Correlation in Accelerated Testing

Principles, Challenges, and Case Studies

Andy Francis	Marketing Director
Dave Duecker	Senior Technical Marketing Specialist
Bill Tobin	Senior Technical Marketing Specialist
Sean Fowler	Senior Technical Director

Q-Lab Corporation

View Recorded Presentation

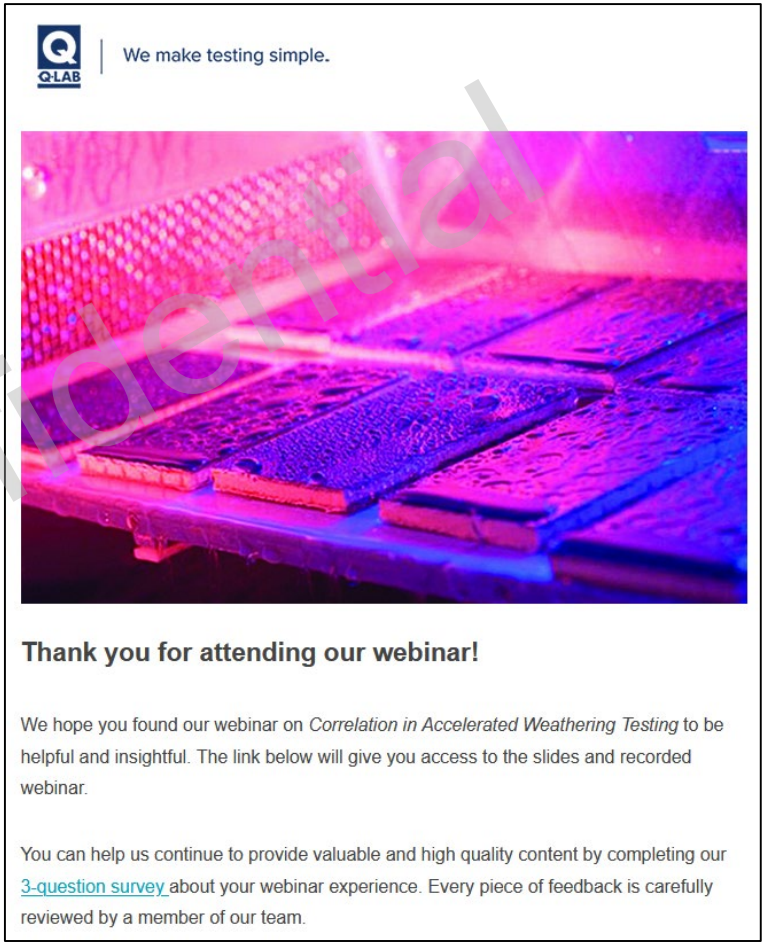
Q-Lab's Weathering Webinar Series

- Today is the last of our five-part webinar series on special weathering testing topics
- Our upcoming and archived webinars are hosted at: q-lab.com/webinars

Date	Topic
14 Apr	Automotive Interior and Exterior Weathering Testing
21 Apr	Modern Automotive Weathering Test: ASTM D7869
28 Apr	Light Stability Testing of Home and Personal Care Products
05 May	Water Delivery in Accelerated Weathering Testing
12 May	Correlation in Accelerated Weathering and Corrosion Testing

Housekeeping

- You'll receive a follow-up email from info@email.q-lab.com with links to a survey, registration for future webinars, and to download the slides
- Use the **Q&A feature in Zoom** to ask us questions today!



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.
- Define goals, set expectations, and from there 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 decide …

- 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?

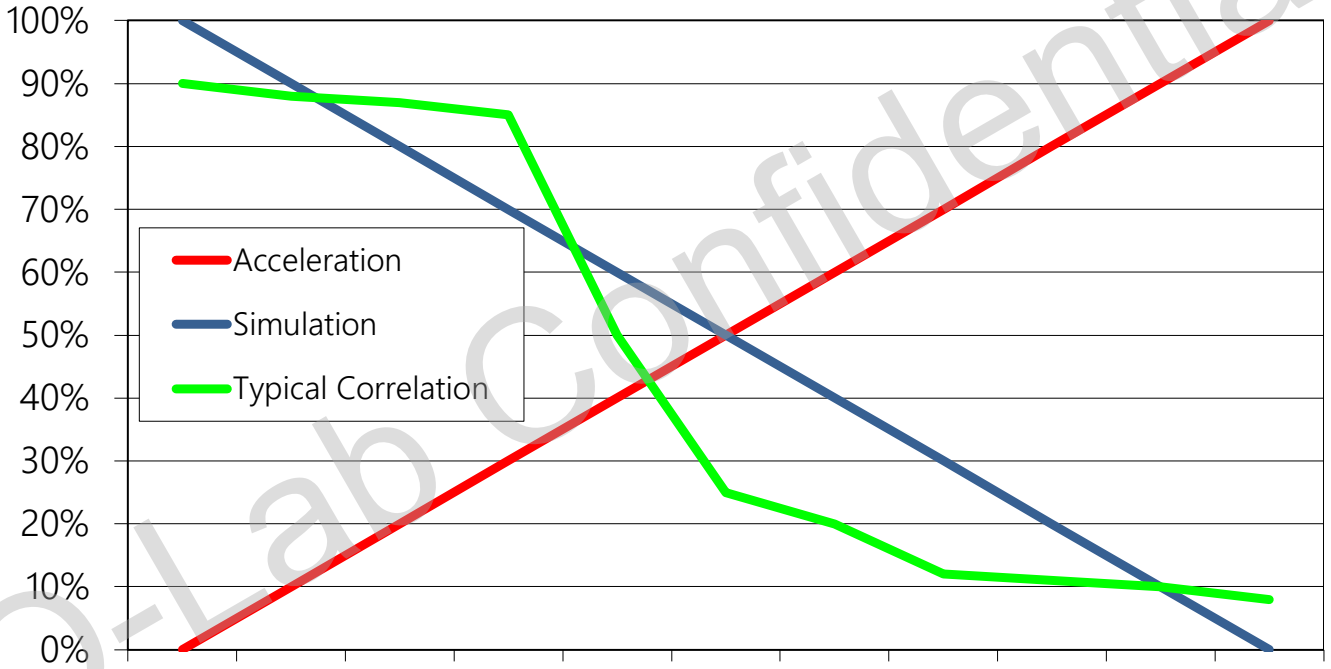
Accelerated Test Type	Result	Test Time	Results compared to
Quality Control	Pass / fail	• Defined • Short	Material specification
Qualification / validation	Pass / fail	• Defined • Medium-long	Reference material or specification
Correlative	Rank-ordered data	• Open-ended • Medium	Natural exposure (Benchmark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

Accelerated Test Types

What do we want to learn?

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)~~

Why is correlation such a challenge?



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

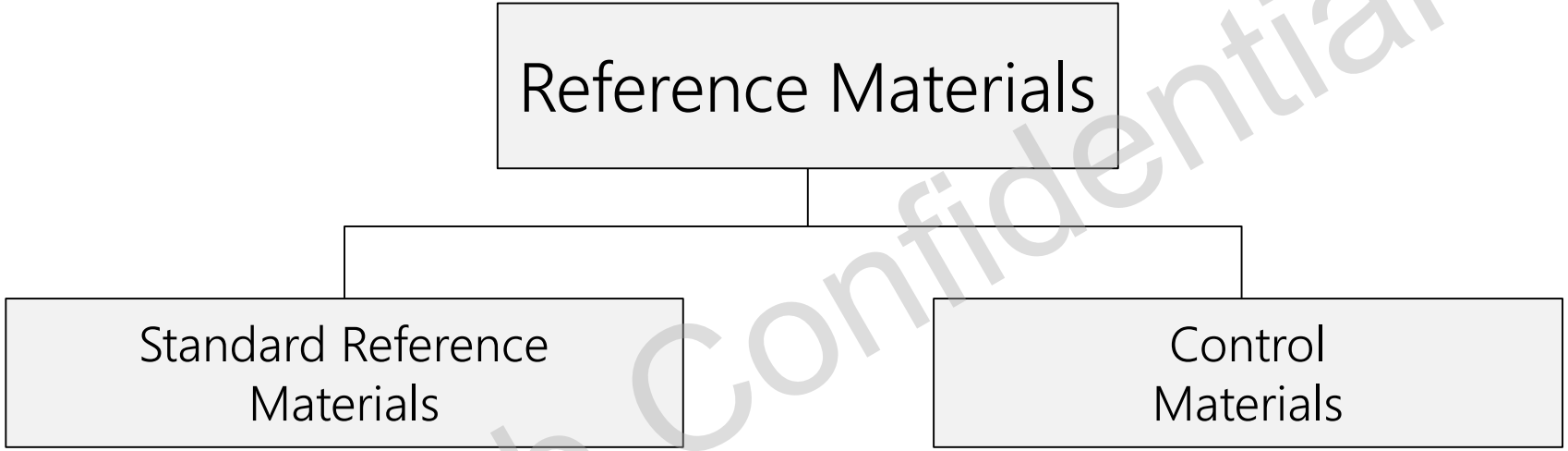
- Decision-making tools need to be validated
- There is an inherent conflict between acceleration and realism
- The only way to validate an accelerated weathering test is with outdoor/real world data
- In other words … Test the Test!

Methods for Establishing Correlation

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

- Reference and Control Materials
- Rank Order Evaluation

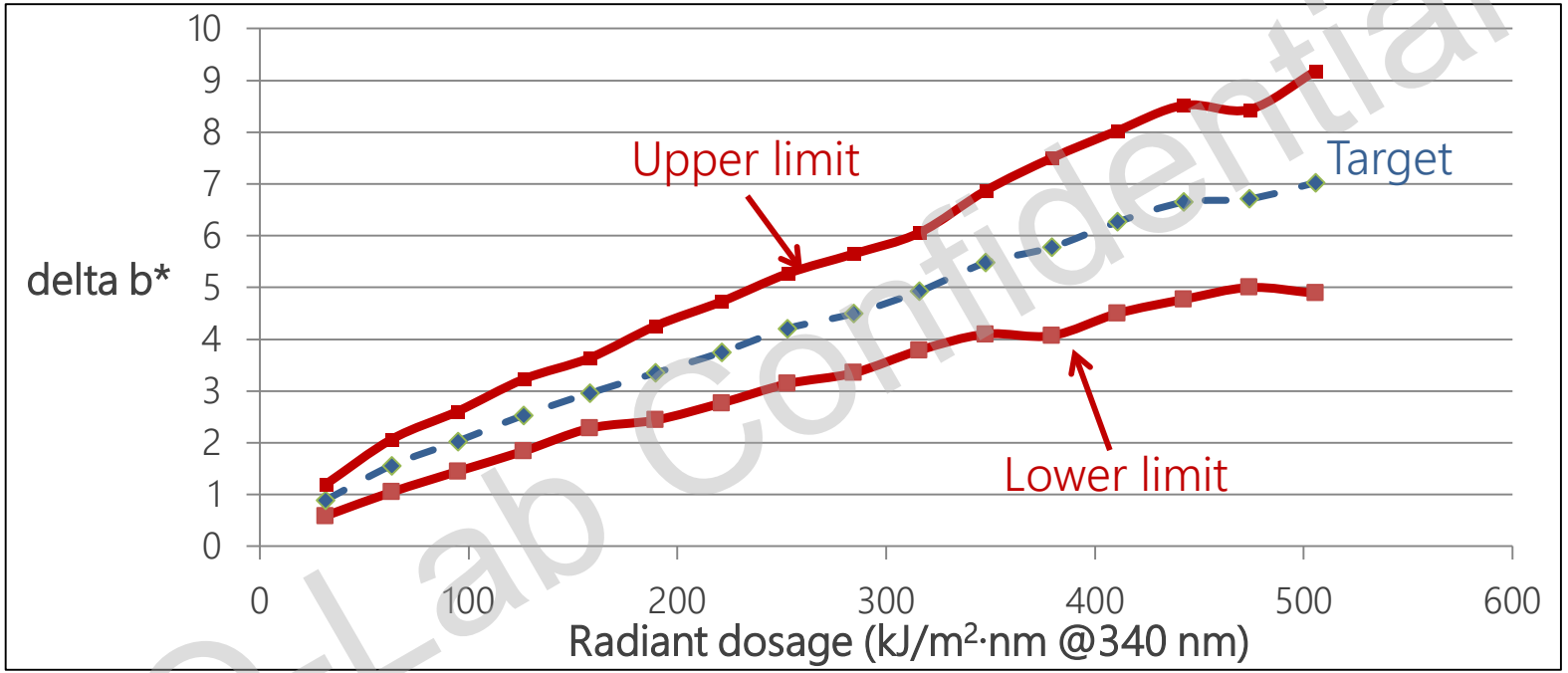
Reference and Control Materials



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

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

Standard Reference Material (Polystyrene)

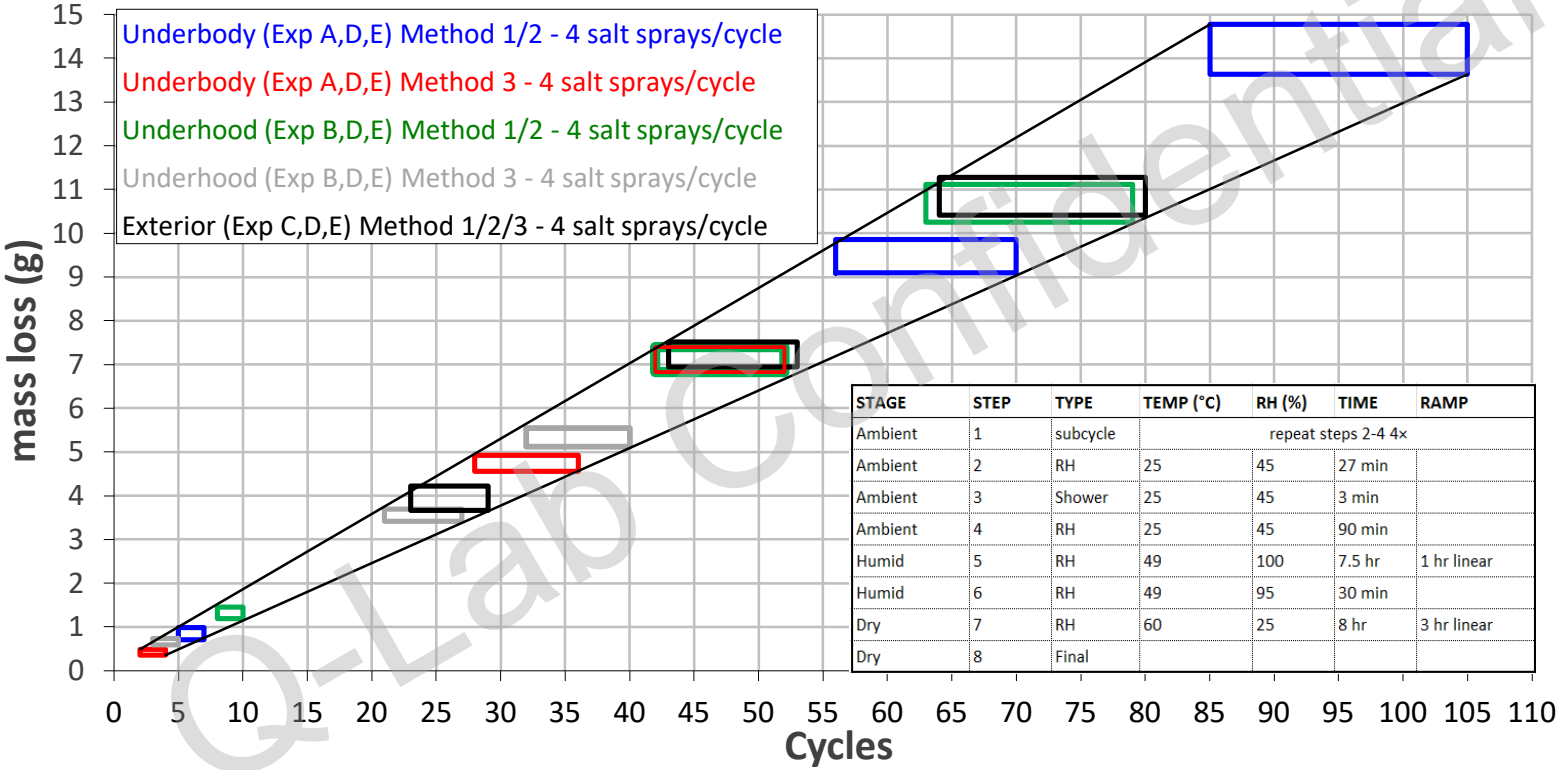


Reference Polystyrene yellowing validates tester performance in SAE J2527

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



STAGE	STEP	TYPE	TEMP (°C)	RH (%)	TIME	RAMP
Ambient	1	subcycle	repeat steps 2-4 4×			
Ambient	2	RH	25	45	27 min	
Ambient	3	Shower	25	45	3 min	
Ambient	4	RH	25	45	90 min	
Humid	5	RH	49	100	7.5 hr	1 hr linear
Humid	6	RH	49	95	30 min	
Dry	7	RH	60	25	8 hr	3 hr linear
Dry	8	Final				

Control Material Guidelines

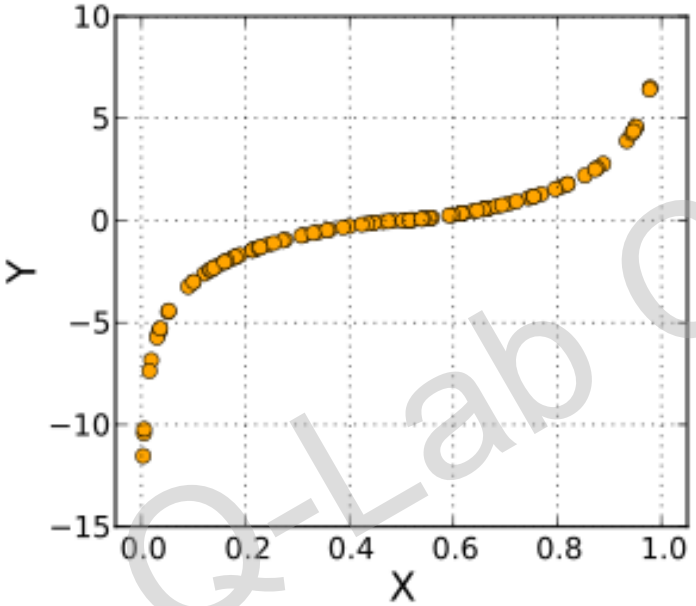
- Control materials must have known durability. This can be from:
  - Outdoor performance
  - Lab performance
  - A combination of these
- Similar composition to test material
- Similar expected degradation mode to test material
- Best practice to include both weak- and strong-performing control materials

Rank Order Correlation

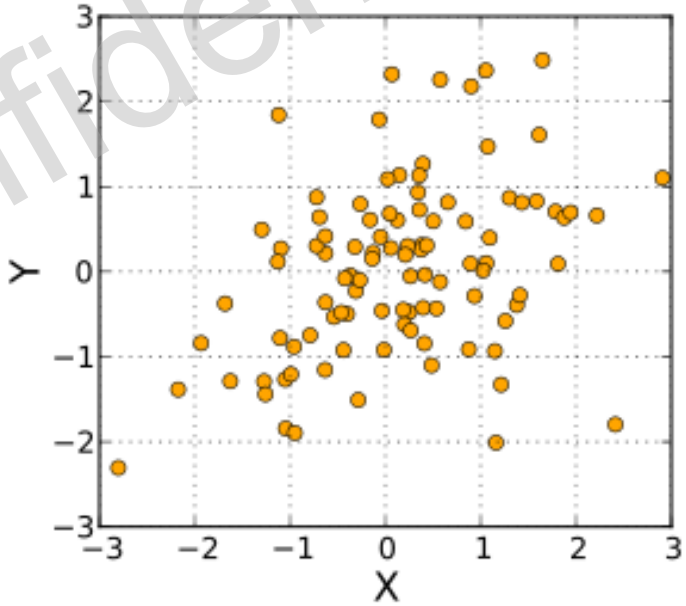
- Rank materials from best to worst outdoors and in lab test
- Calculate correlation coefficient using **Spearman's Rank Correlation Coefficient**
  - Quantitative measure of how well the lab test matches outdoors
  - Correlation of 1 is perfect (so is -1, in a way)
  - Correlation of 0 is random

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 Intermediate Bulk Containers (FIBC)

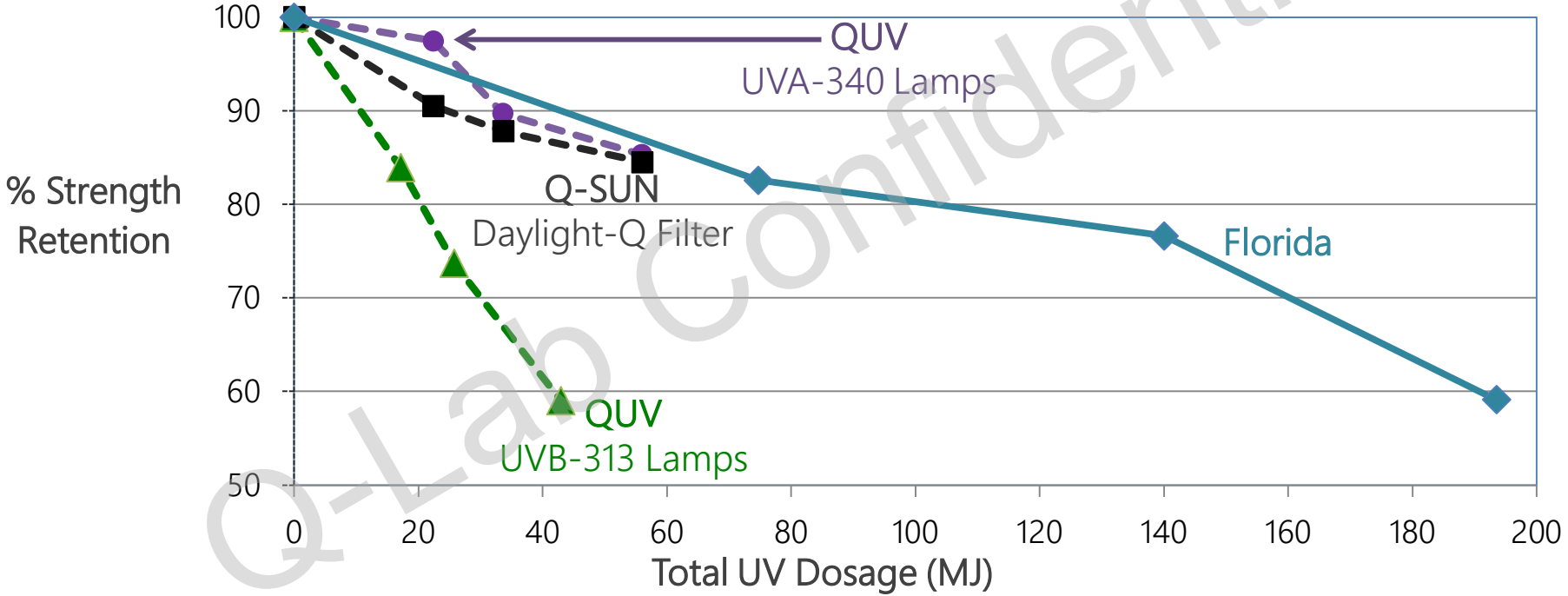
Flexible Intermediate Bulk Containers (FIBC)

Situation

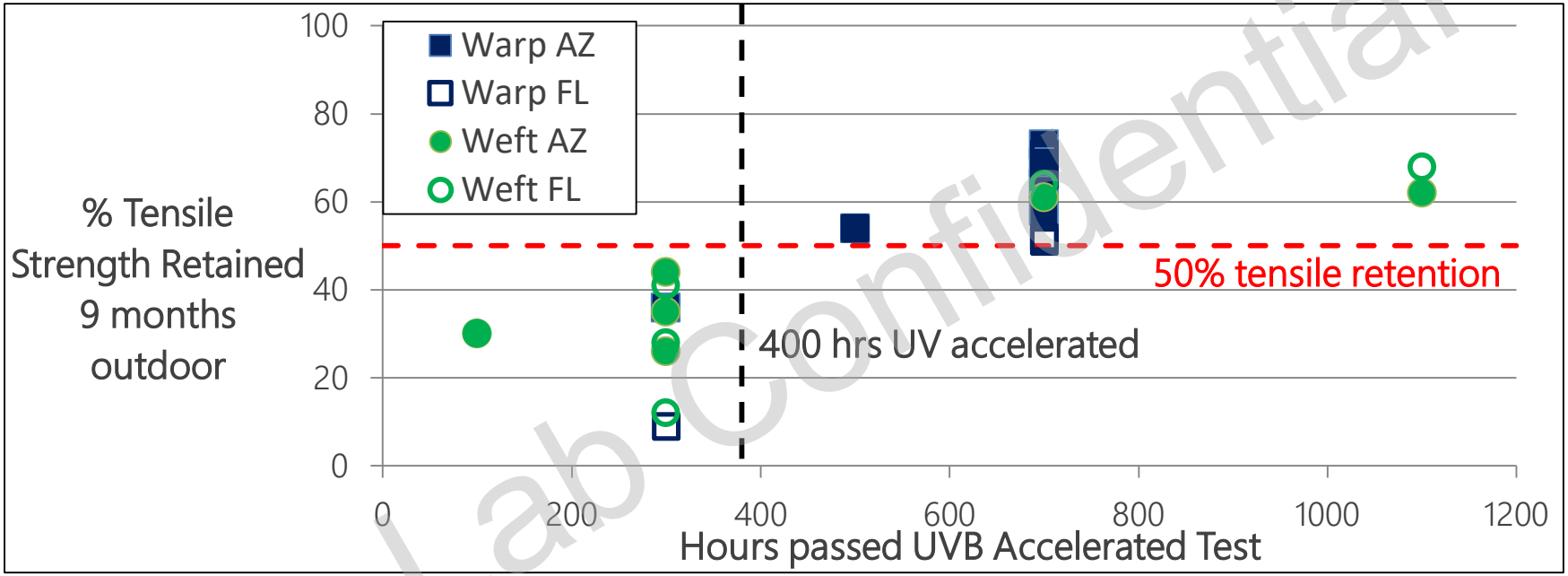
- FIBCs are used to carry goods. They need to survive at a job site for up to 12 months without losing tensile strength.
- Various test methods with Xenon and Fluorescent UV were compared to outdoor performance.

FIBC Correlative Testing

Accelerated and Outdoor testing – Radiant Dosage



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

Color	delta E	Color	delta E	Color	delta E
Red-1	5.7	Yellow	45.6	Blue-1	10.9
Red-1	5.7	Yellow	45.9	Blue-1	11.2
Red-2	26.7	Green-1	6.1	Blue-2	26.8
Red-2	28.5	Green-1	7.0	Blue-2	28.2
Orange-1	79.7	Green-2	5.8	Purple-1	23.0
Orange-1	79.3	Green-2	7.9	Purple-1	22.3
Orange-2	34.8	Green-3	19.3	Purple-2	23.1
Orange-2	34.8	Green-3	19.9	Purple-2	22.9
Beige	19.7	Aqua	5.8	Black	2.7
Beige	19.7	Aqua	5.7	Black	2.1

15 materials – a minimum of 10 (better if 20!) needed for correlation

Colored Pencil Correlation Study

Comparison of accelerated to outdoor

	Arizona Under Glass		Florida Under Glass		Xenon	
Specimen	ΔE	Rank	ΔE	Rank	ΔE	Rank
Red Pigment A	10.9	1	1.3	1	5.7	1
Red Pigment B	45.8	2	36.6	2	27.6	2
Orange Pigment	79.9	3	80.4	3	79.5	3

Results - Rank Order Correlation

Test Rankings Being Compared	Spearman's Rank Coefficient
Arizona – Florida	0.94
Xenon – Arizona	0.95
Xenon – Florida	0.93

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

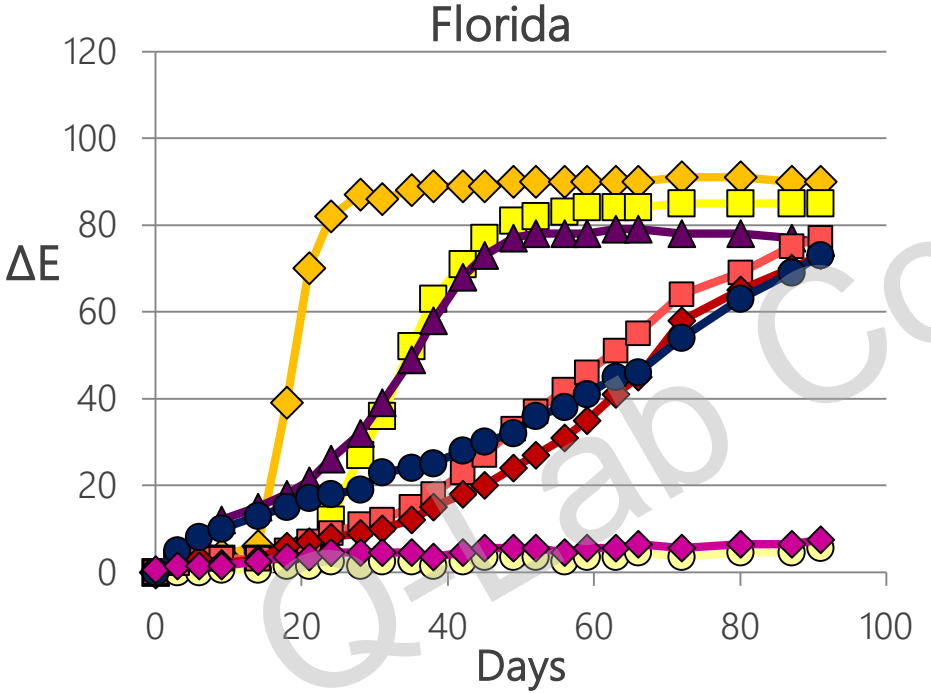
- Evaluate the light stability of lithographic inks

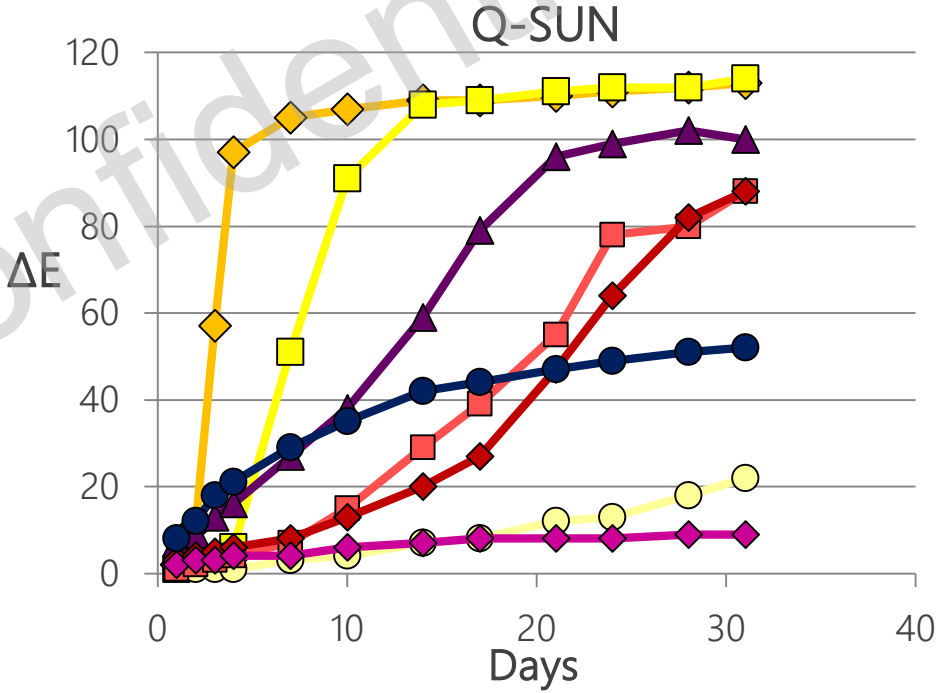
Test Program

- Natural outdoor tests
- Q-SUN Xenon Arc tests

Printing Ink Correlation Study

delta E Color Fade Measurements

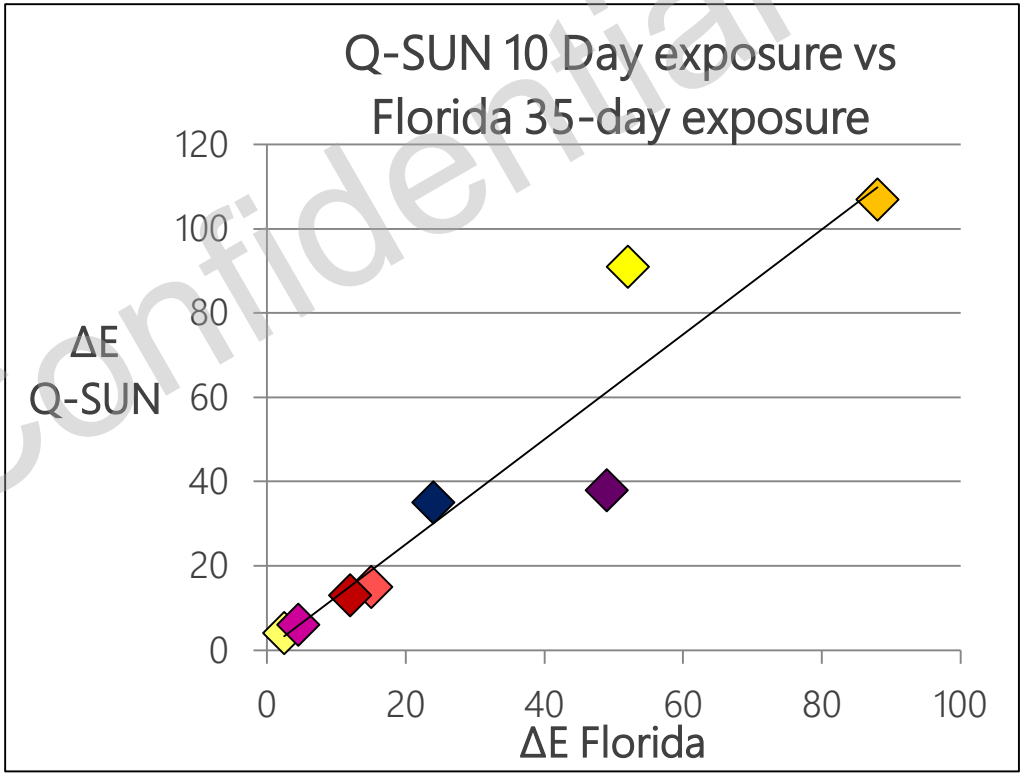




Printing Ink Correlation Study

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:

Colored Plastics

Colored Plastics Correlation Study

Situation

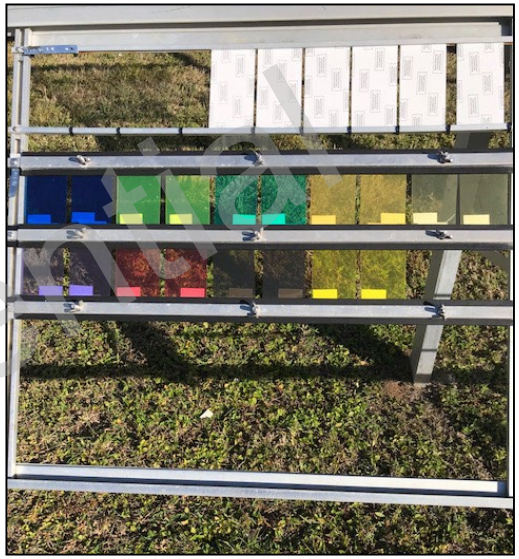
- Inorganic color additives in plastics like PVC are increasingly being replaced by organic additives.
- Better safety, decreased lightfastness performance.
- Need to understand better outdoor light / colorfasteness

Test Program

- Accelerated weathering testing of colored PVC plastics performed, color change (ΔE) measured
- Outdoor exposures for 2 months (Florida)
- Accelerated lab for 200 hours (UV fluorescent and xenon arc)

PVC Weathering Test Program

- Outdoor Exposures
  - Florida
  - Unbacked specimens, 45° south facing
  - 57 days
- Fluorescent UV
  - UVA-340 and UVB-313 lamps
  - 4h light, 0.72 W/m^2/nm, 45 °C
  - 4h condensation, 40 °C
  - 200 hours
- Xenon arc
  - Daylight-Q and Extended UV-Q/B filters
  - 5h light, 0.68 W/m^2/nm, 35-45 °C
  - 20 min spray, 40 °C
  - 200 hours



Results

Green

Results

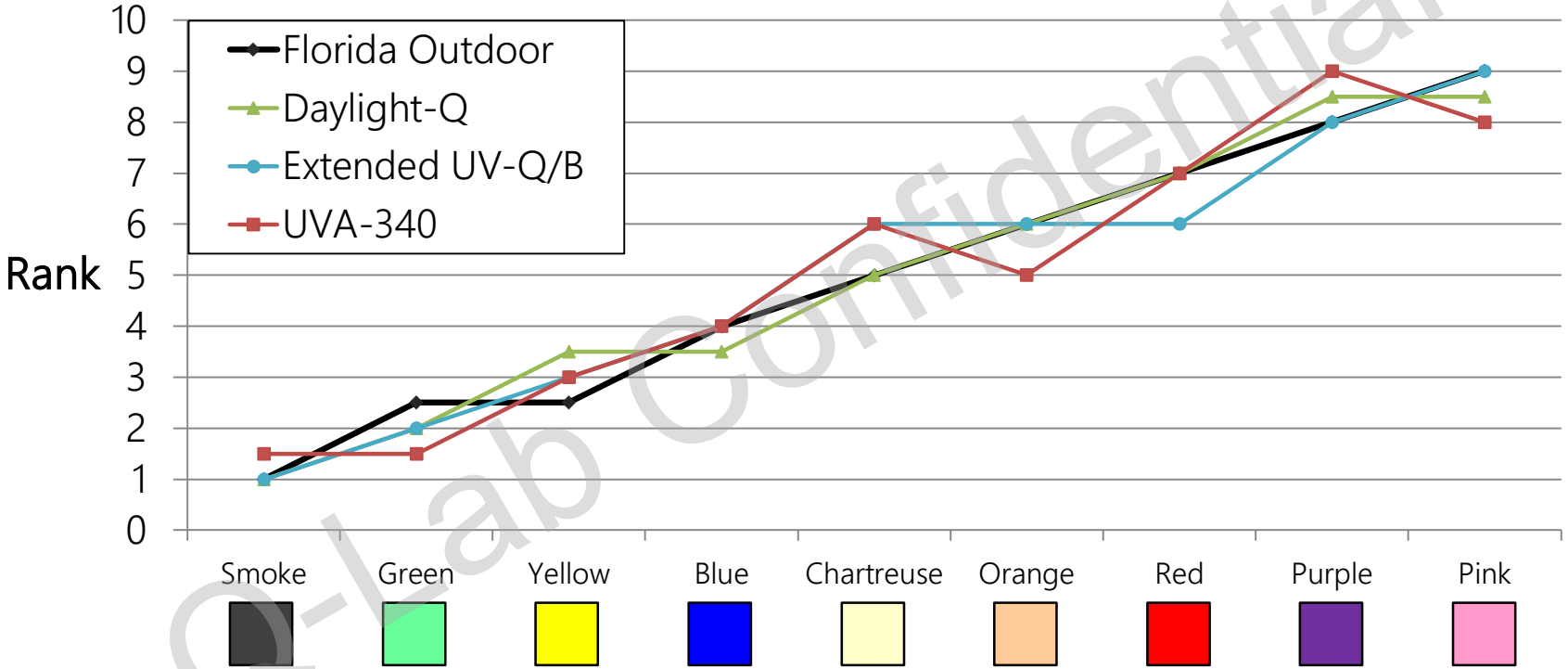
Purple

Correlation: Accelerated vs Outdoor

Color	Florida Outdoor		Daylight		Extended UV		UVA-340		UVB-313	
	ΔE	Rank	ΔE	Rank	ΔE	Rank	ΔE	Rank	ΔE	Rank
Smoke	0.6	1	1.0	1	1.8	1	1.3	1	3.6	1
Green	2.0	2.5	2.0	2	5.6	2	1.8	1	16.7	3.5
Yellow	2.5	2.5	5.0	3.5	6.3	3	4.7	3	43.0	7
Blue	4.7	4	5.2	3.5	7.2	4	5.7	4	21.0	5
Chartreuse	5.6	5	7.7	5	11.0	6	11.9	6	25.5	6
Orange	8.6	6	11.2	6	11.4	6	10.2	5	17.7	3.5
Red	14.0	7	35.0	7	11.8	6	16.8	7	14.3	2
Purple	39.0	8	42.0	8.5	40.7	8	26.6	9	50.7	8.5
Pink	71.9	9	41.3	8.5	65.3	9	19.7	8	49.7	8.5
Rank order correlation with Outdoors --->			**0.98**		**0.96**		**0.95**		**0.54**	

Excellent color change correlation between FL outdoors and accelerated (except UVB-313)

Rank Order Correlation: Accelerated vs Outdoor



Conclusions

- Correlation for color change between accelerated and outdoor tests
  - Excellent rank order correlation for xenon (Daylight or Extended UV filter) and UV fluorescent (UVA-340 lamps)
  - Acceleration factor for 57 days outdoor and 200 h accelerated (7:1)
  - Poor correlation for UV fluorescent UVB-313 lamps

- Different degradation observed for pigments and base plastics
  - Darkening from plastic yellowing from shortwave UV
  - Fade from breakdown of pigments from visible light
  - Differences most pronounced for pink and red specimens
  - Illustrates the need for thorough color characterization beyond ΔE

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 m^2 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

Summary of Correlative Testing

Accelerated Test Types

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)~~

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:

- The specific material tested
- The type of test being correlated to natural outdoor results – fluorescent UV, xenon, accelerated outdoors
- The specific set of lab tester time cycles and temperature
- The specific outdoor exposure site and sample mounting procedure
- The failure mechanism(s) being evaluated

Correlation between accelerated and outdoor testing

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?

Send your inquiry to:
info@q-lab.com