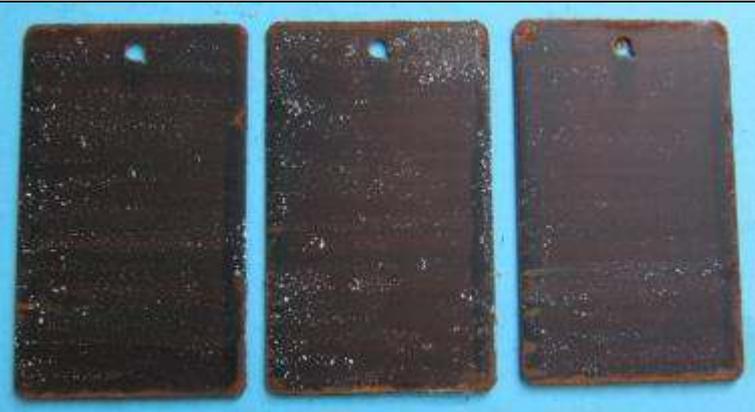
**Table 3.5 – After 525 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 525hrs		
<p>Hammerite Wax-Oyl</p>			
<p>Auson Noxudol 700</p>			
<p>Rustbuster Mil-Spec ASTM Rust Preventive</p>			
<p>EFTEC Dinitrol 3125HS</p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.6 – After 722 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 722hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.7 – After 836 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 836hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.8 – After 1176 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 1176hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.9 – After 1393 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 1393hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.10 – After 1743 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 1743hrs		
<p>Hammerite Wax-Oyl</p>			
<p>Auson Noxudol 700</p>			
<p>Rustbuster Mil-Spec ASTM Rust Preventive</p>			
<p>EFTEC Dinitrol 3125HS</p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

**Table 3.11 – After 1868 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 1868hrs		
<p>Hammerite Wax-Oyl</p>			
<p>Auson Noxudol 700</p>			
<p>Rustbuster Mil-Spec ASTM Rust Preventive</p>			
<p>EFTEC Dinitrol 3125HS</p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

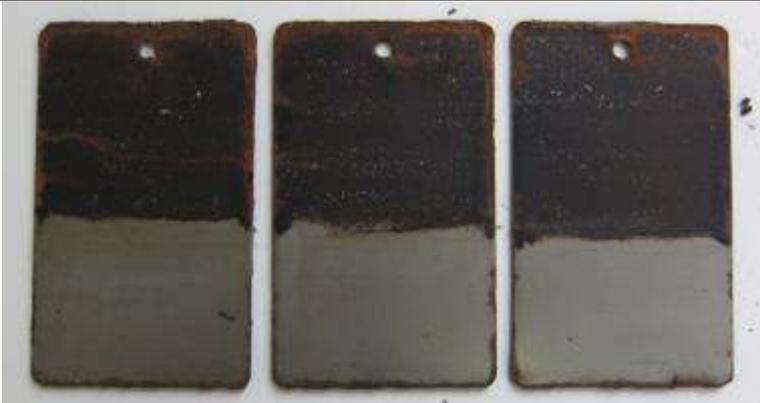
**Table 3.12 – After 2084 hrs**

PRODUCT	COATED SAMPLE APPEARANCE @ 2084hrs		
<p><b>Hammerite Wax-Oyl</b></p>			
<p><b>Auson Noxudol 700</b></p>			
<p><b>Rustbuster Mil-Spec ASTM Rust Preventive</b></p>			
<p><b>EFTEC Dinitrol 3125HS</b></p>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	

The following table (table 3.13) shows the specimens after solvent cleaning  
**Table 3.13 – After 2084 hrs with solvent clean**

PRODUCT	APPEARANCE @ 2084hrs + solvent washed		
<b>Hammerite Wax-Oyl</b>			
<b>Auson Noxudol 700</b>			
<b>Rustbuster Mil-Spec ASTM Rust Preventive</b>			
<b>EFTEC Dinitrol 3125HS</b>			

<p><b>Bilt Hamber DYNAX S50</b></p>	
<p><b>MIKE SANDERS Korrosions- Schutzfett 50µm</b></p>	<p><b>TOO CORRODED</b></p>
<p><b>MIKE SANDERS Korrosions- Schutzfett 500µm</b></p>	
<p><b>UNCOATED</b></p>	<p><b>TOO CORRODED</b></p>

#### 4.0 Discussion

With the exception of the Mike Sanders product each wax was similar in the ease of its application. The Mike Sanders product was applied at 120 degC and as such required additional equipment and also required the specimen to be heated as it instantly solidified on application and could not be spread into a uniform coating. This has significant drawbacks in coating real structures which cannot be heated. The Waxoyl product although easy to apply gave a very non-uniform coating which appeared to have large particles or clumps of particles once applied.

The Mike Sanders product specified a coating thickness of 500µm which is around 10 times the thickness of the nominal 50µm dry film thickness. It is evident from the results that a thickness of 50µm provides very poor protection using this product, and although the 500µm thickness performed well it is not a fair comparison due to the relative excessive thickness of the product.

Both the Rustbuster product and the Noxudol products showed a large difference in the effectiveness of the coating between specimens and for the Noxudol product the protection appeared good for all three specimens up to around 1150 – 1400 hrs after which the rate corrosion increased significantly. This suggests that there is a detrimental interaction between the wax coating and the locally developing products of corrosion which undermine the surrounding coating.

The uncoated sample provides a reference for the severity of degradation through corrosion with which to qualify the effectiveness of each product.

From table 3.13 it is possible to assess the relative performance of the products, section 5.0 concludes the report by stating the relative performance.

## 5.0 Conclusions

With reference to table 3.13 the relative performance of each product in this test, is as follows, the figures in brackets are the dry film thickness of the wax coating:

1. **Bilt Hamber** Dynax S50 (57.6µm)
2. **Mike Sanders** Korrosions-Schutzfett (500µm)
3. **Auson** Noxudol 700 (50 µm)/ **Rustbuster** Mil-Spec ASTM Rust Preventive (49.5µm)
4. **EFTEC** Dinitrol 3125HS (57.6µm)
5. **Hammerite** Waxoyl (60µm)
6. **Mike Sanders** Korrosions-Schutzfett (500µm)

# **APPENDIX 1**

## **– Technical Bulletin Cyclic Corrosion Testing**

# Introduction to Cyclic Corrosion Testing

This paper is intended as a general introduction to cyclic corrosion testing (CCT). It outlines the rationale for cyclic testing, includes some guidelines for using cyclic tests and explains some common CCT 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.<sup>1</sup> This traditional salt spray specifies a continuous exposure to a 5% salt fog at 35°C. During the course of 80 years of use, there have been many modifications and refinements to 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, 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<sup>2,3</sup> 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.<sup>4,5,6,7,8,9,10</sup> Japanese researchers have also developed a number of cyclic corrosion test methods.



## 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.

1. ASTM B 117, Method of Salt Spray (Fog) Testing.
2. Ormet, 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. Timmons, F.D., "Avoiding Paint Failures by Prohesion," J. Oil & Colour Chemists Assoc., Vol. 82, No. 4, p. 131 (1979).
4. M. L. Stephens, "SAE ACAP Division 3 Project: Evaluation of Corrosion Test Method", Paper No. 920571, Automotive Corrosion and Prevention Conference Proceedings, P-238, Society of Automotive Engineers, Warrendale, PA (1992), pp. 157-154.
5. H. E. Townsend, "Status of a Cooperative Effort by the Automotive and Steel Industries to Develop a Standard Accelerated Corrosion Test", Paper No. 920949, Ford, pp. 139-140.
6. F. Bickelhorst, "Hoogovens' Contribution to AISI Program 'Accelerated Corrosion Testing: A Cooperative Effort by the Automotive and Steel Industries' Paper No. 920970, ibid., p. 147-156.
7. M. Pelschel, 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-200, Society of Automotive Engineers, Warrendale, PA (1991), pp. 179-203.
8. R. J. Newell, W.A. Schumacher, D.C. McClure, R.D. Granata and H. E. Townsend, "Progress by the Automotive and Steel Industries Toward and Improved Laboratory Cosmetic Corrosion Test", Paper No. 912075, ibid., pp. 79-96.
9. F. Bickelhorst, "Further Developments Toward a Standard Accelerated Corrosion Test for Automotive Materials", Paper No. 912277, ibid., pp. 99-114.
10. D. D. Davison and W. A. Schumacher, "An Evaluation and Analysis of Commonly Used Accelerated Cosmetic Corrosion Test Using Direct Comparisons with Actual Field Exposure", Paper No. 912284, ibid., pp. 206-220.



Cyclic corrosion testing is intended to produce failures representative of the type found in outdoor corrosive environments. CCT 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, humidity, 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, etc. More recently, microprocessor controlled chambers have been used to 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 CCT 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.

Typically, "ambient environments" are free of corrosive vapors and fumes. There is little or no air movement. Temperature is usually  $25 \pm 5^\circ\text{C}$ . Relative humidity is 50% or less. The ambient conditions should be monitored and recorded for each test.

**Chamber Environments:** Non-ambient environments are usually chamber exposures. Cycling between different non-ambient environments can be performed by physically moving the test specimens from one chamber to another or, in automated chambers, by cycling from one condition to another.

The temperature and relative humidity should be monitored. Whenever possible, automatic control systems should be used. Temperature tolerances should be  $\pm 3^\circ\text{C}$  or better.

**Fog (Spray) Environment:** Salt fog application can take place in a B117 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 2 shows a chamber in the fog mode.

**Humid Environment:** CCT procedures often call for high humidity environments. Typically they specify 95 to 100% RH. These may be achieved by using ASTM D 2247.<sup>11</sup> As an alternative, a B 117 chamber may sometimes be used to apply a pure water fog. Figure 3 shows a Q-Fog operating in the humidity mode.

**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 4 shows Q-Fog dry-off.

**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 D 1193<sup>12</sup> 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  $24^\circ\text{C} \pm 3^\circ\text{C}$ . Conductivity should be  $< 50 \text{ mmho/cm}$  at  $25^\circ\text{C}$ .

11. D 2247, Practice for Testing Water Resistance of Coatings in 100% Relative Humidity.  
12. D 1193, Specification for Reagent Water.

Figure 1  
**Q-Fog Performing the Fog Function**

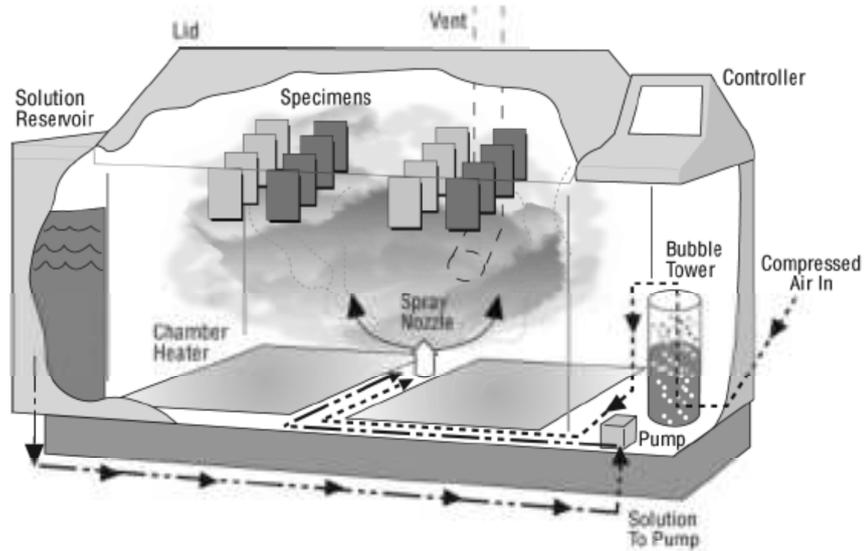


Figure 2  
**Q-Fog Operating the Humidity Function**

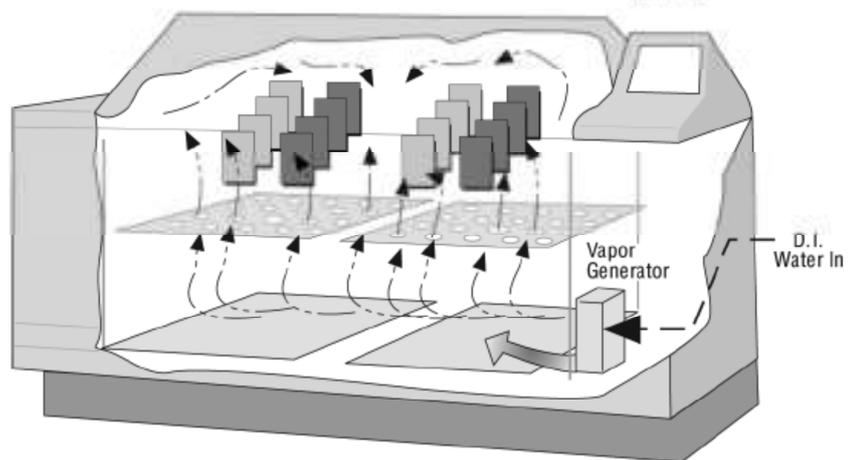
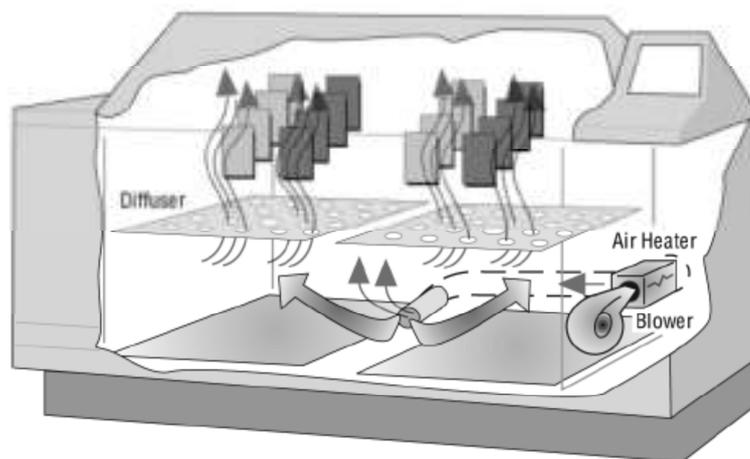


Figure 3  
**Q-Fog Showing the Dry-Off Environment**



## Guidelines for CCT Testing

Because CCT 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 CCT 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 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 CCT. This provides a break in the coating which accelerates corrosion. When a gravelometer is used, the procedure shown in D3170<sup>13</sup> is recommended.

There is a growing body of evidence indicating that differences in scribe depth can significantly affect the CCT 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.<sup>14</sup>

## Exposure Precautions

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

**Chamber Loading Level:** 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.

**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 fall-out at various positions around the chamber. Unlike B117, monitoring of CCT fog deposition rates cannot be accomplished while the test is operating. This is because most CCT exposures specify relatively short fog cycles. Consequently, to determine the fog dispersion uniformity in a CCT tester, it is necessary to collect the fog fall-out between tests in a special continuous spray run of at least 16 hours. See section Method B117 for detailed instructions on fog collection.

**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.

13. D 3170, Standard Test Method for Chip Resistance of Coatings.

14. D 1654, 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, CCT test reports should include:

- Ramp time for all transitions in automated cabinet tests
- Loading (i.e., number samples) of all automated cabinets
- Daily range and mean temperature and relative humidity for the laboratory room where "ambient" conditions are maintained in manual tests

## Advantages of Automated CCT

Cyclic corrosion test methods were originally developed as labor intensive manual procedures. Automated, multi-functional chambers are now available and can perform CCT 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

## 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 J156315 is particularly useful as a source of guidance for cyclic testing.

## 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.

Exposure conditions include:

Electrolyte Solution	0.05% sodium chloride & 0.35% ammonium sulfate
Solution Acidity	pH between 5.0 and 5.4

The Prohesion exposure cycle is:

1 hour	Salt fog application at 25°C (or ambient)
1 hour	Dry Off at 35°C (The dry-off is achieved by purging the chamber with fresh air, such that within 3/4-hour all visible droplets are dried off of the specimens.)
Repeat	

## Corrosion/Weathering Cycle

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

Electrolyte Solution	0.05% sodium chloride & 0.35% ammonium sulfate
Solution Acidity	pH between 5.0 and 5.4.
Typical Duration	2,000 hours.

The Corrosion/Weathering exposure cycle is:

1 hour	Salt fog application at 25°C (or ambient)
1 hour	Dry Off at 35°C (The dry-off is achieved by purging the chamber with fresh air, such that within 3/4-hour all visible droplets are dried off of the specimens.)

Repeat for one week, then manually move the samples to a QUV Accelerated Weathering Tester and expose at the following cycle:

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

Repeat for one week

Manually move the samples to a CCT tester and repeat the whole procedure.

## Automotive CCT Exposures

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

**GM 9540P/B.** According to the research done by the SAE ACAP Committee and the AISI, this is currently considered one of the preferred CCT methods for automotive cosmetic corrosion (painted or precoated metals). GM9540P/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.

The GM9540P/B exposure conditions include:

Electrolyte Solution	0.9% NaCl, 0.1% CaCl <sub>2</sub> & 0.25 NaHCO <sub>3</sub>
Solution Acidity	pH between 6.0 and 8.0.
Typical Durations	80 cycles (1,920 hours)

The GM9540P/B exposure cycle is as follows:

-	Thorough Salt Mist Application
90 minutes	Ambient Conditions (25°C, 30 - 50% RH)
-	Thorough Salt Mist Application
90 minutes	Ambient Conditions (25°C, 30 - 50% RH)
-	Thorough Salt Mist Application
90 minutes	Ambient Conditions (25°C, 30 - 50% RH)
-	Thorough Salt Mist Application
210 minutes	Ambient Conditions (25°C, 30 - 50% RH)
8 hours	Humidity (95 - 100% RH)
8 hours	Dry Off (60°C, <30% RH)
Repeat	

16. Simpson, C.H., Raj, 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. 28-35.

17. Skerry, B.S., Alavi, A., and Lindner, K.L., "Environmental and Electrochemical Test Methods for the Evaluation of Protective Organic Coatings," *Journal of Coatings Technology*, October 1998, Volume 60, No. 765, pp. 97-106.

## Japanese Automotive Cyclic Corrosion Tests

The Japanese 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.

The CCT-1 exposure conditions include:

Electrolyte Solution	5% sodium chloride
Acidity	Not specified
Typical Duration:	200 cycles (1,600 hours)

The CCT-1 exposure cycle is:

4 hours	Salt fog application at 35°C
2 hours	Dry Off at 60°C, <35% RH
2 hours	Humidity at 50°C, >95% RH
Repeat	

**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. CCT-4 exposure conditions include:

Electrolyte Solution	5% sodium chloride
Solution Acidity	not specified
Typical Duration	50 cycles (1,200 hours)

The CCT-4 exposure cycle is:

10 minutes	Salt fog application at 35°C
155 minutes	Dry Off at 60°C
75 minutes	Humidity at 60°C, 95% RH
160 minutes	Dry Off at 60°C
80 minutes	Humidity at 60°C, 95% RH
160 minutes	Dry Off at 60°C
80 minutes	Humidity at 60°C, 95% RH
160 minutes	Dry Off at 60°C
80 minutes	Humidity at 60°C, 95% RH
160 minutes	Dry Off at 60°C
80 minutes	Humidity at 60°C, 95% RH
160 minutes	Dry Off at 60°C
80 minutes	Humidity at 60°C, 95% RH
Repeat	

## Acid Rain CCT

This procedure, intended to simulate an acid rain exposure, is a modification of the Japanese Automobile Standards Organization (JASO) test method M609 for automotive corrosion. Acid Rain CCT exposure conditions include:

Electrolyte Solution 5% (wt) NaCl, 0.12% (vol) HNO<sub>3</sub>, 0.173% (vol) H<sub>2</sub>SO<sub>4</sub>, 0.228% (wt) NaOH

Solution Acidity pH of 3.5

The Acid Rain CCT exposure cycle is:

2 hours	Fog at 35°C
4 hours	Dry-off at 60°C, less than 30% RH
2 hours	Wet/humid at 50°C, over 95% RH

Acid Rain CCT specifies transition times between environments as follows:

Fog to Dry	within 30 minutes
Dry to Wet	within 15 minutes
Wet to Fog	within 30 minutes

## 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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