Laboratory Corrosion Testing: Realism and Reproducibility with Modern Methods

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View Recorded Presentation





Q-Lab's 2023 Webinar Series

- This is the final webinar in our 2023 series
- Thanks to everyone who attended these sessions!
- All the content can be accessed at: <u>q-lab.com/webinars</u>

Title	Grouping		
Outdoor Testing	ab		
Accelerated Outdoor Weathering Testing	Outdoor		
Optimizing your weathering program			
QUV Operator Training - with live content	Operator training		
Q-SUN Operator Training - with live content			
Q-FOG Operator Training - with live content			
How to Run ASTM B117			
Evaluations in weathering testing	Practical / Educational		
MAPS Ask Me Anything			
Essentials of Lab Weathering	E un de se cuntede		
Modern Corrosion	Fundamentals		

Administrative Notes

You'll receive a follow-up email from info@email.q-lab.com with links to take a survey and download the presentation content

Use the **Q&A feature in Zoom** to ask us questions today!



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Thank you for attending our webinar!

We hope you found our webinar on *Essentials of Laboratory Weathering* to be helpful and insightful. The link below will give you access to the slides and recorded webinar.





- Types of Accelerated tests
- Continuous Salt Spray (Neutral & Acidified)
- Wet/Dry Cyclic Tests
- First-Generation Cyclic Automotive Tests
- Modern Corrosion Test Methods
- Verifying Corrosion Test Performance



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Types of Accelerated Tests

Accelerated Test Type	Result 🔍 🤇	Test Time	Results compared to
Quality Control	Pass / fail	 Defined Short	Material specification
Qualification / validation	Pass / fail	DefinedMedium-long	Reference material or specification
Correlative	Rank-ordered data	 Open-ended Medium	Natural exposure (Benchmark site)
Predictive	Service life Acceleration factor	 Open-ended Long	Natural exposure (Service environment)







Continuous Salt Spray ASTM B117

- 5% NaCl salt fog at 35°C
- Neutral pH
- Fine mist (atomized with compressed air) sprayed indirectly onto specimens
- ISO 9227 contains the same test
- When correctly followed, test has reasonable repeatability and reproducibility



- Not a good simulation of most service environments
- Typically produces different corrosion products than natural exposure
- Poor rank order correlation with outdoor corrosion
- A good quality control/screening test, usually not much more





Heater Configurations





* Rapid Ramp Heaters required to meet some fast temperature transition times





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- American Architectural Manufacturers Association recently replaced ASTM B117 with this test in AAMA 2605, "Superior" coatings on aluminum
- Dilute NaCl, (NH₄)₂SO₄
- Development began in England, 1960's
- Alternating spray and dry-off
- Prohesion (Protection is Adhesion)

Wet/Dry Cyclic Tests



Combined Corrosion/Weathering

- As a coating degrades from UV exposure, its ability to protect against corrosion is reduced
- Sherwin Williams developed a UV + Corrosion combined cycle in the 1980's to test this





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Combined Corrosion/Weathering vs Outdoors

QUV + Q-FOG ASTM D5894 2000 hours







Latex

Outdoor 27 months, marine environment









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Wet/Dry Cyclic Test Case Study SSPC (Society for Protective Coatings)

- 15 different systems included
- Outdoor testing (31 months)
- Accelerated tests (2000 hours)
 - Salt spray 5%
 - Prohesion
 - 2 types of cyclic immersion
 - Combined corrosion/ weathering



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SSPC Test Results

	Laboratory Test Method	Correlation w/Severe Marine Environment
	Conventional Salt Spray	-0.11
Q-La	Prohesion O-Lab	© 0.07
	Cyclic Immersion Procedures	0.48
	Cyclic Immersion with UV Procedure	0.61
	Combined Corrosion/ Weathering	0.71

Good correlation from combined test!

Combined Corrosion and Weathering ISO 12944-6 (and -9)



- 4 hours UVA-340, 0.83 W/m²/nm at 340 nm, 60 °C
- 4 hours dark condensation, 50 °C
- 72 hours _ 20



- Continuous salt fog at 35°C
- Rinse panels and put in a freezer for 24 hours
- 72 hours



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Wet/Dry Cyclic Test Limitations

- Poor repeatability and reproducibility
- Poor correlation in some cases
 - Automotive
 - Industrial maintenance coatings on steel
- Attempts to improve correlation & repeatability include...
 - Water retained at chamber bottom
 - Changing temperature of bubble tower
 - Introducing **humid** phases ...



First-Generation Cyclic Automotive Tests Salt Fog \rightarrow Dry-Off \rightarrow Wetting (Humid)

Wetting specimens after dry-off reinitializes corrosion





First-Gen Cyclic Automotive Tests Salt Fog \rightarrow Dry-Off \rightarrow Wetting (Humid)

- **Electrolyte**: NaCl, CaCl₂, others to simulate road salts
- Solution: applied by either direct Spray or Fog
- Cycle: Salt spray applied intermittently in "ambient" conditions
- Verification: Use of corrosion coupons to minimize test variability

First-Gen Cyclic Corrosion test



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Poor Repeatability and Reproducibility!

- Problems observed
 - Different corrosion chambers produced different results

Limitations of First Generation CCT

- Corrosion rates varied among metals from test to test
- Reasons / Areas for Improvement
 - Only full wetting, dry, uncontrolled room/ambient conditions possible
 - No control of RH transition times
 - Variable specimen dry-off rates
 - No RH values in critical transition zones (DRH)
 - Slow application of salt solution from fog
 - Little time for dry-off and re-wetting of specimens

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Deliquescence of salts

Galvanic corrosion Influence of relative humidity

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Automotive Tests & Road Salt

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- Salts **deliquesce** they absorb moisture from the atmosphere until they dissolve and form a solution.
- All soluble salts will liquefy for RH values <100%
- This leads to increased time of wetness and increased corrosion

Deliquescence Relative Humidity (DRH)

Salt	DRH
Potassium Chloride (KCl)	85%
Ammonium Sulfate (NH ₄) ₂ SO ₄	81%
Sodium Chloride (NaCl)	O76%
Sodium Nitrate (NaNO ₃)	74%
Magnesium Chloride (MgCl ₂)	33%
Calcium Chloride (CaCl ₂)	31%

if the environment is above this RH, a liquid salt solution will form

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Galvanic Corrosion



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Relative Humidity & Corrosion

- Corrosion accelerates once it starts
 - Formation of complex oxides
 - Wet time increases as new oxides form
- Salts deliquesce at different RH values
- Formation of liquid solutions affects corrosion by creating a galvanic couple



Relative Humidity and Galvanic Corrosion

Condition	RH Range	Result	
Dry	≤ 50%	Very little corrosion from NaCl	
Electrolytic cells near salt crystals; film formation as RH increases	50-76%	 Corrosion of steel and aluminum AL-Steel galvanic couple broken 	
Uniform Electrolytic Film formation	© Q ≥76%	 Maximum cathode area for steel Deeper non-uniform corrosion Al corrosion in galvanic couple with steel 	

Galvanic Corrosion

- More **anodic** (negative electrochemical potential) metal corrodes (Al, Zn, steel)
- More cathodic metal (stainless, Au, Ag) protected
- Metals must be in **electrolytic** contact







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Relative Humidity and Corrosion

Controlling Step Transition Times

- "Linear" transition
 - Specify Time in test cycle to change test conditions
 - Tester adjusts temperature & RH for linear transition from ramp start to end
- "Less Than" or "Auto" transition
 - Specify Time in test cycle to change test conditions
 - Tester attempts to achieve conditions as fast as possible
 - Used for transition times like JASO M609) designed to minimize test variability...

Fast Transition Times: JASO M609



Uncontrolled Transition Times: SAE J2334

- Transition times are not specified in this standard
- Coupon use is encouraged but no mass loss limits are included
- Some companies have implemented SAE J2334 with their own mass loss limits







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Cosmetic Corrosion LabTest Cycles SAE J2334 - 5 Day/Week - Manual Operation Cosmetic Corrosion LabTest Cycles SAE J2334 - 7 Day/Week - Automatic Operation



Repeat Daily



Slow Dry-off



During the transition, the time above the Deliquescence RH of NaCl is about **1 hour**



Rapid Dry-off

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During the transition the time above the Deliquescence RH of NaCl is about **10 minutes**

SAE J2334 Results



- Green bars represent test under slow dry-off conditions: panels fail
- Blue bars represent test under quick dry-off conditions: panels pass
- Red lines represent tolerance of OEM standard

Under the rapid drying test, the coated panels once again passed the test!

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Poor Repeatability and Reproducibility!

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Faster application of salt solution than Fog



Shower Configurations





Center-Mounted Stationary





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Modern Automotive Corrosion Tests

- FogToyota TSH1555G
- VDA 233-102
- Renault D17 2028 (ECC1)

- GMW 14872
- Volvo ACT 1
- ISO 16701
- Volvo ACT 2/ Ford L-467

No one "right way" to run a test but shower/spray has gained popularity

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Modern Corrosion Tests

Air Preconditioner



- Accurate control of "ambient" conditions
- Accurate ramping of temperature & relative humidity



RH Control with Air Preconditioner



Q-FOG Operational Range: Well-Controlled Lab



Q-FOG Operational Range: Hot, Humid Lab



Modern Corrosion Test Examples

Ford L-467 / Volvo VCS 1027, 1449



Time (hours)



ISO 16701



GMW 14872



Time (hours)

Verifying Corrosion Test Performance

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Corrosion (Mass-Loss) Coupons

- Standardized metal specimens
- Mass loss due to corrosion is measured during a test
- Used by GM, VDA, ISO 16701 standards, and many others
- GMW 14872 requires a specific rate of mass loss throughout a test
- Ensures corrosion chamber is maintaining proper conditions and operator is running the test correctly







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Independent Verification



- Accurate and Precise Temperature/RH Sensor placed in center of chamber to independently verify Q-FOG CRH controller reading
- Chamber is full of steel
 panels

Verify Test in a Full Chamber

- To confirm a chamber is able to satisfy test requirements, validation should be conducted in a full chamber
- Additional thermal mass of a fully-loaded chamber with metal panels will delay reaching temp setpoints



Verify that the test conditions can be reached with a chamber filled with specimens

Q-FOG CRH HSCR Chamber Data

JASO M609 / ISO 14993



Q-FOG CRH HSCR Chamber Data

JASO M609 / ISO 14993



Q-FOG CRH HSCR Chamber Data

JASO M609 / ISO 14993



Conclusions

- Salt spray tests are good pass/fail screening tests
- Wet/Dry tests are good comparative tests for some systems but not repeatable
- Combined weathering / corrosion cycles can provide good outdoor correlation for some materials
- First-generation cyclic automotive tests are comparative tests but not repeatable
- Modern automotive corrosion tests are more realistic and offer better repeatability and reproducibility

Thank you for your time.

Questions? info@q-lab.com



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