Laboratory Corrosion Testing:
Realism and Reproducibility with Modern Methods

Q-Lab Corporation

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Q-Lab's Winter Corrosion Webinar Series

Today is the first of two webinars this winter from Q-Lab focusing on laboratory corrosion testing

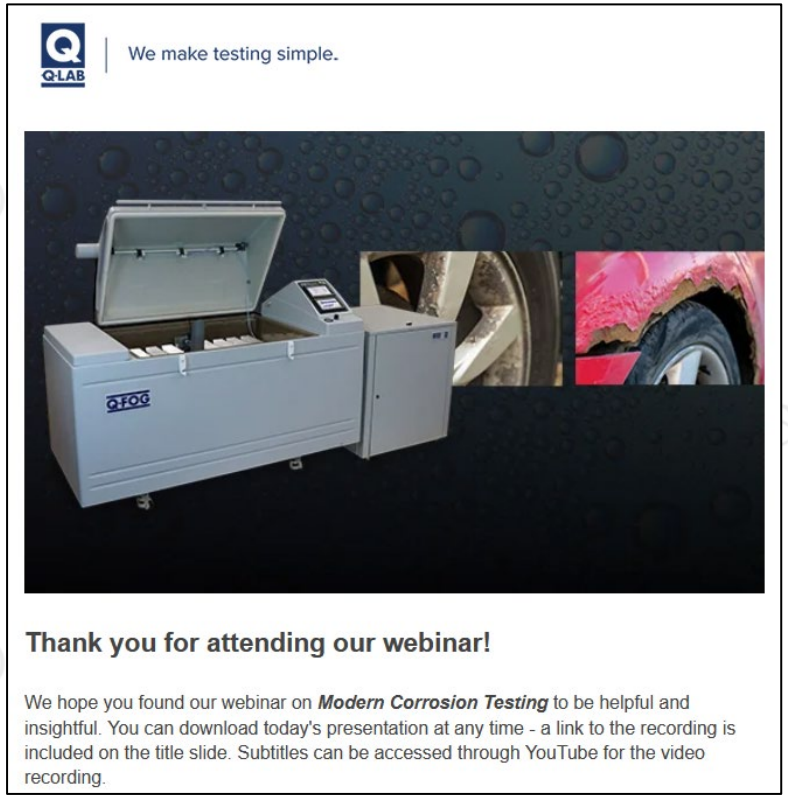
All upcoming and archived webinars can be accessed at:
q-lab.com/webinars

Date	Topic
04 Dec	Modern Corrosion Testing
11 Dec	JASO M 609 – What's New in the 2024 Revision

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!



Topics

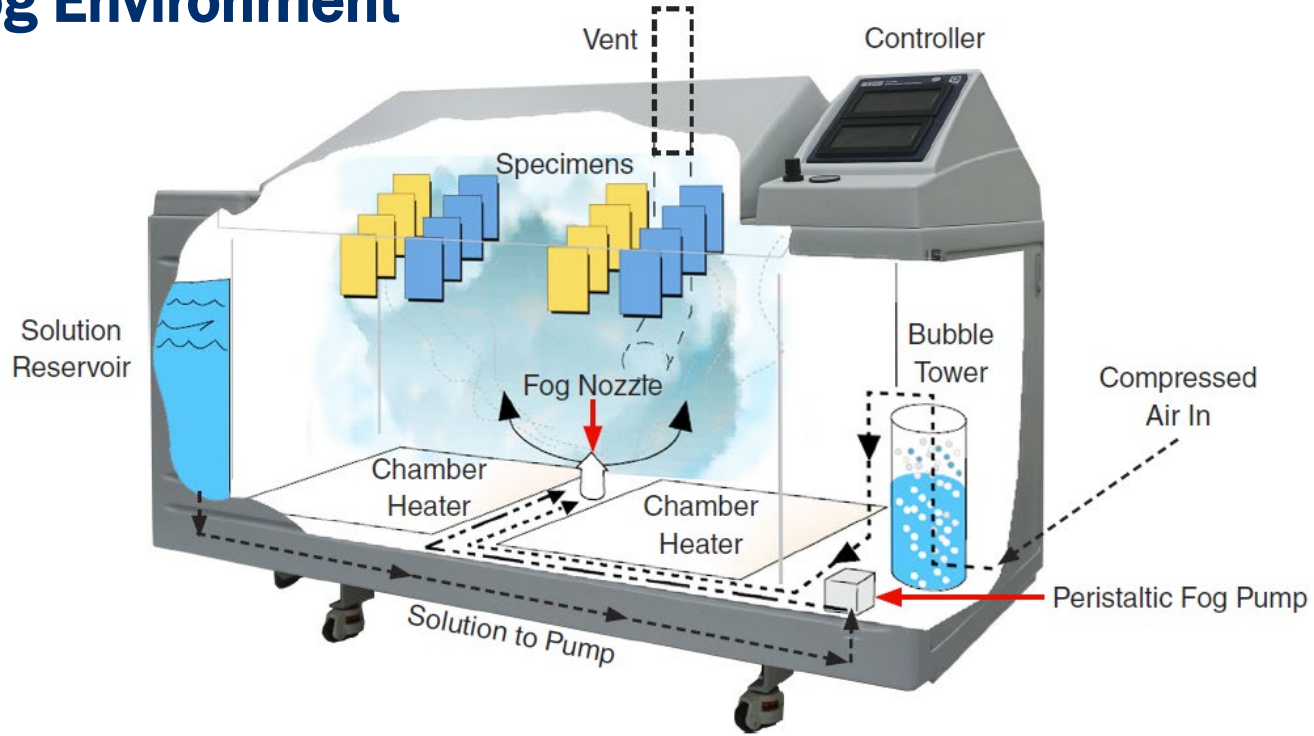
- Types of Accelerated tests
- Continuous Salt Spray (Neutral & Acidified)
- Wet/Dry Cyclic Tests
- First-Generation Cyclic Automotive Tests
- Modern Corrosion Test Methods
- Verifying Corrosion Test Performance

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	• Defined • Medium-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)

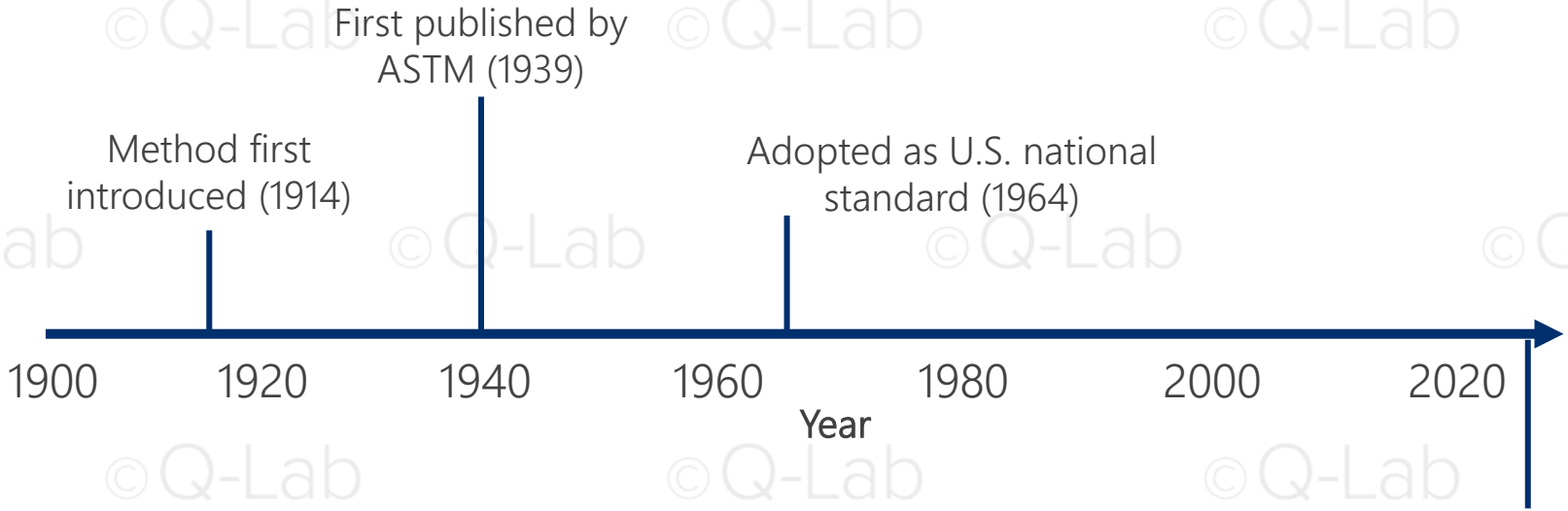
Continuous Salt Spray

Salt Fog Environment



Continuous Salt Spray

ASTM B117



ASTM B117 is the most widely-used corrosion standard today, primarily for quality control and metallic/conversion coatings

Continuous Salt Spray

ASTM B117

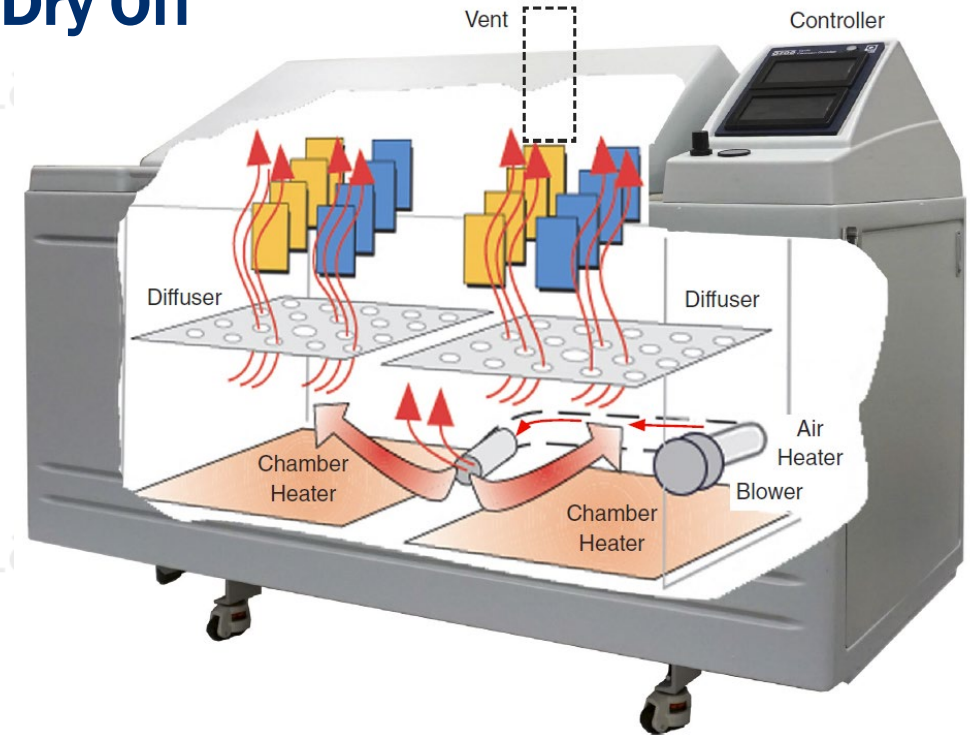
- 5% NaCl salt fog at 35 °C
- Neutral pH
- Fine mist (atomized with compressed air) sprayed indirectly onto specimens
- ISO 9227 contains the same test (and acidified versions)
- When correctly followed, test has reasonable repeatability and reproducibility

Limitations of Continuous Salt Spray

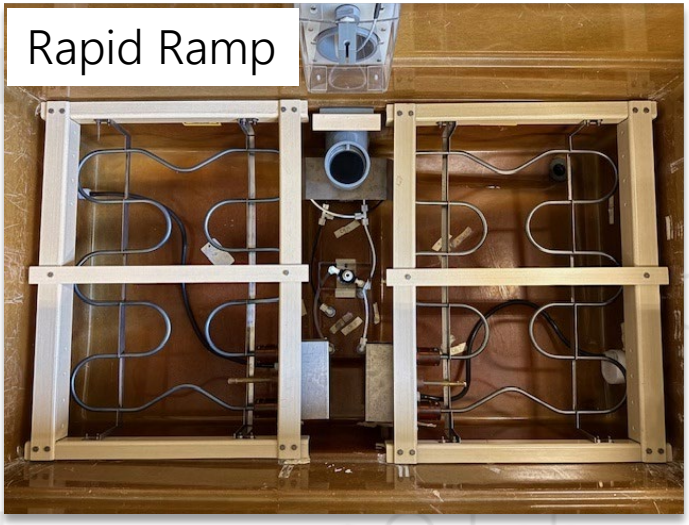
- Not a good simulation of most service environments
- Typically produces different corrosion products than natural exposure
- Poor rank order correlation with outdoor corrosion
- A good **quality control/screening test**, usually not much more

Wet/Dry Cyclic Tests

Salt Fog -> Dry Off



Heater Configurations



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

Wet/Dry Cyclic Tests

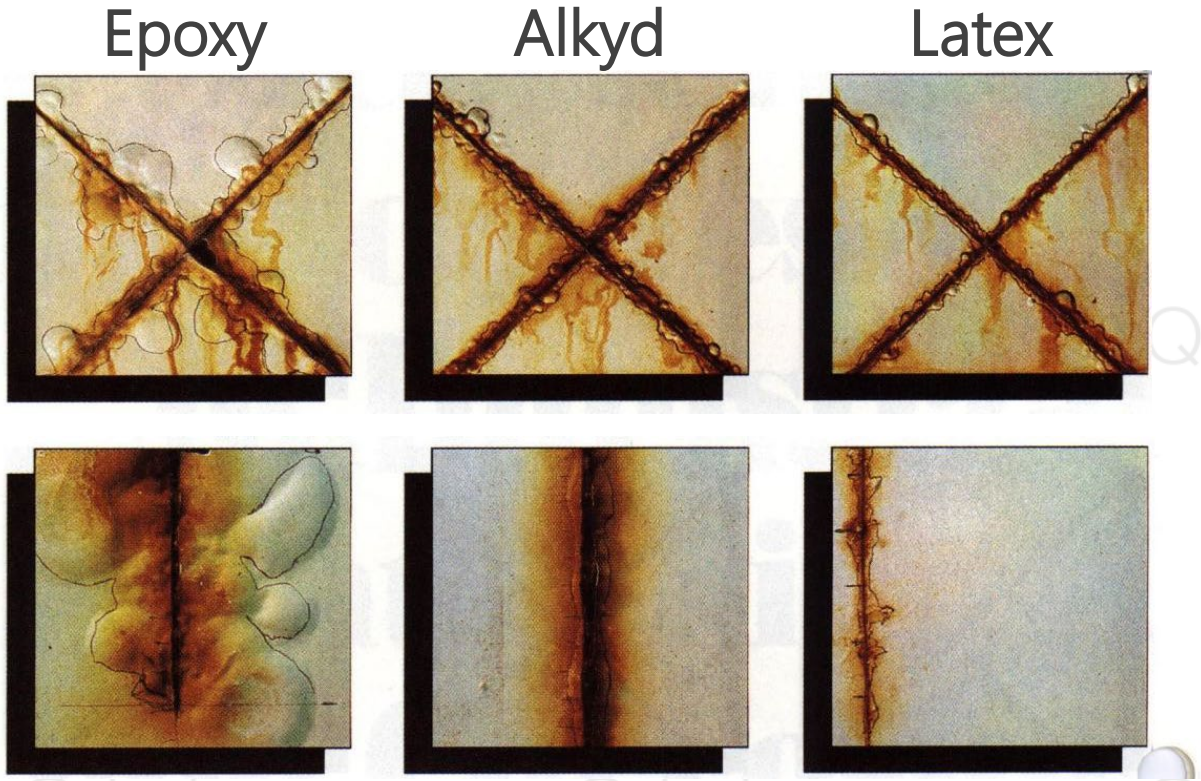
Prohesion (Protection is Adhesion)

- Alternating spray and dry-off
- Development began in England, 1960's
- Dilute NaCl, $(NH_4)_2SO_4$
- American Architectural Manufacturers Association replaced ASTM B117 with this test in AAMA 2605, "Superior" coatings on aluminum

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

Combined Corrosion/Weathering vs Outdoors



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

SSPC Test Results

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

Not good from continuous or simple wet/dry cycles

Combined Corrosion and Weathering

ISO 12944-6 (and -9)

Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
UV/condensation — ISO 16474-3			Neutral salt spray — ISO 9227			Low-temp. exposure at (−20 ± 2) °C

- 4 hours UVA-340, 0.83 W/m^2/nm at 340 nm, 60 °C
- 4 hours dark condensation, 50 °C
  - 72 hours

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

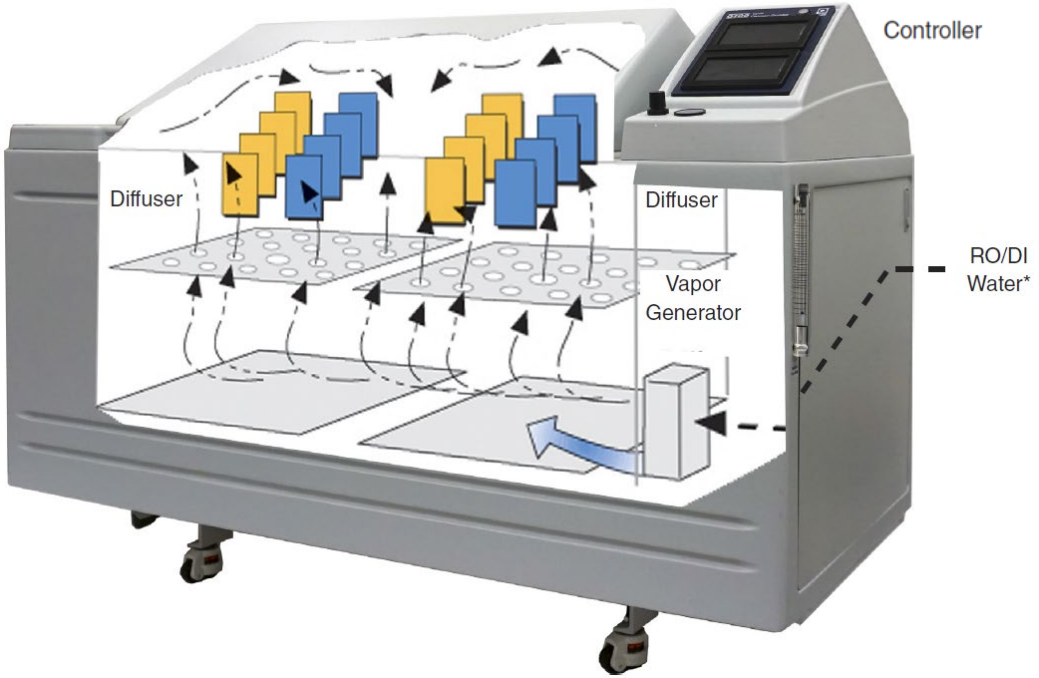
Wet/Dry Cyclic Test Limitations

- 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 → Dry-Off → Wetting (Humid)

Wetting specimens after dry-off reinitializes corrosion



First-Gen Cyclic Automotive Tests

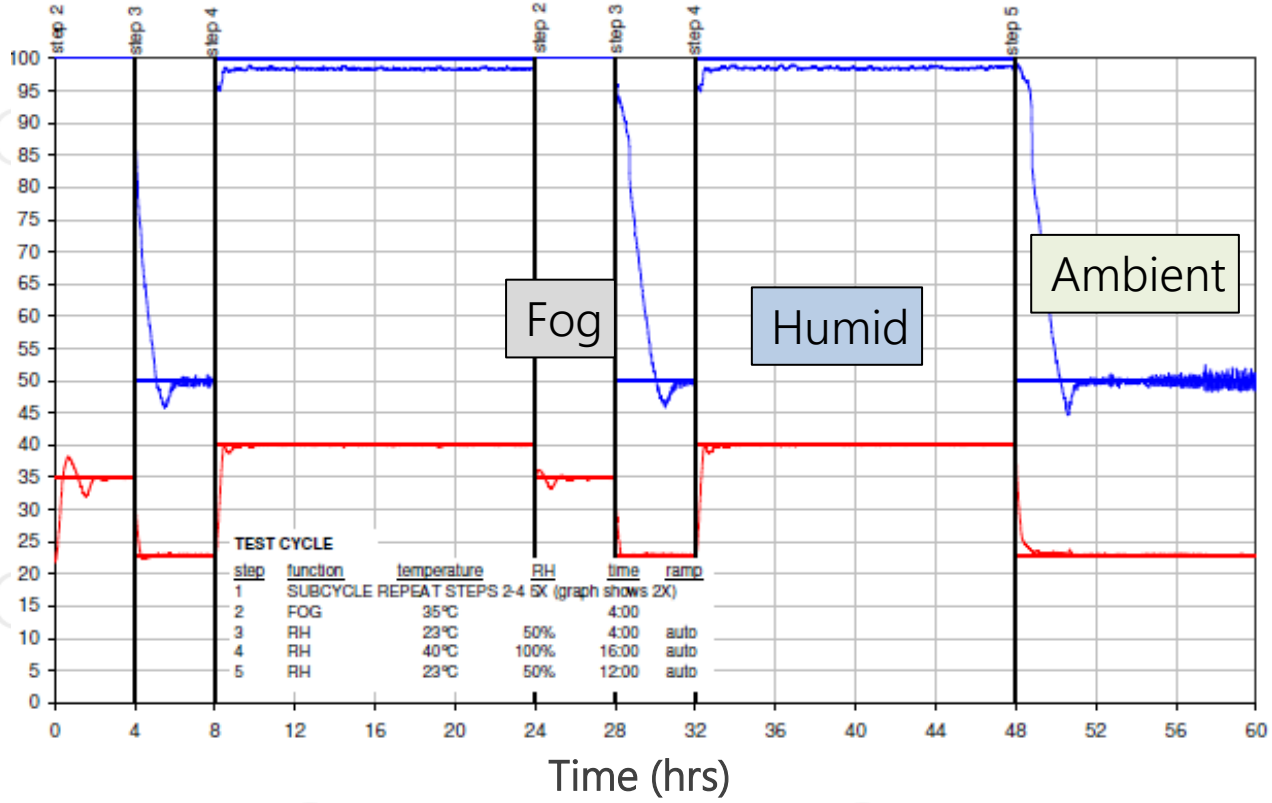
Salt Fog → Dry-Off → Wetting (Humid)

- **Electrolyte**: NaCl, $CaCl_2$, 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

RH (%)

Temp (°C)



Limitations of First Generation CCT

Poor Repeatability and Reproducibility!

- Problems observed
  - Different corrosion chambers produced different results
  - 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

A Deeper Look Into Corrosion Factors

Deliquescence of salts

Galvanic corrosion

Influence of relative humidity

Automotive Tests & Road Salt

- 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 $(NH_4)_2SO_4$	81%
Sodium Chloride (NaCl)	*76%*
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

Galvanic Corrosion

Active (Anode)

Noble (Cathode)

Magnesium
Zinc
Aluminum
Cast Iron/low carbon steel
Steel (low alloy)
Brass
Copper
Nickel
Stainless Steel (passive)
Silver
Gold
Platinum

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	≥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

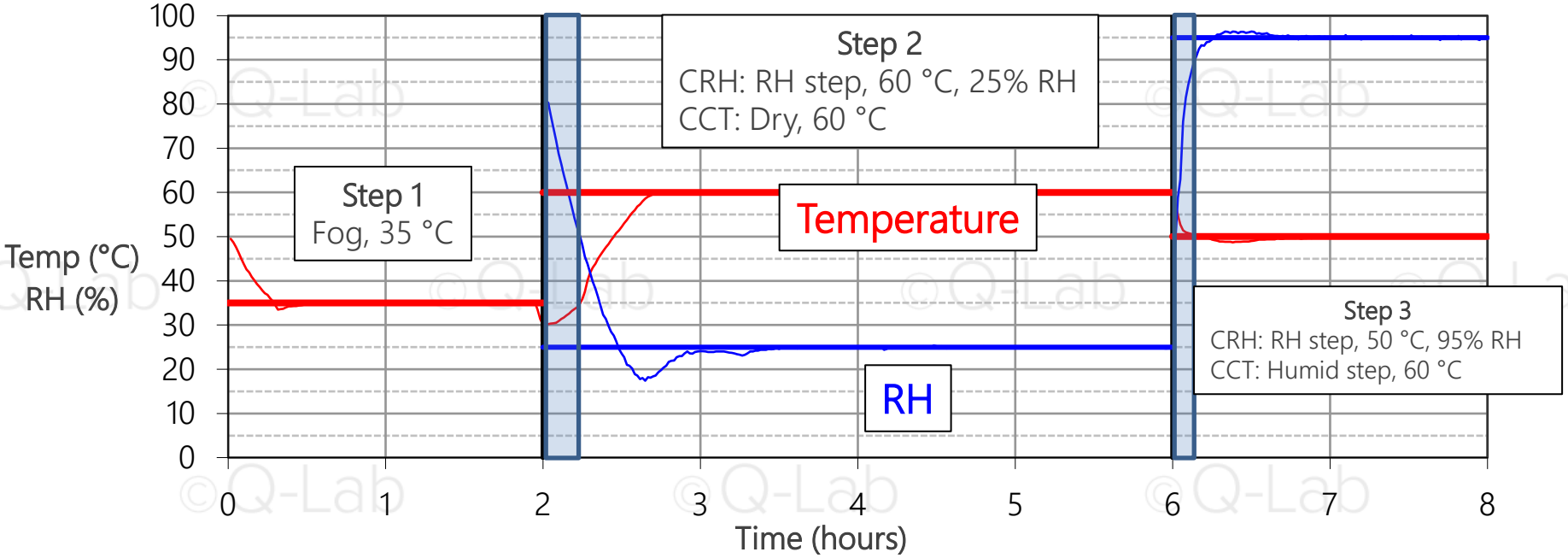
Relative Humidity and Corrosion

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



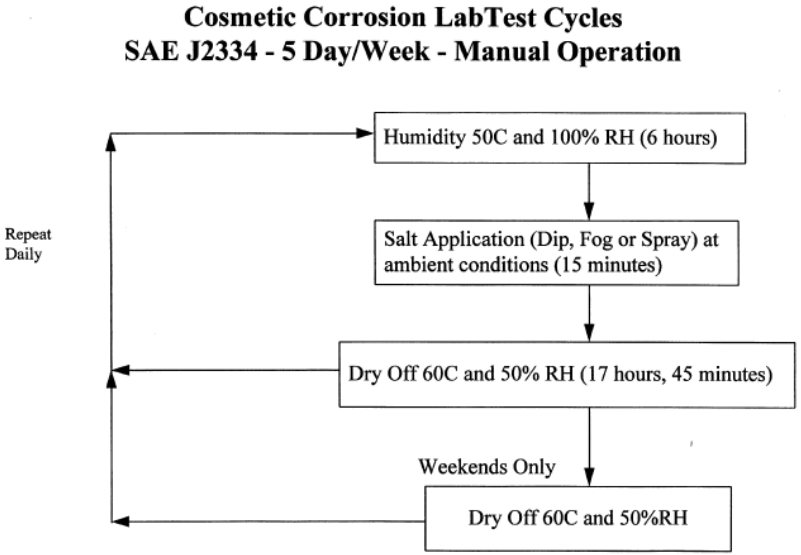
Fast Transition Times Designed to Improve Reproducibility

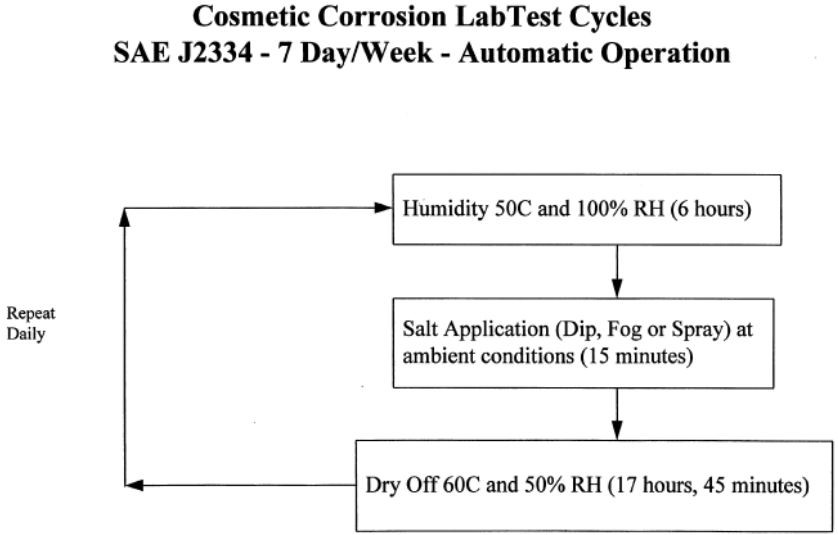
Very limited time in intermediate RH zone of 50-90%!

Uncontrolled Transition Times: SAE J2334

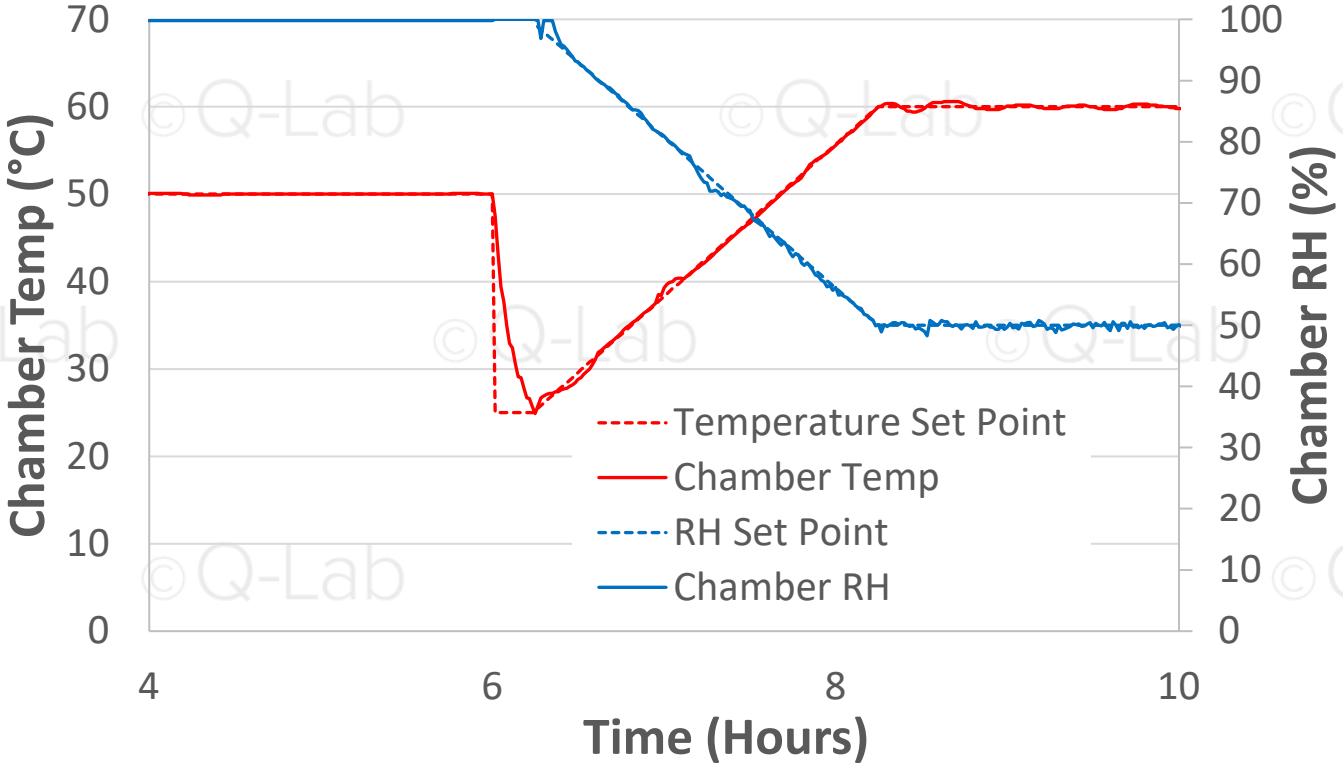
- Transition times are not specified in this standard
- Coupon use is encouraged but no mass loss limits are included
- Some companies have implemented SAE J2334 with their own mass loss limits

SAE J2334 Cycle



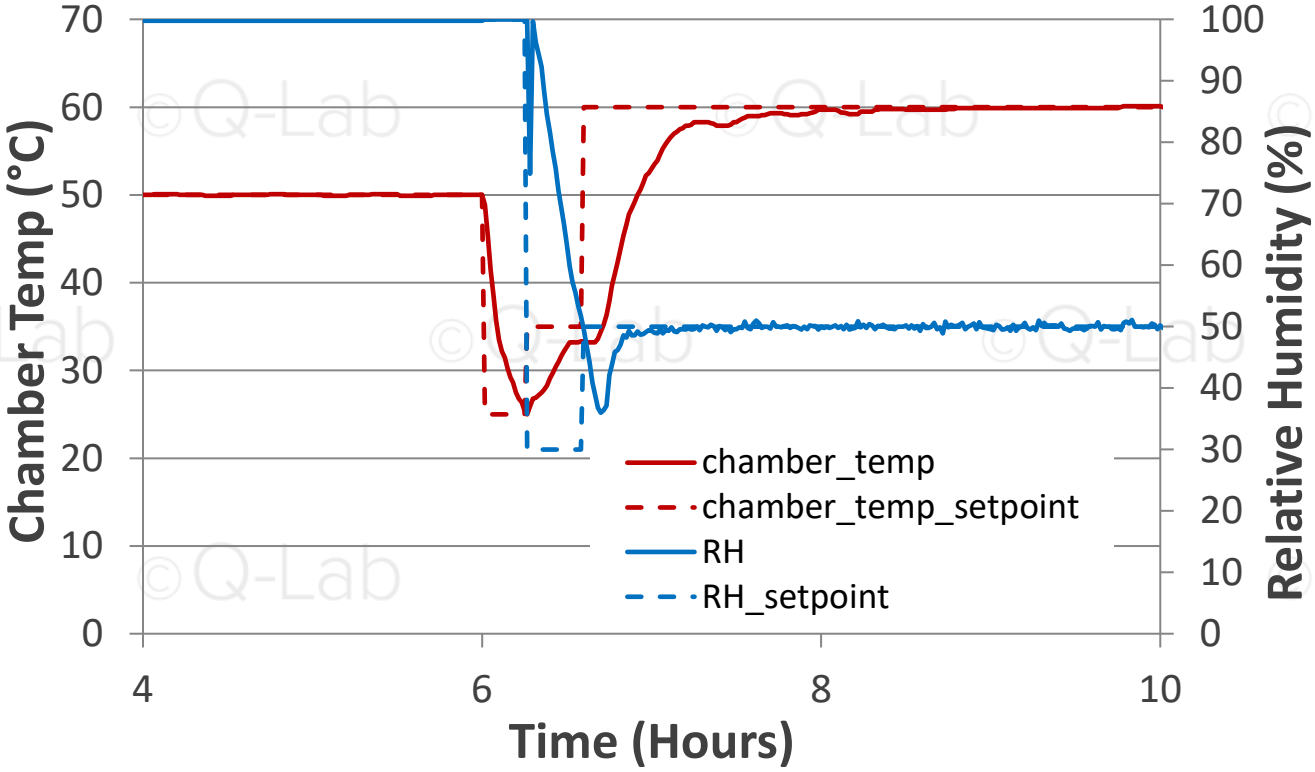


Slow Dry-off



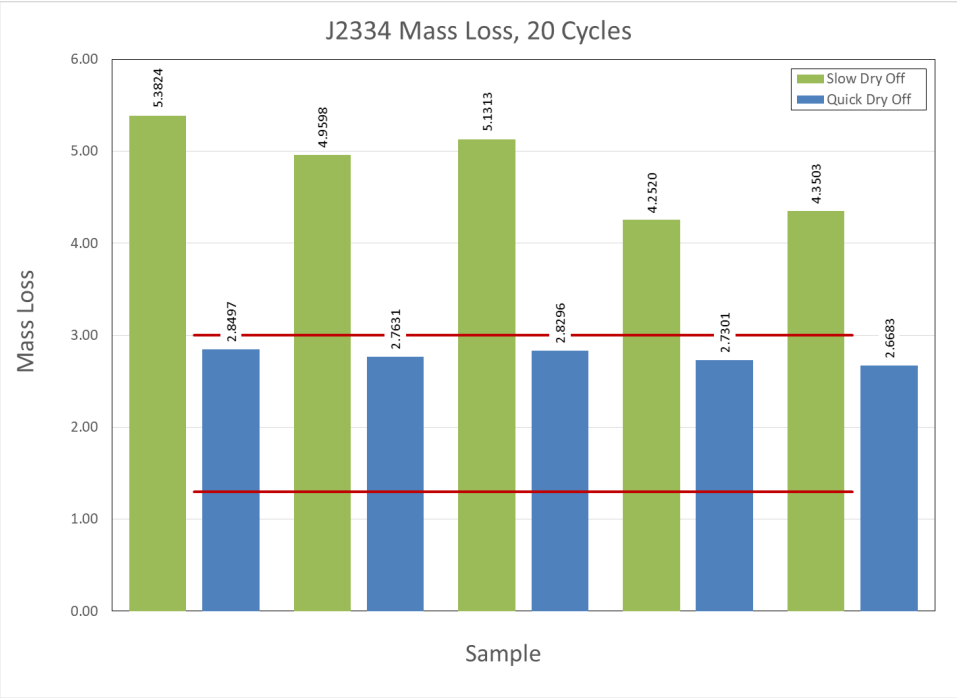
During the transition, the time above the Deliquescence RH of NaCl is about **1 hour**

Rapid Dry-off



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

SAE J2334 Results



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

Limitations of First Generation CCT

Poor Repeatability and Reproducibility!

- Problems observed
  - Different corrosion chambers produced different results
  - 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
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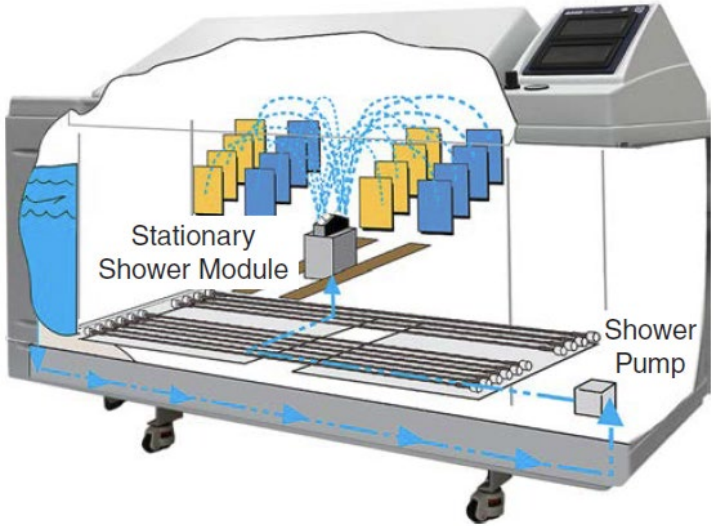
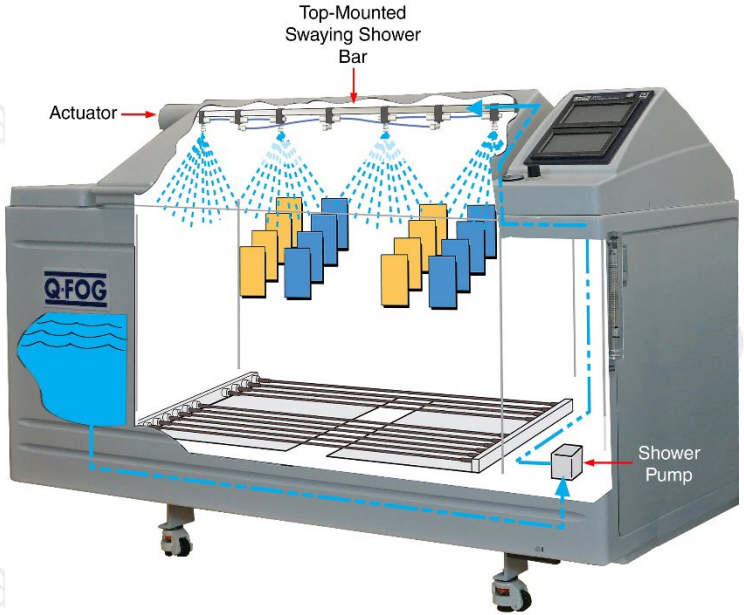
Modern Corrosion Tests

Shower Function



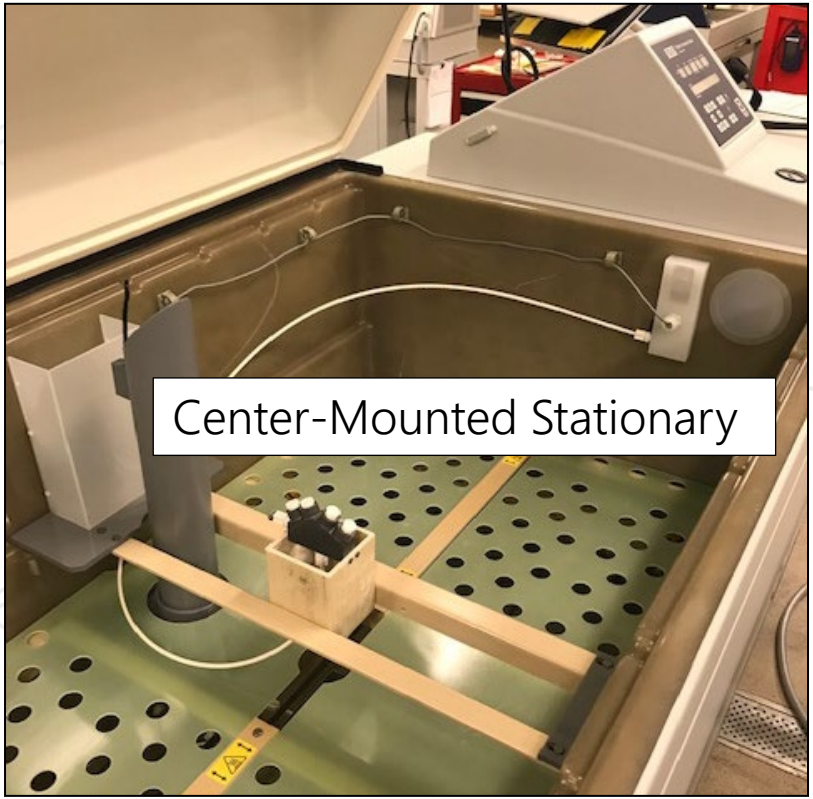
Stationary Shower Module (SSM)



Top-Mounted Swaying Shower Bar (TSSB)

Faster application of salt solution than Fog

Shower Configurations



Modern Automotive Corrosion Tests

Fog

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

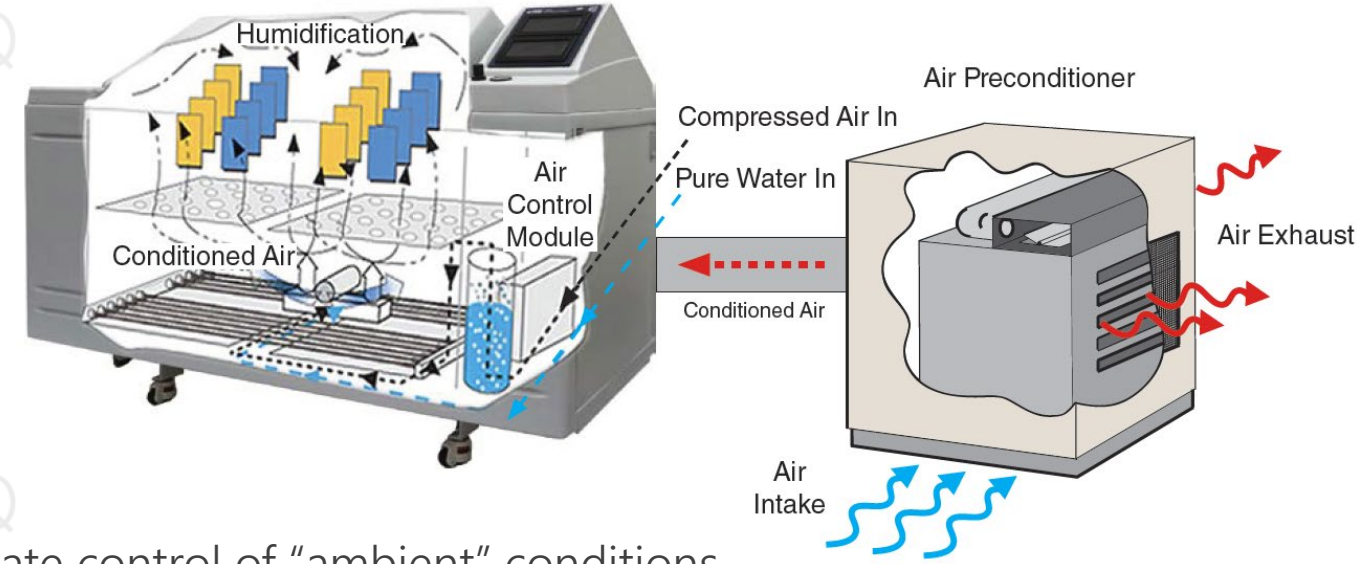
Shower

- 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

Modern Corrosion Tests

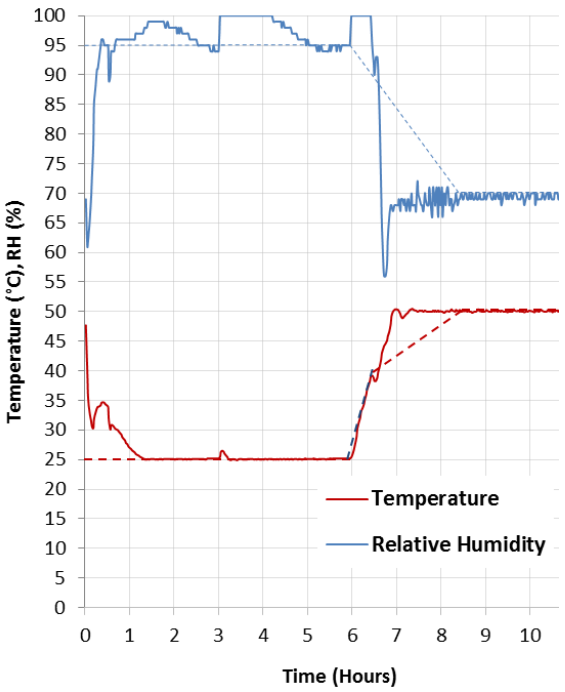
Air Preconditioner



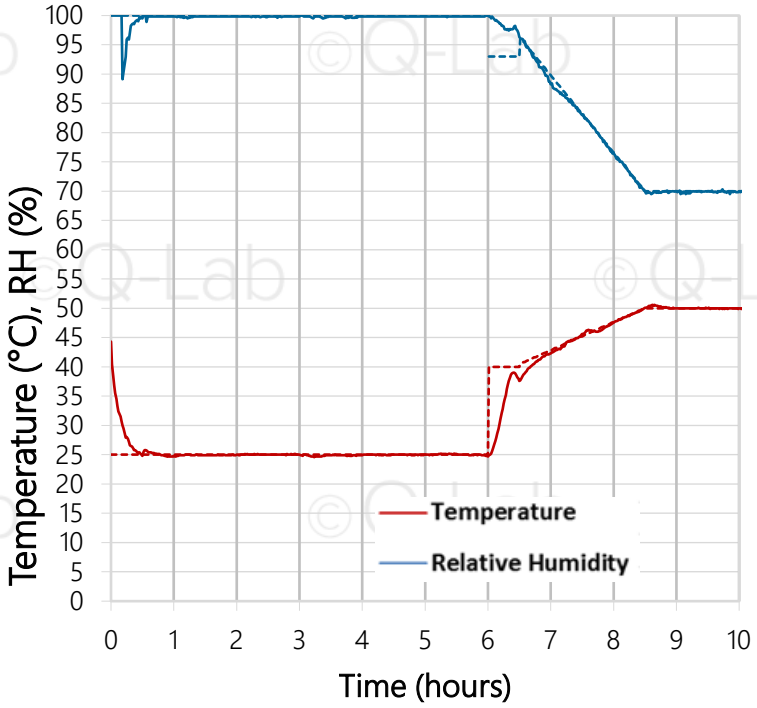
- Accurate control of “ambient” conditions
- Accurate ramping of temperature & relative humidity

RH Control with Air Preconditioner

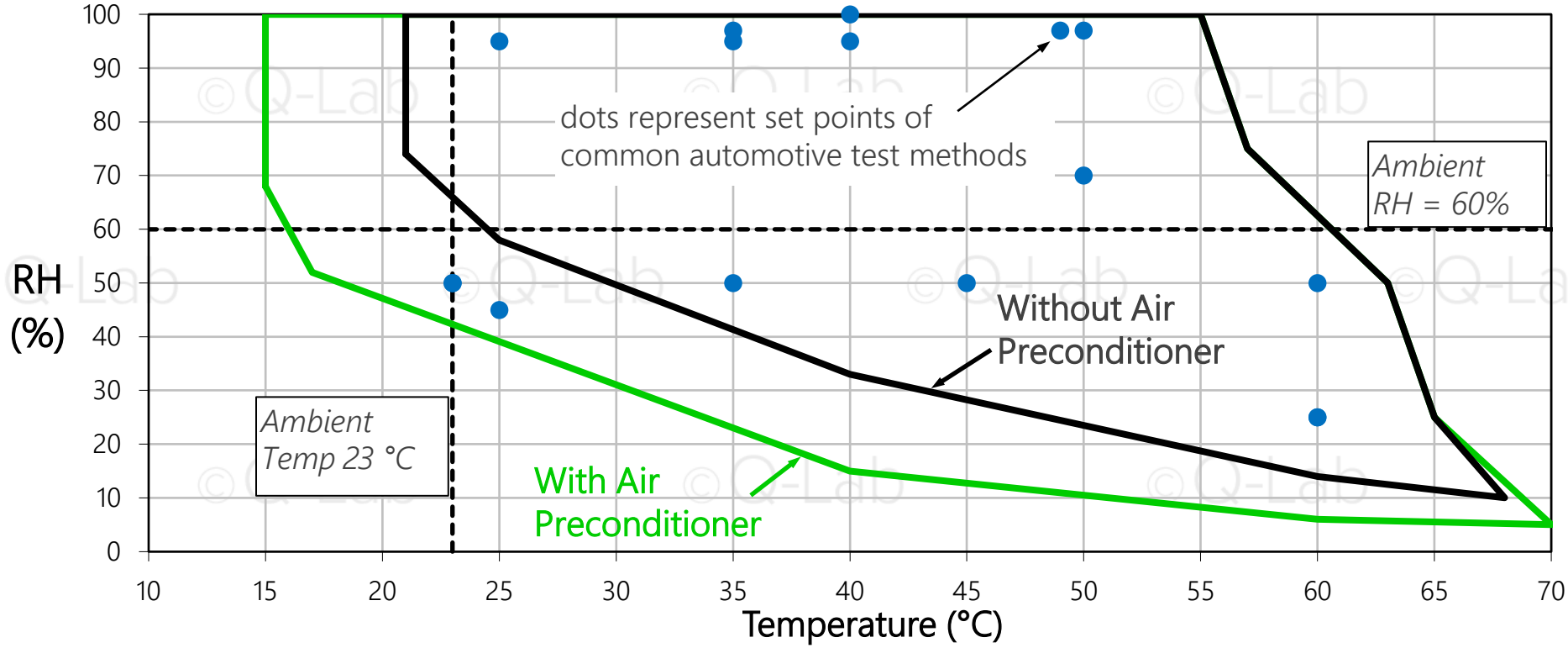
No Air Preconditioner



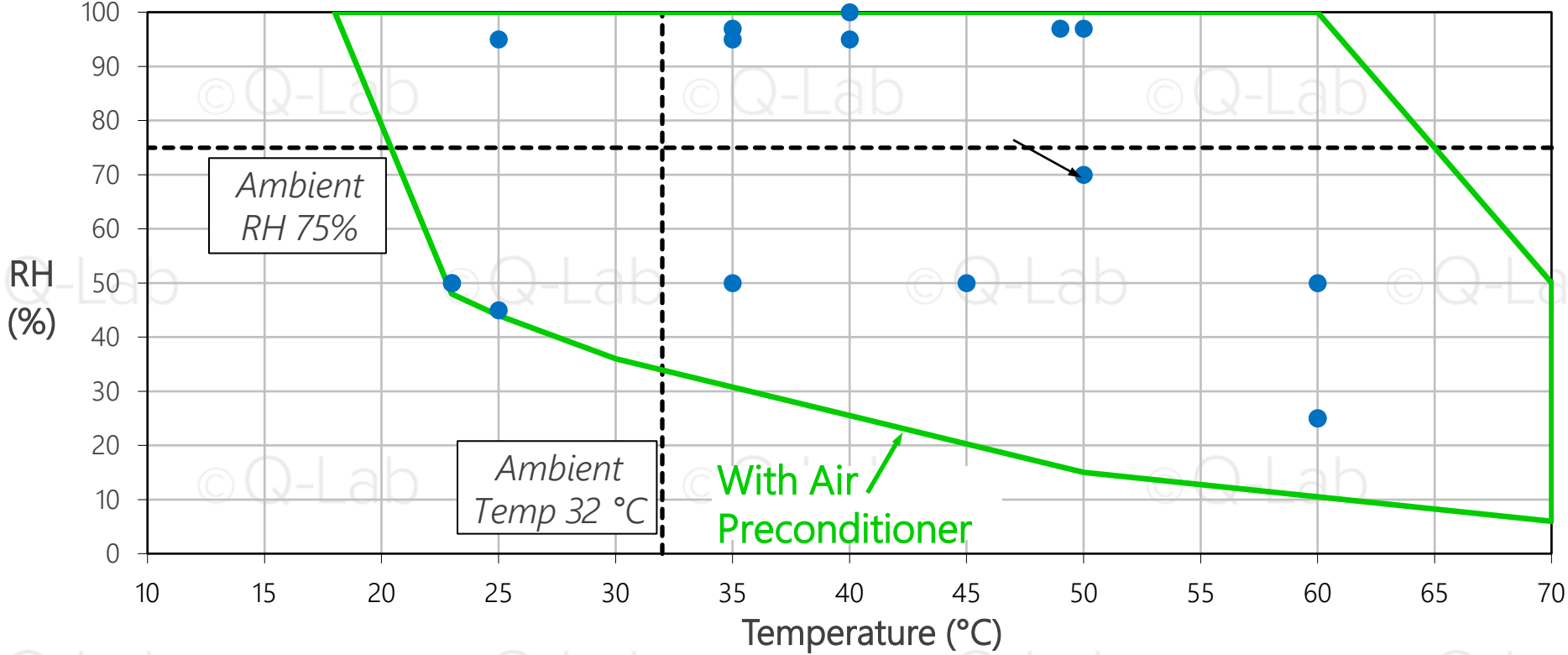
With Air Preconditioner



Q-FOG Operational Range: Well-Controlled Lab

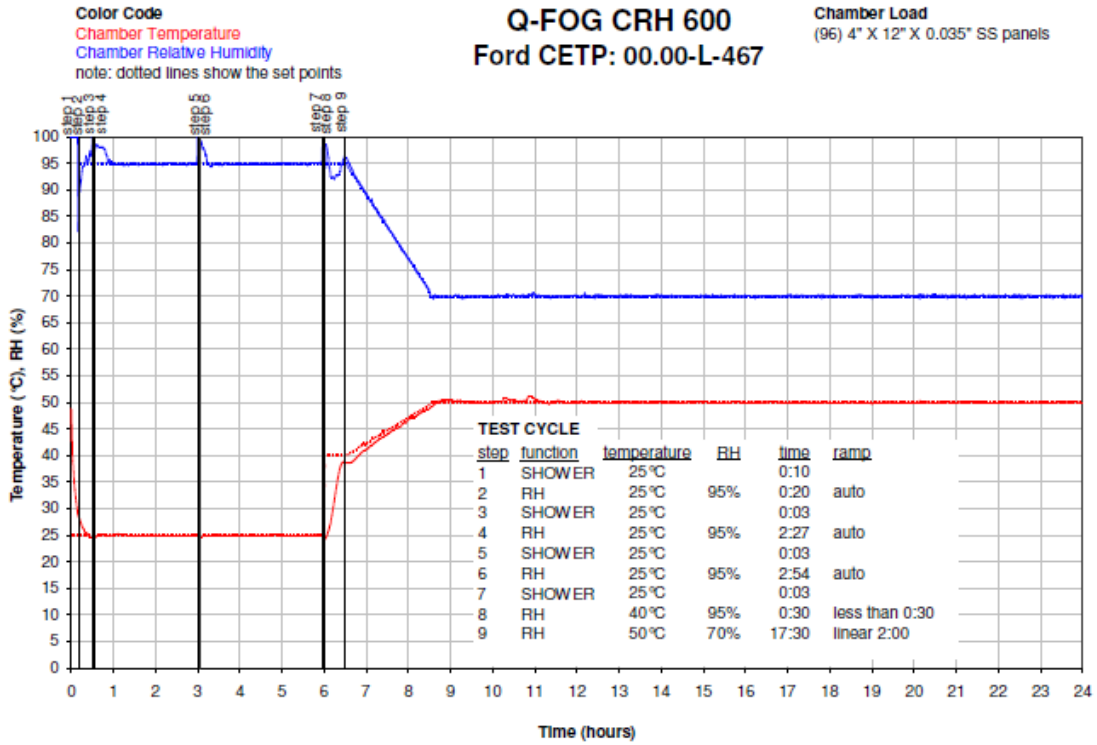


Q-FOG Operational Range: Hot, Humid Lab

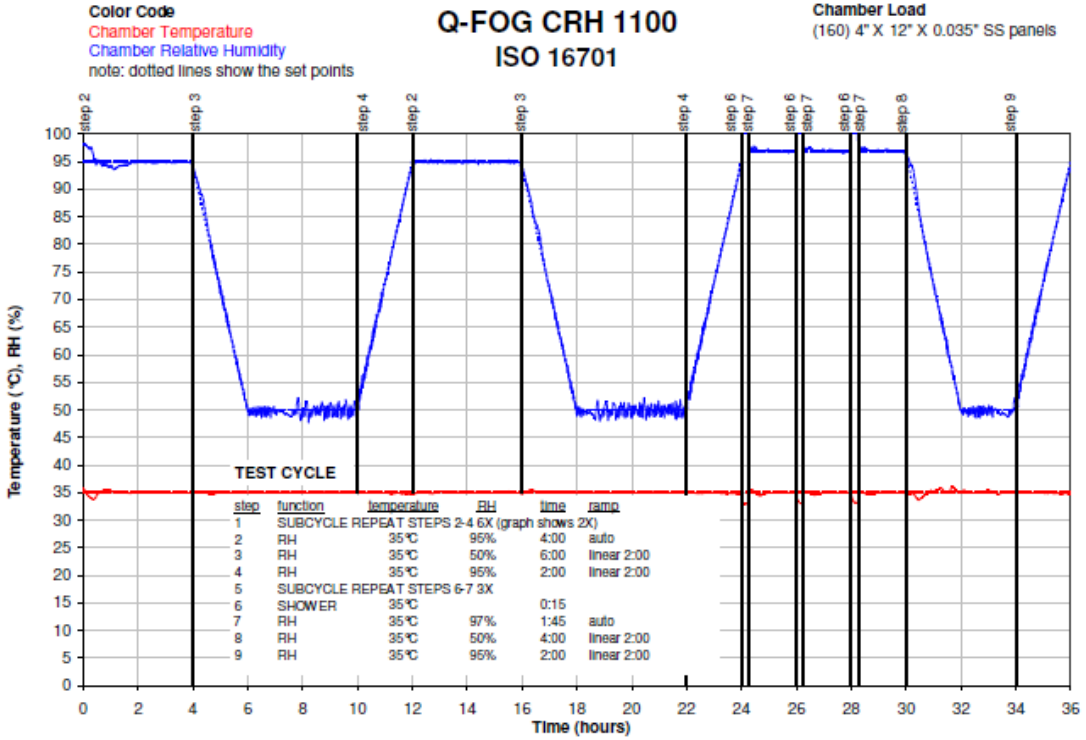


Modern Corrosion Test Examples

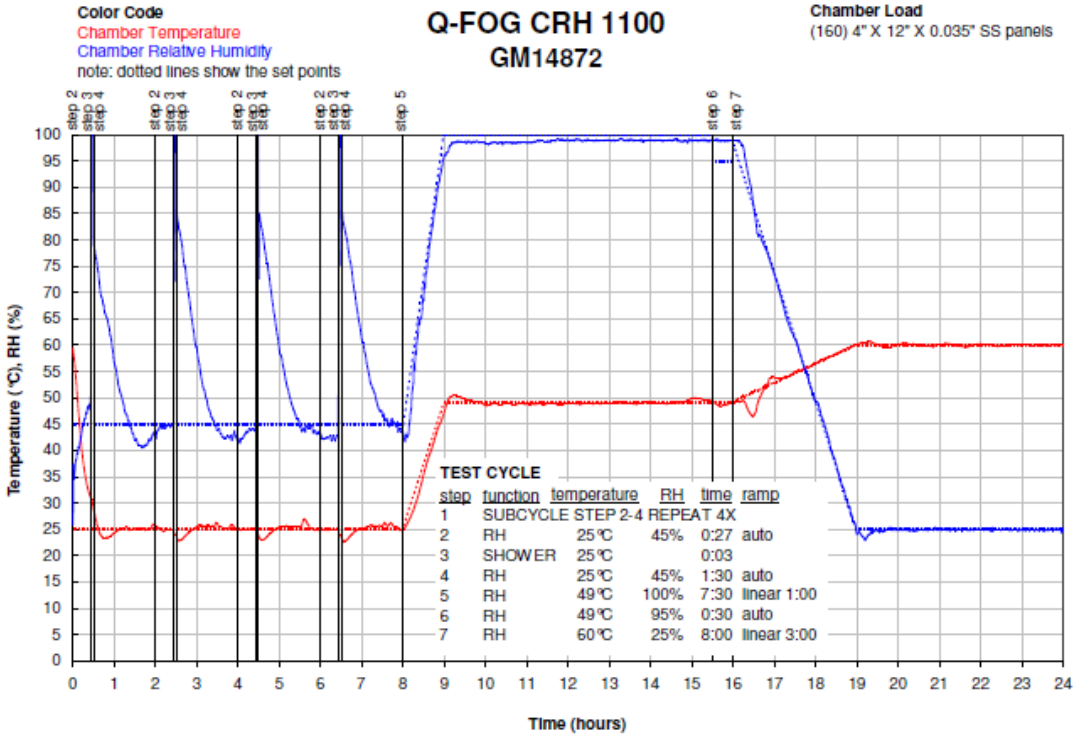
Ford L-467 / Volvo VCS 1027, 1449



ISO 16701



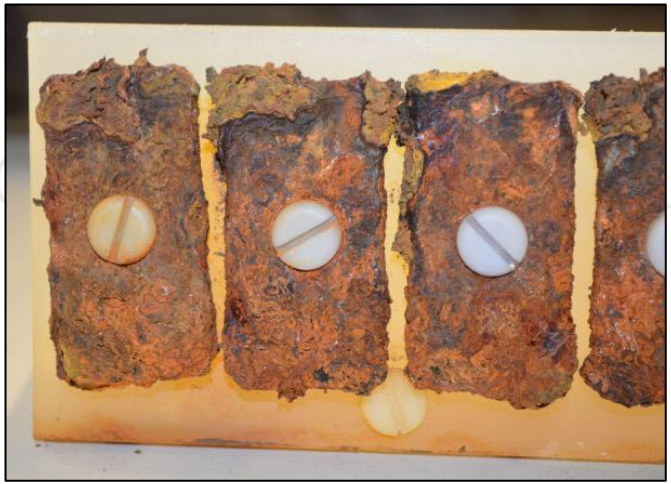
GMW 14872



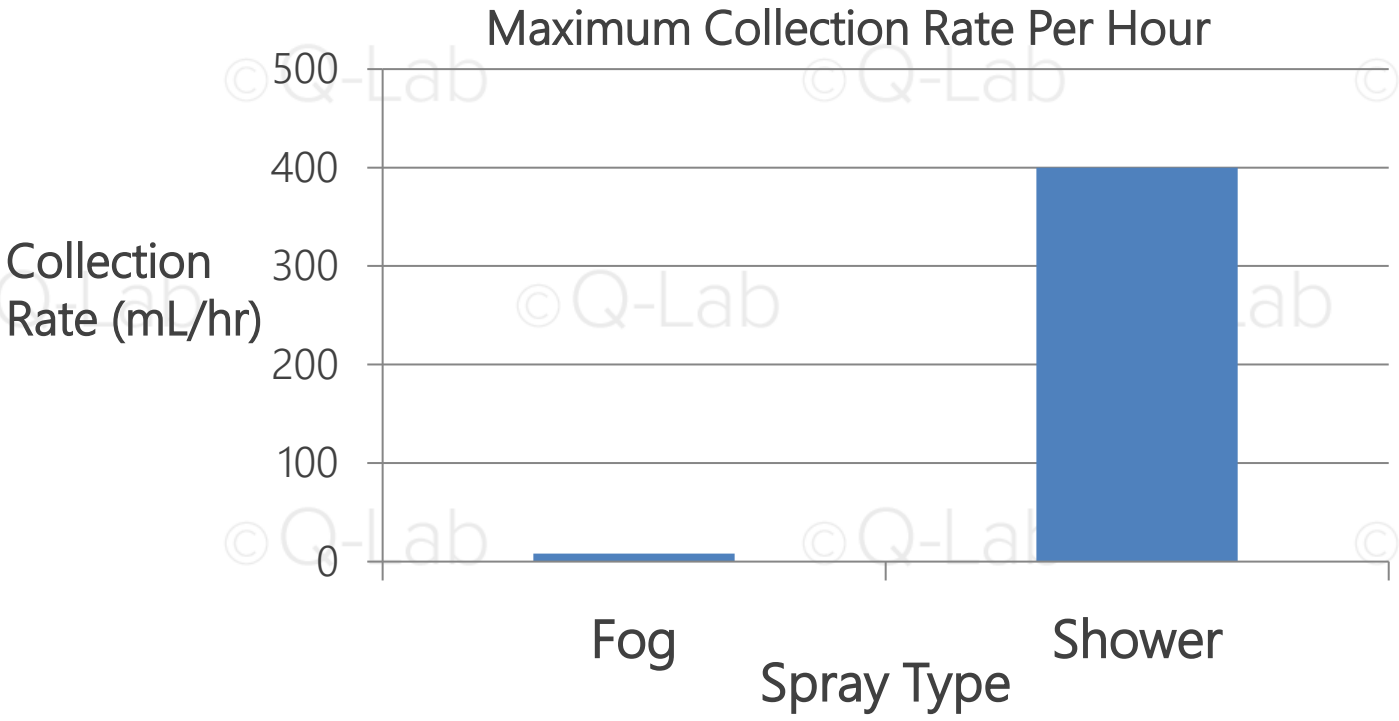
Verifying Corrosion Test Performance

Corrosion (Mass-Loss) Coupons

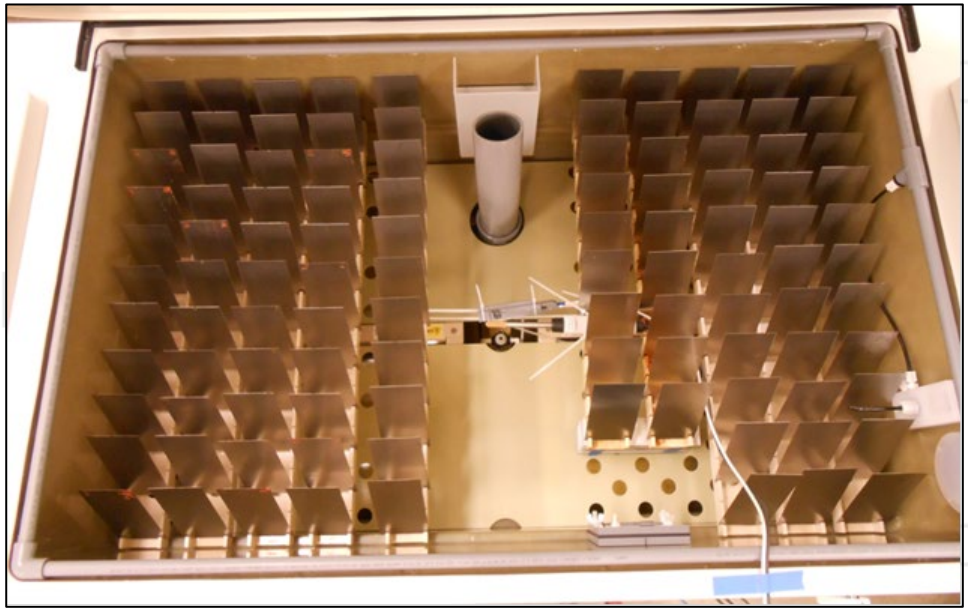
- Standardized metal specimens
- Mass loss due to corrosion is measured during a test
- Used by GM, VDA, ISO 16701 standards, and many others
- GMW 14872 requires a specific rate of mass loss throughout a test
- Ensures corrosion chamber is maintaining proper conditions and operator is running the test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



Pluviometry



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

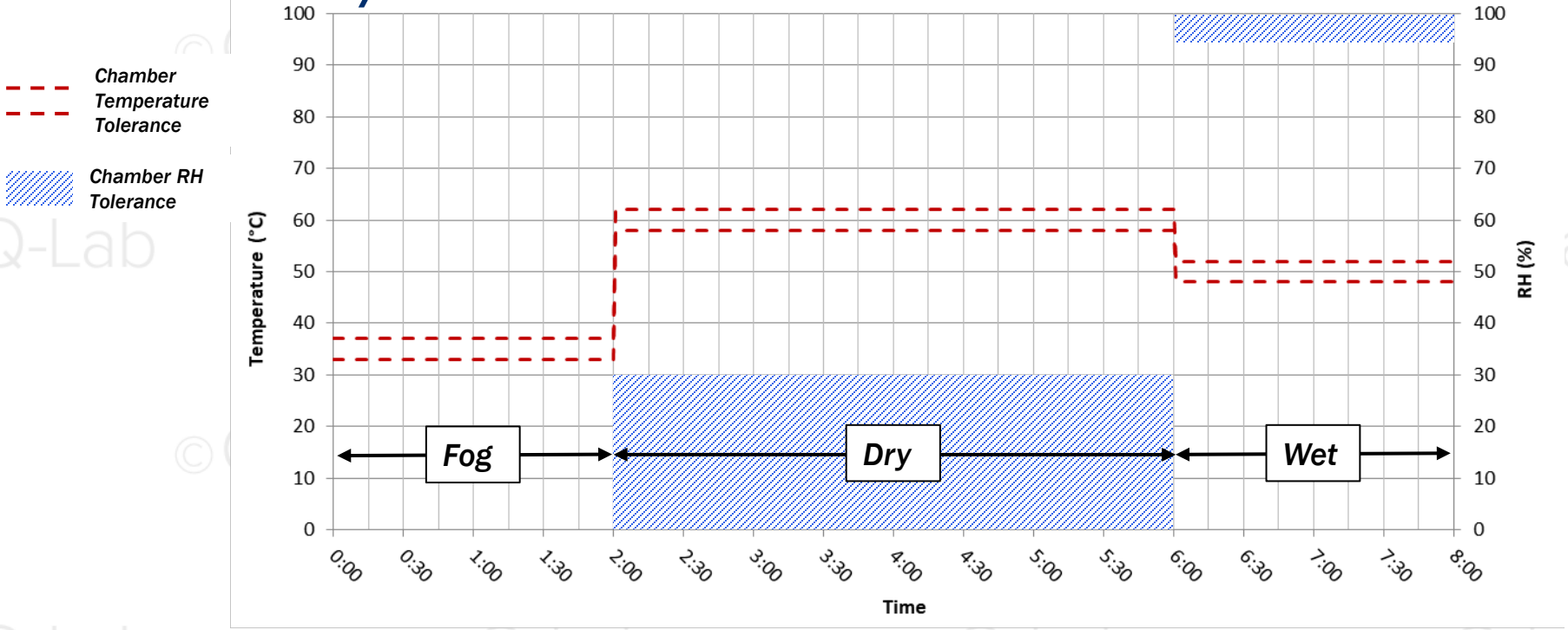
Verify Test in a Full Chamber

- 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

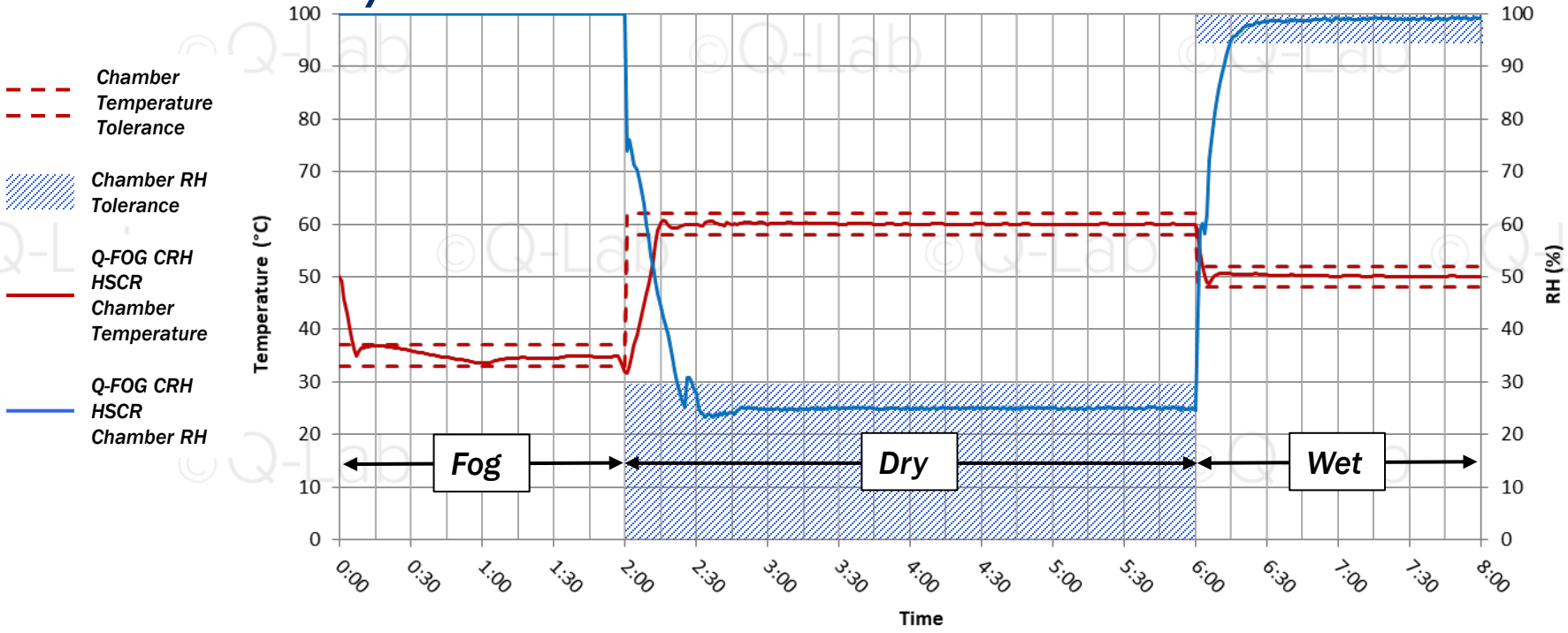
Q-FOG CRH HSCR Chamber Data

JASO M 609 / ISO 14993



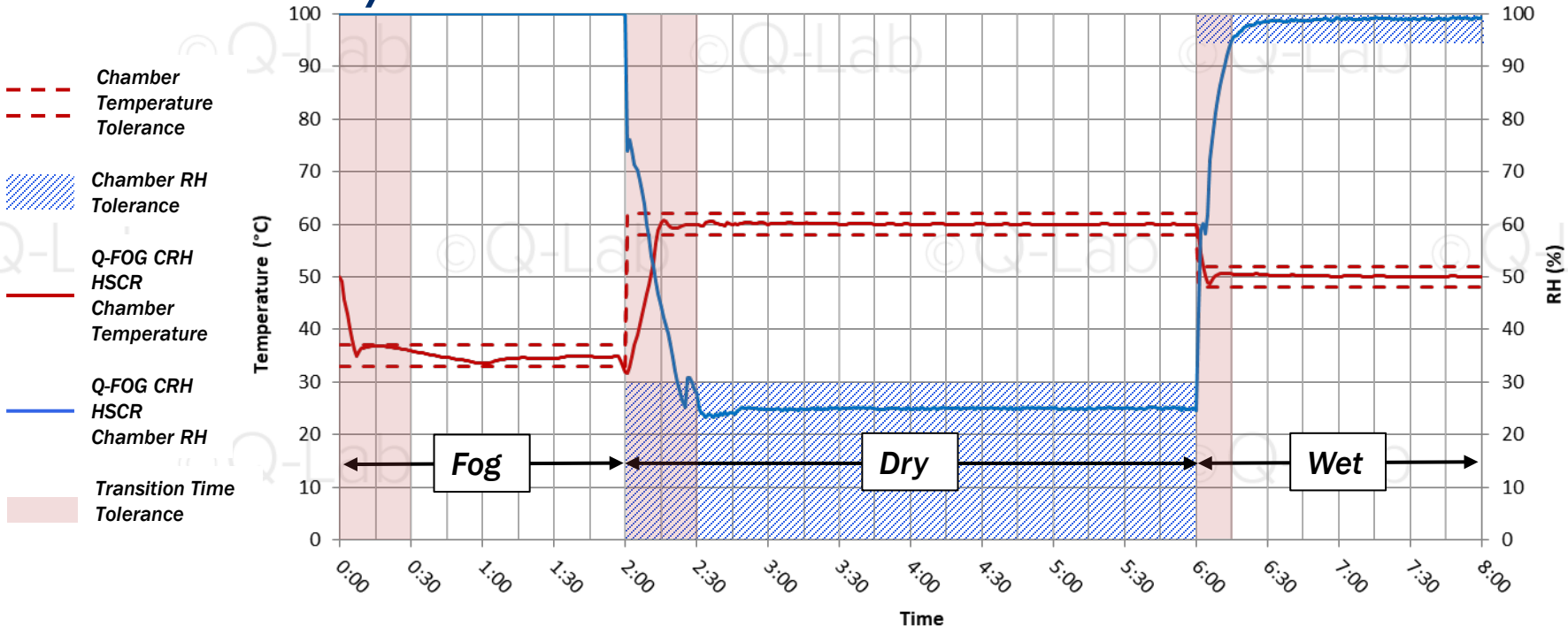
Q-FOG CRH HSCR Chamber Data

JASO M 609 / ISO 14993



Q-FOG CRH HSCR Chamber Data

JASO M 609 / 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

We make testing simple.