Correlation in Accelerated Testing

Principles, Challenges and Case Studies

Click here to view the 8 AM EST presentation

Click here to view the 12 PM EST presentation

Q-Lab Corporation
The Question

In weathering and corrosion, we encounter the same basic question over and over again ...

“How many hours in my accelerated test correlates to ___ years of outdoor service?”
The Hard Truth

There is no Universal Acceleration Factor, or “Magic Number,” between accelerated and outdoor testing

• Different materials in different service environments have different acceleration factors

• Weathering and Corrosion Tests do not give quantitative predictions of Service Life
Why is this such a challenge?

• The problem is not that we just haven’t developed the perfect weathering tester yet.

• The biggest problem is the inherent variability and complexity of outdoor exposures. Consider just some of the many factors in relationships between outdoor and accelerated tests:

  
  **Outdoor factors**

  1. Latitude
  2. Altitude
  3. Geography
  4. Year-to-year variations
  5. Seasonal variations
  6. Specimen Orientation
  7. Environmental particulates

  
  **Laboratory factors**

  8. Specimen insulation
  9. Test cycle
  10. Water delivery
  11. Test temperatures
  12. Light source

  
  **And of course…**

  13. The particular materials system tested
What Can Be Done

• Weathering and corrosion testing can have many goals other than determining acceleration factors and service life.

• Determine what you need to know for your materials and select an appropriate test program.

• Although weathering and corrosion tests usually are not *predictive*, they can often be *correlative*.

• Weathering and corrosion tests are comparative, and comparative data can be powerful.
Accelerated Testing is a Tool for Decision Making

Accelerated tests can help you ...

– What ingredients to include or not include in a product

– Whether a lot or batch is OK to ship to customers

– What vendors to buy from

– What processing and manufacturing parameters should be selected

– Make better, faster decisions
# Accelerated Test Types

## What do we want to learn?

<table>
<thead>
<tr>
<th>Accelerated Test Type</th>
<th>Result</th>
<th>Test Time</th>
<th>Results compared to</th>
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<tbody>
<tr>
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Why is correlation such a challenge?

Simulation and Acceleration

Degree

- Acceleration
- Simulation
- Typical Correlation
Correlation

The degree to which sets of data from separate tests agree with one another

– Accelerated vs outdoor weathering
– One accelerated test method vs another
– One outdoor environment vs another
Why Correlation Matters

• Decision-making tools need to be validated

• There is an inherent conflict between acceleration and realism

• The only way to validate an accelerated weathering test is with outdoor/real world data

• In other words ... Test the Test!
Methods for Establishing Correlation

Two main methods for correlating two tests (usually outdoor and accelerated)

- Reference and Control Materials
- Rank Order Evaluation
Reference and Control Materials

Reference Materials

Standard Reference Materials
- Known performance in test environments
- Not necessarily similar to test specimens
- Performance may not match test specimens
- Verify that lab tester is operating properly

Control Materials
- Similar characteristics to test specimens
- May be your products or competitors’
- Give confidence in lab exposure

Correlation in Accelerated Testing
We make testing simple.
Standard Reference Material
Example: Polystyrene (PS) yellowing for SAE J2527

Reference Polystyrene yellowing validates tester performance
Control Material Guidelines

• Control materials must have known durability
  – Outdoor performance
  – Lab performance

• Similar composition to test material

• Similar degradation mode to test material

• Best practice to include both weak- and strong-performing control materials
Corrosion Coupons

- Standardized metal specimens
- Mass loss due to corrosion is measured during a test
- 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
Mass Loss Tolerances in GMW 14872

- **Underbody (Exp A,D,E) Method 1/2 - 4 salt sprays/cycle**
- **Underbody (Exp A,D,E) Method 3 - 4 salt sprays/cycle**
- **Underhood (Exp B,D,E) Method 1/2 - 4 salt sprays/cycle**
- **Underhood (Exp B,D,E) Method 3 - 4 salt sprays/cycle**
- **Exterior (Exp C,D,E) Method 1/2/3 - 4 salt sprays/cycle**

<table>
<thead>
<tr>
<th>STAGE</th>
<th>STEP</th>
<th>TYPE</th>
<th>TEMP (°C)</th>
<th>RH (%)</th>
<th>TIME</th>
<th>RAMP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>1</td>
<td>subcycle</td>
<td>repeat steps 2 &amp; 4 4x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ambient</td>
<td>2</td>
<td>RH</td>
<td>25</td>
<td>45</td>
<td>27 min</td>
<td></td>
</tr>
<tr>
<td>Ambient</td>
<td>3</td>
<td>Shower</td>
<td>25</td>
<td>45</td>
<td>3 min</td>
<td></td>
</tr>
<tr>
<td>Ambient</td>
<td>4</td>
<td>RH</td>
<td>25</td>
<td>45</td>
<td>90 min</td>
<td></td>
</tr>
<tr>
<td>Humid</td>
<td>5</td>
<td>RH</td>
<td>49</td>
<td>100</td>
<td>7.5 hr</td>
<td>1 hr linear</td>
</tr>
<tr>
<td>Humid</td>
<td>6</td>
<td>RH</td>
<td>49</td>
<td>95</td>
<td>30 min</td>
<td></td>
</tr>
<tr>
<td>Dry</td>
<td>7</td>
<td>RH</td>
<td>60</td>
<td>25</td>
<td>8 hr</td>
<td>3 hr linear</td>
</tr>
<tr>
<td>Dry</td>
<td>8</td>
<td>Final</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Rank Order Correlation

• Rank materials from best to worst outdoors and in lab test

• Calculate correlation coefficient using **Spearman’s Rank Correlation Coefficient**
  – Quantitative measure of how well the lab test matches outdoors
  – Correlation of 1 is perfect (so is -1, in a way)
  – Correlation of 0 is random
Rank Ordering
Spearman Coefficient

Spearman coefficient: 1.0

Spearman coefficient: 0.35
Rank Order Correlation Benefits

• Determines or confirm relationship between different exposure techniques

• Develops confidence in realism of lab techniques

• Provides a basis for directional decision-making in research and development
Why not Pearson’s Product-Moment Correlation?

- Pearson’s compares two variables for fit (e.g. exposure length and degradation)

- Since most degradation mechanisms are non-linear, Pearson’s coefficient is usually poor

- May still be useful in reformulation, once a test is verified with Rank Order Correlation!
Perfect Correlation

Perfect correlation between Accelerated and Outdoor performance is rarely observed
Correlation Case Study #1:

Flexible Bulk Intermediate Containers (FIBC)
Flexible Intermediate Bulk Containers (FIBC)

Situation

FIBCs are used to carry goods. They need to survive at a job site for up to 12 months without losing tensile strength.

Various test methods with Xenon and Fluorescent UV were compared to outdoor performance.
FIBC results: Outdoor/Accelerated Correlation

Very good pass/fail correlation between accelerated and outdoor weathering
Every specimen that survived >400 hours accelerated survived 9 mo outdoors
FIBC Correlation Conclusions

• Xenon arc and fluorescent accelerated testing both provided good correlation to outdoor evaluation

• Realistic light sources (UVA fluorescent, Daylight filtered xenon arc) gave strength retention results that can be correlated to outdoor exposure on a radiant dosage basis
  – Acceleration factor ~7: >250 hours xenon testing correlated to 2.5 months in Florida

• Pass/fail behavior of FIBC over 6-9 months predicted well by UVB-313 fluorescent test
  – Acceleration factor ~16: >400 hours lab testing correlated to 9 months outdoors. Pass/fail testing can often be faster!
Correlation Case Study #2:

Artists’ Colored Pencils
Colored Pencils Correlation Study

Background

– There was no standard to distinguish colored pencils’ light stability

Objective

– Develop standard and determine correlation between natural and accelerated exposures
– Property measured is delta E – total color change
Colored Pencils Correlation Study
Xenon accelerated test data

<table>
<thead>
<tr>
<th>Color</th>
<th>delta E</th>
<th>Color</th>
<th>delta E</th>
<th>Color</th>
<th>delta E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red-1</td>
<td>5.7</td>
<td>Yellow</td>
<td>45.6</td>
<td>Blue-1</td>
<td>10.9</td>
</tr>
<tr>
<td>Red-2</td>
<td>26.7</td>
<td>Green-1</td>
<td>6.1</td>
<td>Blue-1</td>
<td>11.2</td>
</tr>
<tr>
<td>Orange-1</td>
<td>79.7</td>
<td>Green-2</td>
<td>5.8</td>
<td>Blue-2</td>
<td>26.8</td>
</tr>
<tr>
<td>Orange-1</td>
<td>79.3</td>
<td>Green-2</td>
<td>7.9</td>
<td>Blue-2</td>
<td>28.2</td>
</tr>
<tr>
<td>Orange-2</td>
<td>34.8</td>
<td>Green-3</td>
<td>19.3</td>
<td>Purple-1</td>
<td>23.0</td>
</tr>
<tr>
<td>Orange-2</td>
<td>34.8</td>
<td>Green-3</td>
<td>19.9</td>
<td>Purple-2</td>
<td>23.1</td>
</tr>
<tr>
<td>Beige</td>
<td>19.7</td>
<td>Aqua</td>
<td>5.8</td>
<td>Purple-2</td>
<td>22.9</td>
</tr>
<tr>
<td>Beige</td>
<td>19.7</td>
<td>Aqua</td>
<td>5.7</td>
<td>Black</td>
<td>2.7</td>
</tr>
</tbody>
</table>

15 materials – a minimum of 10 (better if 20!) needed for correlation
**Colored Pencil Correlation Study**
Comparison of accelerated to outdoor

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Arizona Under Glass</th>
<th>Florida Under Glass</th>
<th>Xenon</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ΔE</td>
<td>Rank</td>
<td>ΔE</td>
</tr>
<tr>
<td>Red Pigment A</td>
<td>10.9</td>
<td>1</td>
<td>1.3</td>
</tr>
<tr>
<td>Red Pigment B</td>
<td>45.8</td>
<td>2</td>
<td>36.6</td>
</tr>
<tr>
<td>Orange Pigment</td>
<td>79.9</td>
<td>3</td>
<td>80.4</td>
</tr>
</tbody>
</table>
Results - Rank Order Correlation

<table>
<thead>
<tr>
<th>Test Rankings Being Compared</th>
<th>Spearman’s Rank Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arizona – Florida</td>
<td>0.94</td>
</tr>
<tr>
<td>Xenon – Arizona</td>
<td>0.95</td>
</tr>
<tr>
<td>Xenon – Florida</td>
<td>0.93</td>
</tr>
</tbody>
</table>

Excellent rank order correlation between natural and accelerated exposure results of all of the specimens.
Correlation Case Study #3: Lithographic Inks
Printing Ink Correlative Study

Purpose

- Evaluate the light stability of lithographic inks

Test Program

- Natural outdoor tests
- Q-SUN Xenon Arc tests
Printing Ink Correlation Study
delta E Color Fade Measurements

Florida

Q-SUN
Conclusions

• Excellent **rank order correlation** between outdoor & lab results

• Test technique can be applied to any ink, ink/substrate combination

• Acceleration factor ~3.5 for these materials under these test conditions
Correlation Case Study #4:

Automotive Coatings tested with ASTM D7869
Transportation Coatings Correlation Study

Situation

- Widely-used accelerated test standard for auto coatings, SAE J2527, did not reproduce all common outdoor failures. Poor correlation!
- ASTM D7869 developed to provide better correlation between outdoor results and accelerated laboratory tests
- More realistic acceleration of light, heat, water

Test Program

- Over 100 automotive and aerospace coating systems exposed outdoors for two years (~16000 hours)
- Accelerated testing conducted to 3000 kJ (~1800 hours ASTM D7869, ~2300 hours SAE J2527)
Correlation results: Control material

Florida Exposure

Expected Failure Mode: None – positive control
Observed: Excellent performance in all tests
Correlation results: Coating removal

**Florida Exposure**

**J2527**

**ASTM D7869**

**Expected Failure Mode:** Slight BC/E-coat pick off

**Observed:** Slight BC/E-coat pick-off ASTM D7869, not SAE J2527
Correlation results: Delamination

Expected Failure Mode: Blistering, gloss loss, adhesion loss
Observed: Gloss and adhesion loss on both. Blistering ASTM D7869
Correlation results: Blistering

Expected Failure Mode: Blistering, gloss loss, adhesion loss

Observed: Gloss loss and adhesion loss seen on all panels. Blistering on ASTM D7869 mimics that seen on Florida Exposure.
**Correlation Study for Transportation Coatings**

- ASTM D7869 reproduced all major outdoor failure mechanisms – critical for correlation

- **Correlating** degradation between exposures is only valid when the type of degradation is the same!

- 1800 hour accelerated laboratory test results matched well two-year Florida exposures for many coatings systems (acceleration factor ~10)

- May be applicable to other materials systems but **outdoor data has to be collected to verify this**
Correlation Case Study #5:

Vinyl Siding
What is Vinyl Siding?

• Co-extruded building cladding material
  – Manufactured mostly from Polyvinyl Chloride (PVC)
  – Top layer (capstock) is durable and UV-stabilized
  – Also known as uPVC Weatherboarding in some regions

• Developed in the 1960’s, became popular in the 1970’s

• Most common residential exterior cladding material in US & Canada – about 20 million m² used per year
Vinyl Siding Institute
Outdoor test program

• Large-scale, long-term study
• Outdoor data collection ongoing since 1984
• New tests started every 5 years; thousands of specimens and replicates tested
• Long-term material degradation mechanisms are now well understood

Correlation here is between short- and long-term outdoor testing
Vinyl Siding Institute
Service Life Certification

• Accurate service life *estimate* based on 2-year outdoor testing
  – If after 2 years of exposure, color change is <1, then after 25 years it has a high probability of color change <4
  – Acceleration for service life prediction of 12:1

• 2 year outdoor certification program
  – Administered by ISO 17025-accredited, independent 3rd party
  – Exposures in FL, AZ, OH
  – Tests performed in accordance with ASTM test standards
  – Receive a VSI stamp, gives credibility to a 25-year warranty
Qualification / Correlation Case Study
Vinyl Siding Institute (VSI)

- **New Goal**: Correlate accelerated test to 2-year outdoor results
- Six rounds of accelerated testing conducted by multiple labs – examined test cycles of both UV fluorescent and xenon
- Unique Fluorescent UV cycle provided best correlation for PVC siding material
  - Hot condensation best for accelerating realistic moisture attack synergistically with UV
  - Long wave and visible had little impact
  - Reduced UV temps and increase condensation temps gave better results
- UV fluorescent test not adopted for certification program, but used by members for product development
Correlation Case Study #6:

Corrosion of Protective Coatings
Correlation in Corrosion Testing of Coatings

- Conducted by SSPC (Society for Protective Coatings)
- 15 different systems
- Outdoor vs. accelerated
  - 27 months outdoor, 2000 hours lab
- Accelerated tests
  - Salt spray 5%
  - Prohesion
  - Two (2) types of cyclic immersion
  - Combined corrosion/ weathering
Combined Corrosion/Weathering

- Developed in the 1980s by Sherwin Williams
- ASTM D5894
- ISO 11997-2
Combined Corrosion/Weathering

As a coating degrades from UV exposure, its ability to protect against corrosion is reduced
Correlation in Accelerated Testing

Corrosion / Weathering Test Conditions

1 week “Prohesion” corrosion test
- 1 hour salt fog application at 25°C (or ambient)
- 1 hour dry-off at 35°C

1 week QUV exposure
- 4 hours UV exposure, UVA 340, at 60°C
- 4 hours condensation (pure water), at 50°C

Specimens are moved manually between testers.
Corrosion/weathering testing
ISO 12944-6 and -9

<table>
<thead>
<tr>
<th>Day 1</th>
<th>Day 2</th>
<th>Day 3</th>
<th>Day 4</th>
<th>Day 5</th>
<th>Day 6</th>
<th>Day 7</th>
</tr>
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</table>

Similar to the Sherwin-Williams/ASTM D5894 method, except:

- There is more frequent changing of exposure conditions
- The method uses continuous salt spray instead of Prohesion
Combined Corrosion/Weathering vs Outdoors
 Excellent correlation

QUV + Q-FOG
ASTM D5894
2000 hours

Outdoor marine environment
27 months
## SSPC Test Results

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<tr>
<th>Laboratory Test Method</th>
<th>Correlation w/Severe Marine Environment</th>
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<tr>
<td>Conventional Salt Spray</td>
<td>-0.11</td>
</tr>
<tr>
<td>Prohesion</td>
<td>0.07</td>
</tr>
<tr>
<td>Cyclic Immersion Procedures</td>
<td>0.48</td>
</tr>
<tr>
<td>Cyclic Immersion with UV Procedure</td>
<td>0.61</td>
</tr>
<tr>
<td><strong>Combined Corrosion/Weathering Cycle</strong></td>
<td><strong>0.71</strong></td>
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**Good correlation from combined test**
Summary of Correlative Testing
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<td>• Medium</td>
<td></td>
</tr>
<tr>
<td>Predictive</td>
<td>Service life</td>
<td>• Open-ended</td>
<td>Natural exposure (Service environment)</td>
</tr>
<tr>
<td></td>
<td>Acceleration factor</td>
<td>• Long</td>
<td></td>
</tr>
</tbody>
</table>
What did we learn from those correlation case studies?

All of the acceleration factors were different! They are not general or universal and they depend on:

1. The specific material tested.
2. The type of test being correlated to natural outdoor results – fluorescent UV, xenon, accelerated outdoors
3. The specific set of lab tester time cycles and temperature.
4. The specific outdoor exposure site and sample mounting procedure
5. The failure mechanism(s) being evaluated
Correlation between outdoor and accelerated testing can be determined for a variety of materials systems. However...

- Acceleration factors are not general and often only valid for one type of degradation
- Comparative testing usually gives rank-ordered data, which can be powerful data
- It is critical to perform outdoor testing to validate accelerated testing - “Test the Test”
Thank you for your attention!

Questions?

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