Accelerated Testing

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Abstract

Over the years there have been many accelerated test methods developed to gauge the performance of anticorrosive coatings and coatings in general. Many of the initial test methods were developed for solvent borne systems, whose cure mechanisms were well understood. With the advent of newer resin technologies and also more recently, tighter restrictions on VOC (Volatile Organic Content), many of these older test methods fall short of truly gauging how these systems will perform in an exterior real world environment. Some of the keys to more accurately assessing the real world performance of your coating is choosing a method which closely mimics the end use environment of your coating, properly conducting the test, and knowing how to interpret the results. Understanding these test methods is the first step towards successfully optimizing your coatings for the real world.

OVERVIEW

1. Background on Corrosion
2. Static & Cyclic Test Methods
3. Exterior Exposure
4. Comparison and Evaluation
5. Summary and Q&A

Corrosion

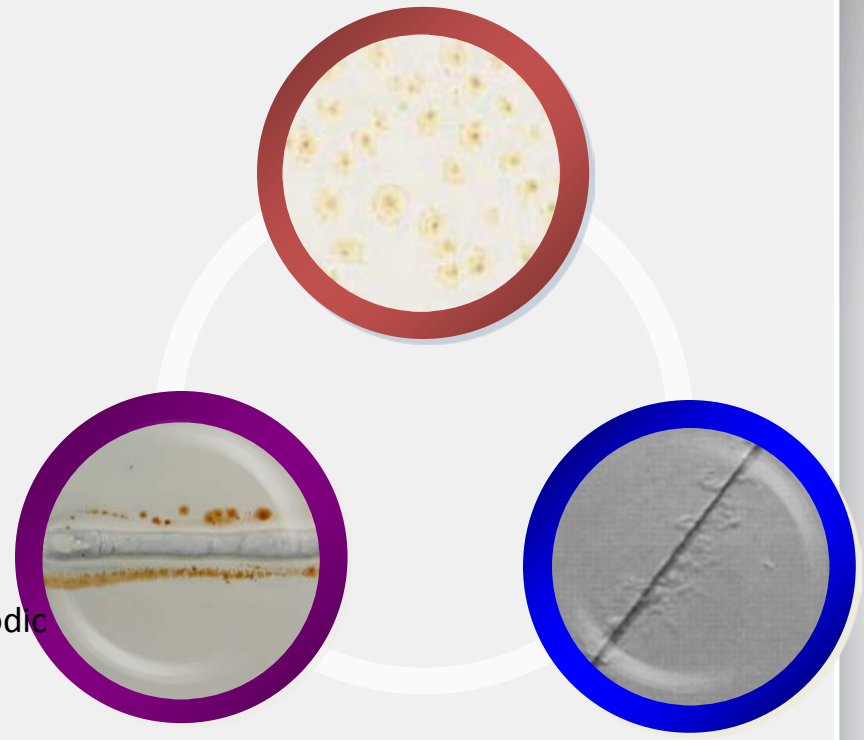
Of concern in coatings…

Flash Rust — Rapid, widespread corrosion seen during initial application.

Galvanic — Contact between two alloys which promotes oxidation of the less noble metal.

Filiform — Differential aeration promotes this unique form of corrosion.

Uniform — Metal degradation due to shifting anodic and cathodic positions.



Lambourne, R. (1999); *Paint and Surface Coatings: Theory and Practice,* 2 ed. (Lambourne, R. and Strivens, T.) England: William Andrew Publishing

Background

- What comes to mind when you hear the phrases:
  - Accelerated Testing?
  - Coating Durability?

Background

Experimenter Requires

- Rapid info
- Reproducible data
- Accurate data
- Numerous test samples

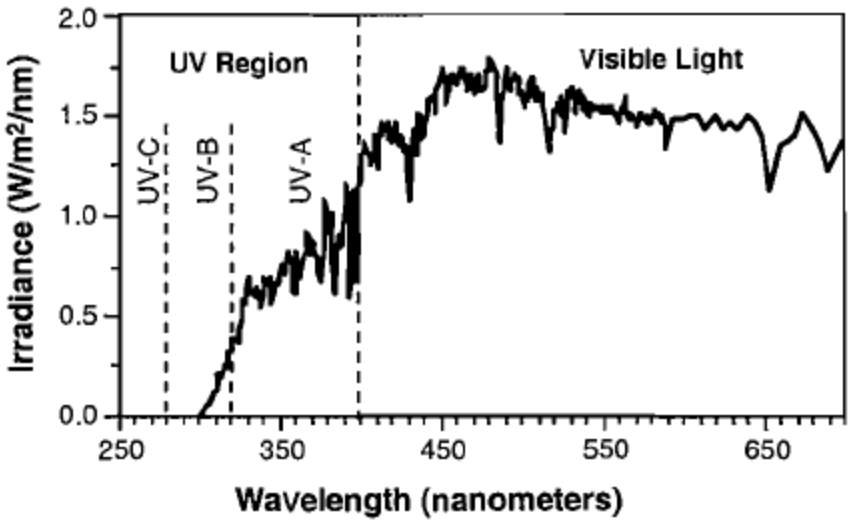
Accelerated Devices

- Artificial light
- Corrosion
- Humidity
- R&D
- QC
- Material selection

Real World Data

- Realistic?
- Not always reproducible
- Takes too long to acquire data
- Too many variables

Background



Spectral power distribution (SPD)
Summer June 1986 (Cleveland, OH)

WAVELENGTH REGIONS OF THE UV

UV-A **400 to 315 nm**	Causes polymer damage
UV-B **315 to 280 nm**	Includes the shortest wavelengths found at the earth's surface; responsible for severe polymer damage; absorbed by window glass.
UV-C **280 to 100 nm**	Found only in outer space; filtered out by earth's atmosphere; germicidal

Background

- Radiation – UV (short wavelength)
- Temperature
- Moisture permeation – dew, rain, vapor
- Oxygen permeation
- Pollutants e.g. volcanic ash, acid, SO_2
- Human error – batching, mixing, painting
- Microbes – biofilm, foulants
- Inadequate surface preparation
- Deficient coating thickness
- Others: marine environments, wind, vibration, abrasion, impact, mechanical damage, altitude

Qualification

STATIC TESTS

- Salt Spray (ASTM B117)
- Humidity Testing (ASTM D2247)
- Immersion Test (ASTM D870)
- Electrochemical Impedance (ASTM G106)

Qualification

CYCLIC TESTS

- Volvo outdoor SCAB test Simulated Corrosion Atmospheric Breakdown
- SAE J2334 Cosmetic Corrosion Lab Test, 80 cycles = 5 yrs on vehicle testing
- GMW14872 Cyclic Accelerated Corrosion Testing Formerly GM9540P 60 cycles = 10 years)
- CASS (Copper Accelerated Acetic Acid Salt Spray)

Qualification

CYCLIC TESTS

- Filiform Corrosion Test (ASTM D2803)
- QUV-A Test (ASTM D4587)
- QUV Condensation, QUV/Prohesion (ASTM D5894)
- Xenon Arc (ASTM D2568, G26)
- EMMAQUA (ASTM G90)
- South Florida Test

Salt Spray

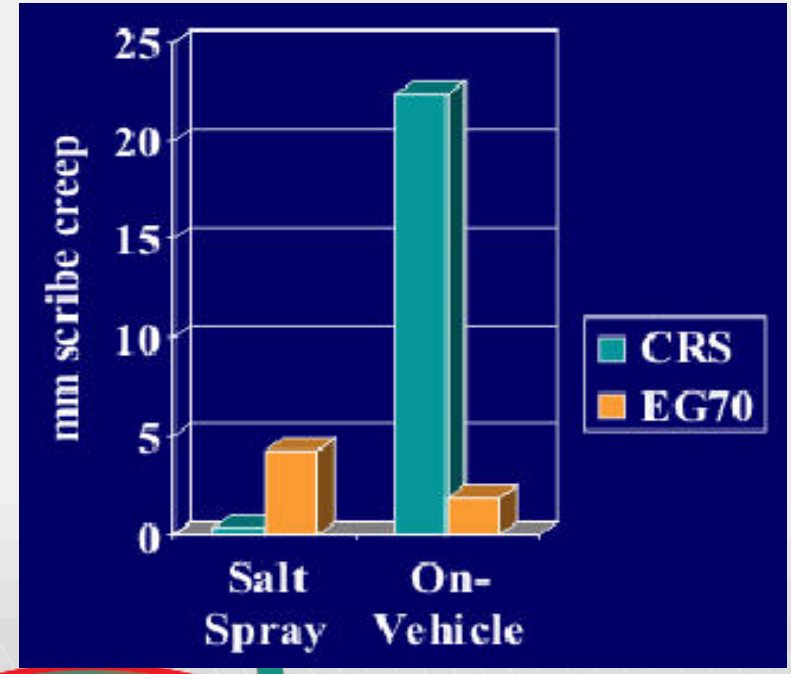
- Samples are placed in racks at an acute tilt angle.
- A 5% NaCl solution (pH 6.50-7.20) is mixed with humidified air at a spray nozzle in center of cabinet
- The temperature of the cabinet is maintained at 95°F
- Panels are subjected to static fog until failure or the desired number of hours are obtained

Salt Spray – The Good

- Widely used and requested in numerous industries
- Can be used as a quality control (QC) method
- Low cost
- Process of elimination – can eliminate coatings which have poor anticorrosive properties sooner

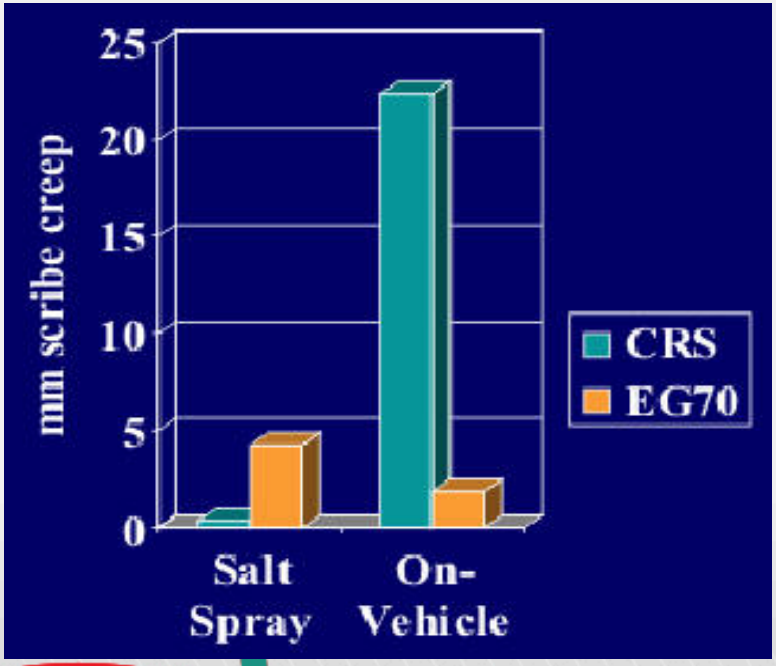
Salt Spray – The Bad

- High concentration of salt (5%)
- Corrosion rates differ among substrates (galvanized steel in sulfide environment)
- Cabinet to cabinet reproducibility
- Static fog and temperature

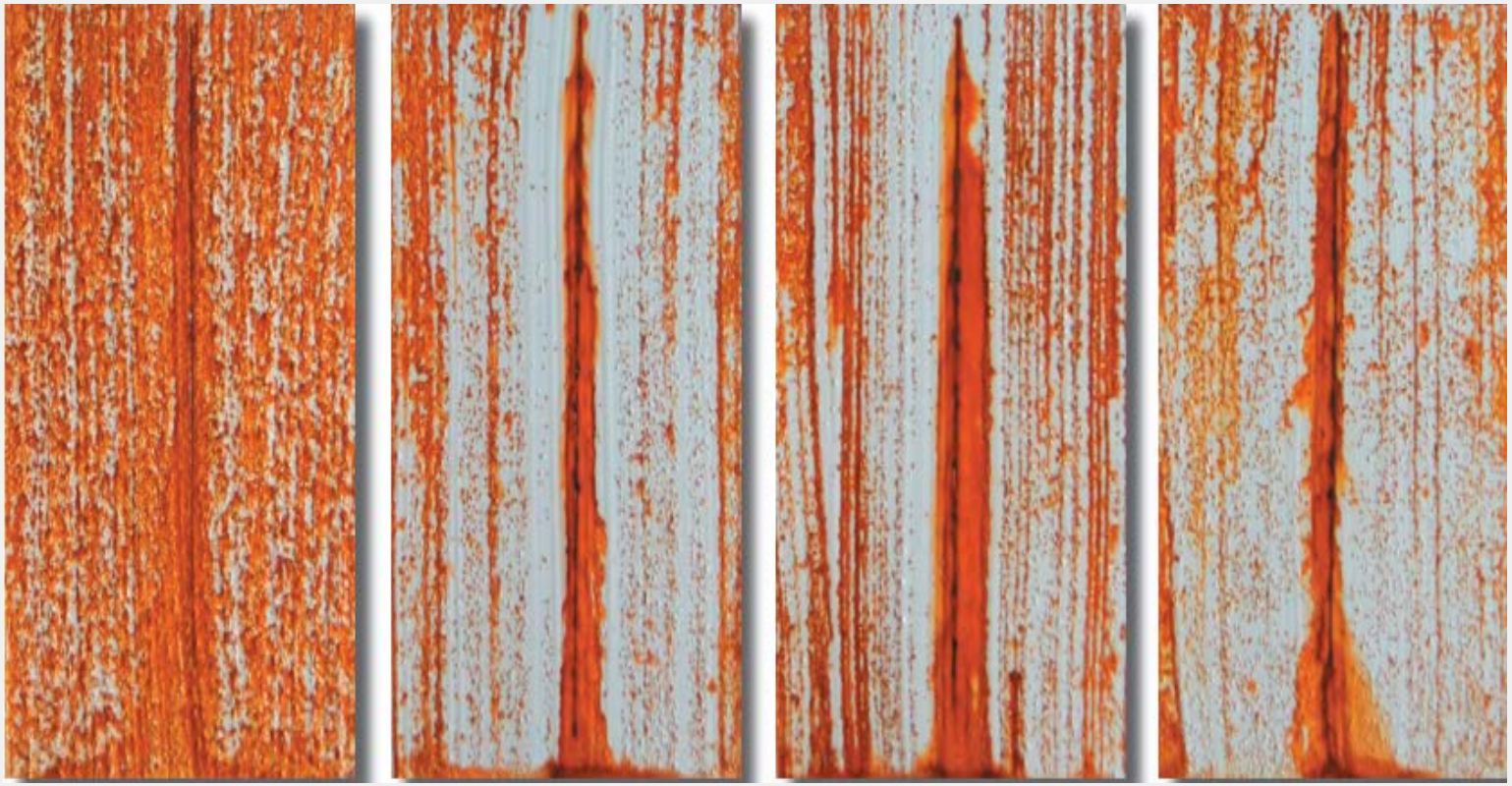


Salt Spray – The Ugly

- No exposure to UV light
- Variance between replicates
- No correlation to outdoor exposure



Salt Spray - Failures

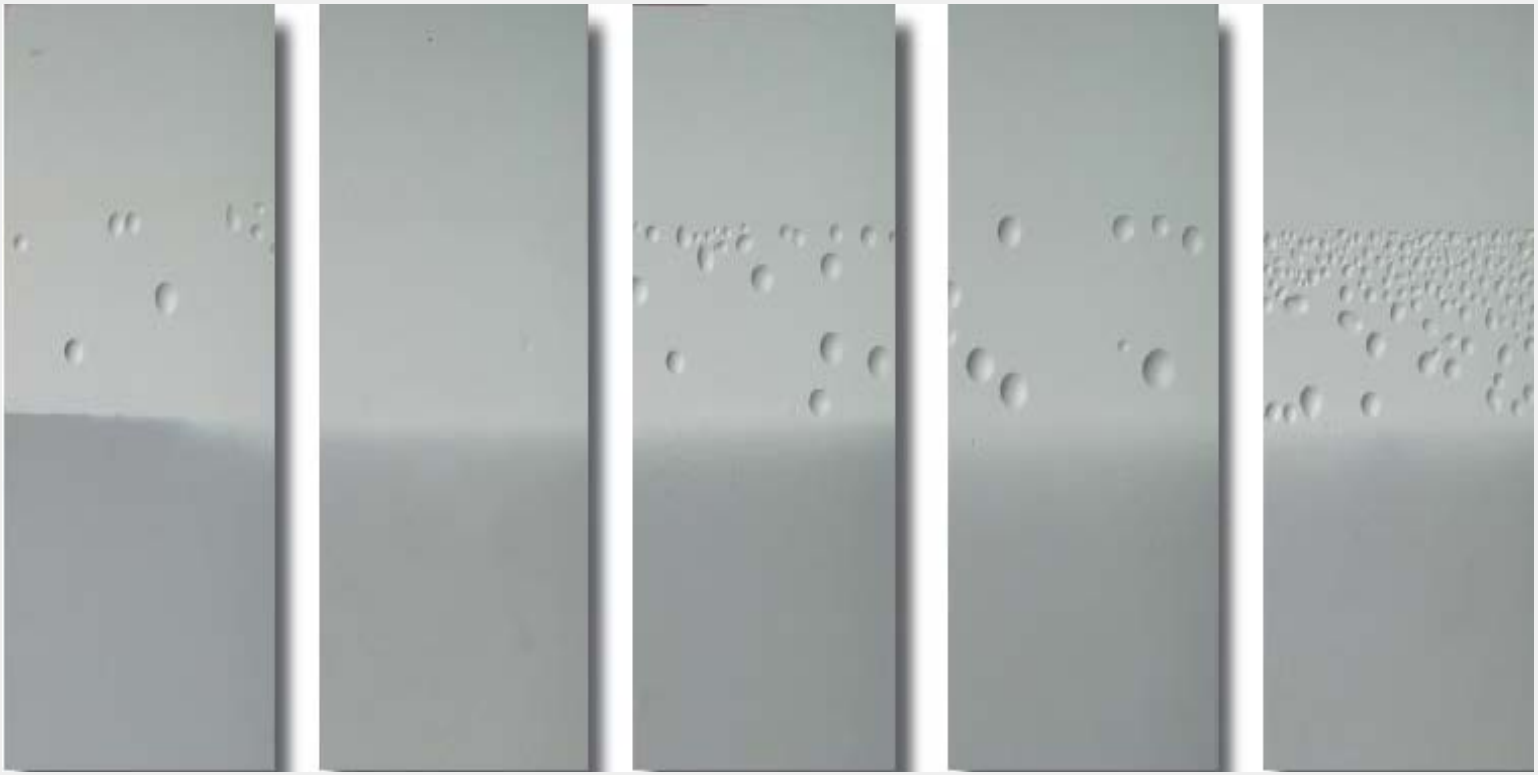


100 hours salt spray – Water based Acrylic – Substrate: CRS – 2.0 mils D.F.T

Humidity Test (ASTM D2247)

- Also referred to as Water Resistance Test
- Samples are exposed to 100% relative humidity
- Water vapor condenses on surface
- Failure can occur in many forms

Humidity Testing



Long Oil Alkyd – CRS - 2.0 mils D.F.T

336 Hours Humidity

Humidity Testing

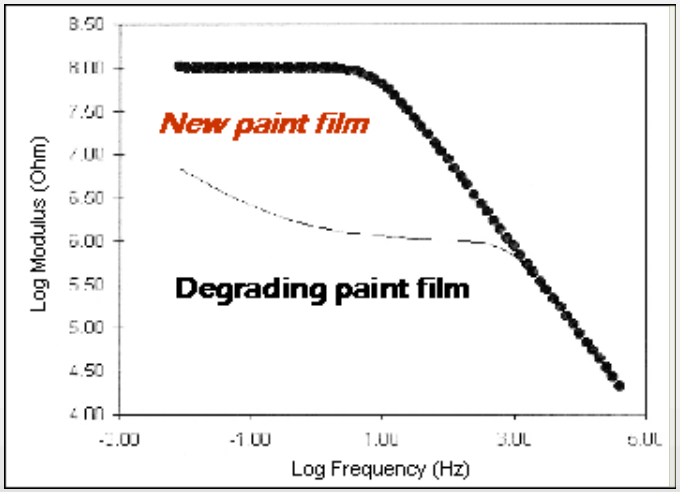
500 Hours Humidity – 2K High Solids Epoxy
Blasted Hot Rolled Steel – 3.0 mils D.F.T

Immersion Test (ASTM D870)

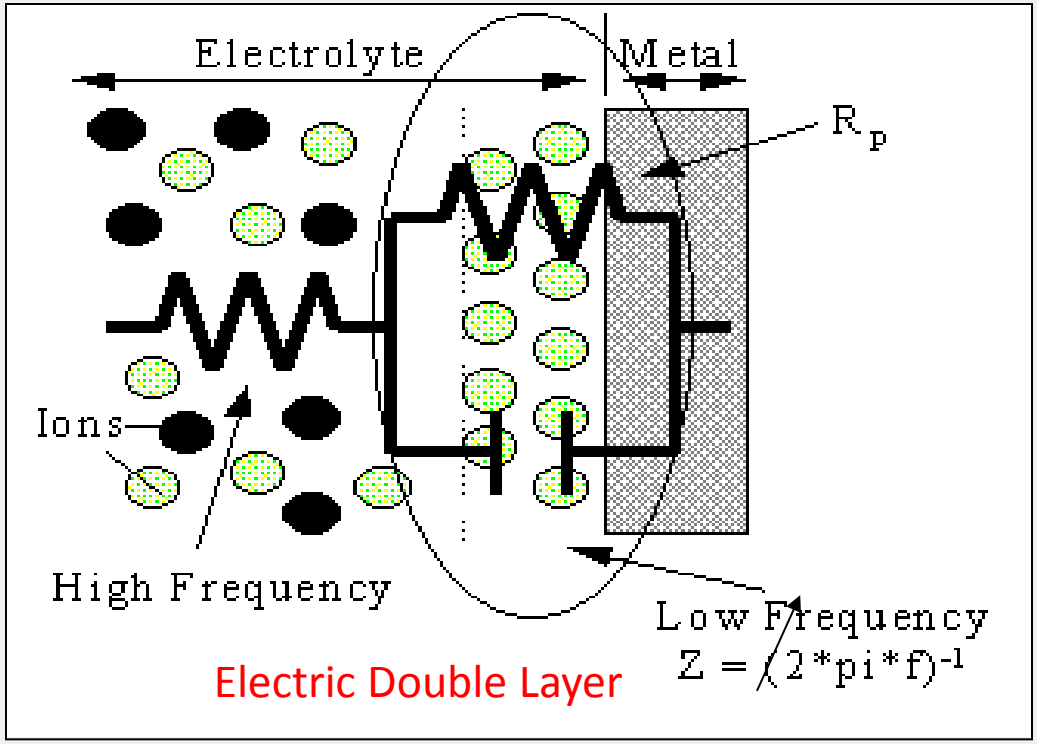
- Samples are immersed in 100°F de-ionized water bath
- Failure can be in the form of blistering, loss of adhesion or coating breakdown
- Other methods include using salt water, acids, bases, etc.
- Also be conducted at varying temperatures

Electrochemical Impedance Spectroscopy (EIS)

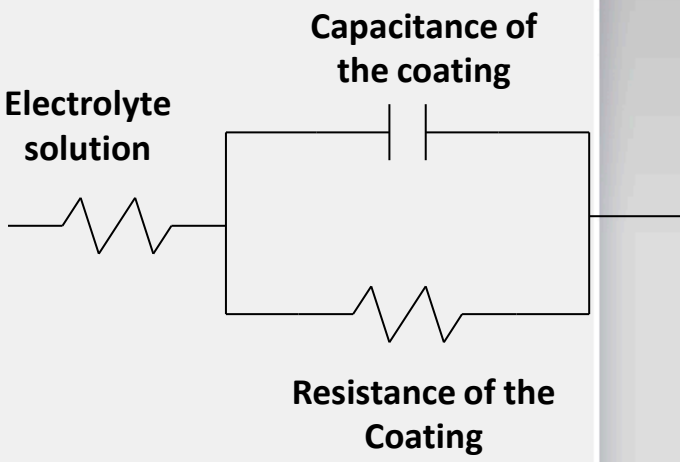
- Can detect changes in a coating before damage is observed
- Measures the breakdown of a coating due to electrolyte attack
- Measures the resistance (charge transfer) and capacitance (how the coating behaves when exposed to an electrolyte)



EIS- How does it work?



Equivalent Circuit intact coated metal



S. Koka, A. Shi and J.S. Ullett, S & K Technologies, Dayton, OH

Reference: J.M. Fildes, P. Chen, and X. Zhan, North Western University, IL.

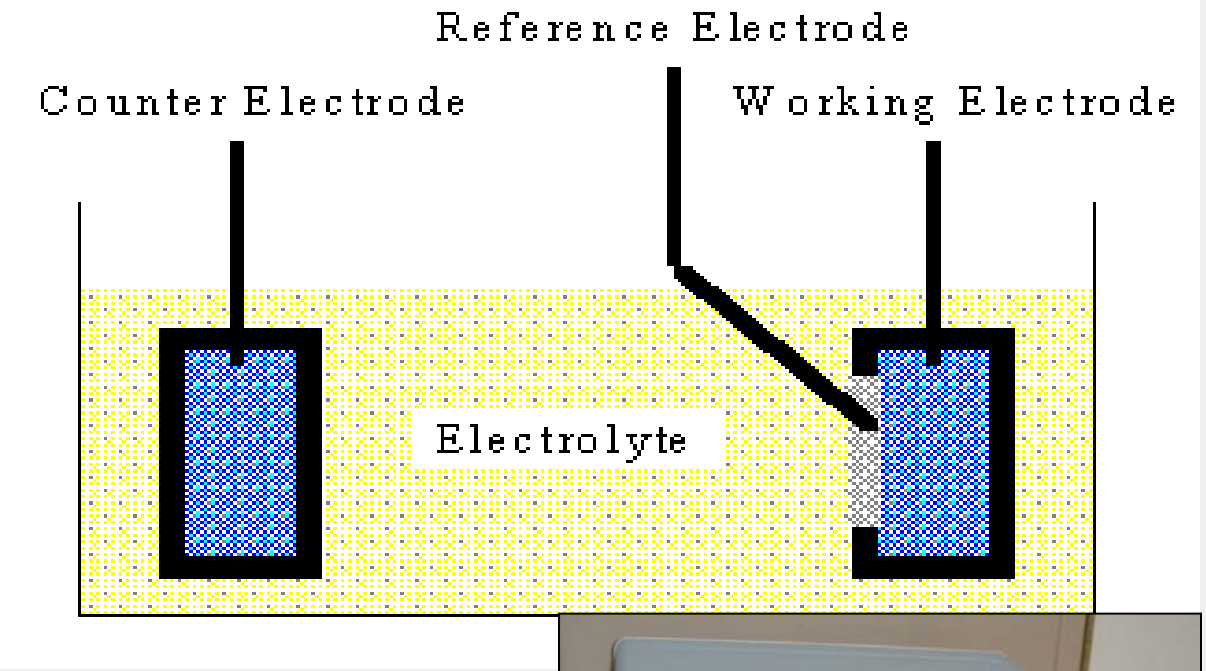
Higher Coating Resistance

or

Lower Capacitance is desired

EIS Equipment

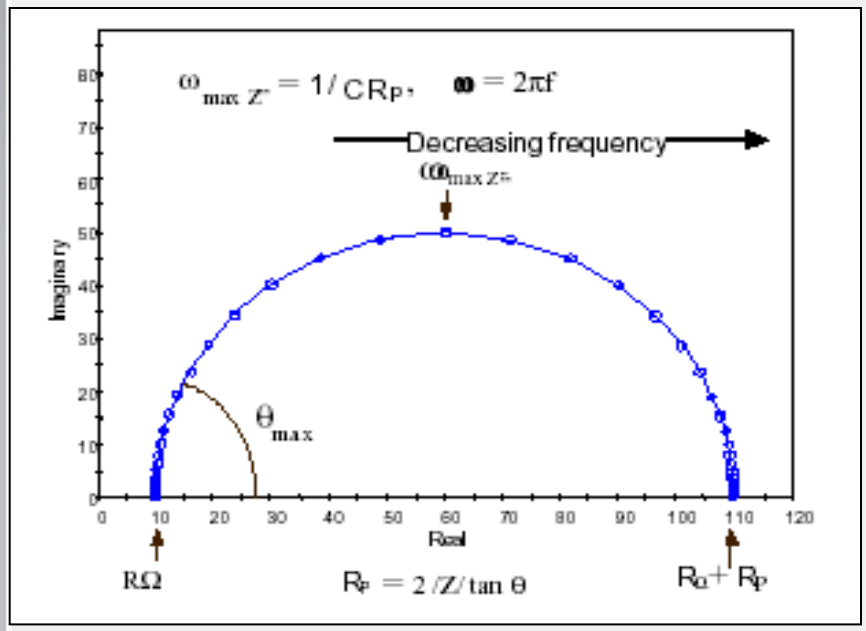
Reference Electrode - Ag / AgCl
Working Electrode - Coating Sample
Counter Electrode - Platinum Mesh



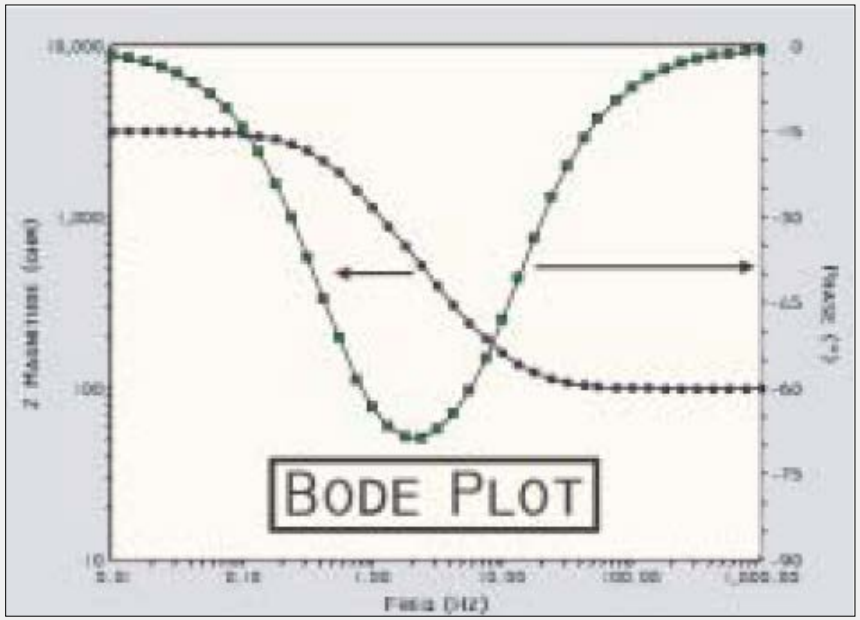
Pictures courtesy of S. Koka, A. Shi and J.S. Ullett, S & K Technologies, Dayton, OH

Result Plots

Nyquist Plot

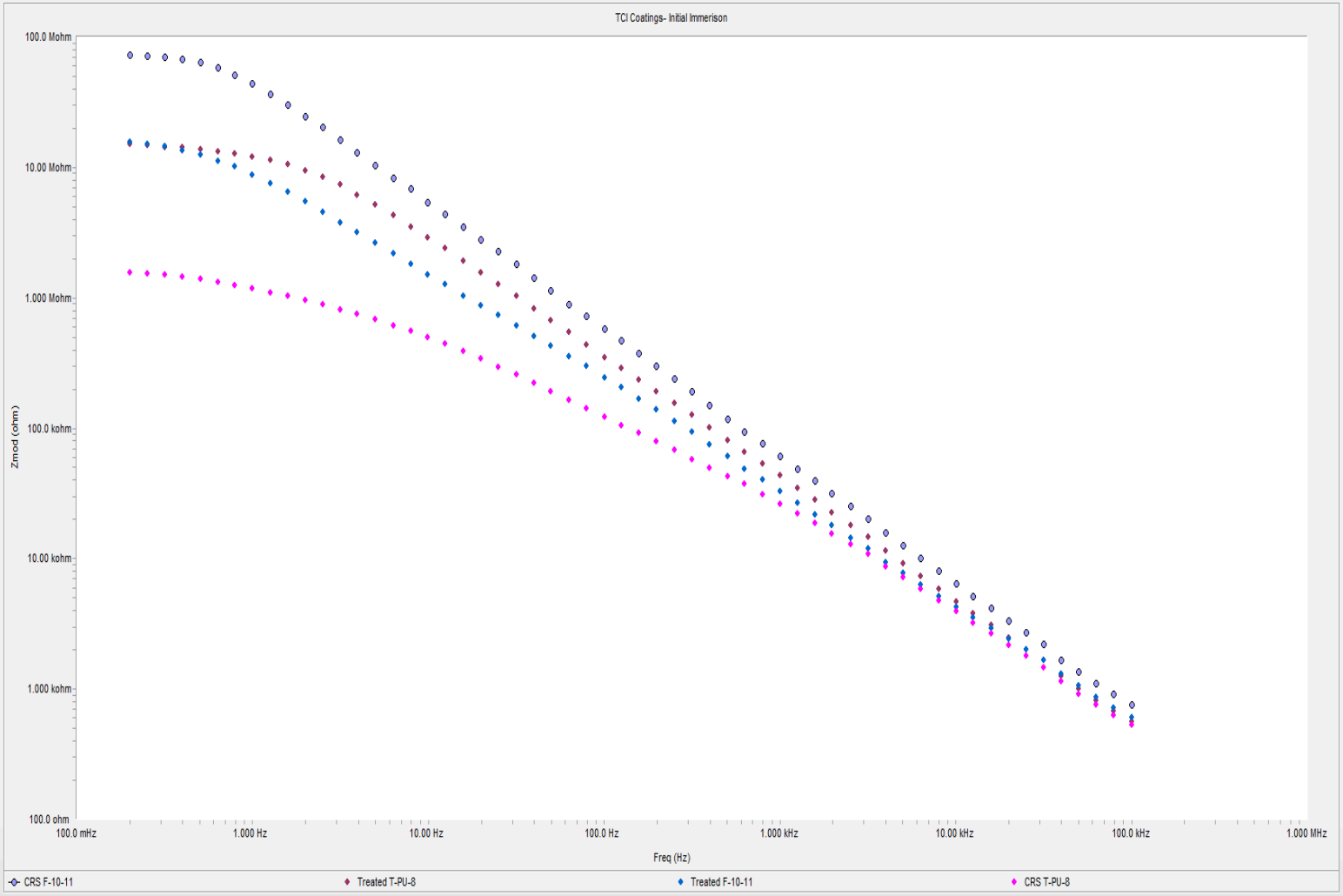


Bode Phase & Magnitude Plot



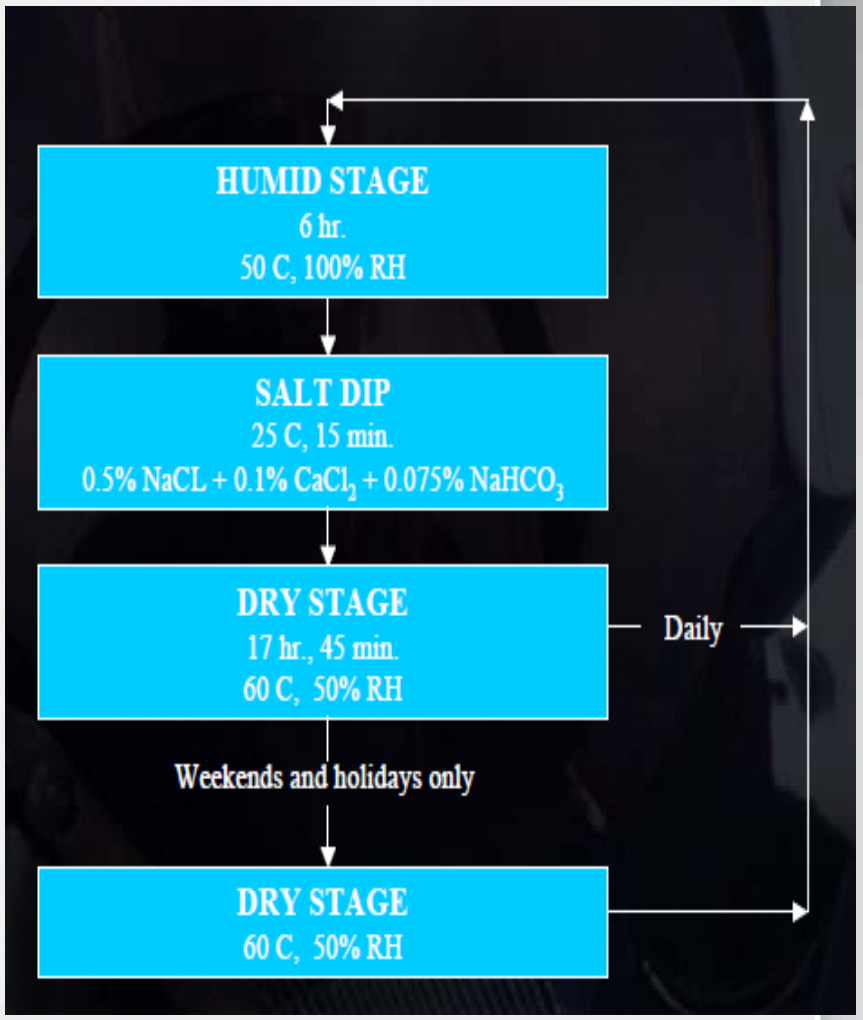
Result Plots

Bode Plot



SAE J2334

- 1 test cycle is 24 hrs
- Three stage test
- 6 hours exposure to a water fog/condensing humidity climate of 100% RH at 50°C.
- 15 minutes direct spray salt solution at ambient temperature.
- 17 hours 45 minutes of air drying in a climate of 50% RH at 60°C



http://www.ascott-analytical.com/SAEJ2334/saej2334testconditions.htm and
http://www.leymanlift.com/images/Army_Corrosion_Summit_SAEJ2334.pdf

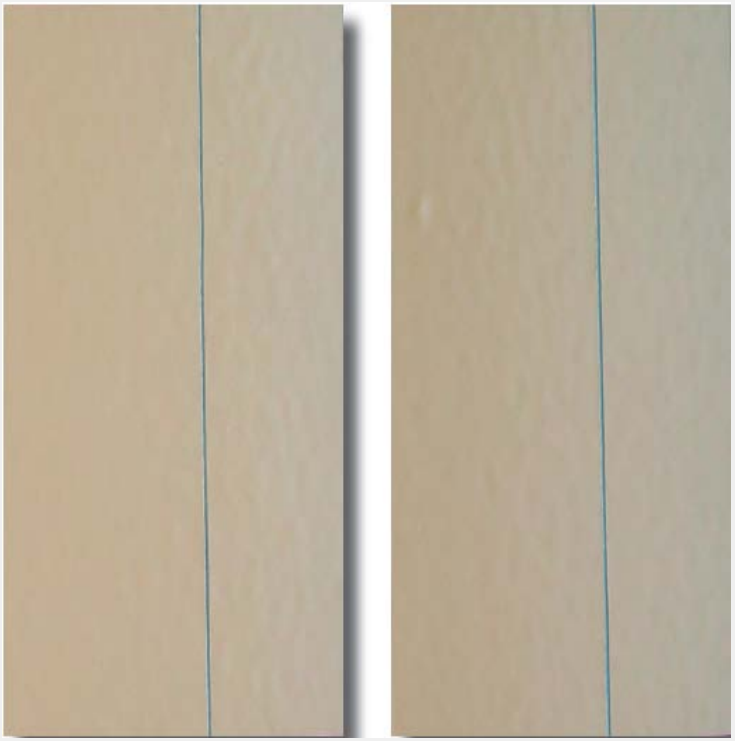
CASS (ASTM B368)

- CASS = 5% NaCl + 0.25 g $CuCl_2 \cdot H_2O$ acidified with acetic acid
- Useful in the testing of anodized, chromated or phosphated aluminum, but is primarily used for the rapid testing of chromium plating on steel and zinc die-castings
- The solution is then adjusted to a pH range of 3.1 to 3.3 by adding acetic acid, and the temperature of the salt spray chamber is controlled

Cyclic Weathering(ASTM D5894-5)

- Cyclic corrosion test consisting of one week in QUV and one week in Prohesion
- Cyclic, panels exposure to wet/dry periods
- UV exposure
- Correlates more to real world exposure

Cyclic Weathering Failure



Initial

5 Cycles – 1680 Hours

Corrosion Tests

Perfect Correlation $C+R^2 = 2.00$

Test	Duration	C	R^2	$C+R^2$
SAE J2334	80 cycles	0.97	0.96	1.93
Acid Rain CCT	45 cycles	0.97	0.78	1.75
CCT-IV	35 cycles	0.86	0.74	1.6
GM9540P(B) (GM)	50 cycles	0.59	0.84	1.43
JASO M610	45 cycles	0.98	0.44	1.42
AISI-A	50 cycles	0.75	0.46	1.21
AISI-C	50 cycles	0.41	0.74	1.15
Michigan Suburban	24 months	0.51	0.61	1.12
GM9540P(B) (ACT)	50 cycles	0.67	0.44	1.11
B117 Salt Spray	4 weeks	0.05	0.19	0.24
QUV-Prohesion	12 weeks	0.2	0.62	0.82

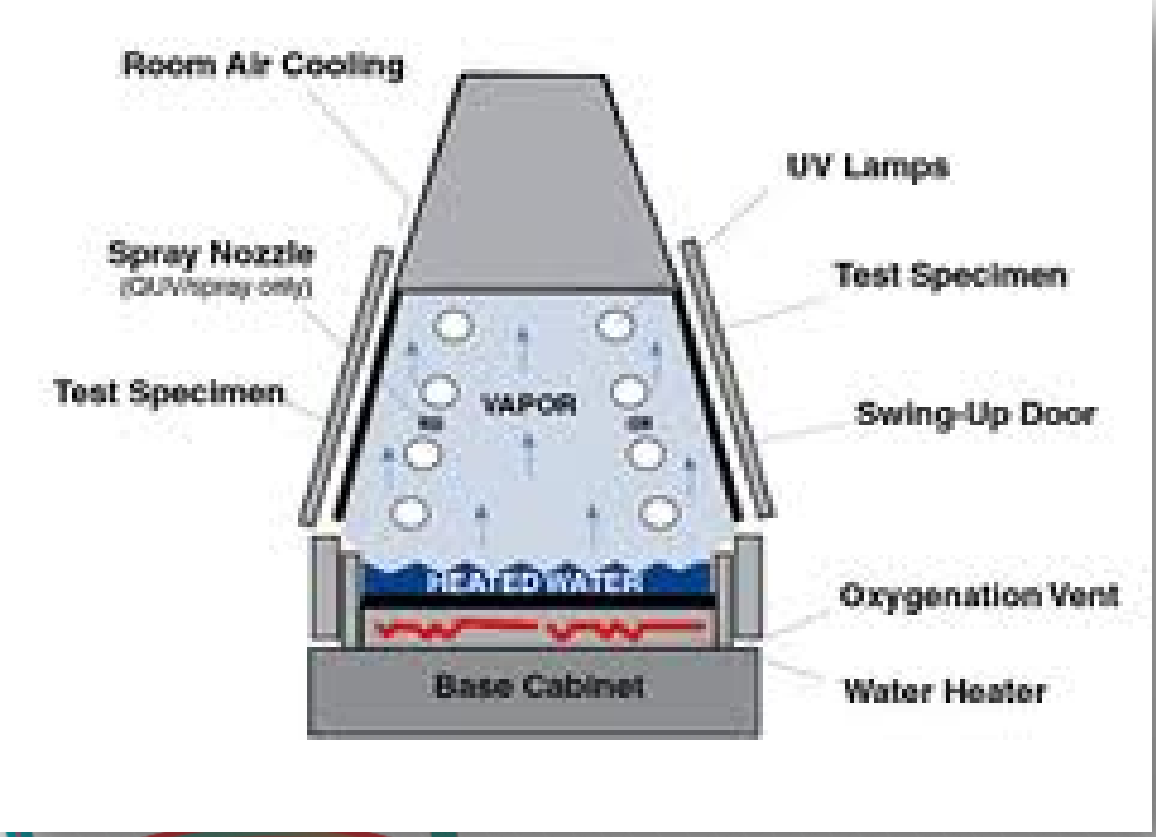
Reference: John Repp – Corrpro Companies, Inc - US Army Corrosion Summit 2002
Correlation Coefficient, C and r^2 Scribe creep compared for 5-yr exterior exposure data

QUV Test Method

- Cyclic ultraviolet weathering introduced in 1969 by the Q-Panel Company.
- 40-W fluorescent lamps provide UV sours with an emission range of 280 to 350 nm, with continuous peak output at 310 nm.
- UV radiation emulates the intensity of mid-day June sunlight in Florida.
- Oxygenated water is applied to the test samples by condensation or spray. The water supply is heated in a vented tank below the test racks and lamps.
- A timer alternates the UV and the condensation at 43°C followed by 8 hours UV radiation at 60°C and high humidity

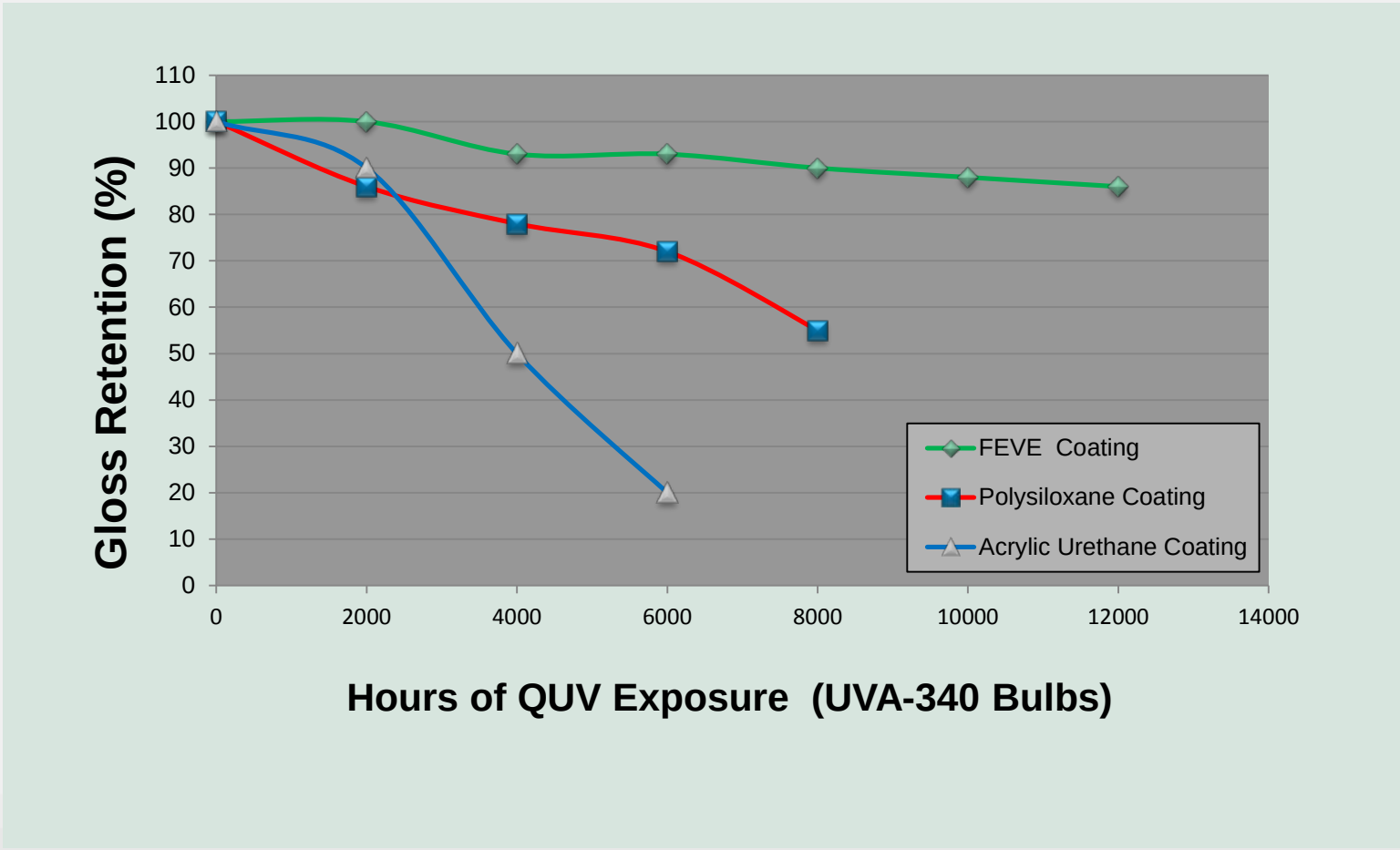
QUV-A Test (ASTM D4587)

- Exposes coated steel panels to a ultraviolet light at a constant wavelength of 340 nm
- Gloss retention is measured for 5,000 hours



QUV-A Test (ASTM D4587)

- Gloss retention is measured



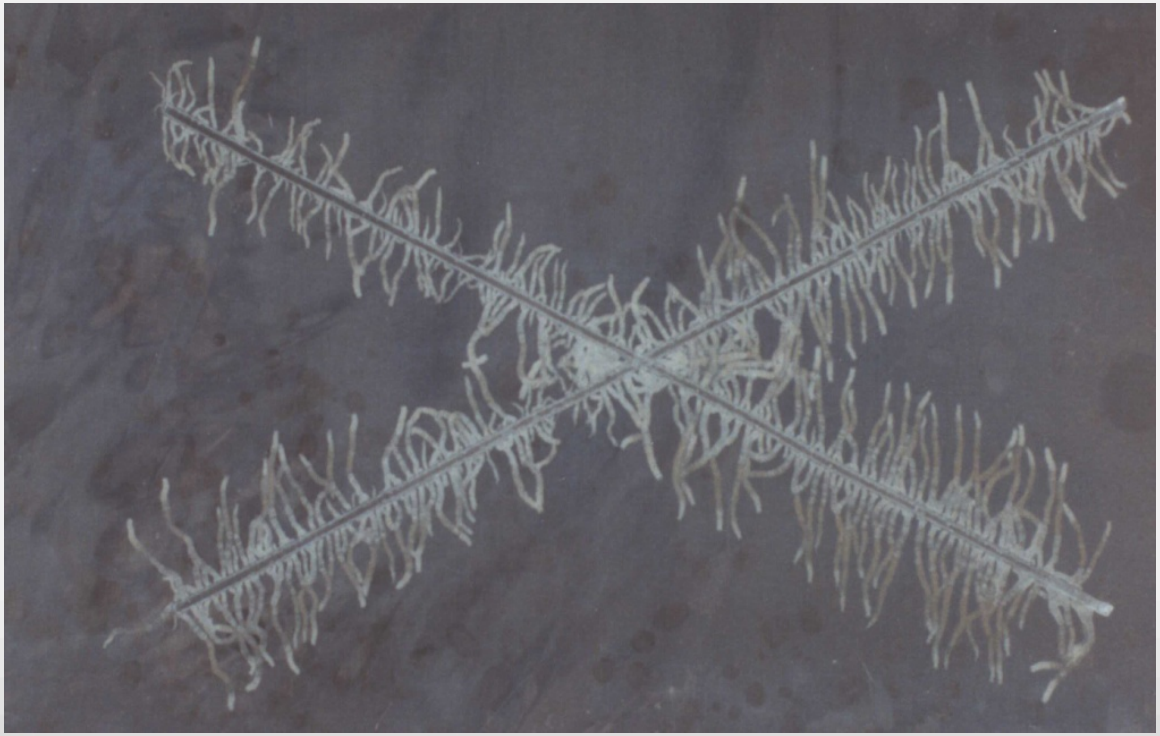
Data courtesy of: Bob Parker, AGC Chemicals Americas, Inc.

Filiform Corrosion (ASTM D2803)

- Filiform Corrosion is a unique differential oxygen cell occurring on coated substrates. FFC leads to development of thin thread-like filaments full of corrosion products beneath the coating.
- It occurs on Aluminum, Steel, Magnesium and other metals. Filaments grow by anodic propagation or undermining.
- De-lamination: corrosion products push up coating causing it to delaminate. The head is anodic and tail end tail cathodic
- What conditions cause it to occur?

Filiform Corrosion

- Scribed panels placed in corrosive atmosphere (salt spray ASTM B117 for 4 to 24 hours) or immersed in a NaCl solution
- Panels exposed to humidity (77^{o}F & 85% RH)



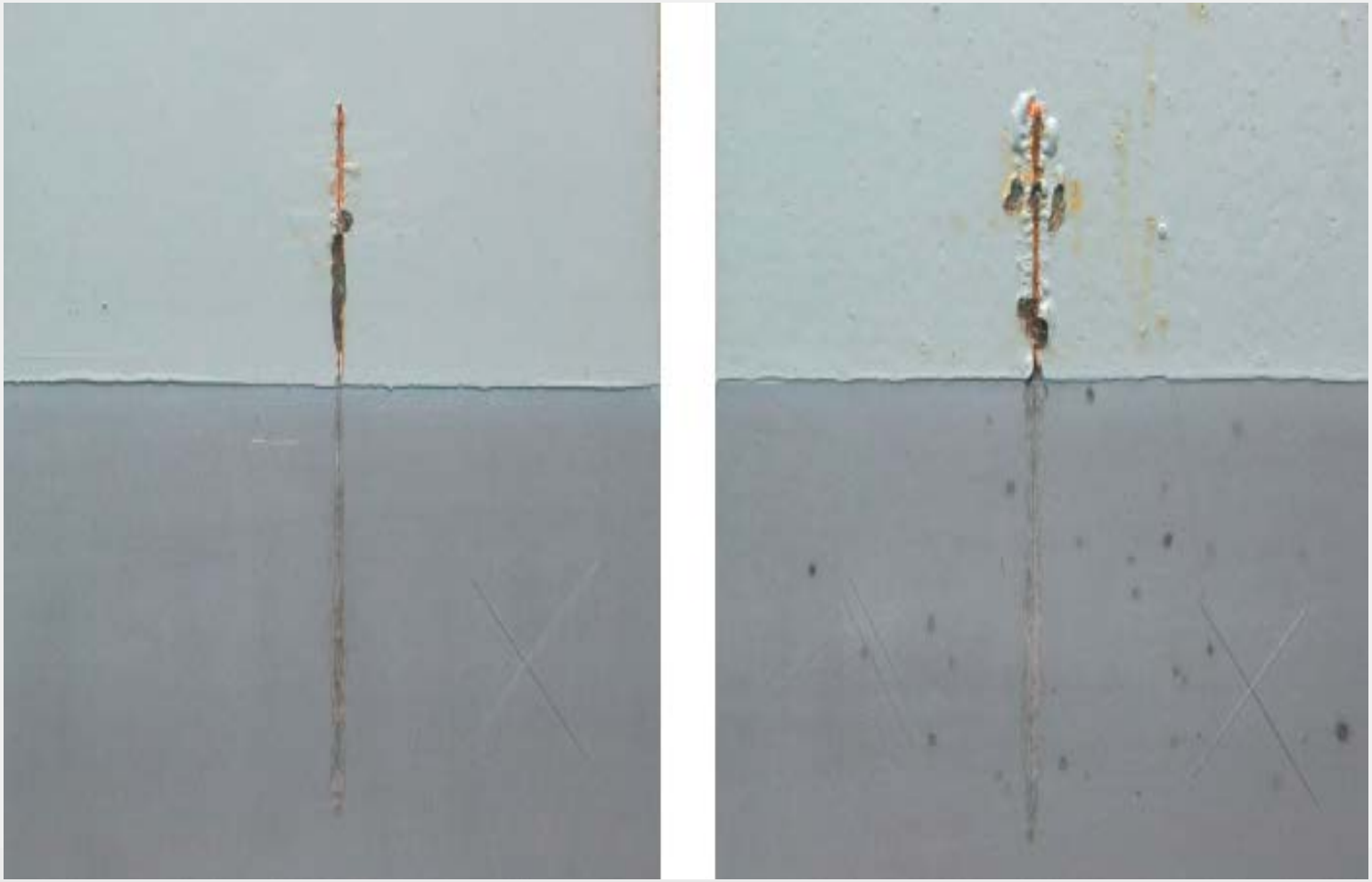
QUV (ASTM G154)

- QUV cycle – UV light –4 hr followed by condensation cycle – 4 hr
- Fluorescent UV lamps
  - **UVA-340**
  - **UVB-313**
- Condensation Cycle – chamber maintains 100% RH, 50°C

Prohesion Cycle (ASTM G85 A5)

- Prohesion Cycle –Samples exposed to an electrolyte solution (0.05% NaCl + 0.35% ammonium sulfate) at 35°C for one hour then dried at 40°C for one hour, the cycle repeats

Prohesion Failure



1080 Hours of Prohesion

Xenon Arc (ASTM D2568, G26)

- Simulates full spectrum solar radiation-UV, visible, and infrared
- Xenon arc spectrum must be filtered
  - **Daylight**
  - **Window Glass**
  - **Extended UV**
- **Water spray**
- **Humidity controlled**

Photo courtesy of Q-Lab Corporation

Exterior Exposure

- Location – Worst Case Environments : South Florida, and Arizona
- Type of test rack (unbacked, backed, and under glass
- Orientation of test sample (examples 90^{o} South, 45^{o} South)
- Natural Marine Atmospheric Conditions – very aggressive environments. Example: Kure Beach N.C. (increases in temperature, chloride content, moisture, wind).

EMMAQUA Test (ASTM G90)

- Uses 10 mirrors to focus natural light onto coated panels, exposing the panel to all the wavelengths found in natural light (intensity of ~ 8 suns)
- Panels are sprayed periodically with water to simulate rain
- Results reported in units of energy exposure per unit area (MJ/m^2) and percentage of gloss retention
- Correlates to subtropical S. Florida & Arizona

EMMAQUA®: Equatorial Mount with Mirrors for Acceleration with Water

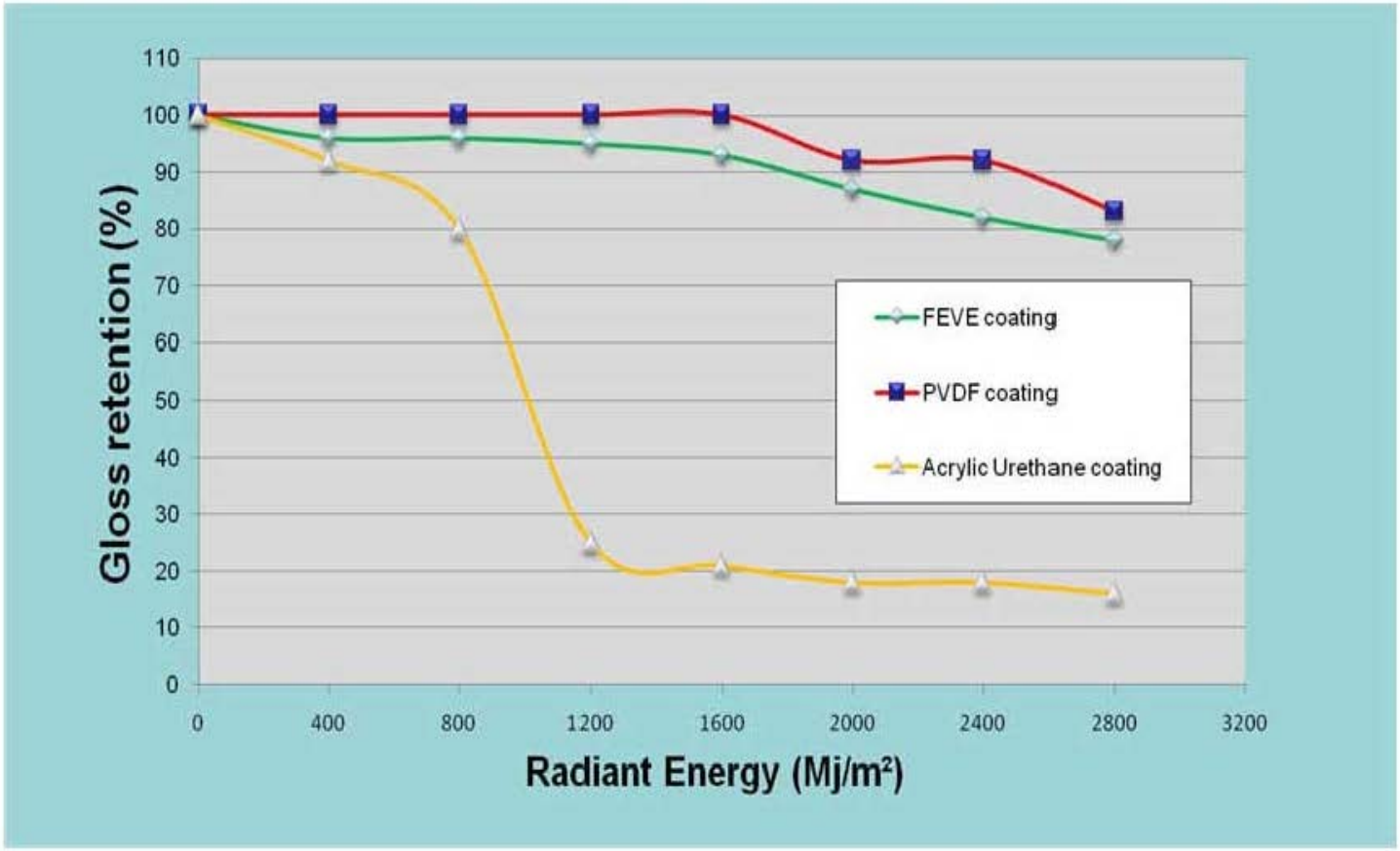
EMMAQUA

- 1,000 MJ/m^2 corresponds to 10 yrs of exposure
- Check gloss retention after exposure

Data courtesy of: Bob Parker, AGC Chemicals Americas, Inc.

EMMAQUA

EMMAQUA Testing (Outside Test Fence – Arizona)



Data courtesy of: Bob Parker, AGC Chemicals Americas, Inc.

South Florida Test

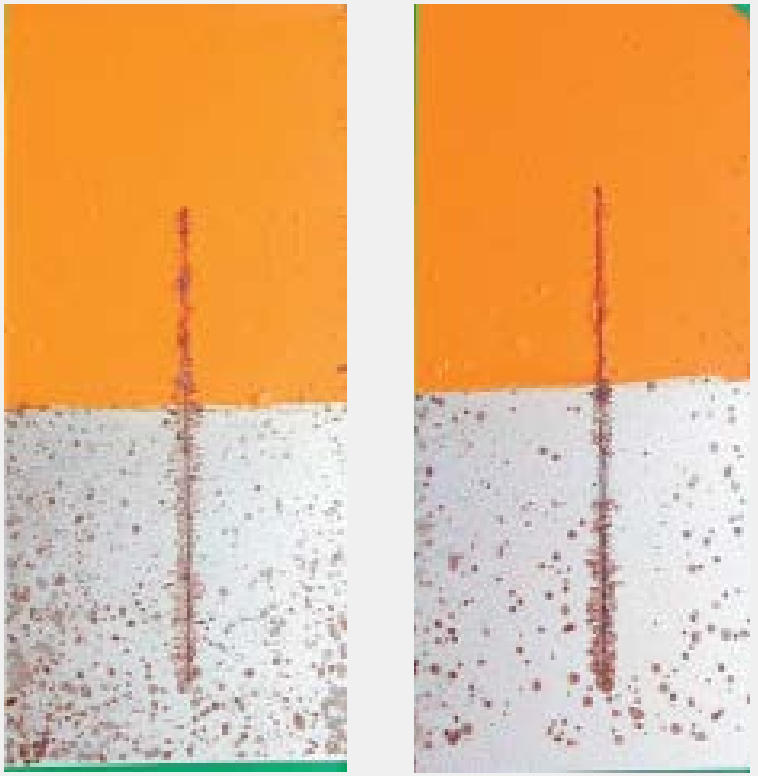
- Outdoor coated panel testing farm
- Harsh environment consisting of:
  - **High wavelengths of ultraviolet light**
  - **Marine atmosphere**
  - **Heavy rain**

Data courtesy of: Bob Parker, AGC Chemicals Americas, Inc.

Test Comparisons

Salt Spray vs. QUV/Prohesion

336 Hours Salt Spray



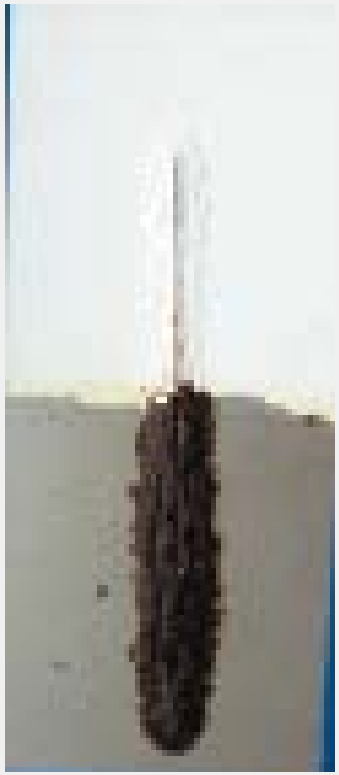
2 Cycles QUV/Prohesion

Waterborne Acrylic – CRS- 2.25 mils DFT

Test Comparisons

Salt Spray vs. QUV/Prohesion

500 Hours Salt Spray

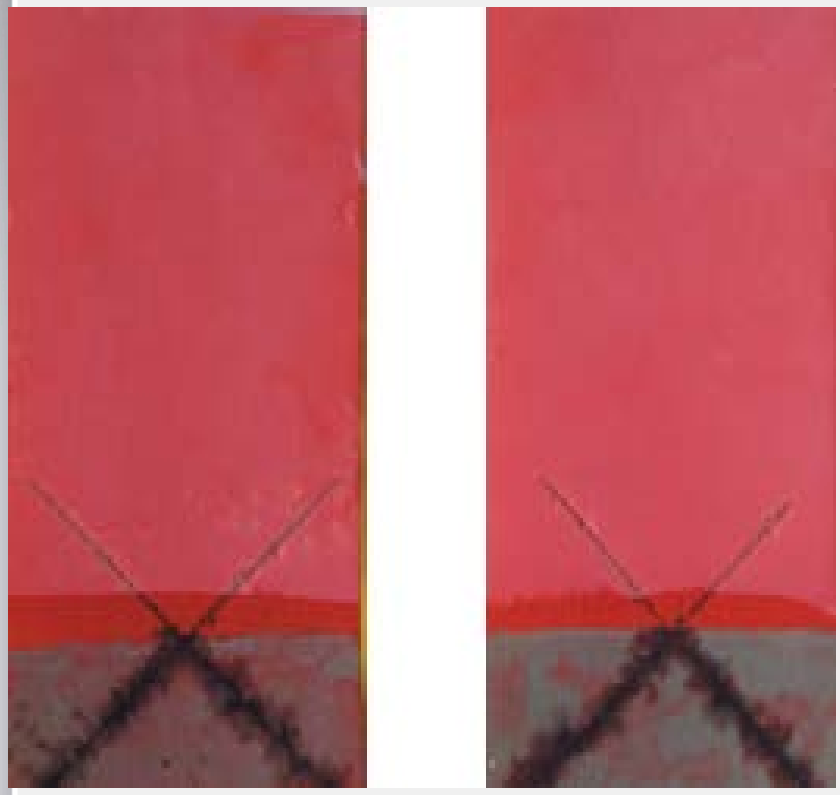


2 Cycles QUV/Prohesion

2K Water Based Polyurethane– CRS- 2.25 mils DFT

Laboratory vs. Real World Exposure

Exterior Exposure vs. Salt Spray

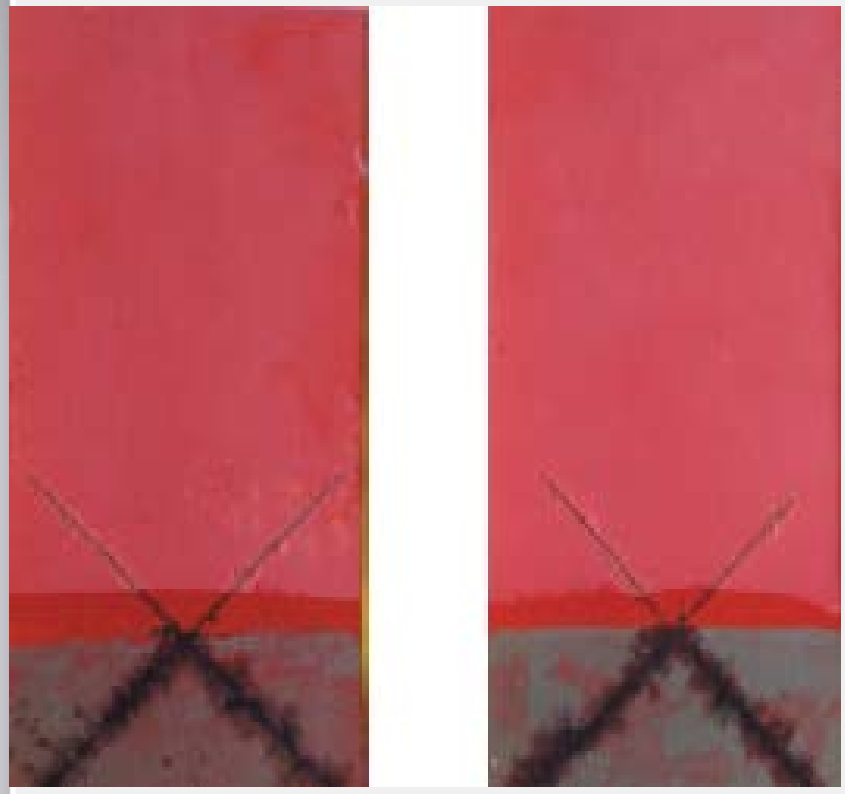


2.5yrs Industrial Site

500 Hours Salt Spray

Test Comparisons

Prohesion vs. Industrial Exposure



2.5yrs Industrial Site

500 Hours

1600 Hours

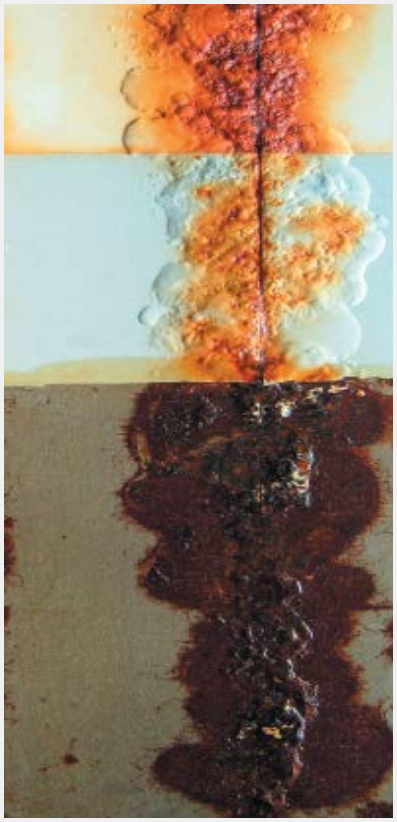
Prohesion

Comparison of Corrosion

Solvent Based Polyurethane – CRS- 2.0 mils D.F.T

2K Hrs SS

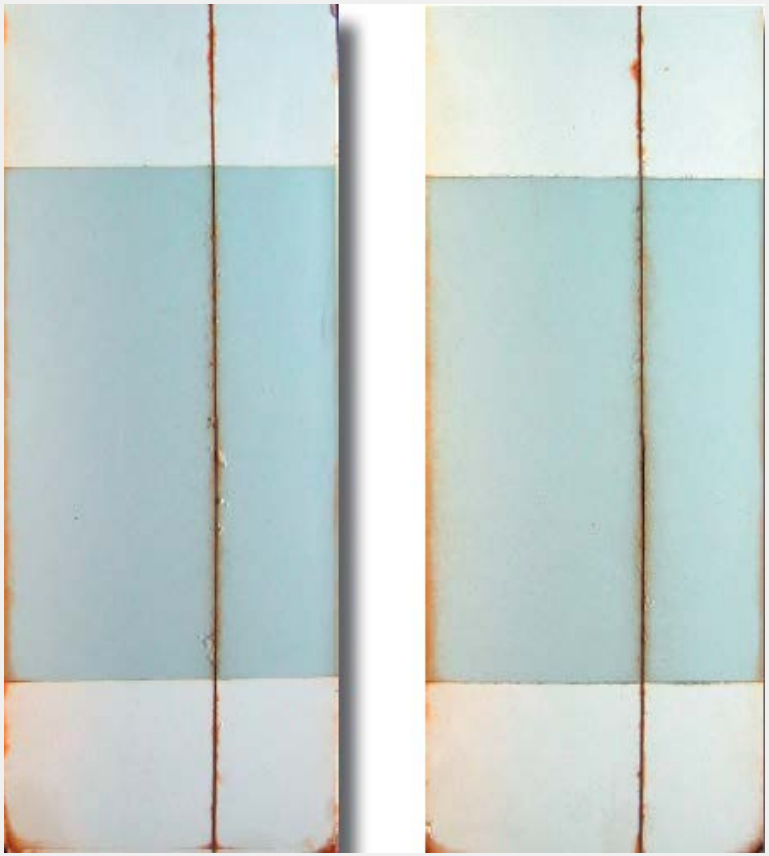
2K Hrs Prohesion



2 Years
Ocean City, FL 45°S

Industrial vs. Marine Environment

3yrs Exposure – CRS- 2.0 mils D.F.T

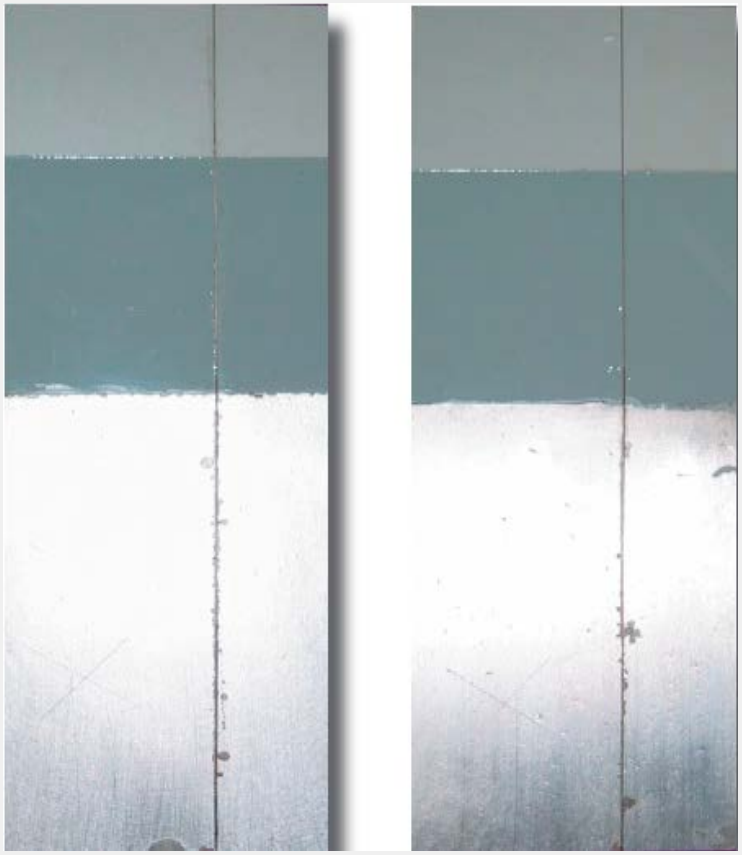


Industrial Site 45º South

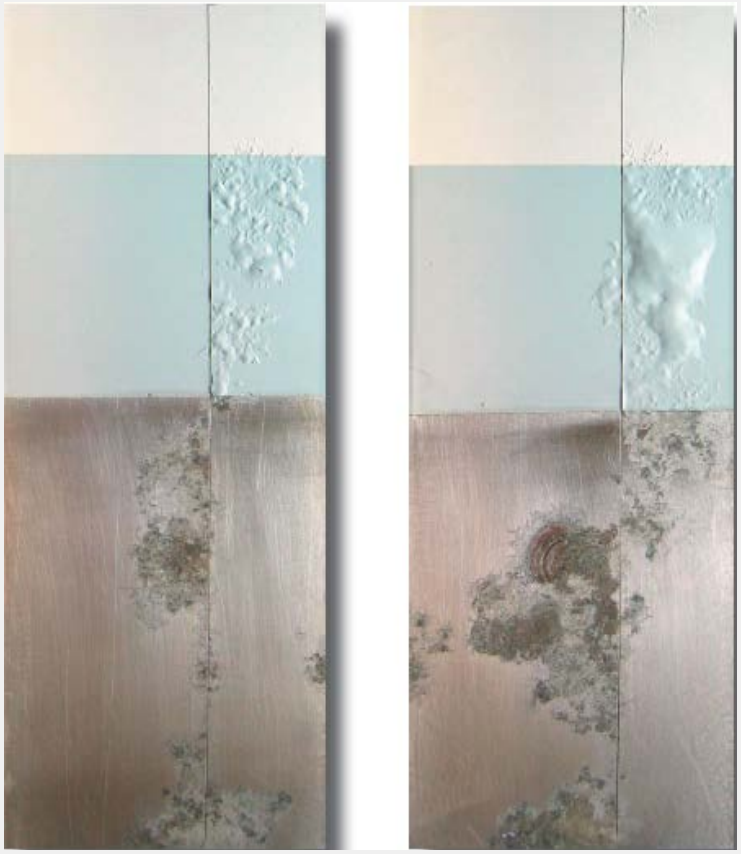
Ocean City, Florida 45ºSouth

Salt Spray vs. Marine Exposure

Solvent Based 2K Polyurethane –Aluminum – 2.0 mils D.F.T



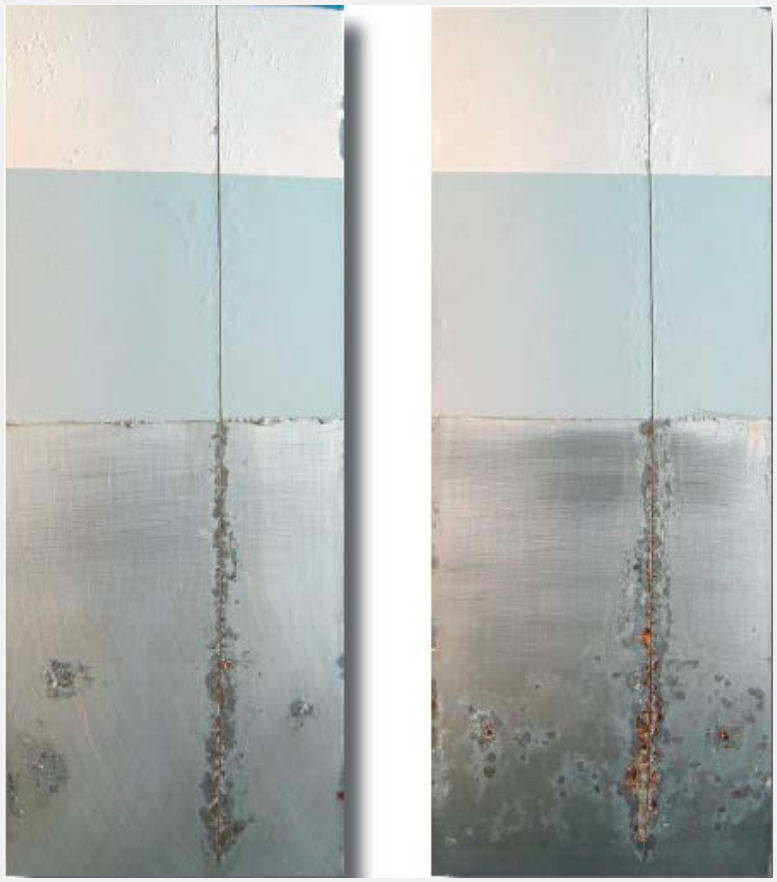
2K Hours Salt Spray



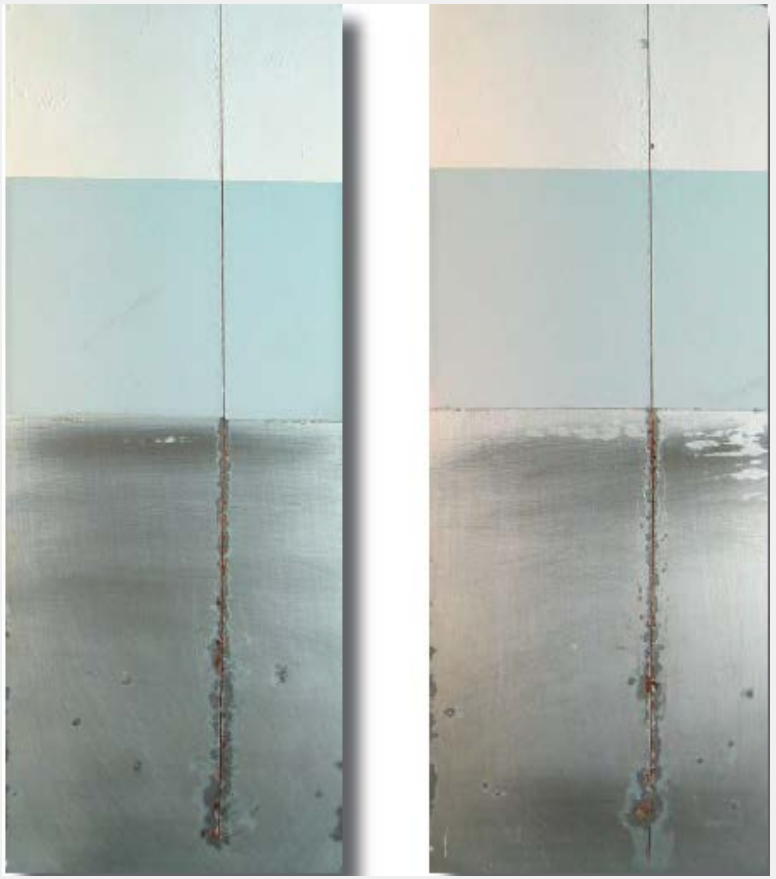
3 Years Ocean City, FL

Angle of Orientation Comparison

Ocean City, FL – 3yrs Exposure – Galvanized Steel



45° SOUTH



30° EAST

Accelerated Testing vs. Exposure?

- Predicting the unpredictable- the actual weather
- Multiple weather factors influence the life of the coating
- Laboratory tests are more controlled
- Sample size

Standard Tests

- Evaluation of painted or coated samples subjected to corrosive environment (ASTM D1654)
- Solvent Resistance (ASTM D5402)
- Gloss Retention (ASTM D523)
- Coating Hardness (ASTM D4366 and D2794)
- Adhesion (ASTM D3359)
- Water Resistance using Immersion (ASTM D870)
- Water Resistance using Condensation (ASTM D4585)

Evaluation of Scribed Panels

- ASTM D1654 Procedure A - Evaluation of Scribed Panels
  - Air Blow off
  - Scraping
  - Rating

Rating the Scribe

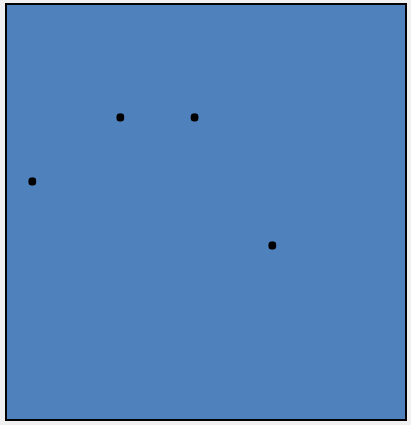
- Corrosion or loss of paint is measured at the scribe
- The measurement either in millimeters or inches will receive a rating number
- Rating numbers range from 10 to 0
- Example – no corrosion at scribe = 10
- 16.0 or more millimeters of corrosion or 5/8 inch or more = 0

Evaluation of Field Area

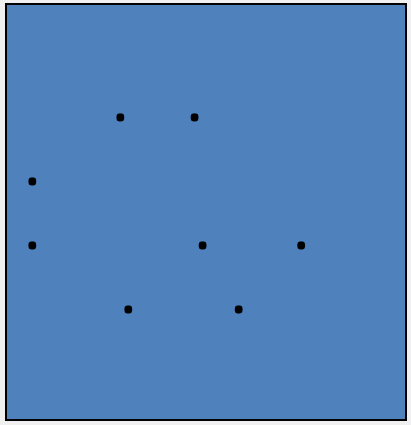
- Percentage of field failure will correspond to a rating number.
- Numbers range from 10 to 0
- Example – no corrosion in field area =10
- over 75% of panel corroded =0

Evaluation of Panel Rusting

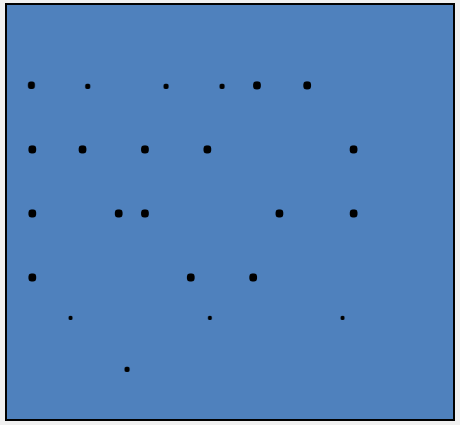
- ASTM D610-85
- Using standard pictures of panel rust to compare to the test panel to determine the degree of rusting



9 (0.03%)



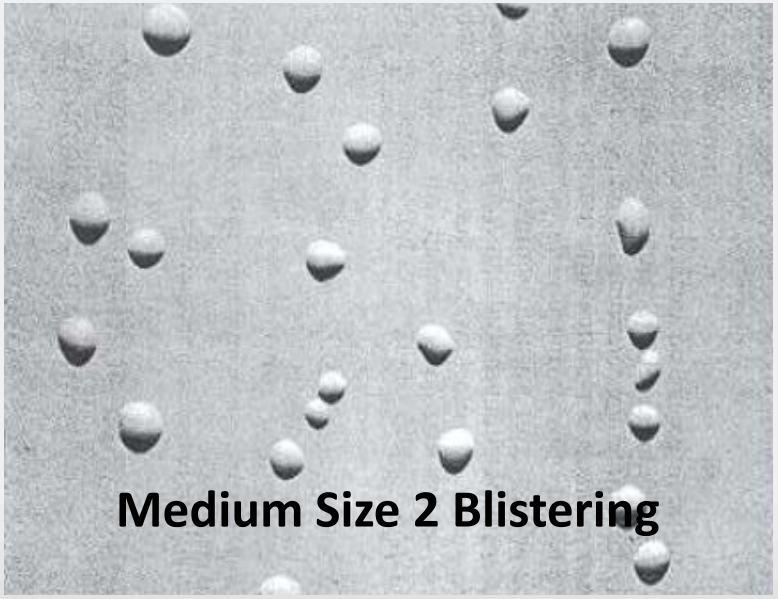
8 (0.1%)



7 (0.3%)

Determining Blistering

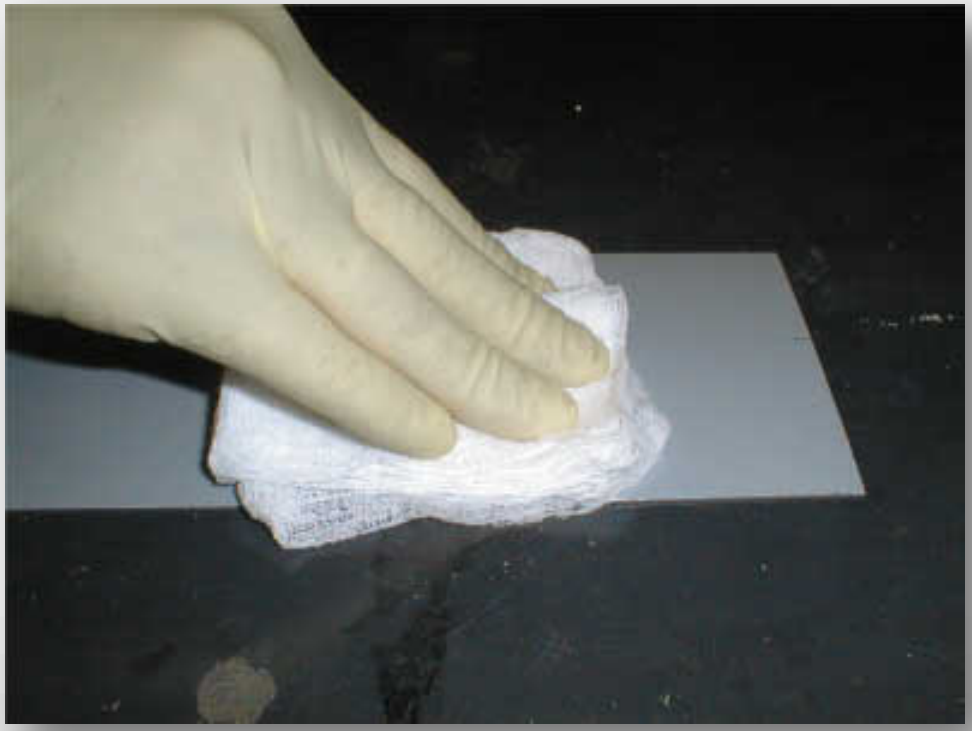
- ASTM D714-87 - Pictures of blistering are used to compare test panels to determine the size of blistering and the frequency
- Blister size range from 10 to 2
- Frequency of blistering from “few” to “dense”



Medium Size 2 Blistering

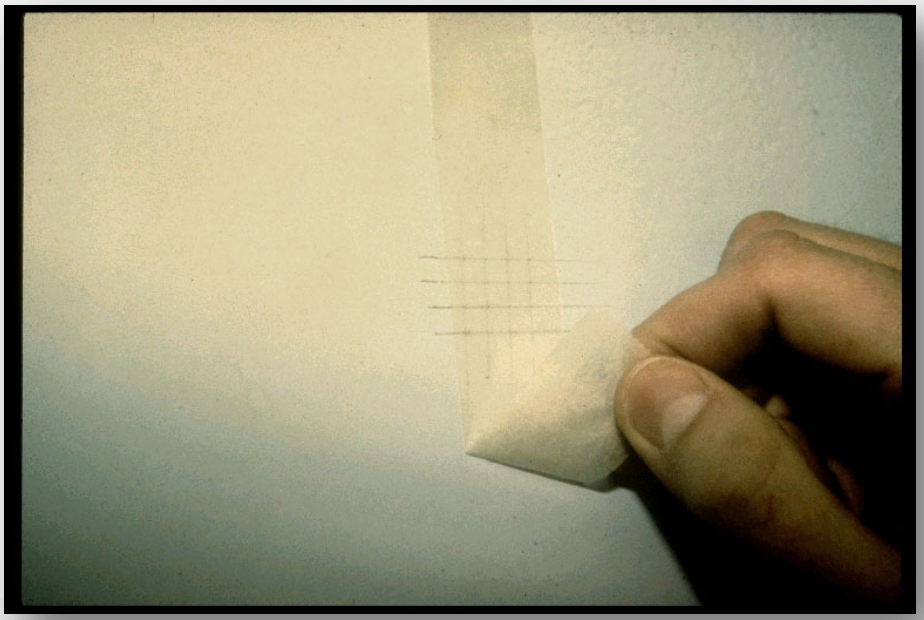
Solvent Resistance ASTM D5402

- A solvent rub technique for assessing the solvent resistance of an organic coating that chemically changes during the curing process
- 100 MEK Solvent Double Rubs



Cross Hatch Adhesion ASTM D3359

- Covers procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film



Classification	Surface of Cross-Cut Area From Which Flaking Has Occurred
5B	None
4B	(1% To 5%)
3B	(6% To 15%)
2B	(16% To 35%)
1B	(36% To 65%)
0B	Greater Than 65%

Summary of Test Methods

Test	Method
Filiform Corrosion Test	**D2803**
Salt Spray	**B117**
QUV	**G154**
Prohesion	**G85-A5**
QUV/Prohesion	**D5894**
Immersion	**D870**
Xenon Arc	**D2568, G26**
Humidity	**D2247**
EIS	**G106**

Summary of Test Methods

Test	Method
Cosmetic Corrosion Lab Test	**SAE J2334**
SCAB Corrosion	**GM9511P**
CASS	**B368**
QUV-A	**D4587/4329**
Solvent Resistance	**D5402**
Adhesion	**D3359**
Pendulum Hardness	**ISO 1522**
Impact	**D2794**
EMMAQUA	**G90**

So many tests…..

- Which test is the best one?
- The more accelerated tests, the better
- Cyclic testing more indicative of “real world”
- There is little correlation of test results between lab test methods and natural conditions
- Natural weathering is not a controlled process
- Lab tests are simply an effort to incorporate natural stresses (temp, time, humidity, UV, salts) into a controlled environment

Acknowledgements

- S. Koka, A. Shi and J.S. Ullett, S & K Technologies, Dayton, OH
- GalvInfo Center, GalvInfoNote 15, The Salt Spray Test, www.Galvinfo.com
- Atlas Testing Solutions, Ch.10 Weathering Test Methods, http://www.atlas-mts.com/en/client_education/weathering_library/weathering_test_methods/index.shtml
- Douglas M. Grossman, The Right Choice – UV Fluorescent Testing or Xenon Arc Testing? PCI Paint and Coatings Industry March 10, 2006
- Gamry Instruments www.gamry.com

THANK YOU