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*by N. D. Cremer, Managing Director
C. & W. Specialist Equipment Ltd., Shropshire, England*

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Introduction

With the continual development of paint systems, there are many coatings available today which are capable of standing the most severe of environments. However their performance is essentially dependent on the adhesion of a primer to the base metal. Laboratory tests such as ASTM B117 Salt Spray, Humidity and Sulphur Dioxide influence the development of coatings yet they still allow coatings into the market place which then fail in practice. These accelerated tests consequently bear little or no resemblance to natural weathering.

Foremost among these tests is the hot Salt Spray for example ASTM B117. This test method has been and is still widely used and accepted as the definitive accelerated test to assess reliability. However, it is in reality totally unrealistic, as the majority of products are not exposed to the conditions of this test in their working environment.

When a chemist is looking at his results after Salt Spray testing, he often decides a coating with good salt spray performance is accepted over a coating with poor salt spray performance. Consequently if a coating passes its laboratory examination, then it is considered suitable and often introduced to the market place.

If a coating fails its laboratory examination then it is discarded. With this philosophy a chemist could have thrown away an ideal product for the natural world and a winner in the market place!

The average coating, or indeed the above average coating, is after application to a substrate, subjected to all the elements of natural weathering. These elements attack the coating increasingly, each one in turn causing more corrosion. Yet in the laboratory, these same coatings are assessed after undergoing individual test procedures.

However, evidence is all around us showing that these forms of individual testing are not providing the answer. Natural weathering is cyclic, so laboratory tests should correspond.

The History of Prohesion

During the 1970's considerable work was carried out to find an alternative to the continuous accelerated test ASTM B117. This test was and still is widely used in all spheres of industry, yet it was initially developed over the period 1910—1920¹ and first standardized in 1939.

This test is perhaps useful for the testing of coating performance in marine environments, however it is now used to predict the performance of coatings in every type of environment some fifty years later.

A pioneer in this early work was F.D. Timmins² who decided that although conventional salt spray was qualifying coatings in the laboratory, failures were still occurring in practice. The whole concept of accelerated testing was to be questioned. Why was a hot salt spray at 35° C to be used? How many environments could this be related to? Why use a continuous salt spray, and why use a 5% sodium chloride solution?

A 5% sodium chloride solution was originally used possibly because it relates to a marine environment. As to why a continuous spray was used or why a constant 35° C was preferred, I do not know. Again, the question must be, are they realistic conditions?

As early as 1962, Dr. J. B. Harrison and T. C. K. Tickle³ noted that although the behaviour of zinc phosphate primers were generally excellent outdoors in an industrial environment, their performance during accelerated salt spray testing was poor.

As a result of this observation, Harrison used as a spray solution a mixture of the commonly occurring atmospheric salts ammonium sulphate and sodium chloride, 3.25% by weight ammonium sulphate and 0.25% sodium chloride.

Timmins however, decided that a weak solution of Harrison's mixture should be used, consisting of 0.40 wt % ammonium sulphate and 0.05 wt % sodium chloride. It was also decided that spray at *ambient* temperature would correspond closely to natural weathering. Timmins dubbed his test Prohesion, which is an acronym for *Protection is Adhesion*.

*Presented at Federation of Societies for Coatings Technology 1989 Paint Show

Development

Early results suggested that this approach to testing was providing more realistic results. So Timmins, through Mebon Paints Ltd., approached our company to ask us to manufacture a cabinet that could provide reproducible conditions time after time, so that they had a stable base from which to work.

After further consultation with Timmins it was decided that the following test conditions would be required in a production test chamber.

Prohesion Test Requirements

- 1) A spray cycle at ambient temperature was selected and a weak solution of Harrison's mixture would be used.
- 2) An elevated temperature drying cycle was required, with an air temperature within the chamber variable from 23° C to 55° C.
- 3) Air introduction to the test chamber was required during the drying cycle.
- 4) A facility to allow cyclic wetting and drying cycles from a minimum of one hour to a maximum of ten hours was essential.
- 5) The test samples should be placed on racks on the cabinet walls so that the surfaces of all panels were exposed to the spray.

These essential conditions were incorporated together with other features which we considered vital in any form of spray chamber. The Mebon Prohesion Cabinet was then produced.

Cabinet Construction and Operation

The inner cabinet is constructed of glass reinforced plastic using high temperature resin. Between the inner and outer cabinet are electric heaters which allow much more rapid temperature cycling than possible in conventional salt spray cabinets with water jackets. This special capability for rapid cycling is essential for the Prohesion test. The outer plastic cabinet houses the control panel, which incorporates a proportional band temperature controller with a range of 23° C to 55° C. Also incorporated are timers which allow cyclic wetting and drying from periods of 10 minutes to 1000 hours.

Two or three sample galleries are located on each internal wall for the positioning of test samples. Samples are held at an angle of 15° to the vertical.

The cabinet should be situated ideally in a temperature controlled room maintained at $24 \pm 2^\circ$ C. This requirement is essential to maintain consistency of test from laboratory to laboratory.

Spray System for Salt Solution

The salt solution is stored in an external plastic reservoir, so that the temperature of the solution remains at ambient. A peristaltic pump delivers the solution to the spray nozzle via an inline solution filter. The pump provides a well controlled flow rate selectable between 0.5 ml and 1.5 ml per hour under a constant pressure. A flowmeter allows monitoring of the solution flow rate. At the spray nozzle the controlled flow of solution is mixed with compressed air for atomisation. The dispersion of

the spray pattern can be adjusted by altering the pressure of the compressed air. Thus the spray flow rate and spray dispersion can be varied independently.

The spray nozzle is positioned in the centre of the cabinet and is made out of a non-corrodible material. The nozzle does not require a baffle to break up the solution, because the solution is atomised completely by the nozzle itself to supply a particle size no greater than 50 microns.

The Air Supply

The supply of air to the tester is maintained at 28 psi. Controls on the chamber reduce air pressure to approximately 15 psi for salt solution atomization.

During the drying cycle, the full 28 psi is introduced into the test chamber via directional nozzles which create a whirl-pool effect. This air introduction provides an air enriched environment and assists the drying out of the test panels.

Typical Tests

It has been found through extensive contact with companies using this equipment that a typical test consists of a one hour spray cycle at ambient temperature followed by a one hour drying cycle at 35° C.

Using this procedure the specimens under test become visibly wet within a few minutes of the commencement of the spray cycle and visibly surface dry at the end of a dry cycle.

S. B. Lyons⁵ carried out extensive work using this method as did B. S. Skerry.⁹ Timmins also carried out later work using this method.⁴

Comparison with Conventional Salt Spray

These two tests can easily be compared and several fundamental differences can be seen.

ASTM salt spray has a saturated atmosphere (100% RH), and the samples under test are subjected to continual spray. However in the Prohesion test there is a cyclic approach with wetting and drying of test pieces. With this cyclic test, samples have an opportunity to absorb, via an osmotic effect, more water than in a continuous spray test.

Although samples in a Prohesion test are subjected to salt spray for half the time period compared with ASTM, it has been found by W. T. Shieh⁷ that if a wet/dry cycle is continually used, a highly corrosive concentrated solution will eventually build up on the surface, despite the use of a relatively dilute spray solution.

M. Stratmann⁸ also found that during a wet/dry cyclic test, the corrosion of iron occurs most rapidly during drying and rewetting episodes, not during the actual wet period. The reason for this is that the concentration of a dilute salt solution will be exceeded during surface drying, with an eventual salt precipitation. This build up of salts on the surface coating will result in general in an increase in the rate of corrosion.

The ASTM salt spray test uses a solution which has a relatively high 5% concentration, with straight sodium chloride as the salt. Prohesion uses a solution of low concentration with a mixture of naturally occurring atmospheric salts, consequently it would appear realistic to incorporate these in a test solution.

However, it should be emphasized that the two salts mentioned refer to atmospheric salts in an industrial environment. Other salts can be used. The choice of salts to be used in a solution for accelerated corrosion testing should be determined in general by the environment in which the coating is to be exposed in practice.

In Prohesion during a dry cycle, the high air flow combined with the elevated temperature changes the levels of humidity considerably in the chamber. Not only do the humidity levels change by this drying effect, but the introduction of air may enhance the oxidation process and consequently increase the rate of corrosion.

In ASTM salt spray, with a saturated environment, the humidity levels are always high. However in Prohesion, from a condition of high relative humidity at the start of a dry cycle, the relative humidity drops to between 40% and 45% by the end of a one hour dry cycle.⁹ This level of humidity is below the level at which Barton¹⁰ considers atmospheric corrosion to occur.

Consequently during the Prohesion dry cycle an air enriched atmosphere and a temperature of 35° C results in a changing humidity condition. Throughout this cycle the rate of corrosion is changing from a period of high corrosion attack to a condition of little or no corrosion attack at the end of the cycle. This type of condition is what must occur in nature.

Conclusion

Salt spray testing provides answers which are unrealistic in the natural world, yet Prohesion provides realistic results which correlate with long term exterior exposure. These results also show that with a change in raw material input, the long term performance of a coating can be effected exactly opposite to what is predicted by salt spray testing. Results obtained from Prohesion testing suggest that as an accelerated corrosion test method, it correlates with natural weathering consequently providing realistic results.

No single laboratory test can provide all the answers to the corrosion problems throughout the world. The choice of electrolyte, the selected test temperature and the type of cycle are all relevant points to take into consideration. Application malpractice will still produce failures, sub-standard raw materials will provide other failures, and extremes in the environment will cause more, but the Prohesion test could provide a benchmark for correlation with natural weathering. □

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Prohesion® is the trademark of BP Chemicals Inc. Cleveland, Ohio USA.

C. & W. Specialist Equipment Ltd., Shropshire, England, is registered user of trademark No. 1166171 G.B.

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Correlation Test Results

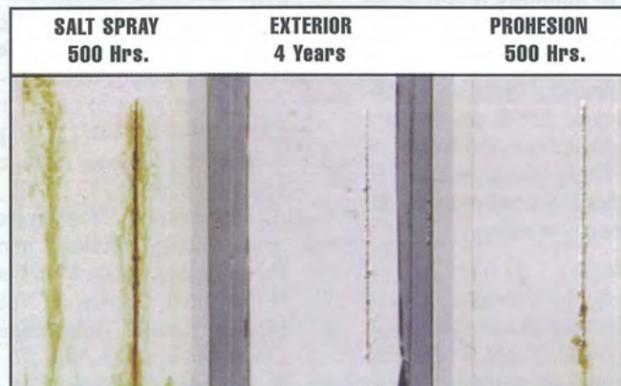
Salt Spray, Prohesion, Outdoors

These figures show the correlation of test results between Prohesion, Salt Spray (ASTM B117) and outdoor exposures at an industrial site in northwestern Indiana. The Prohesion results were more representative of outdoor corrosion than the salt spray results.

The Prohesion cycle consisted of one hour spray at ambient temperature and one hour dry off at 35°C, using a solution of 0.40% ammonium sulphate and 0.05% sodium chloride.

Two Coat Latex

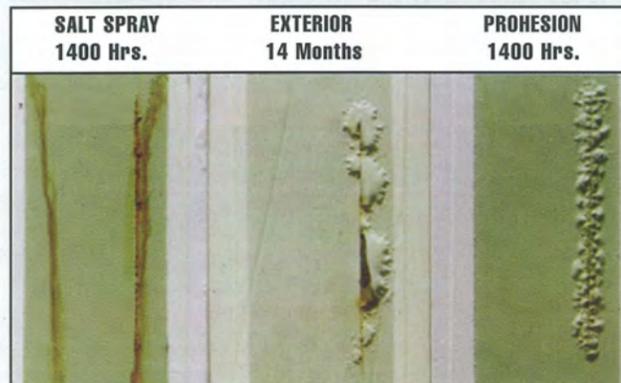
Poor correlation between salt spray and industrial exposure. Fairly good correlation between Prohesion and exterior exposure.



Acrylic Latex Primer/Topcoat System; PVC 34%, Volume Solids 40%; Inhibitor Loading 0.75 lbs/gal; applied 2 mils per coat (4 mils total) to ground test panels.

High Solids Epoxy

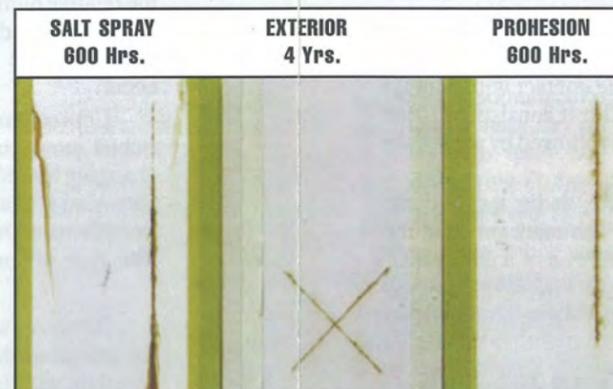
Excellent performance in salt spray with little blistering, no scribe creepage or undercut corrosion. Exterior exposure shows severe delamination from scribe and no correlation with salt spray. Prohesion shows blistering and delamination, correlating with exterior exposure.



High Solids Epoxy System; PVC 30.7%; Volume Solids 74%; Inhibitor Loading 1 lb/gal; applied 3.5 mils dry film thickness to ground test panels.

Medium Oil Alkyd, Inhibitor A

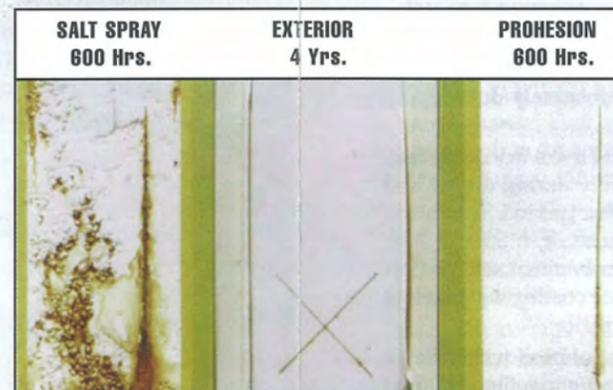
All panels exhibit good performance.



Medium Oil Alkyd System; PVC at 45%; Volume Solids 42%; Inhibitor Loading 1.5 lb/gal; primer applied to ground test panels at 1.5 mils film thickness.

Medium Oil Alkyd, Inhibitor B

A sharp contrast between industrial site exposure and salt spray. Salt spray shows complete failure. Prohesion and Exterior exposures show good performance.



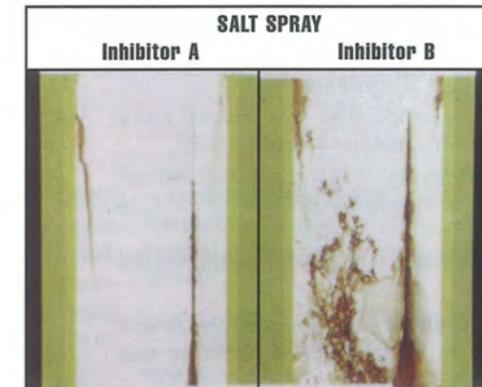
Medium Oil Alkyd System; PVC at 45%; Volume Solids 42%; Inhibitor Loading 1.5 lb/gal; primer applied to ground test panels at 1.5 mils dry film thickness.

A Comparison of Two Inhibitors

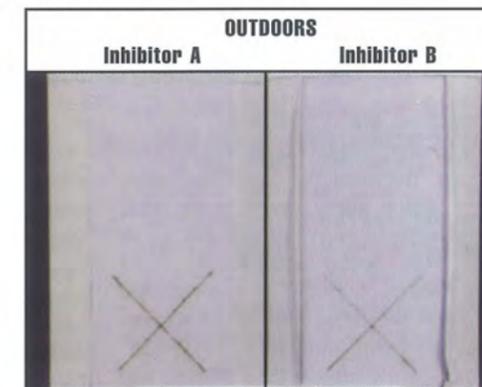
(Medium Oil Alkyd System)

Medium Oil Alkyd System; PVC at 45%; Volume Solids 42%; Inhibitor Loading 1.5 lb/gal; applied to ground test panels at 1.5 mils dry film thickness.

After 600 hours at salt spray exposure, Inhibitor A is vastly superior to Inhibitor B.



After 4 years industrial exposure, both systems exhibit good performance, with Inhibitor B slightly superior. Poor correlation with salt spray results.



After 600 hours Prohesion exposure, Inhibitor B is marginally better than Inhibitor A. Prohesion results more closely correlate to the industrial exposure.

