# Laboratory Corrosion Testing: Realism and Reproducibility with Modern Methods

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Q-Lab Corporation

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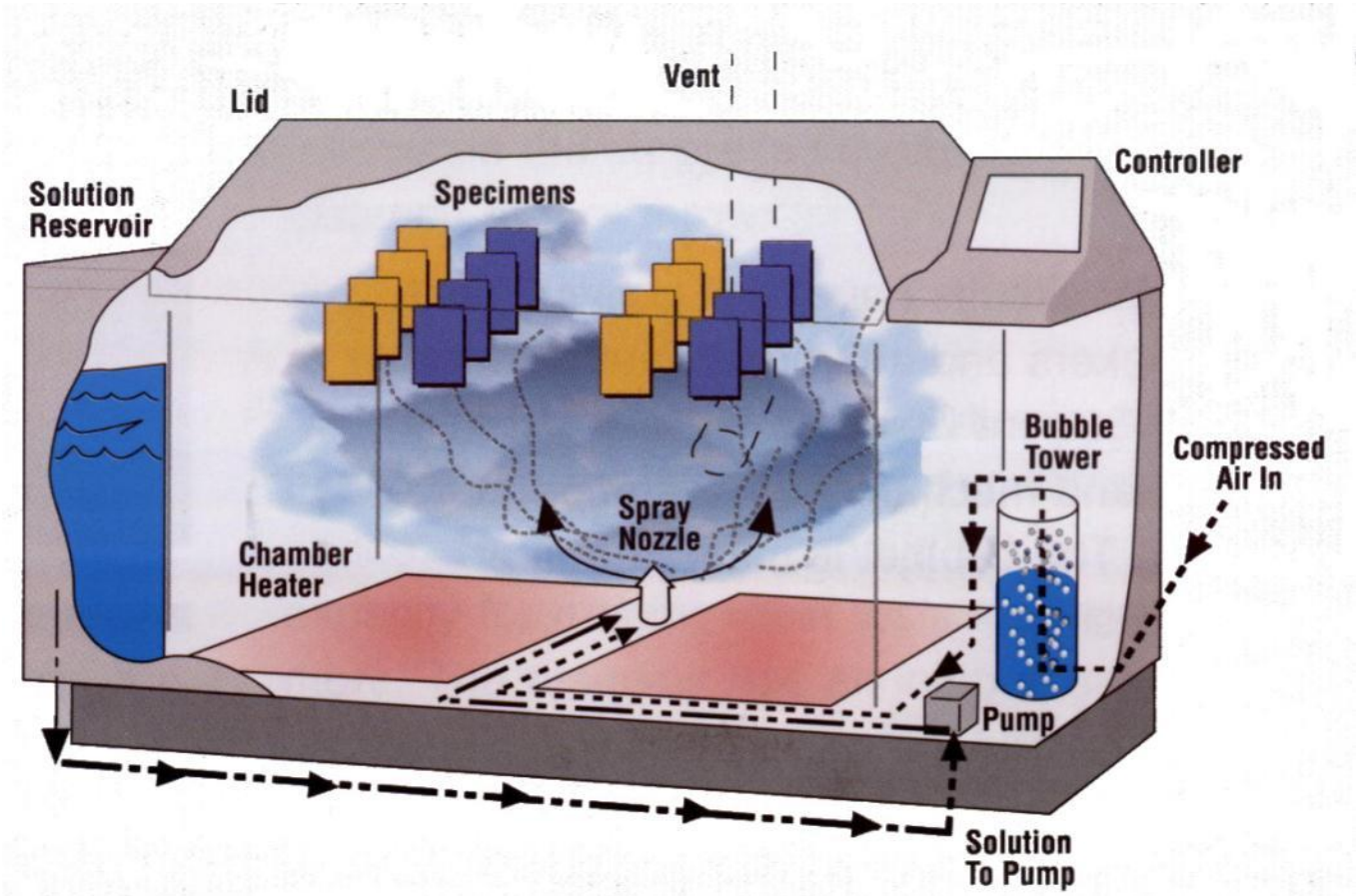
# Topics

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

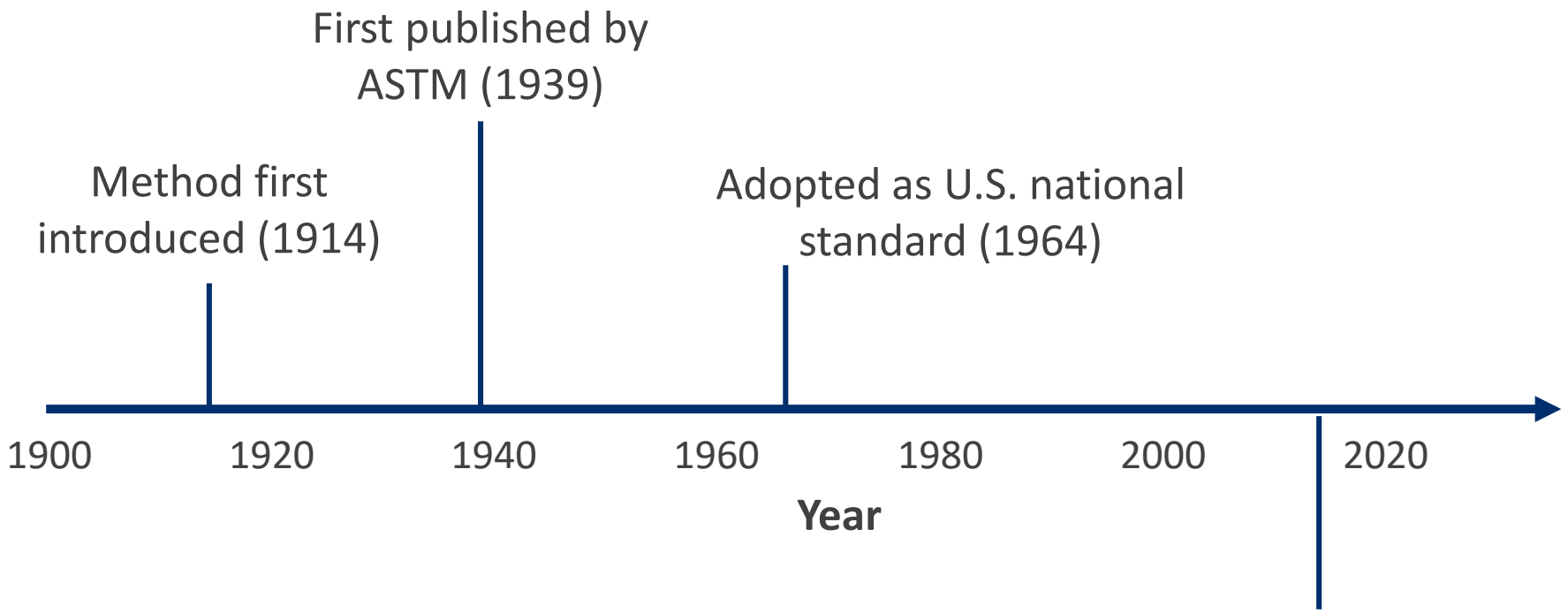
# Types of Accelerated Tests

| Accelerated Test Type | Result | Test Time | Results compared to | Research? Development? Certification? |
|---|---|---|---|---|
| Quality Control | Pass / fail | • Defined<br>• Short | Material specification | Certification & Research |
| Qualification / validation | Pass / fail | • Defined<br>• Medium-long | Reference material or specification | Certification & Development |
| Correlative | Rank-ordered data | • Open-ended<br>• Medium | Natural exposure (Benchmark site) | Development |
| Predictive | Service life<br>Acceleration factor | • Open-ended<br>• Long | Natural exposure (Service environment) | Development & Warranty Contracts |

# 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
- When correctly followed, test has reasonable repeatability and reproducibility

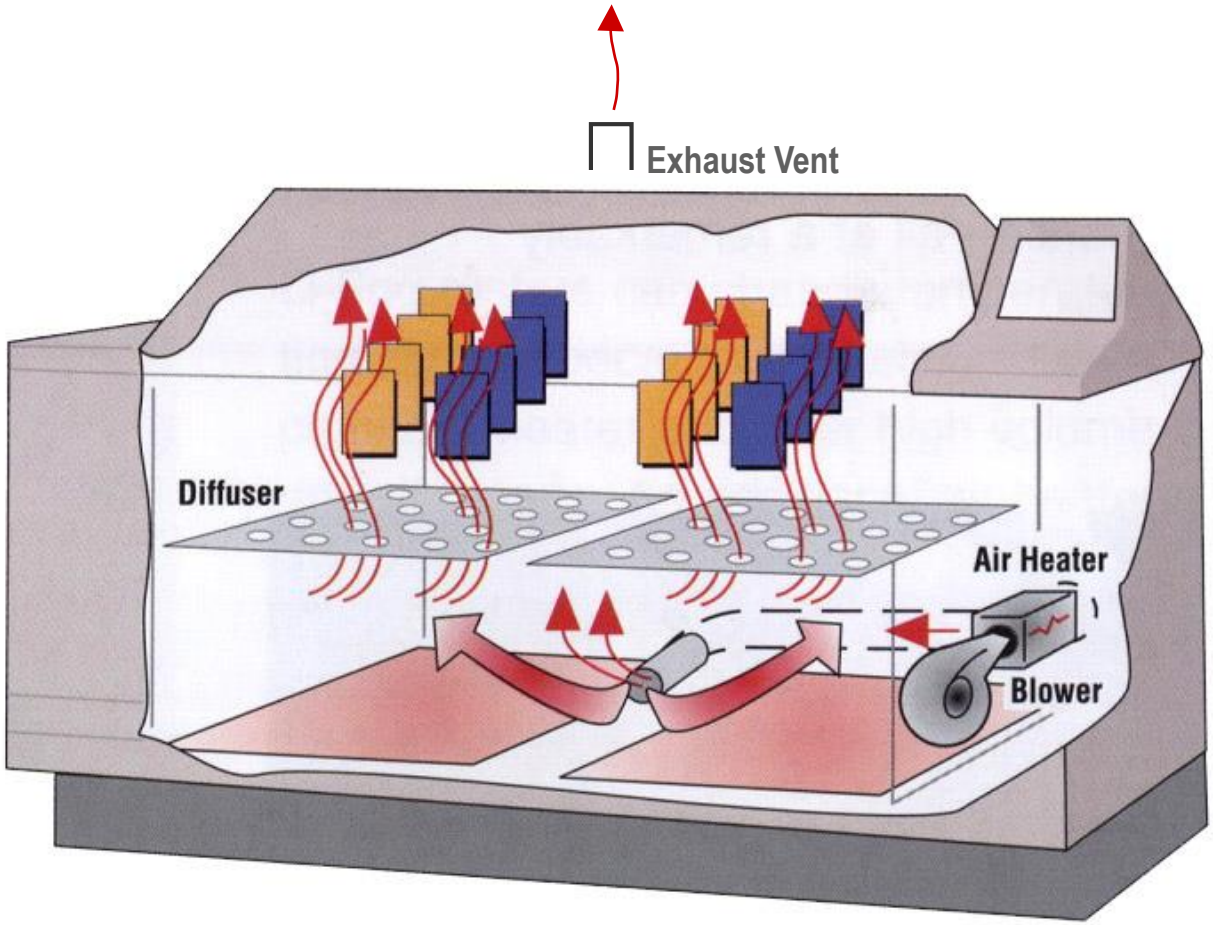
# Limitations of Salt Spray

- Not a good simulation of most service environments
- Typically produces different corrosion products than natural exposure
- Poor rank order correlation with outdoor corrosion

***Q: What type of accelerated test is this?***

# Wet/Dry Cyclic Tests

## Salt Fog -> **Dry Off**



# Wet/Dry Cyclic Tests

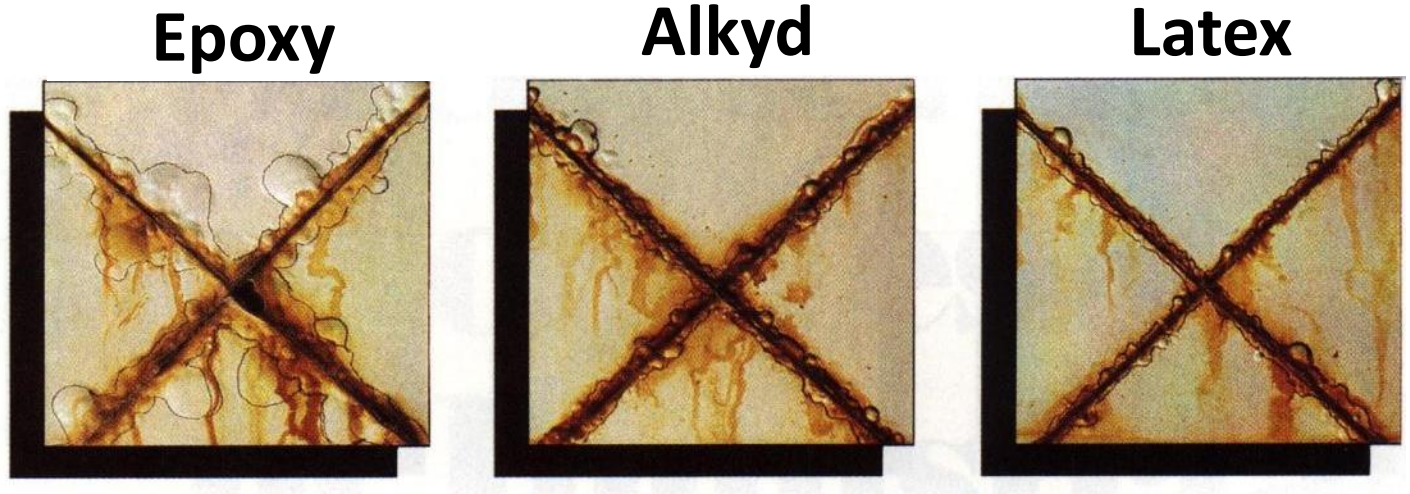
## Prohesion (***Pro***tection is Ad***hesion***)

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

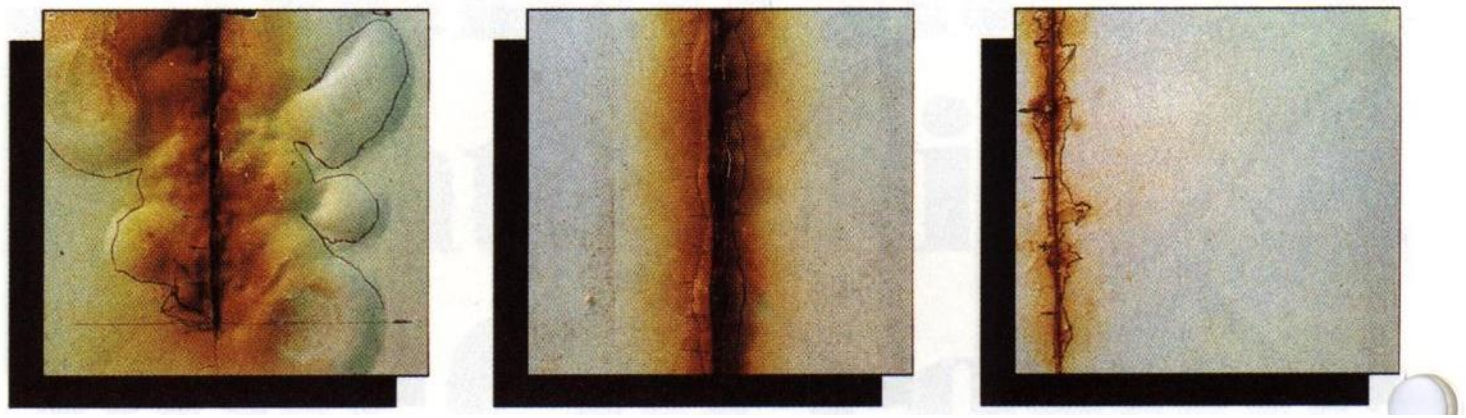
# Combined Corrosion/Weathering

- 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




*ASTM D5894 - QUV & Q-FOG for 2000 hrs*




*27 months outdoor marine environment*

# Wet/Dry Cyclic Test Case Study

## SSPC (Society for Protective Coatings)

- 15 different systems
- Outdoor vs. accelerated
  - 31 months
- Accelerated tests
  - 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 Cycle** | **0.71** |

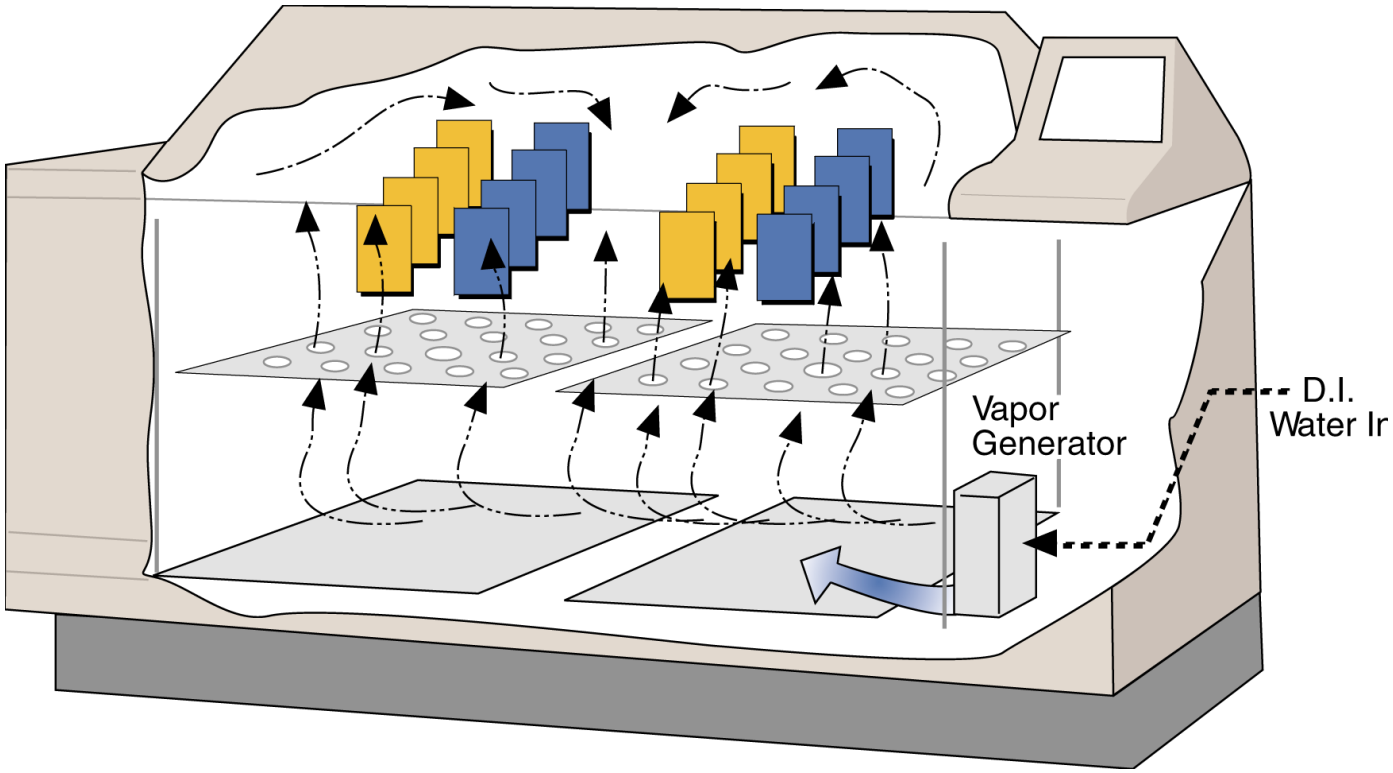
- **Conventional salt spray and Cyclic results effectively random!**
- **Good correlation from combined test**

# Wet/Dry Cyclic Tests
## Limitations

- Poor repeatability and reproducibility
- Poor correlation in some cases
  - Automotive
  - Industrial maintenance coatings on steel
- Attempts to improve correlation & repeatability include…
  - Wet bottom (water retained at chamber bottom)
  - Changing temperature of bubble tower
  - ***Both are crude "workarounds" for poor RH control technology***

# First-Generation Cyclic Automotive Tests

Salt Fog → Dry-Off → **Wetting (Humid)**




Wetting specimens after dry-off reinitializes corrosion

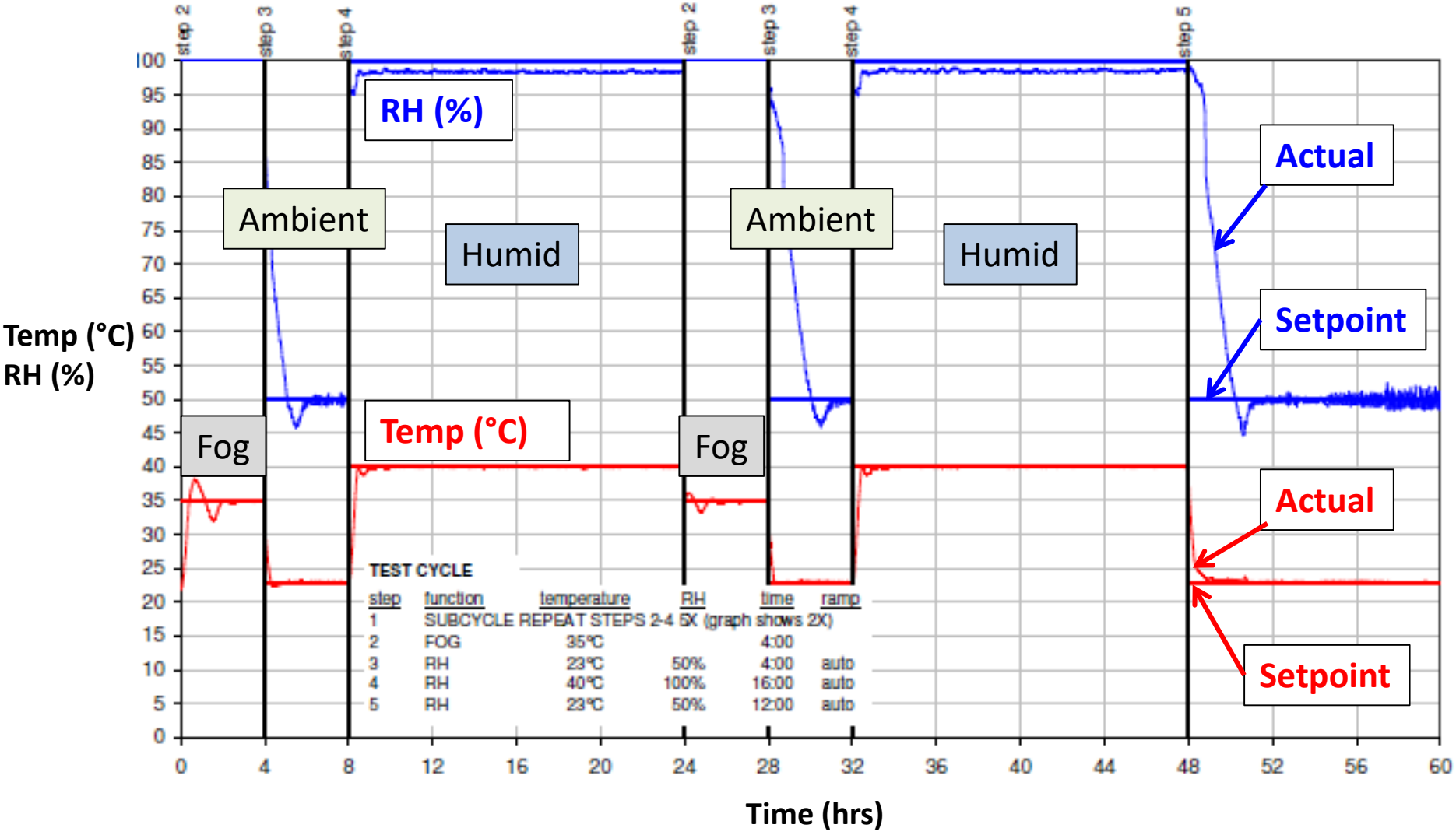
# First-Generation Cyclic Automotive Tests

Salt Fog → Dry-Off → **Wetting (Humid)**

## Example: GM 9540P

- NaCl and $CaCl_2$ to simulate road salts
- Solution applied by direct Spray, not Fog
- Salt spray applied intermittently in “ambient” conditions
- Use of **corrosion coupons** to minimize test variability
- SAE & American Iron & Steel Institute rated this method best predictor of outdoor performance in 1991

# First-Gen Cyclic Corrosion test



# Limitations of First Generation CCT

## Poor Repeatability and Reproducibility!

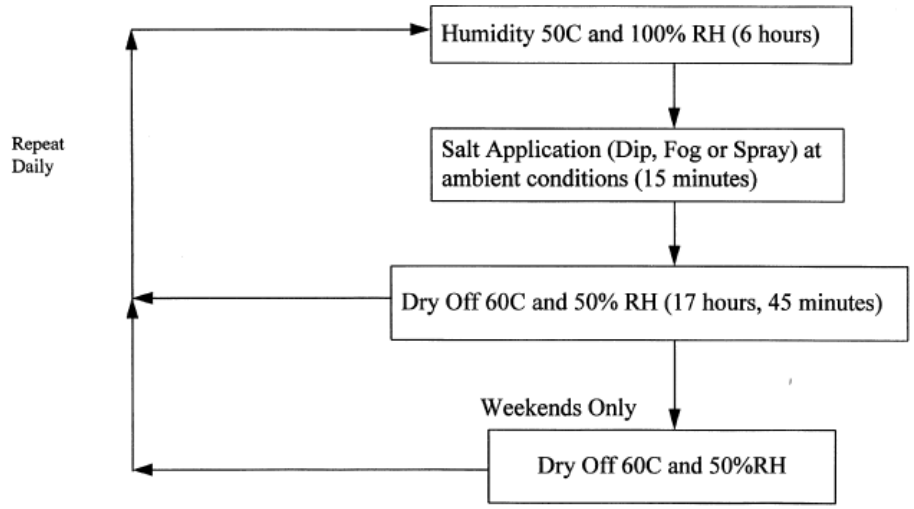
- Different corrosion chambers give different results
- Huge variations in corrosion rates between different metals from test to test

# Case Study  SAE J2334

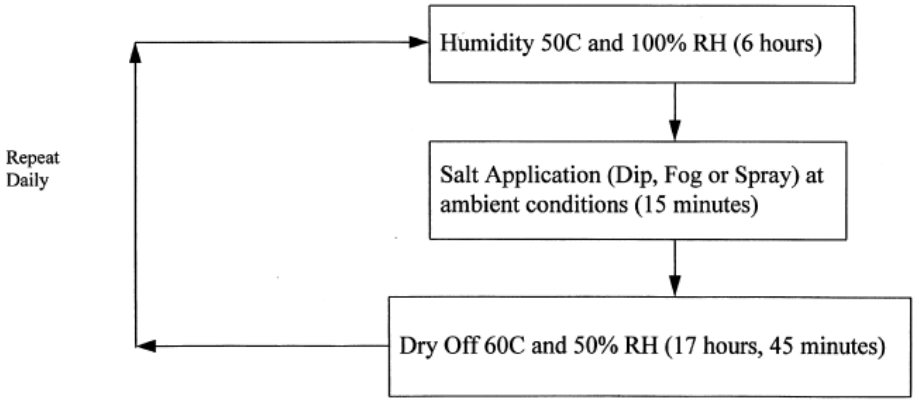
- Transition times are not specified
- Coupon use is encouraged but no mass loss limits are included
- Some companies have implemented J2334 with their own mass loss limits

# SAE J2334

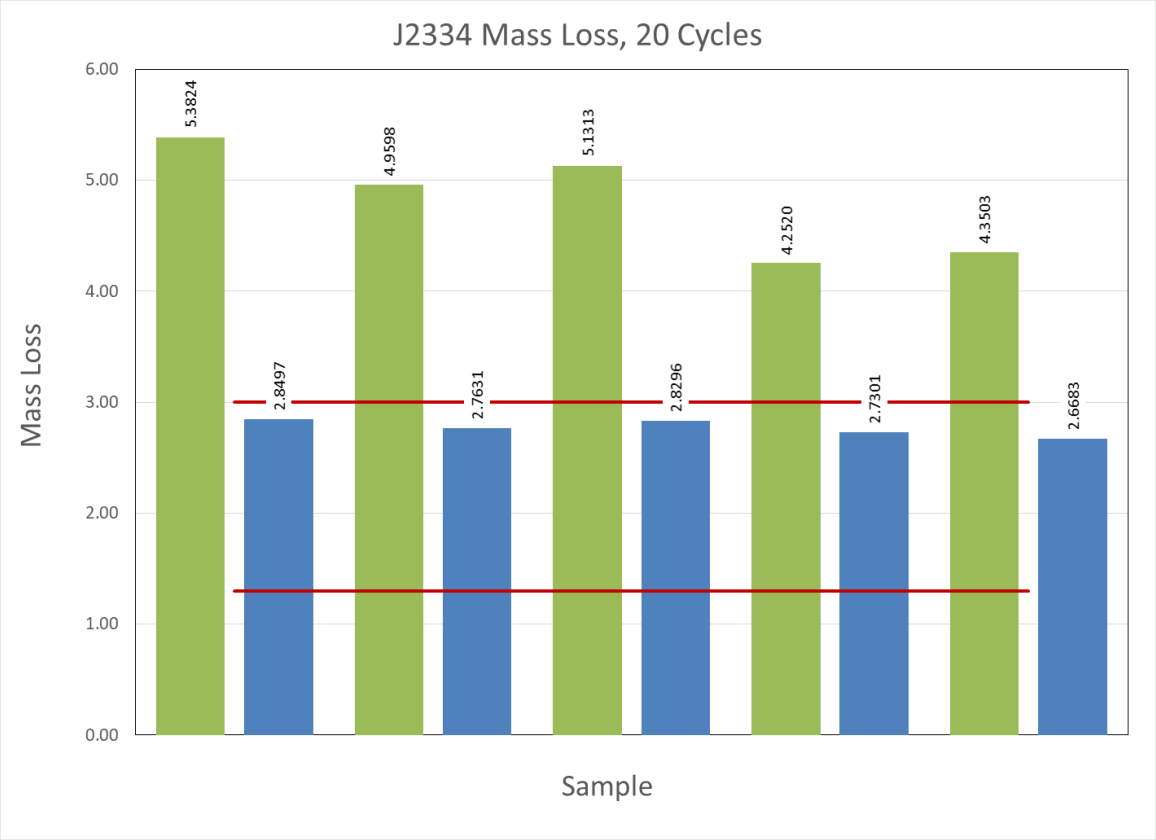
**Cosmetic Corrosion LabTest Cycles**
**SAE J2334 - 5 Day/Week - Manual Operation**




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



# SAE J2334 Coupon Mass Loss




*Green and blue bars represent two different J2334 tests*

# Corrosion rates

## Galvanic corrosion

## Deliquescence of salts

## 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) Examples

| Salt | DRH |
|---|---|
| Potassium Chloride (KCl) | 85% |
| ***Sodium Chloride (NaCl)*** | ***76%*** |
| ***Calcium Chloride ($CaCl_2$)*** | ***31%*** |

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

# 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**

# 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 |

- Affects products made from metals
  - Steel
  - Aluminum
  - Magnesium alloys
- Organic & Inorganic Protective Coatings

# Galvanic Corrosion

# Relative Humidity and Corrosion
## Stainless steel and Al example

| Condition | RH Range | Result |
|---|---|---|
| **Dry** | **≤ 50%** | Very little corrosion from NaCl |
| **Electrolytic cells around salt crystals; film formation as RH increases** | **50-76%** | • Corrosion of **steel** (maximum corroded area ~70% RH) and **aluminum**<br>• AL-Steel galvanic couple broken |
| **Uniform Electrolytic Film formation** | **≥76%** | • Maximum cathode area for steel; deeper non-uniform corrosion<br>• **Al corrosion** in galvanic couple with steel |

# Relative Humidity and Corrosion

## Controlling Step Transition Times

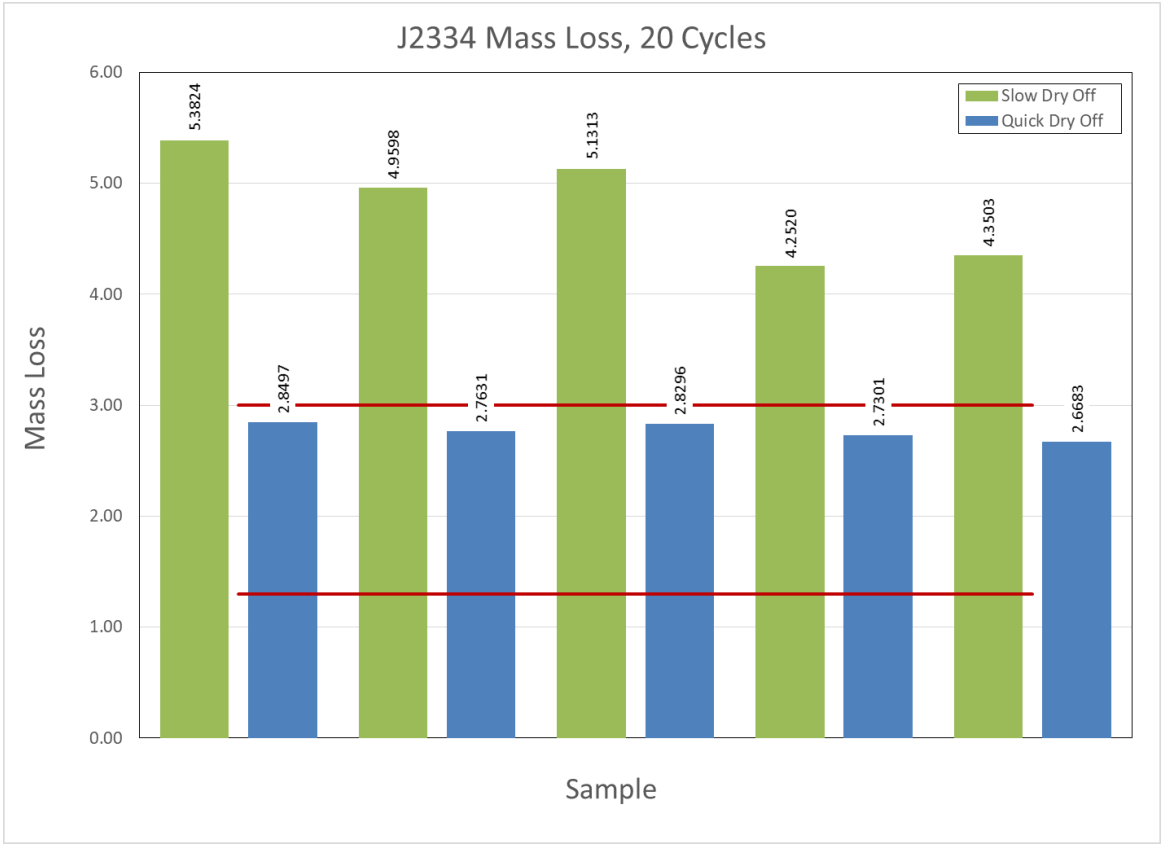
### “Linear” transition

- Specify Time in test cycle to change test conditions
- Tester adjusts temperature & RH for linear transition from beginning to end of ramp time

### “Less Than” transition

- Specify Time in test cycle to change test conditions
- Tester attempts to achieve conditions within specified time – effectively as fast as possible
- Fast “less than” transition times (e.g. JASO M609) designed to **minimize test variability…**

# SAE J2334 Results

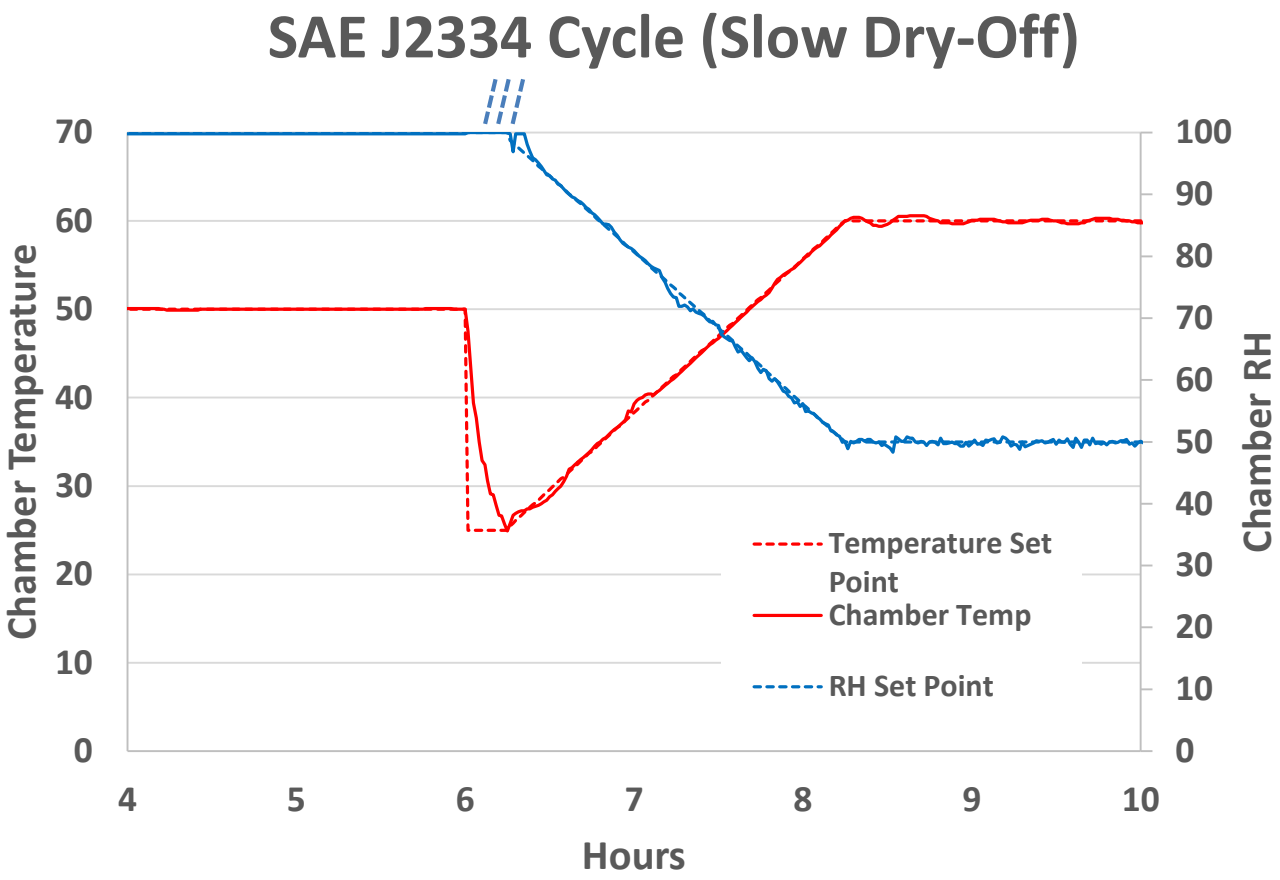



*Green bars represent test under slow dry-off conditions*

*Blue bars represent test under quick dry-off conditions*

*Red lines represent tolerance of OEM standard*

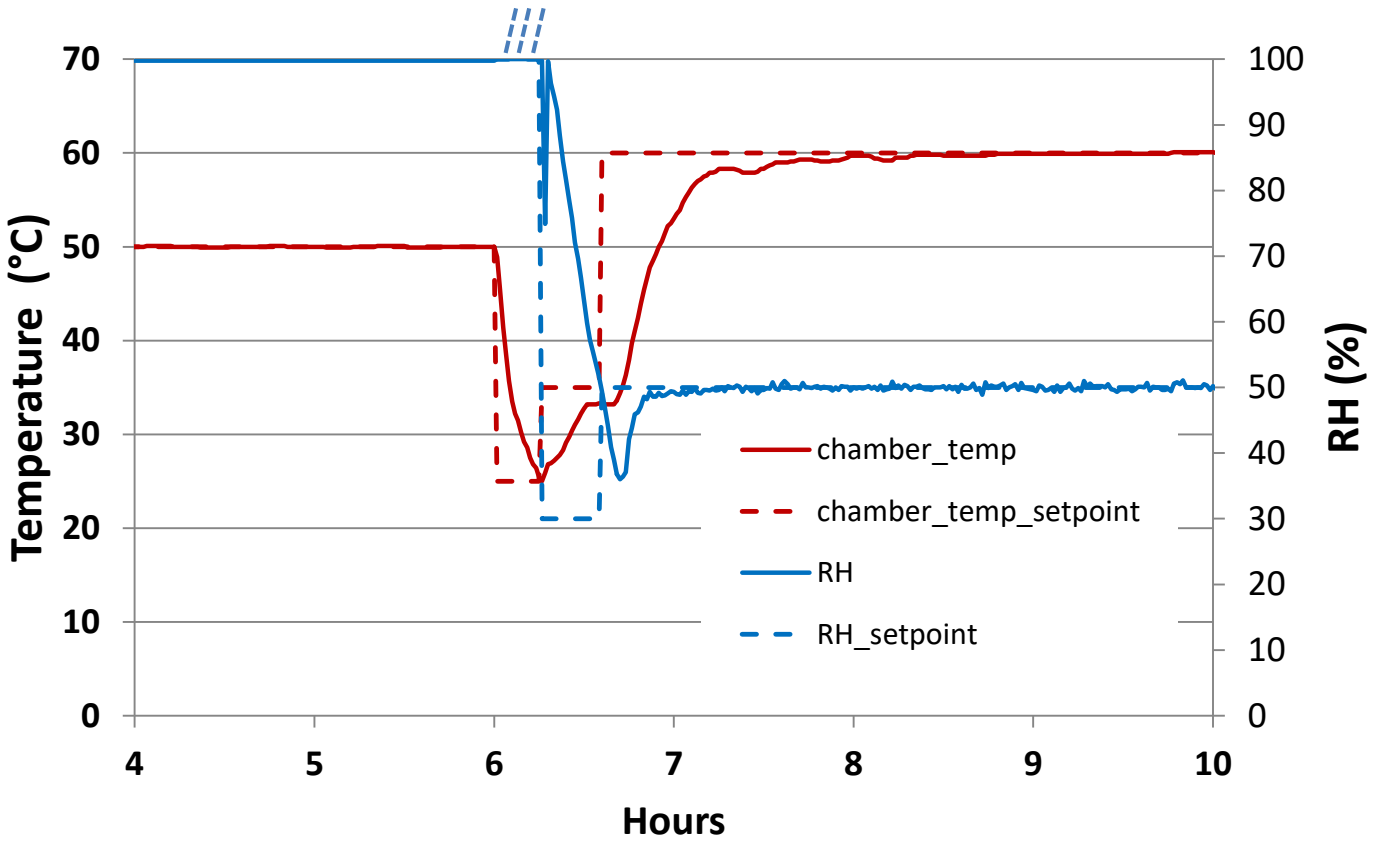
*Under the quick dry test, the coated panels once again passed the test*




*Zoomed in view of the transition*

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

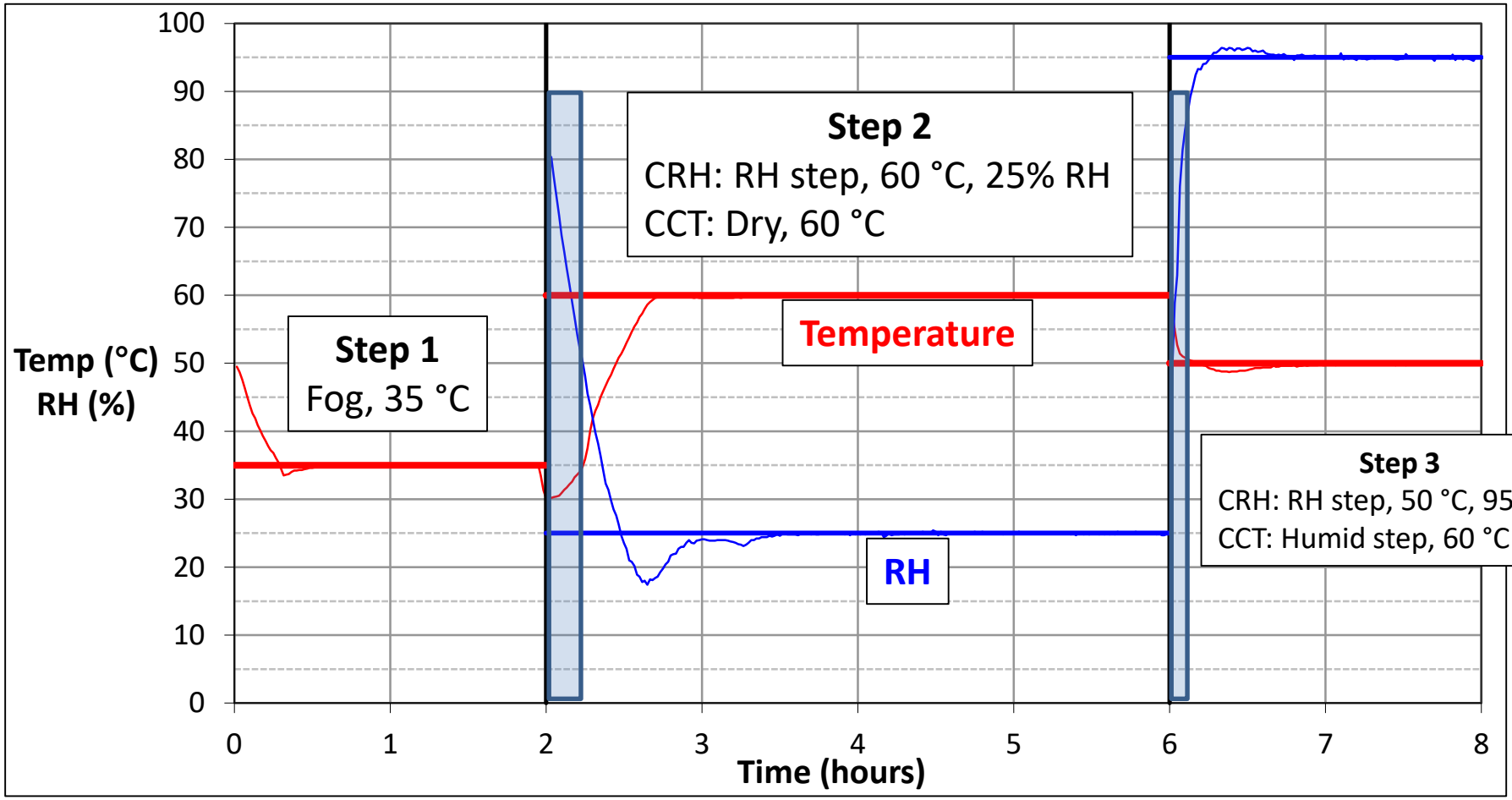
# SAE J2334 Quick Dry-Off




*Zoomed in view of the transition*

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

# JASO M609 (Rapid Transitions)




**Fast transition times designed to improve Reproducibility but…**

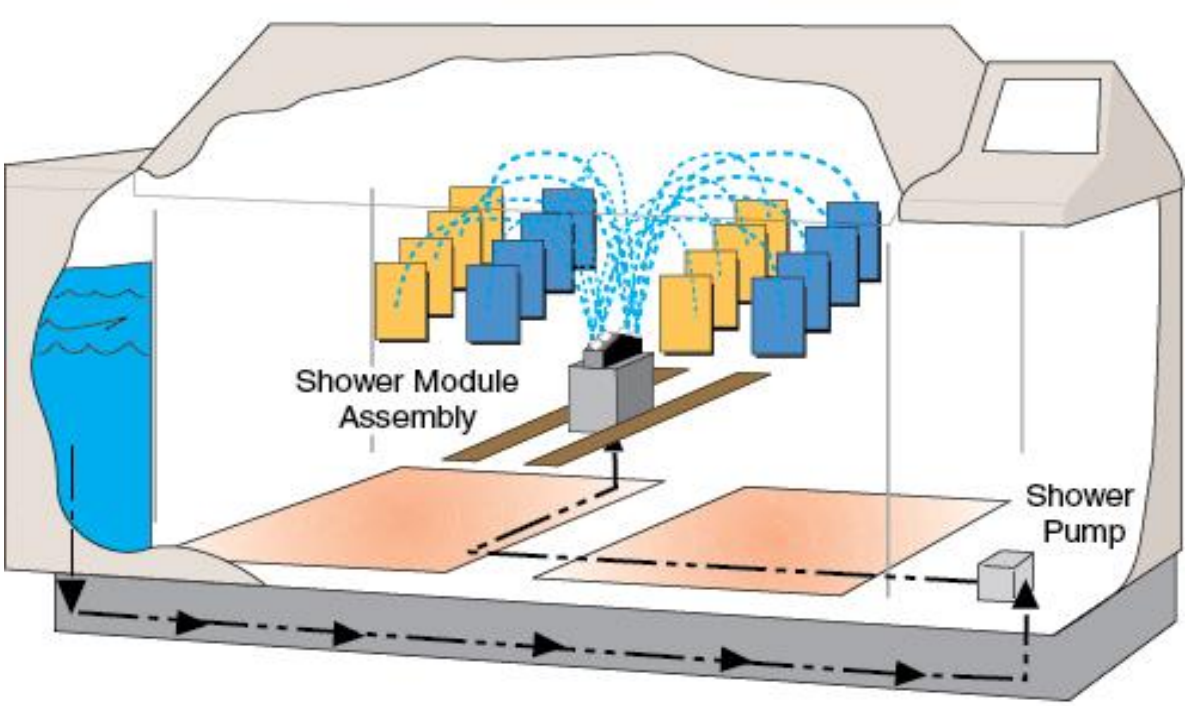
**Very limited time in critical RH zone of 50-90%!**

# First generation cyclic automotive methods: what was missing?

- Lack of comprehensive RH control
  - Conditions limited to full wetting, dry, uncontrolled room/ambient
  - No control of RH transition times – used "workarounds" like fast transition times
  - Variable specimen dry-off rates
  - No RH values in critical transition zones (DRH)
- Slow application of salt solution (fog)
  - Little time for dry-off and re-wetting of specimens

# Modern Corrosion Tests

## Salt Fog/Shower; Dry-Off; Controlled RH




- High-volume spray for faster, controllable specimen wetting
- Precise control of transition times
- **Precise control of Relative Humidity**

# Modern Automotive Corrosion Tests

## Fog

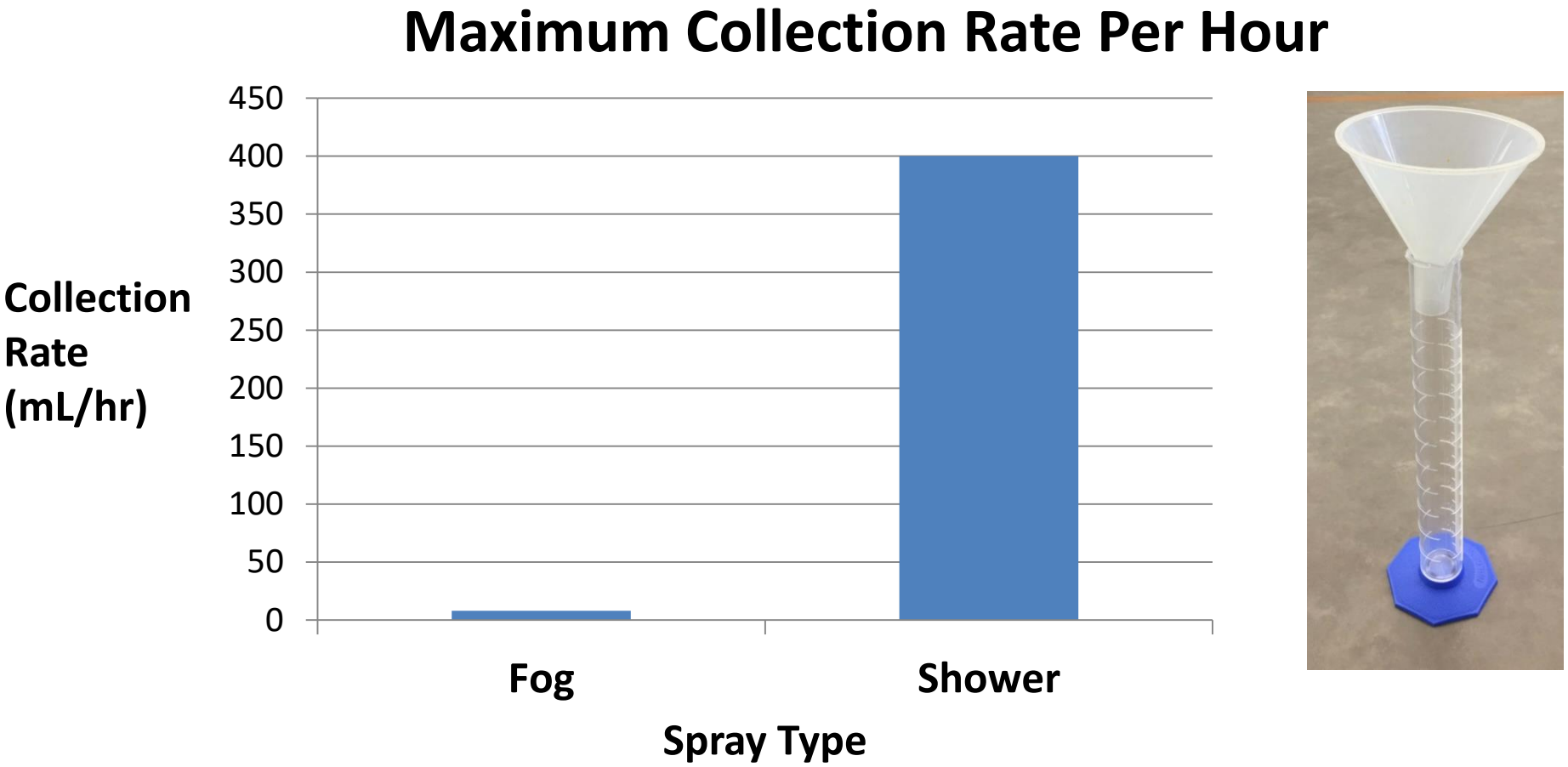
- Toyota TSH1555G
- VDA 233-102
- Renault D17 2028 (ECC1)

## Shower

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

# Corrosion Test Control

## Pluviometry



# Environmental Transitions in Today's Standards: Two Approaches

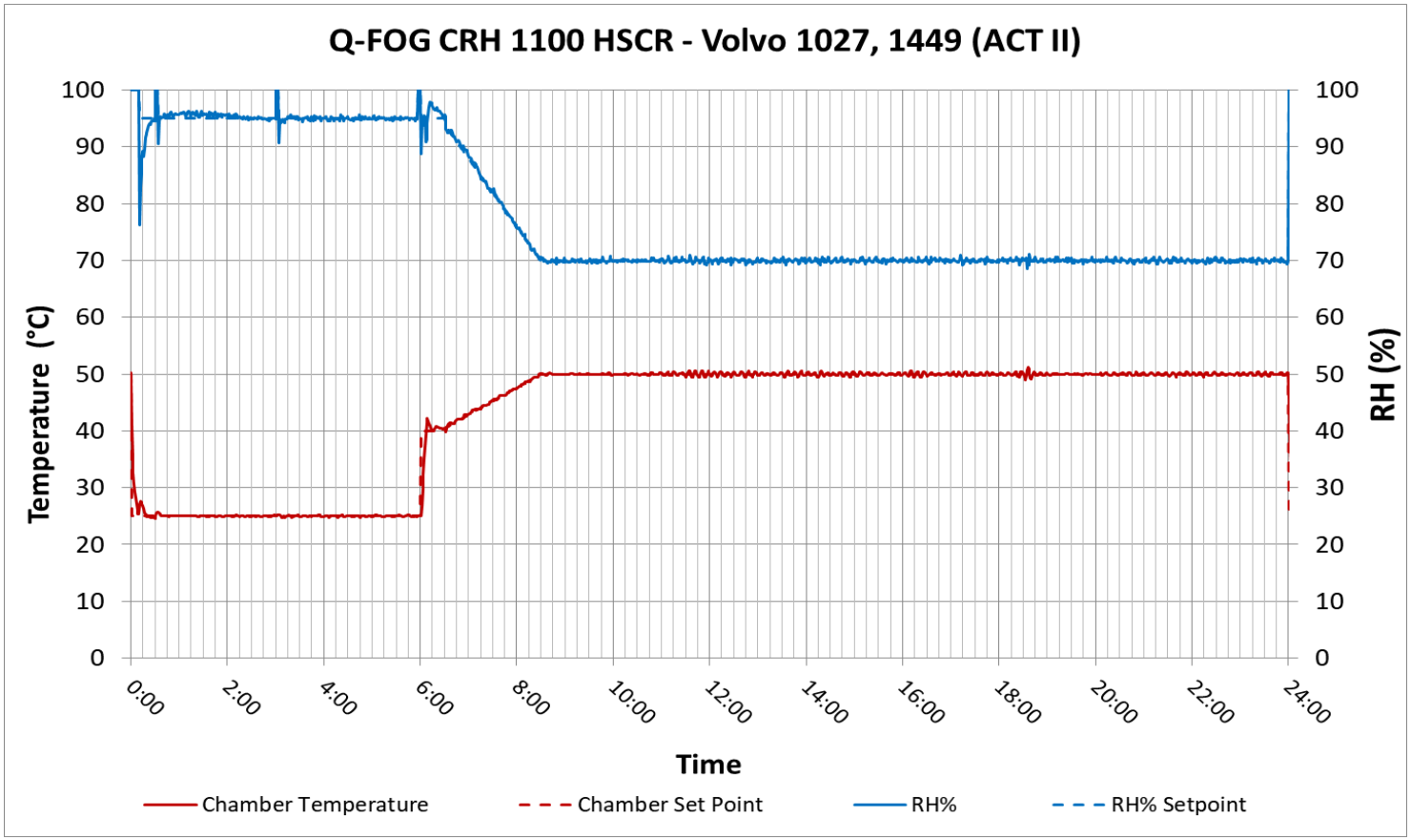
Rapid (e.g. <30 minutes wet to dry)

- Japanese Car Companies
- CCT I, II, IV, JASO M609
- Renault ECC1

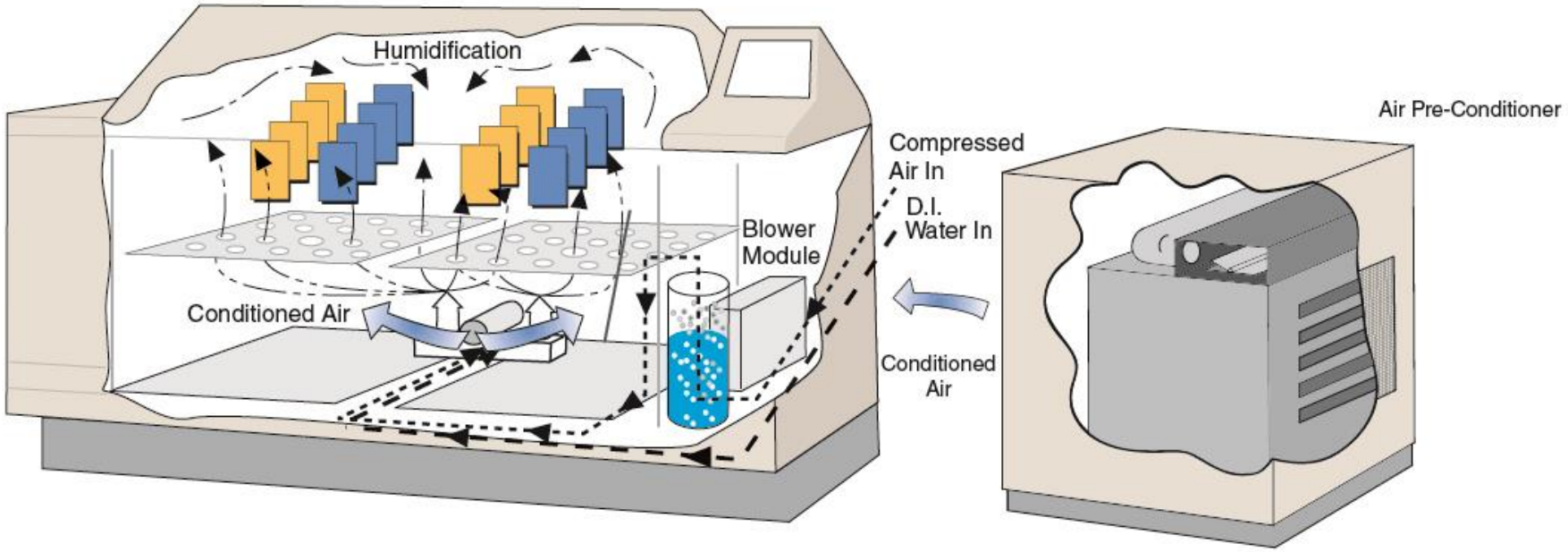
Controlled/Linear

- Volvo ACT1
- Volvo ACT2/Ford L-467
- GMW 14872
- Renault ECC1
- VDA 233-102

# Volvo VCS 1027, 1449 (ACT-II)/Ford L-467



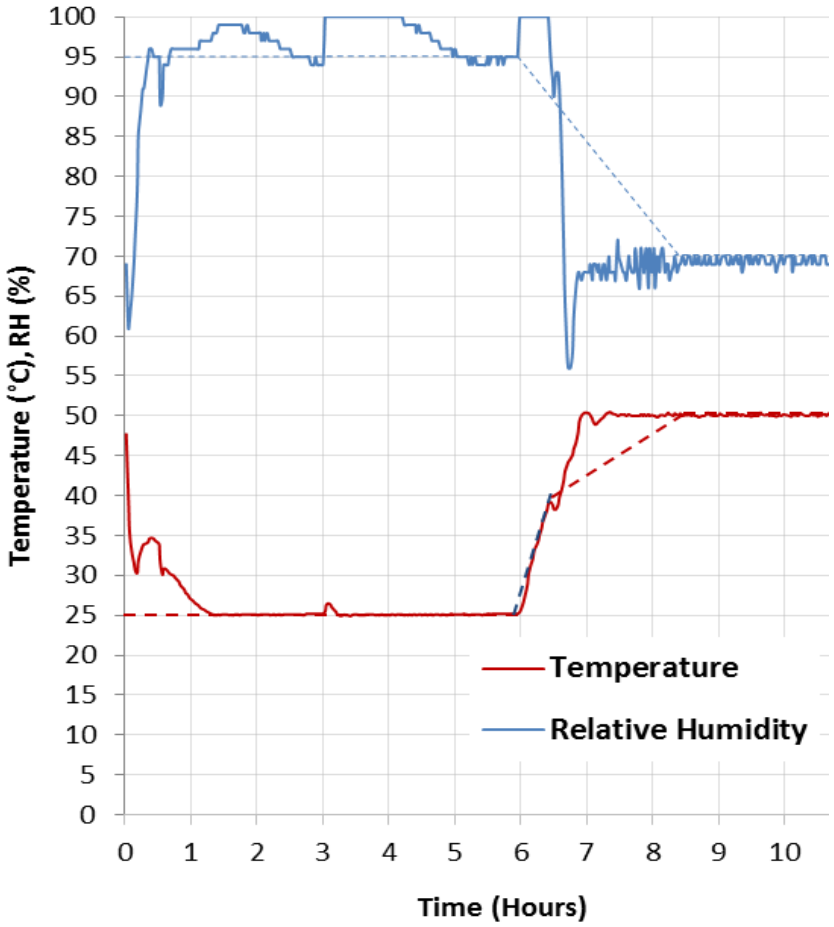
# Corrosion Test Control
## Air Pre-Conditioner



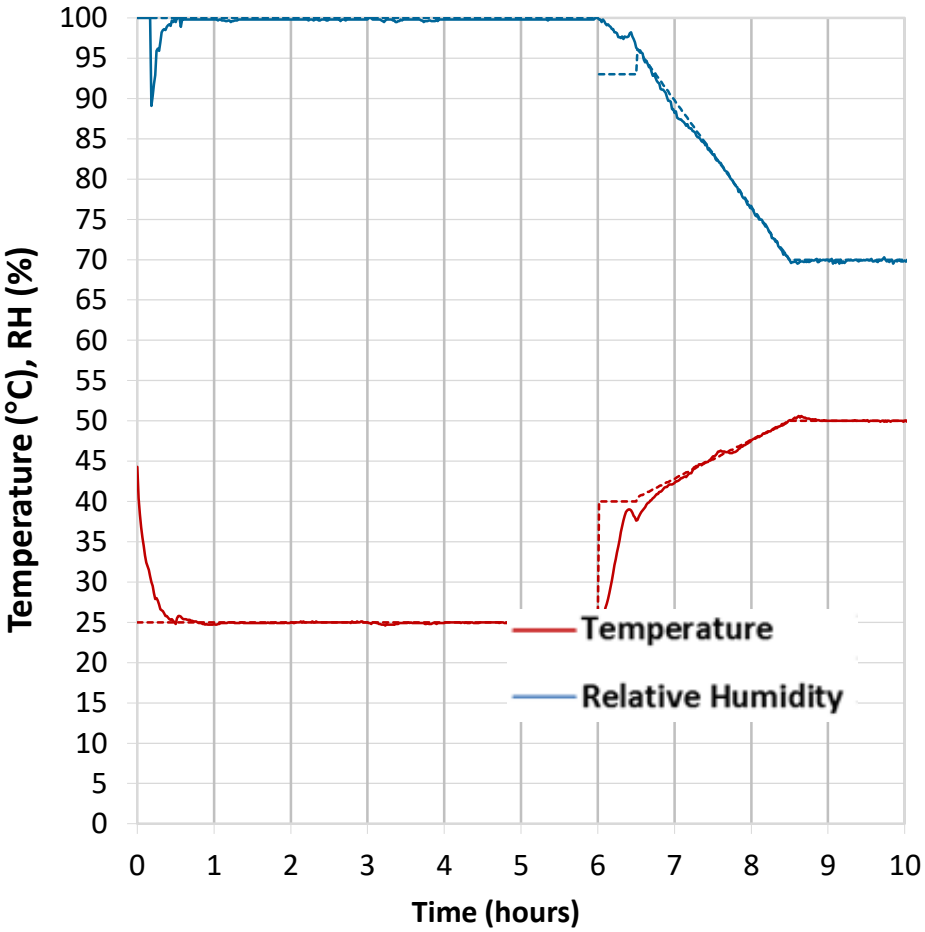

- Accurate control of “ambient” conditions
- Accurate Ramping of Temperature & Humidity

# Performance Improvement with Air Preconditioner

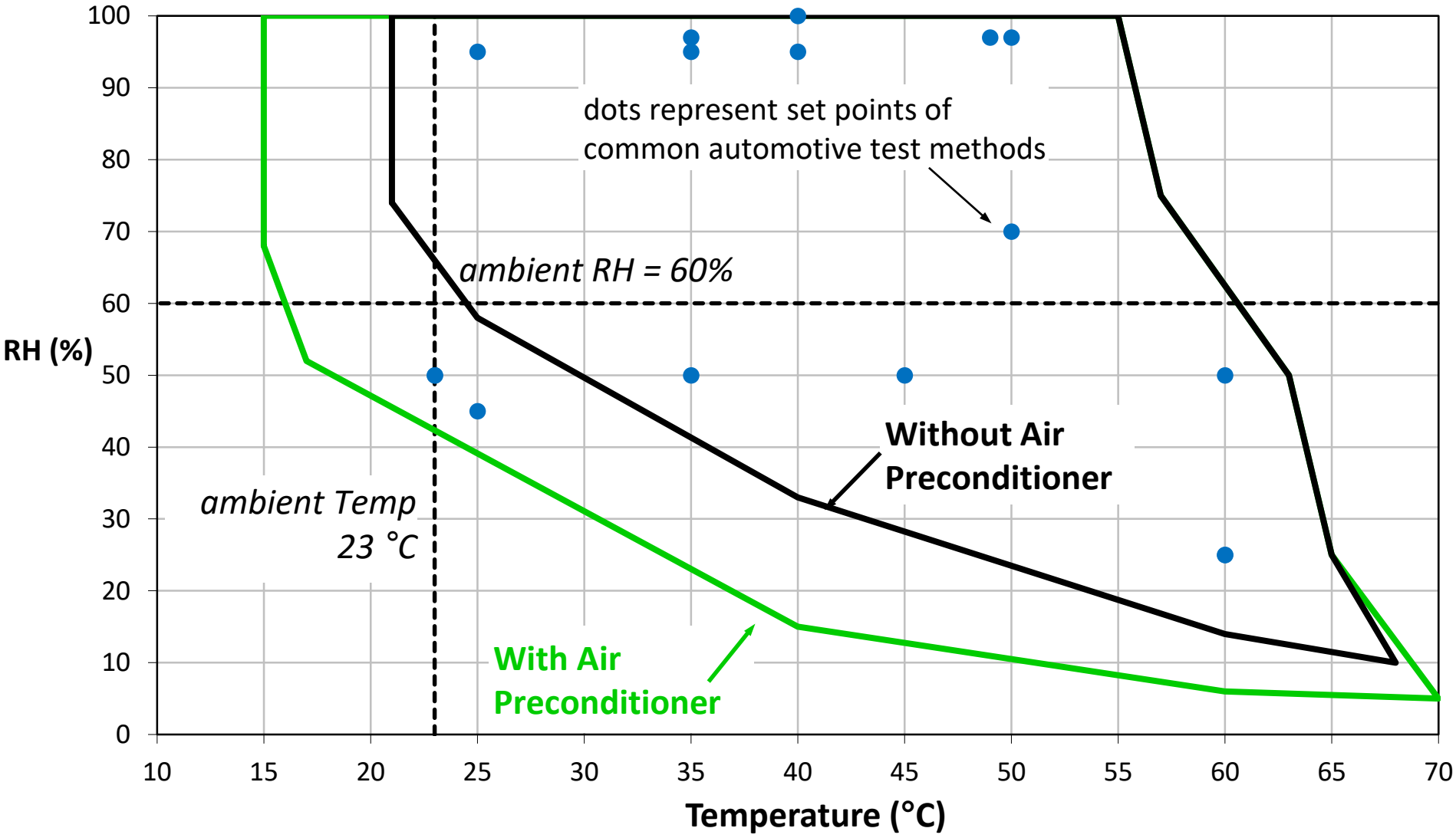
## No Air Preconditioner




## With Air Preconditioner



# Corrosion Test Operational Range



# 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 attention!

# Questions?

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