

MATERIAL SCREENING



SERVICE LIFE SOLUTIONS



OUTDOOR WEATHERING TESTING



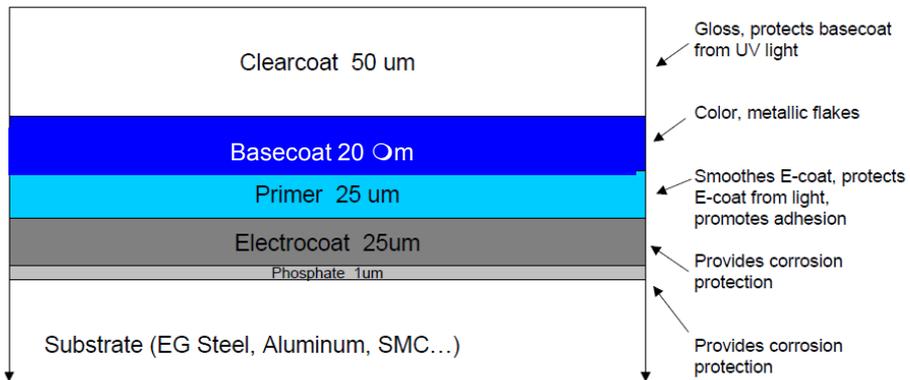
Lessons learned from other industries: A consortium of automotive stake holders' approach in the development of a science based accelerated weathering test standard

NREL PVMRW, February 24, 2015

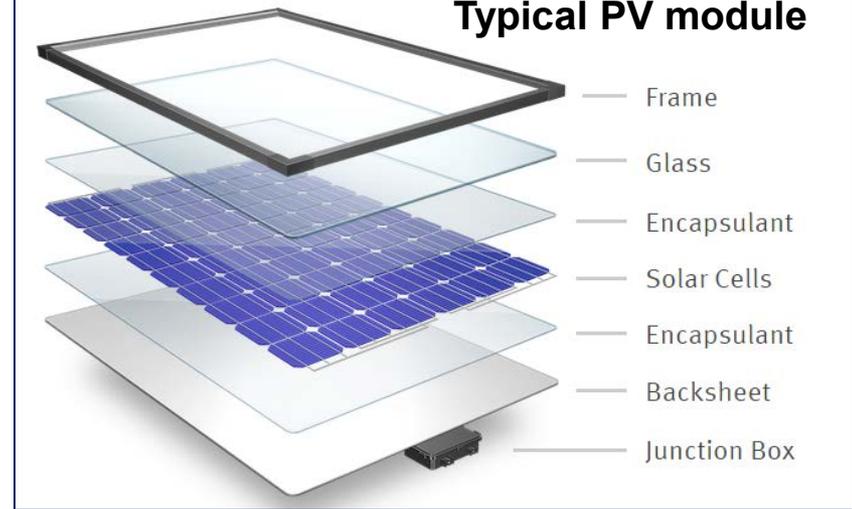
Allen Zielnik, Senior Consultant – Weathering Science
Kurt Scott – Global Manager, Solar Energy Competence Center
Atlas Material Testing Technology LLC

Why should we care? Automotive paint and PV, more similar than you think!

Automotive Paint System BC/CC



Typical PV module



M.Nichols, Ford Motor Co.

<http://www.dupont.com/products-and-services/solar-photovoltaic-materials/what-makes-up-solar-panel.html>

- ❑ Similar multilayer (built-up v. laminate) structure with UV filtering top layer
- ❑ Multiple material-material (diffusion and adhesion) interfaces
- ❑ Photo-oxidative and moisture sensitive materials
- ❑ Yes, each has different materials and some unique degradation mechanisms (“Denver cracking”, EVA acetic acid generation and corrosion)
- ❑ PV has two weather exposed surfaces (three including edges) vs. one
- ❑ Similar global service environments (not all vehicles are garaged)
- ❑ Main difference is scale -- μm vs. mm and total service lifetime (10 vs 30 yr)

Automotive industry and weather durability



Exterior and
Interior
Environmental
Durability



- Long lived high value product with high customer expectations.
- Need to withstand any and all climates; product is mobile.
- Variable BOM – many suppliers, changing materials, cost pressures.
- A few international weather testing standards, but most OEM's and Tier One suppliers create their own test methods and specs.
- Short product development cycle (short time to test and validate).
- Extensive long term use of accelerated weathering testing for interim acceptance coupled with real-time (5+ yr) outdoor weathering

Paint sells the car . . . or not!

Value of Automotive Coatings

- Coating appearance is a key driver of customer perception of quality.
- Functional Lifetime should be >10 years



Source: M.Nichols, *et al*, "Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods", Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

Must Reduce the Risk to an Acceptable Level

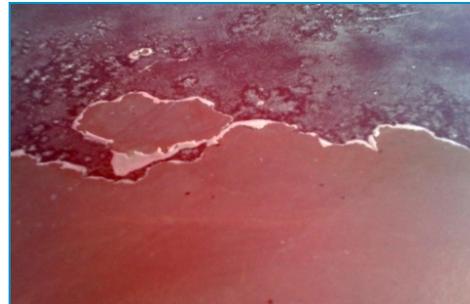
Even a poor performing paint system will last 2 years or more; that can mean production of 520,000 – 940,000 vehicles from a single plant before you even realize there is a problem.

Modern automotive paint failure

- The 1977 (Ford) Lincoln Versailles was the first production vehicle to offer clearcoat paint, followed by the VW “Sun Bug” Beetle and, in 1981, the Corvette. By mid-80’s most U.S. made cars and light trucks featured BC/CC.



- In the 80's and 90's new cars on dealer lots needed to be re-painted before they could be sold (initially blue & silver metallics were the worst)
- Today, most passenger cars by all manufacturers use BC/CC systems; failures are still seen by most OEM's and are often not predicted by current tests.



Natural Weathering Test – Automotive Coatings

45° (or 5°) Direct in a reference climate



5° Black Box automotive exposure

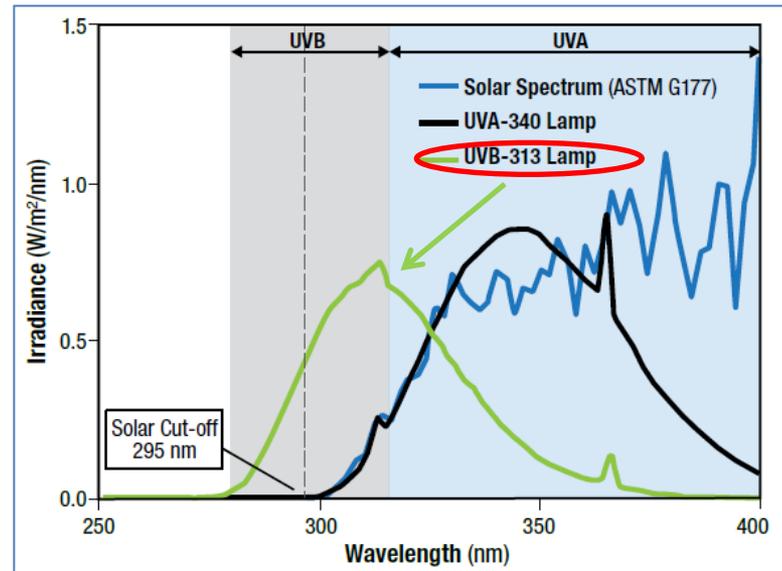
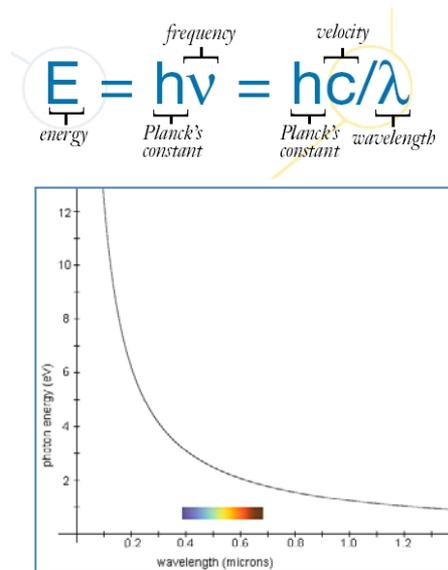


- Natural weathering exposures typically performed in **subtropical climate**
 - Hot, humid (maximum wet time), high annual UV dose – South Florida
- **Black Box** better simulates *in-situ* automotive temperature / moisture profiles due to dead air space volumes (e.g., underhood, trunk and passenger cabin)

Today, long 5 yr minimum to 10 yr exposure required to approve a new coating formulation or pigment system is burdensome, but the costs of a paint failure are huge.

So what happened?

- The weathering performance of clearcoat/basecoat paint systems was assessed on the basis of gloss loss and color change traditionally used for monocoat paints.
- The testing philosophy used the “bigger hammer” harsher-is-better way to highly accelerate testing using UVB-313 fluorescent lamps with condensation.

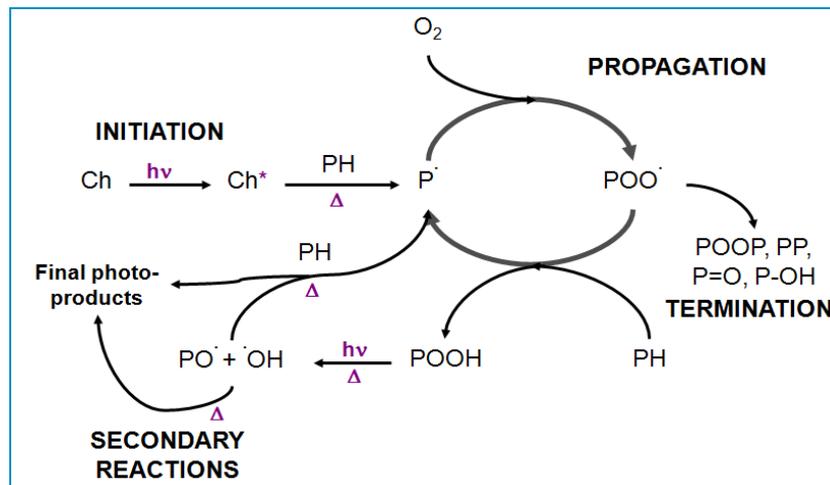


- “Slower” xenon (SAE J1960), EMMAQUA (ASTM G90) testing was still ongoing, but showed some “anomalies”; real-time outdoor testing was begun.
- **But based on UVB-313 testing the decision was made to “start painting cars”.**
- **Note: Fluorescent UV testing is no longer used for paint system qualification although an SAE J2020 test is still “on the books” (original 1989, updated 2003)**

What went wrong?

- But, this was inadequate to correctly assess the weathering performance of clearcoat/basecoat paint systems which are entirely capable of failing suddenly and catastrophically by clearcoat cracking and/or peeling with little or no gloss loss indication that failure is imminent.
- Loss of UV absorber (UVA) or HALS, clearcoat oxidation, localized hydrolysis, etc., all contribute to failure.

Polymer Autocatalytic Photooxidation Cycle



Note: Hydrolysis reactions not shown

- In attempt to be fast and severe, the accelerated test significantly *altered* the natural photo-degradation chemistry -- and in this case did not result in the delamination and other failures soon seen in the field!

Accelerated weathering tests – automotive coatings

Fresnel solar concentrator

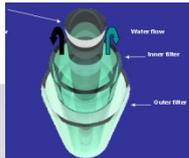


General standard ASTM G-90 EMMA® / EMMAQUA® (with water)

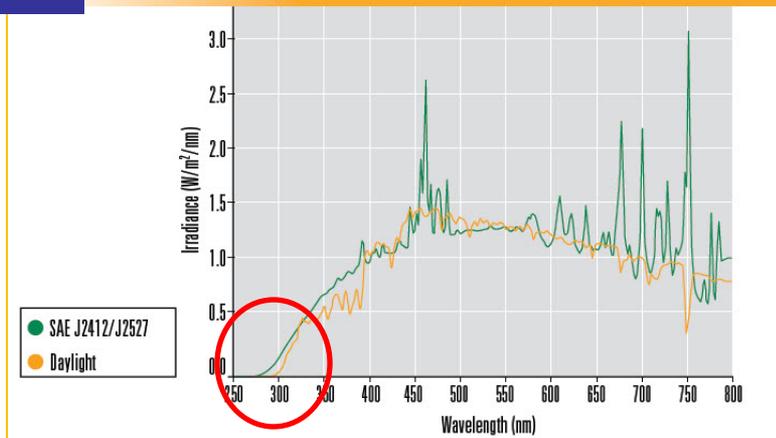
Laboratory – xenon arc light source



Weather-Ometer®



SAE J2412/J2527 vs. Daylight



Many other methods also used

OEM	Test Method
Ford	SAE J1960-Daylight Filters
	SAE J2527
	FLTM BO 116-1
General Motors	FLTM EU BO 050-1
	GMW 14650
	SAE J2527
	ISO 4892-2
	GMW 14170
Volkswagen	SAE J1960
	PV3929
	PV3930
Hyundai	MS-300-31
PSA (Peugeot, Renault)	D27 1380
	D27 1389
	D27 1911 / --D (2007)
	D27 1911 / --D (2007)
Fiat	50451
Porsche	ISO 4892-2
Daimler	DBL 7399
	DBL 5555
SAAB	STD 3159
Volvo	STD 1027, 337
	ISO 11341
International	ISO 4892-2
	JASO M351
	JIS D0205
	SAE J1960/J2527
	SAE J1960/J2527

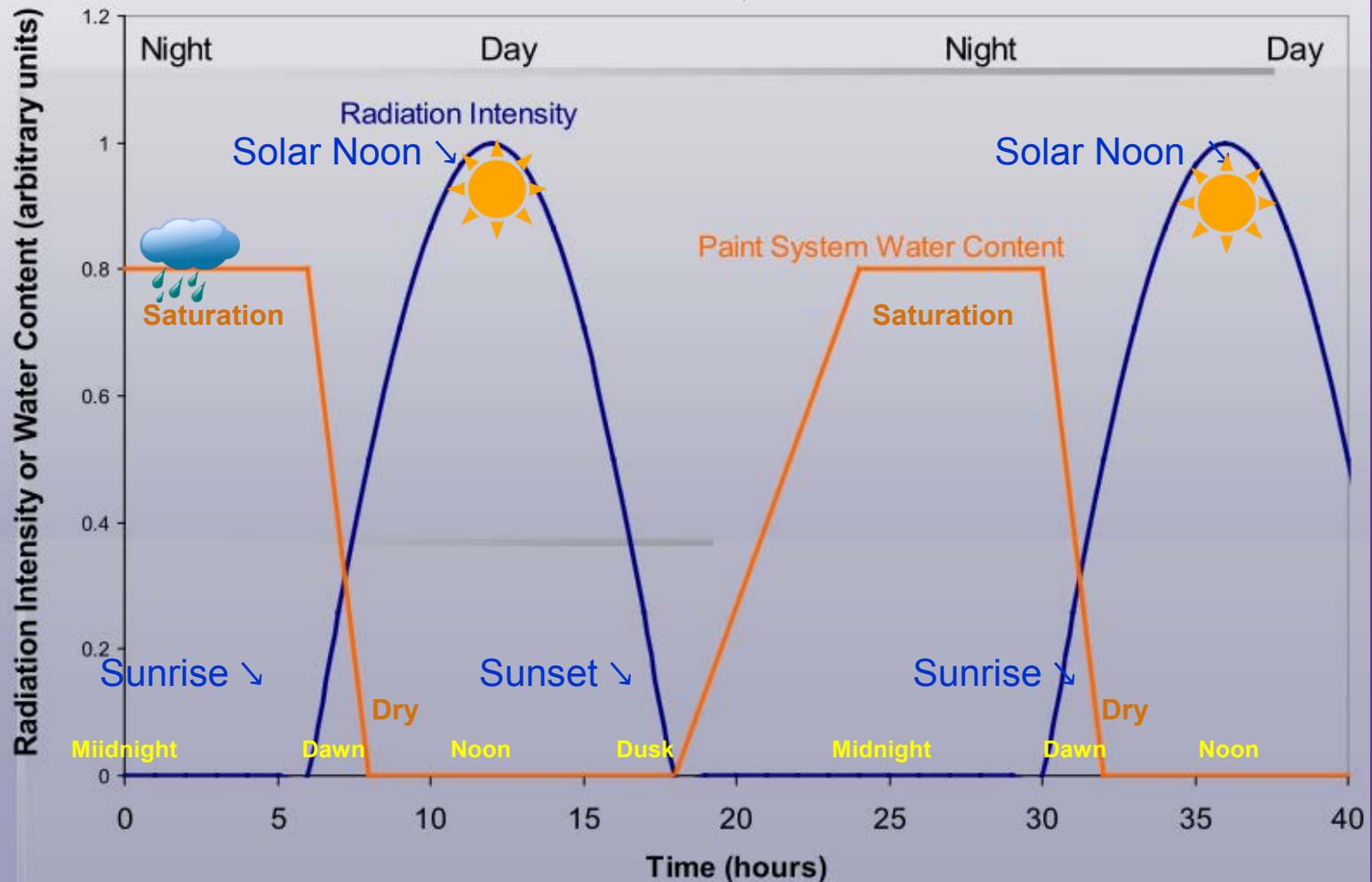
SAE J1960 (now J2527) brief background

- For over 20 years the *de facto* standard for xenon testing of automotive exterior materials by Detroit “Big 3” & Tier 1 suppliers (but not by European or Asian OEMs)
 - Mid 1980’s – original test method development, primarily by GM
 - SAE J1960 (1989) standard using “extended UV” filters; UV more severe than sunlight (lower UV cut-on λ ~270nm)
 - Late 1990’s - Ford adopts a “modified” SAE J1960, using “Daylight” filters – less unrealistic UV (cut-on ~285nm)
 - 2004 – SAE J2527 replaces J1960 – “Performance-based version” with choice of “extended UV” or “daylight” filters, *but still excessive UV*
 - **Frequent lack of correlation with South Florida 2 & 5 year results (Note – this is not unique to automotive coatings!)**

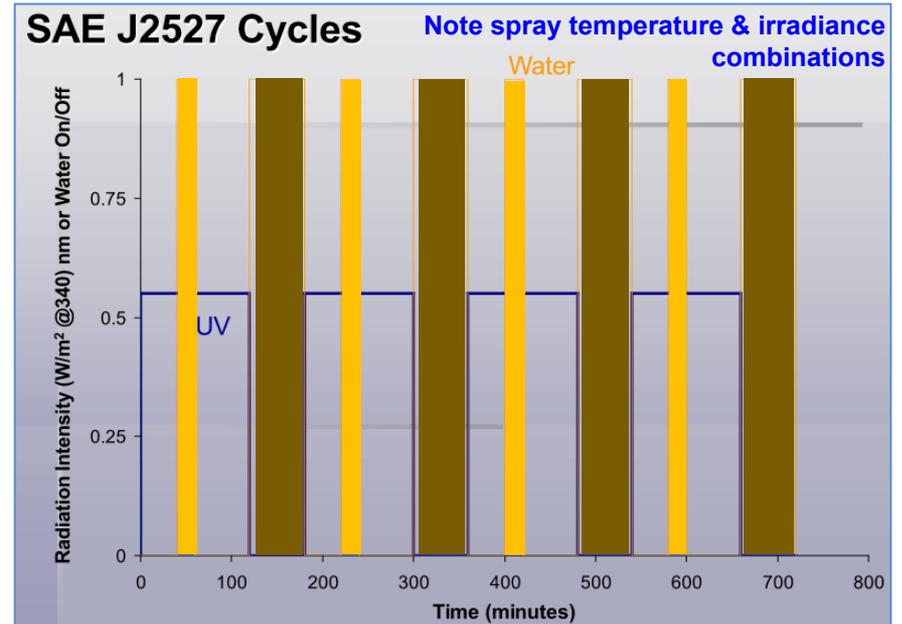
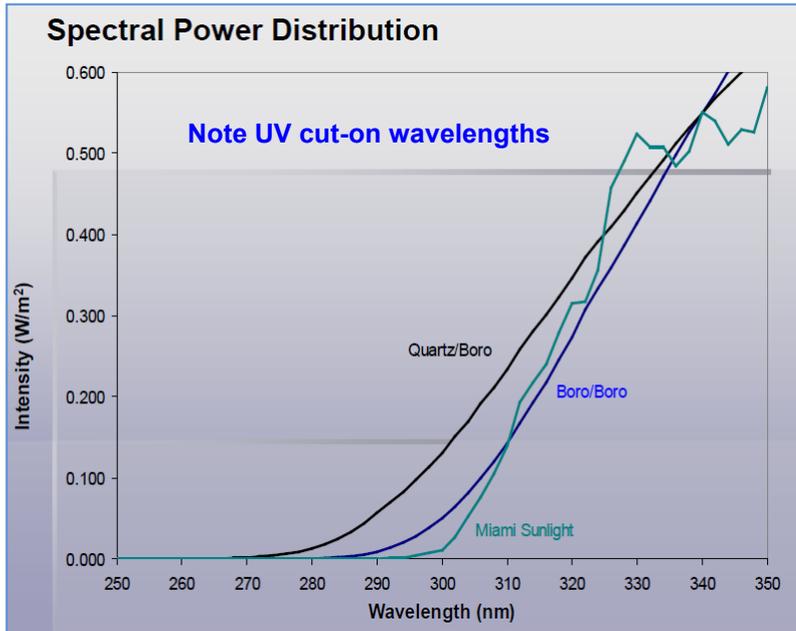
So.Florida Diurnal Cycle – Solar Radiation & Moisture

Florida Panel Exposure

Source: M.Nichols, *et al*, "Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results.", 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011



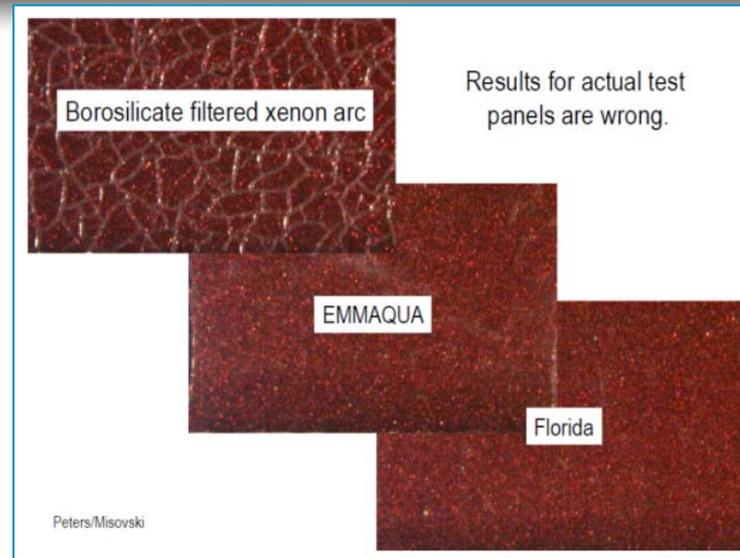
SAE test cycle



Step#	Water Spray	Irradiance (W/m ² @340 nm)	Humidity %	Chamber Temperature (°C)	Black Panel Temperature (°C)	Duration (minutes)
1	Off	0.55	50	47	70	40
2	Front Spray	0.55	50	47	70	20
3	Off	0.55	50	47	70	60
4	Back Spray	0	95	38	38	60

Source: M.Nichols, *et al*, "Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods", Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

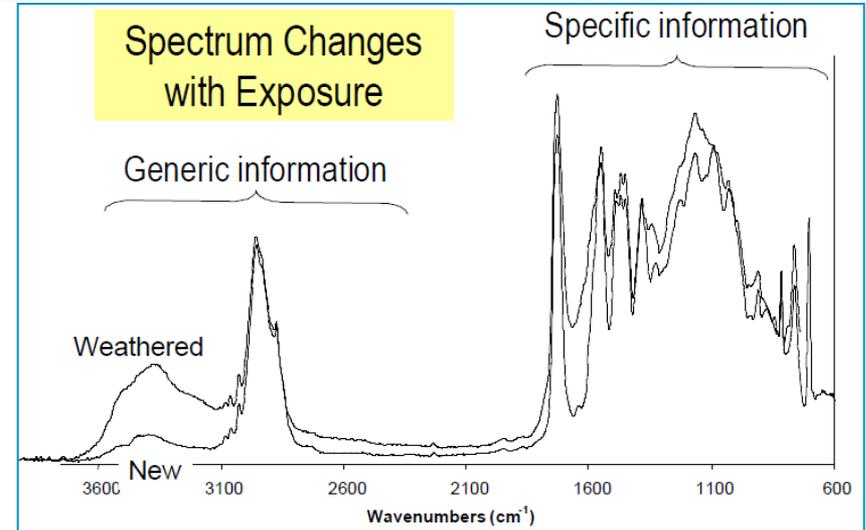
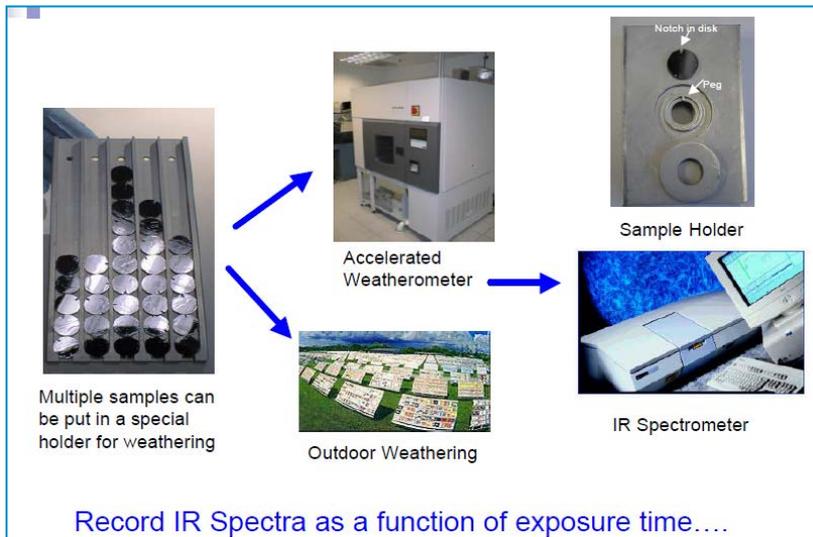
SAE test cycle



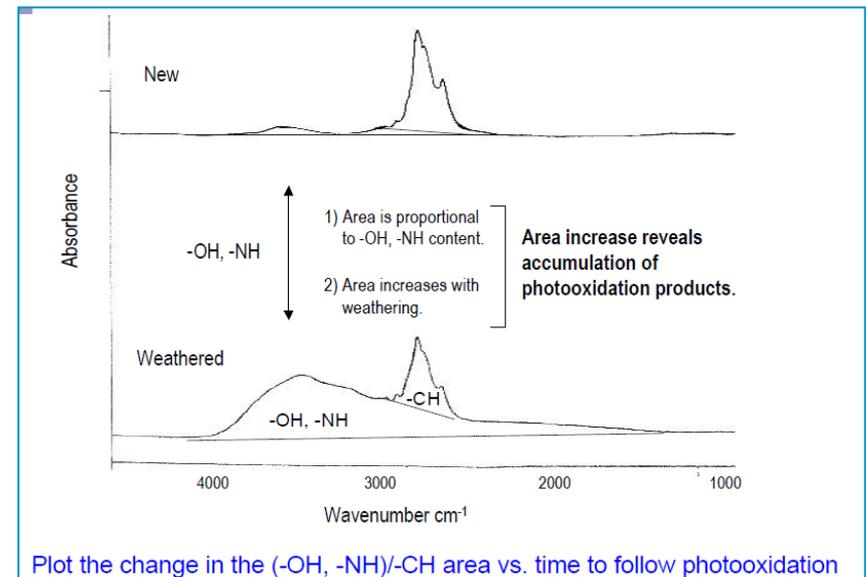
What is Wrong with the Current Tests?

- Light source – output from lamp/filters does not match sunlight
- Temperature – water spray when the light is on
- Time of wetness – panels are wet for a long time outdoors
- Humidity – rarely reach temperature/humidity conditions outside

Weather-Ometer & Outdoor Findings



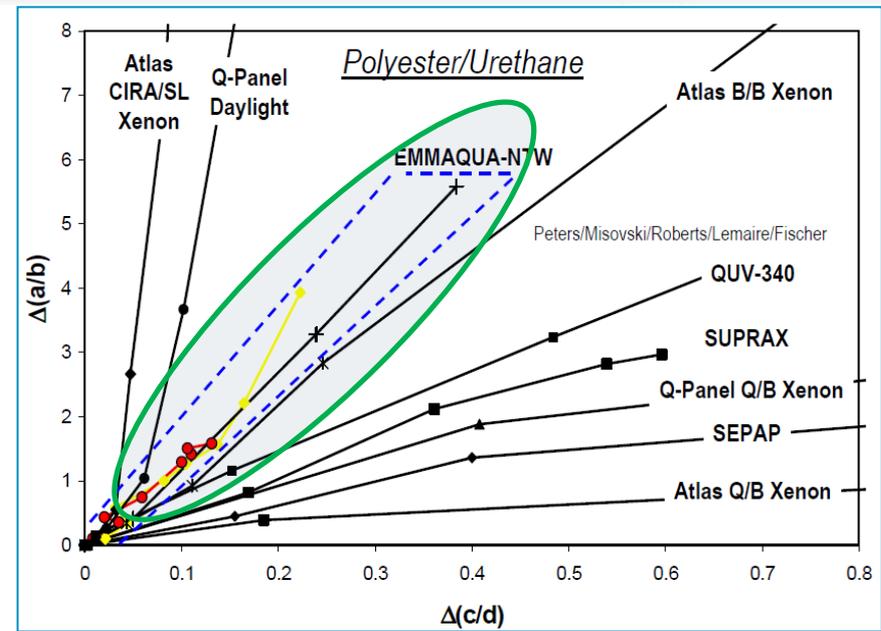
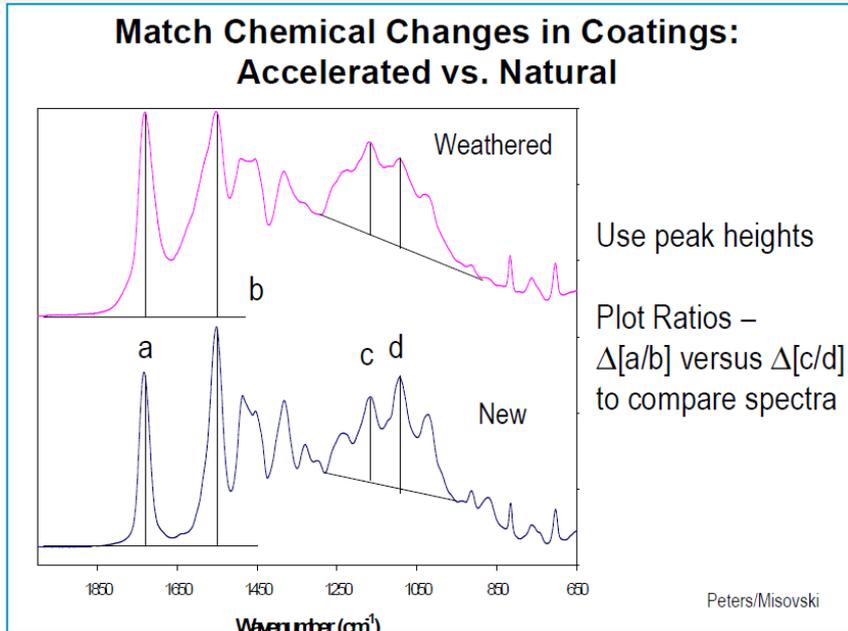
- Accumulation of photooxidation products by (Δ -OH, -NH / -CH) ratio with PAS-FTIR correlates well with outdoor
- CC's that crack or loss gloss rapidly tend to accumulate higher levels at shorter exposure times.
- PAS-FTIR helps predict systems that will lose gloss or crack



Source: M.Nichols, *et al*, "Test Methods to Determine the Long Term Weathering Performance of Coatings Systems – Chemical and Mechanical Testing of Paint Systems, October 6, 2005,

Plot the change in the (-OH, -NH)/-CH area vs. time to follow photooxidation

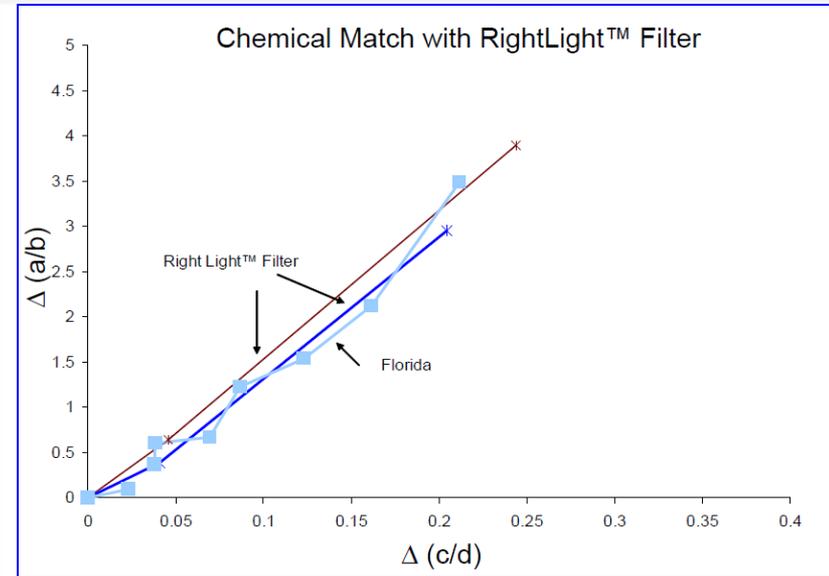
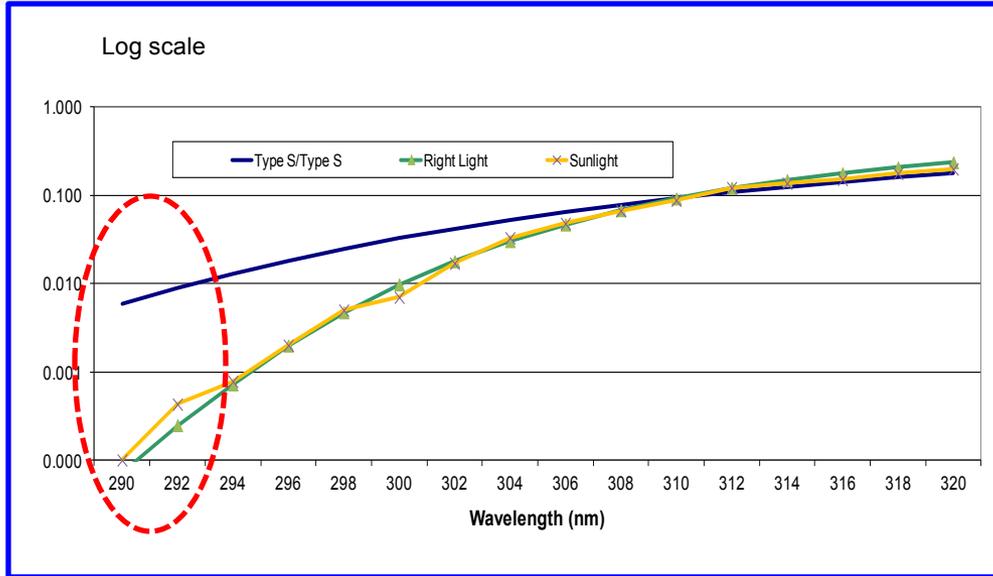
Weather-Ometer & Outdoor Findings



- Ratio plots of more specific FTIR chemical marker peaks showed a good photochemical match of EMMAQUA to outdoors. Various lab accelerated systems with various spectral power distributions (SPD) all skewed the photochemistry.
- Critical need to “**Get the light right**” in accelerated tests to reproduce the photochemistry. None of the existing lab systems match TSR SPD well enough.

Source: M.Nichols, *et al*, “Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods”, Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

“Getting the Right Light”



Source: M.Nichols, *et al*, “Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods”, Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

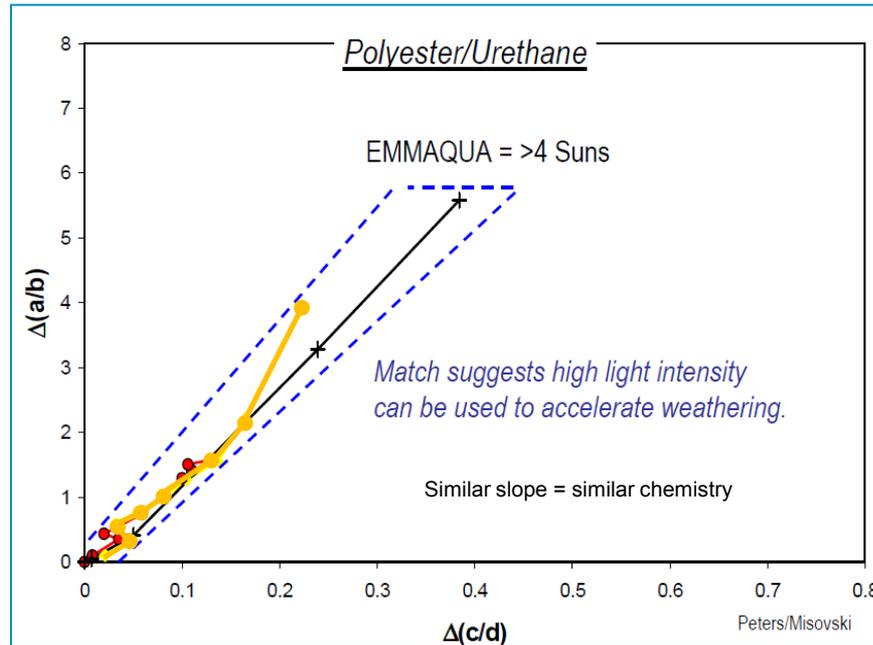
- A new xenon lamp filter set correctly matches the UV cut-on of terrestrial solar radiation *and* correctly reproduces the photochemistry.

“The precise simulation of terrestrial sunlight, particularly in the short UV range, is even more critical for reliable weatherability testing of automotive coatings than originally thought. Xenon light used in conjunction with the Right Light™ filter provides the appropriate ultraviolet spectrum for weathering of today’s complex automotive coatings.”

Dr. Mark Nichols, Ford R&D, Exterior Coatings

Source: Quoted in Atlas Sunspots, Vol. 38, Issue 81; <http://atlas-mts.com/technical-information/sunspots>

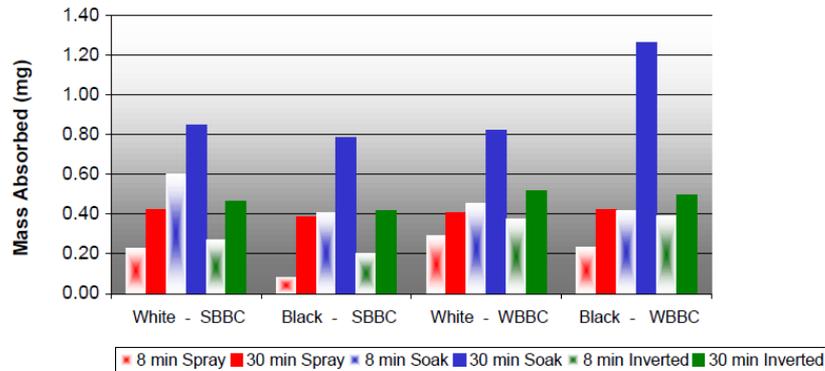
Weather-Ometer & Outdoor Findings



- With the “right light” the photochemistry matches outdoor weathering.
- **BUT**, the paint panels DON'T always or completely reflect that.
- So, there may be chemical effects not detected by FTIR, or a physical effect occurring in addition to the photochemistry known to cause cracking and blistering.

Findings About Water

EMMAQUA Water Absorption



Note: J2527 has a 20 minute front spray with the lights on and panels at 70°C.

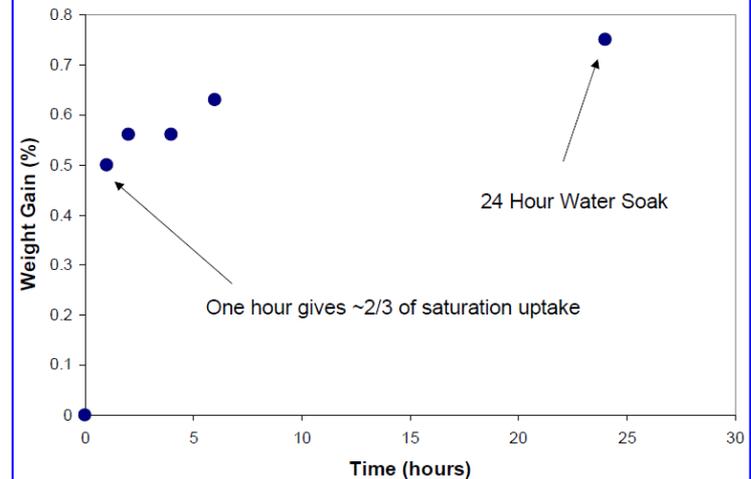
Source: M.Nichols, *et al*, "Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods", Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

Effects of water on weathering

- Plastication – reduced modulus
- Swelling – induced stresses due to differential stresses
- Blistering – localized swelling and rupture
- Adhesion – accumulation of water at interface, breakdown of interfacial bonds
- Mass transport – movement of small molecules and reaction products through film
- Mass loss – removal of film (degradation products) from surface of the coating due to erosion

Source: M.Nichols, *et al*, "Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results.", 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011

Water Uptake in Atlas Ci4000 WOM



5.5 hours to reach acceptable water uptake (near saturation) based on average Florida wet time

Additional studies have shown that temperature is the most significant factor. Sprays v. immersion – or orientation (vertical/horizontal) has no effect

Water Implications and Approaches

Implications for Accelerated Test Cycles

SAE J2527

- 2 Hrs light (w/ 20 minute H₂O spray), 1 Hr dark w/ back spray
- ~4kJ/m² @340 nm per 3hr. Cycle (0.55 W/m² @340nm)
- One wet-dry interval/cycle

Florida Exposure

- 7.5 kJ/m² @340nm per day
- 1 wet-dry cycle per day
- 3600 Hrs. wetness/year (~10 hrs/day)

Possible Approaches

Heat water to accelerate diffusion and shorten saturation time

- Must ensure we do not alter chemistry

Disregard water scaling and accelerate with long light and long but infrequent water soaks

- Must introduce correct mechanical stresses

Source: M.Nichols, *et al*, "Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results.", 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011

- After considerable experimentation and many iterations, a solution to the problem of long water saturation times, the need for frequent cycling for mechanical stress, and the desire not to modify existing commercial instrument hardware was developed

Xenon arc devices accelerate weather stresses



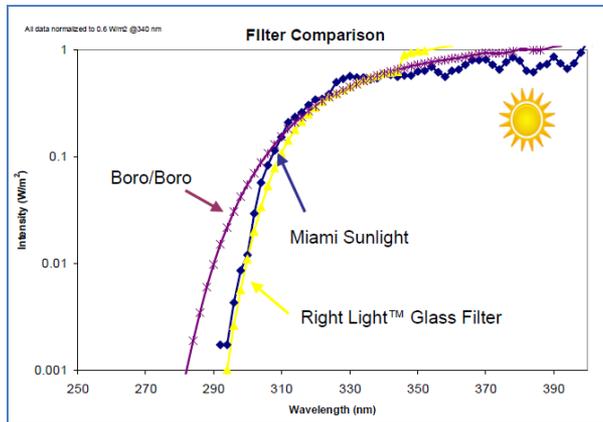
Solar spectrum, daily intensity & cycles



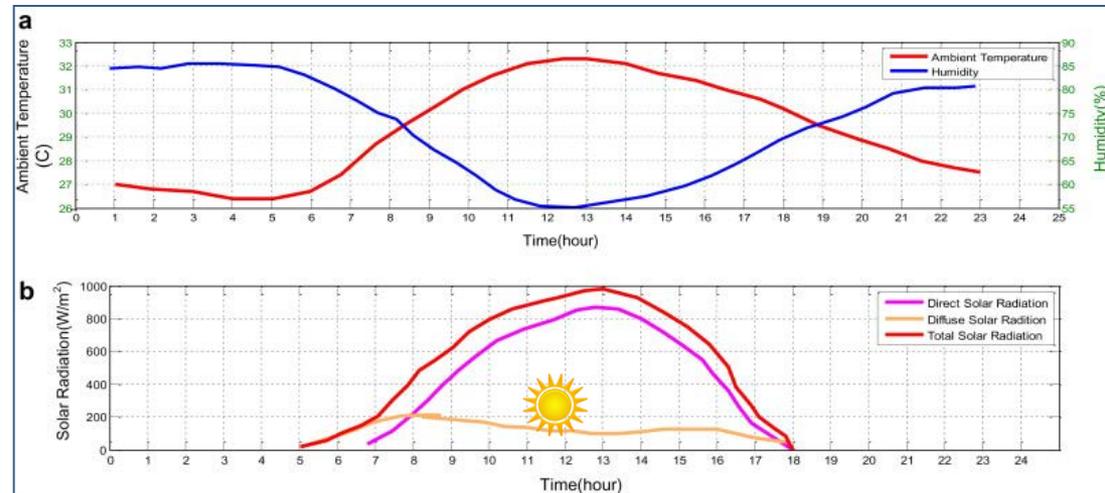
Solar load, heating and cycles



RH%. Dew, rain, cycles



Source: M.Nichols, *et al*, "Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods", Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.



However, current test methods have very limited, if any, cycling.

The seeds of a better, predictive test

20 years of experimental research findings:

- Much of the failure was traced to a spectral mismatch between the lab light source and outdoors. Fluorescent UV abandoned for qualification tests.
- Ford & GM add automotive glazing UV filtering to interior xenon test methods
- Various tests with ozone-filtered xenon led Ford *et al* to use Boro-S-Boro-S daylight xenon filters rather than SAE J1960 Quartz/Boro “extended UV”.
- FTIR spectroscopy showed only EMMAQUA produced the same weathering chemical marker changes as real time outdoor testing.
- Clearcoat UV Absorbers depleted from the top down (microtomy and ATR spectroscopy)
- Search to “Get the light right” led to iterations of improved spectral match filters, especially in UV cut on wavelength and IR heat reduction.
- Extended water soak and EMMAQUA spray cycles revealed inadequate water uptake of coatings with current methods. And it doesn’t rain when the sun is brightly shining!

Collaborators

Volvo, Mazda, **BASF**, DuPont, PPG, Akzo, RedSpot, Visteon, Ciba, Cytec, General Electric, Sabic, Momentive, Henkel, Atlas, Q-Panel, Suga, Bruker, 3M, Exatec, Bayer, AOC, Ashland, Dow, **Fusion UV**, NIST, U. of Michigan, Eastern Michigan U., U. Blaise Pascal, U. Mulhouse France, **NDSU**, Swedish National Testing Institute, **GM**, Chrysler.

The seeds of a better, predictive test

Followed by 10 years of consortium effort leading to ASTM D7869:

- The methodology described is the result of a multi-year collaborative effort between researchers at the following companies:
 - Ford Motor R&D
 - Boeing Commercial Aircraft
 - BASF, Bayer MaterialScience
 - Atlas Material Testing Technology,
 - Q-Lab
 - and later, Honda R&D Americas

Paint Systems Tested

- Automotive
 - ~20 systems, multiple colors
 - All systems were BC/CC
 - Fortified and unfortified
 - Positive controls and known Florida exposure failure mechanisms
- Aerospace
 - Four systems, two colors (blue and white)
 - Two monocoat systems, two BC/CC systems
 - Florida, and in-service performance known

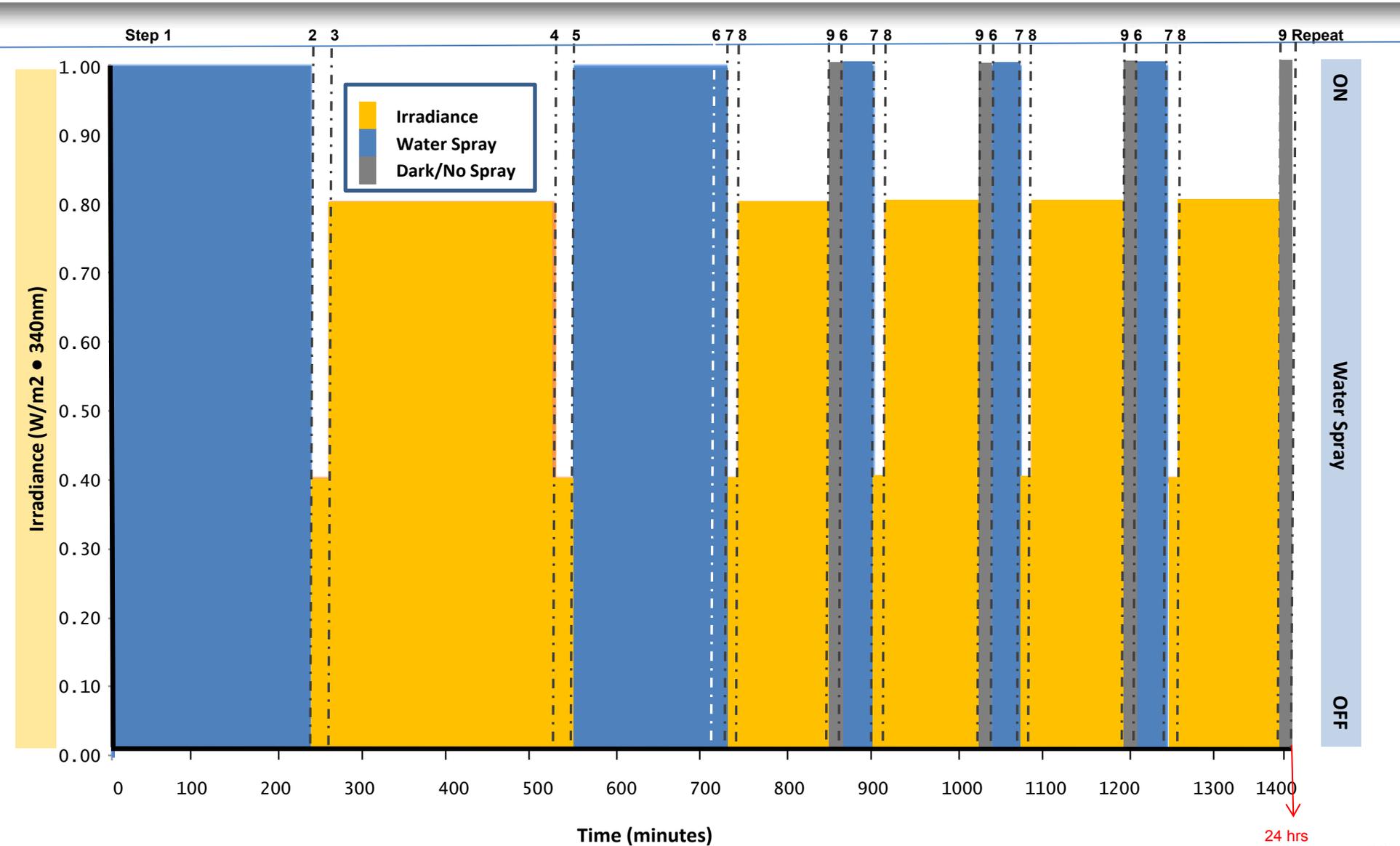
Fast Forward 10 Years -- Putting it all together: A New ASTM Test Method D7869

For reference only, this will be explained in the following slides . . .

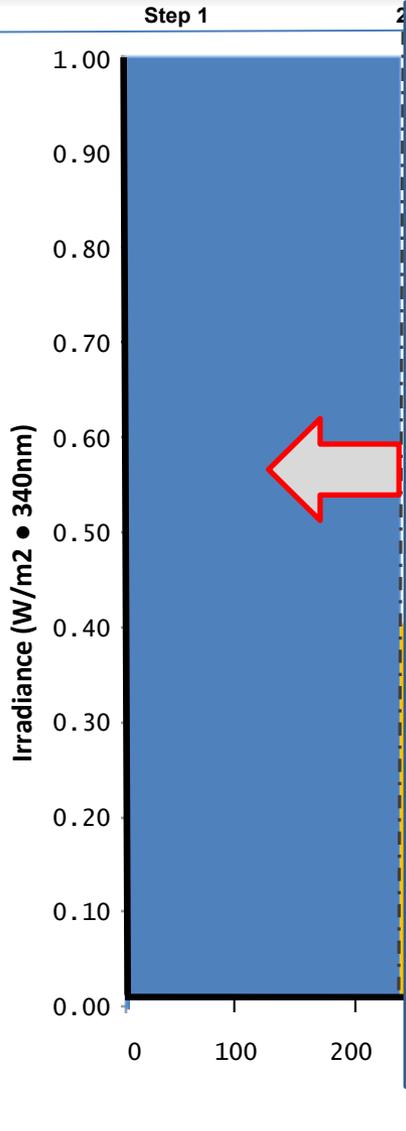
ASTM D7869 Test Cycle Sequence

Step Number	Step Minutes	Function	Irradiance Set Point ¹ @340nm (W/m ² /nm)	Black Panel Temperature Set Point ¹	Chamber Air Temperature Set Point ¹	Relative Humidity Set Point ¹
1	240	dark + spray	-	40°C	40°C	95%
2	30	light	0.40	50°C	42°C	50%
3	270	light	0.80	70°C	50°C	50%
4	30	light	0.40	50°C	42°C	50%
5	150	dark + spray	-	40°C	40°C	95%
6	30	dark + spray	-	40°C	40°C	95%
7	20	light	0.40	50°C	42°C	50%
8	120	light	0.80	70°C	50°C	50%
9	10	dark	-	40°C	40°C	50%
10	Repeat steps 6-9 an additional 3 times (for a total of 24 hours = 1 cycle)					

Final Iteration ASTM D7869 Test Cycle



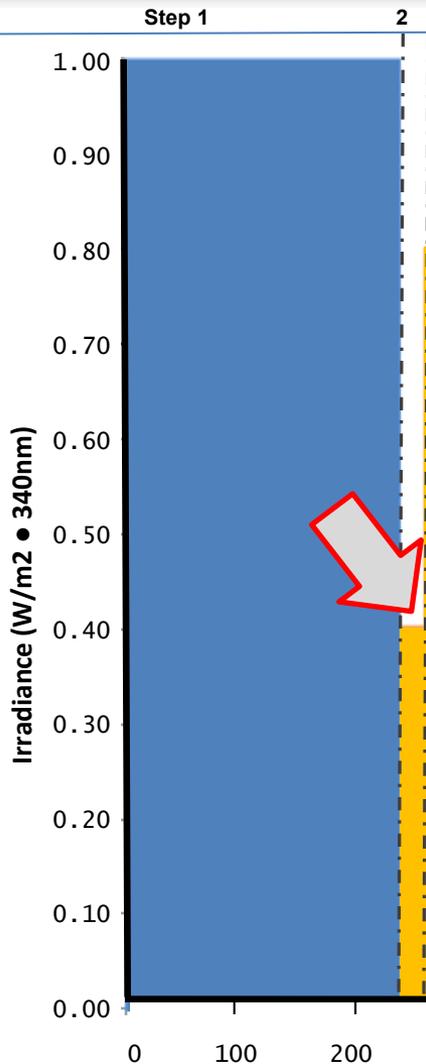
Step 1 – Deep Moisture Saturation



240 min, Dark + Spray, BPT 40°C, CHT 40°C, 95% RH

- **Purpose** - to produce water uptake within the coating that is similar to the maximum uptake in a normal day outdoors in south Florida
- Water uptake *more than* what is achieved in Step 1 did not produce significant changes in test results. However, water uptake *less than* what Step One achieves did fail to produce degradation of the types found in Florida
- Dark cycle, because almost all wetness in Florida occurs when there is no sunshine. The vast majority of wet time is caused by nighttime dew.
- Outdoor data show that natural specimen wet temperature is lower in Florida, typically 20°C to 25°C. But outdoor wet periods were also much longer, ranging from 8h to 16h. ***So 4h at 40°C produces similar water uptake to the much longer, but cooler, Florida wet periods***

Step 2 – Removing the water

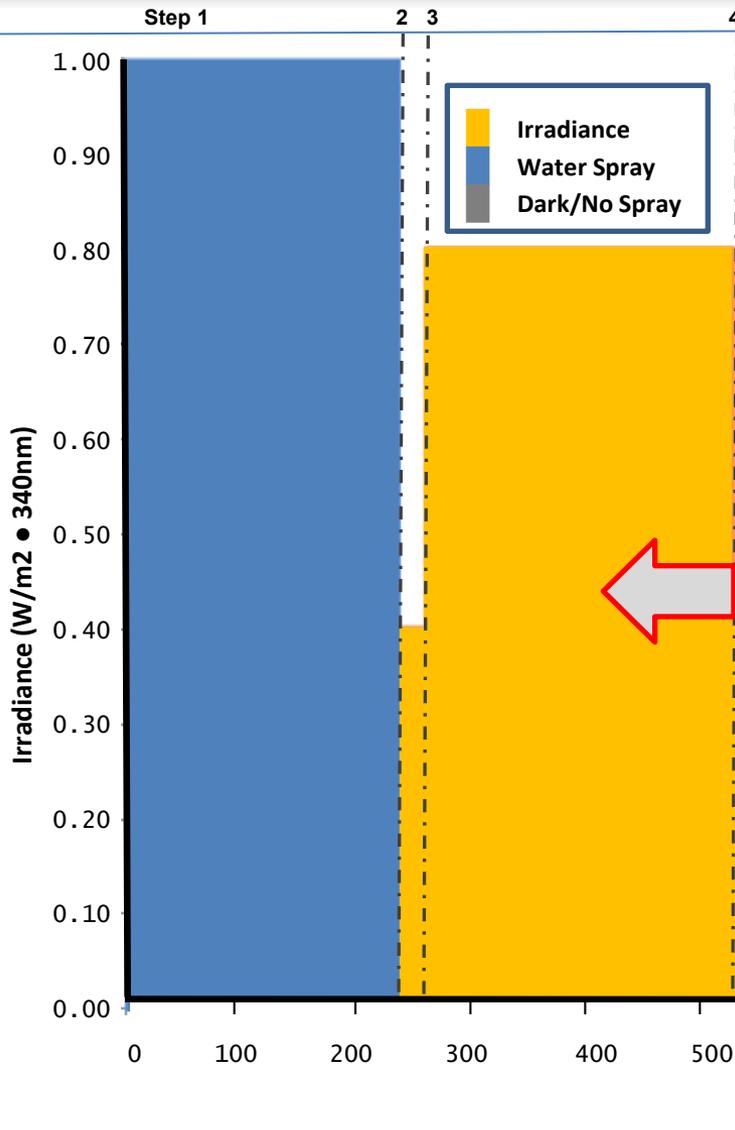


30 min, Light, 0.40 W/m² @ 340nm with new filter, BPT 50°C, CHT 42°C, RH 50%

- **Purpose** - to remove all of the water from within the coating layers
- The irradiance is set at a relatively low level, 0.40 W/m²/nm, **because Florida data has shown that all the water was driven out from the coating before the sun ever got high enough in the sky to produce higher irradiances**
- The Black Panel Temperature is set at 50°C, because Florida data has shown that by the time the sun heats the specimen to 50°C, almost all of the water has been removed from the coating
- A time of 30 minutes was chosen because data has shown that **30 minutes at 50°C is the time required to take the water content to near zero.**

Time (minutes)

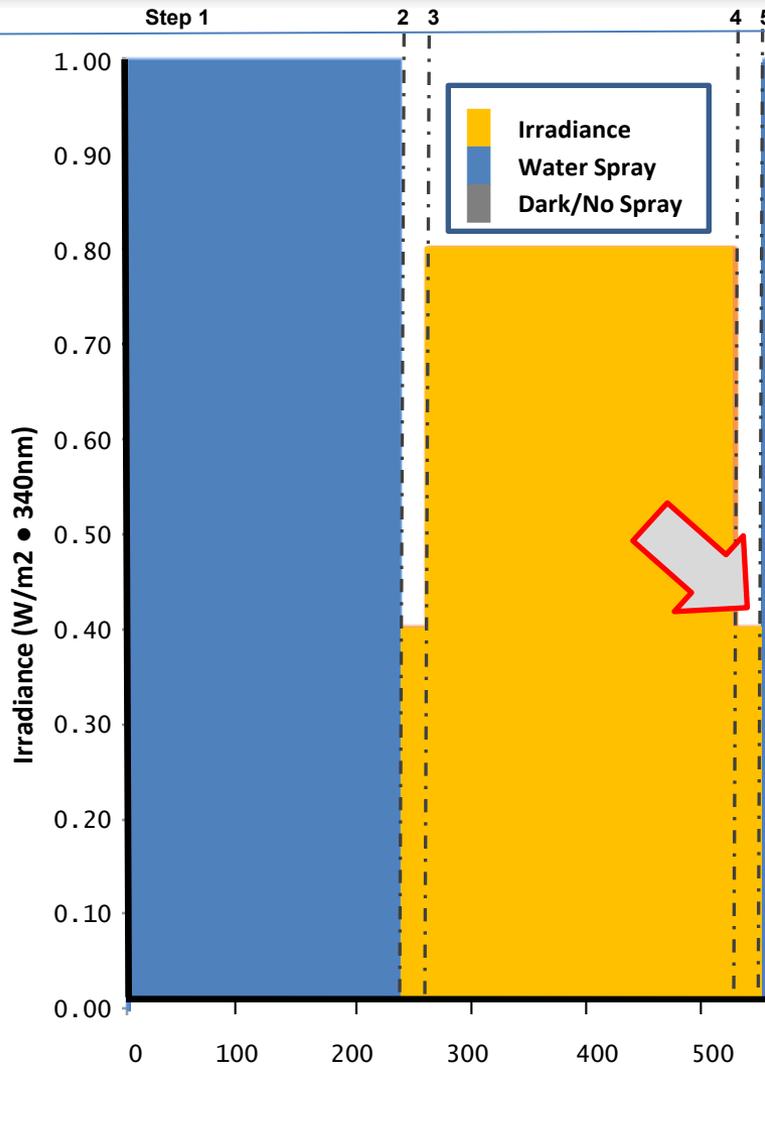
Step 3 – Exposure to the Right Light



270 min, Light, 0.80 W/m² @340nm, BPT 70°C, CHT 50°C, RH 50%

- **Purpose** – to simulate the effects of bright sunlight on the coatings.
- Most Florida sunlight exposure occurs at much lower irradiances than noon midsummer sunlight. So this irradiance can be expected to produce significant acceleration.
- **The irradiance is set at somewhat higher than the maximum irradiance seen in Florida with noon midsummer sunlight.**
- **The Black Panel Temperature is set at 70°C, because this approximates the maximum specimen temperature averaged across the color palette.**

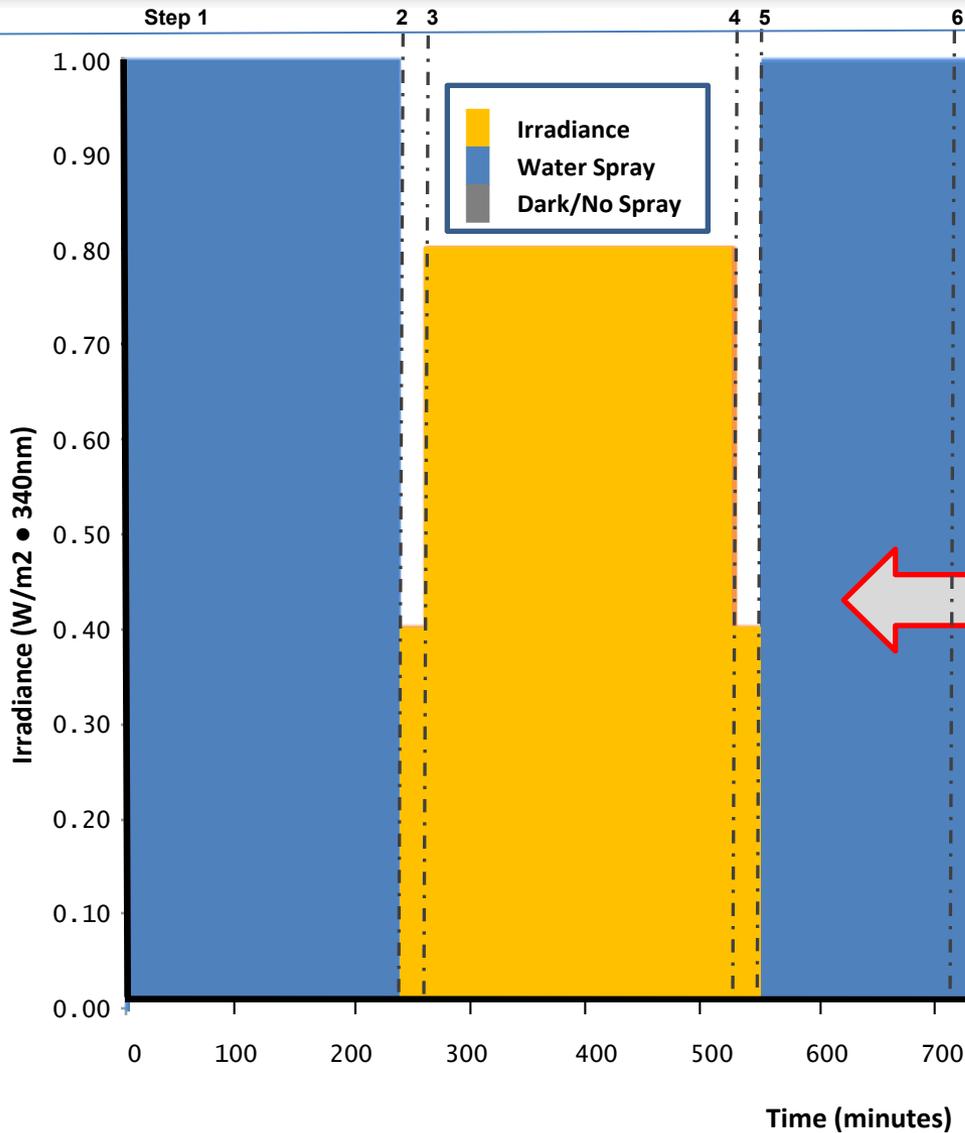
Step 4 – “Relaxation”



30 min, Light, 0.40 W/m² @ 340nm, BPT 50°C, CHT 42°C, RH 50%

- **Purpose - to transition** between the hot, high-irradiance “daytime” step and the dark, cool, wet “night time” step.
- This step gradually **reduces thermal stresses** within the coating, similar to what occurs as the sun gets lower in the sky during the evening.
- Unnatural effects can be produced if the test does not cool down the specimens before water is introduced. For instance, excessive cracking and micro cracking can be produced if cold water is sprayed onto a hot specimen.
- **The relatively low set points for irradiance and temperature are typical of what has been measured in FL late afternoon and early evening.**

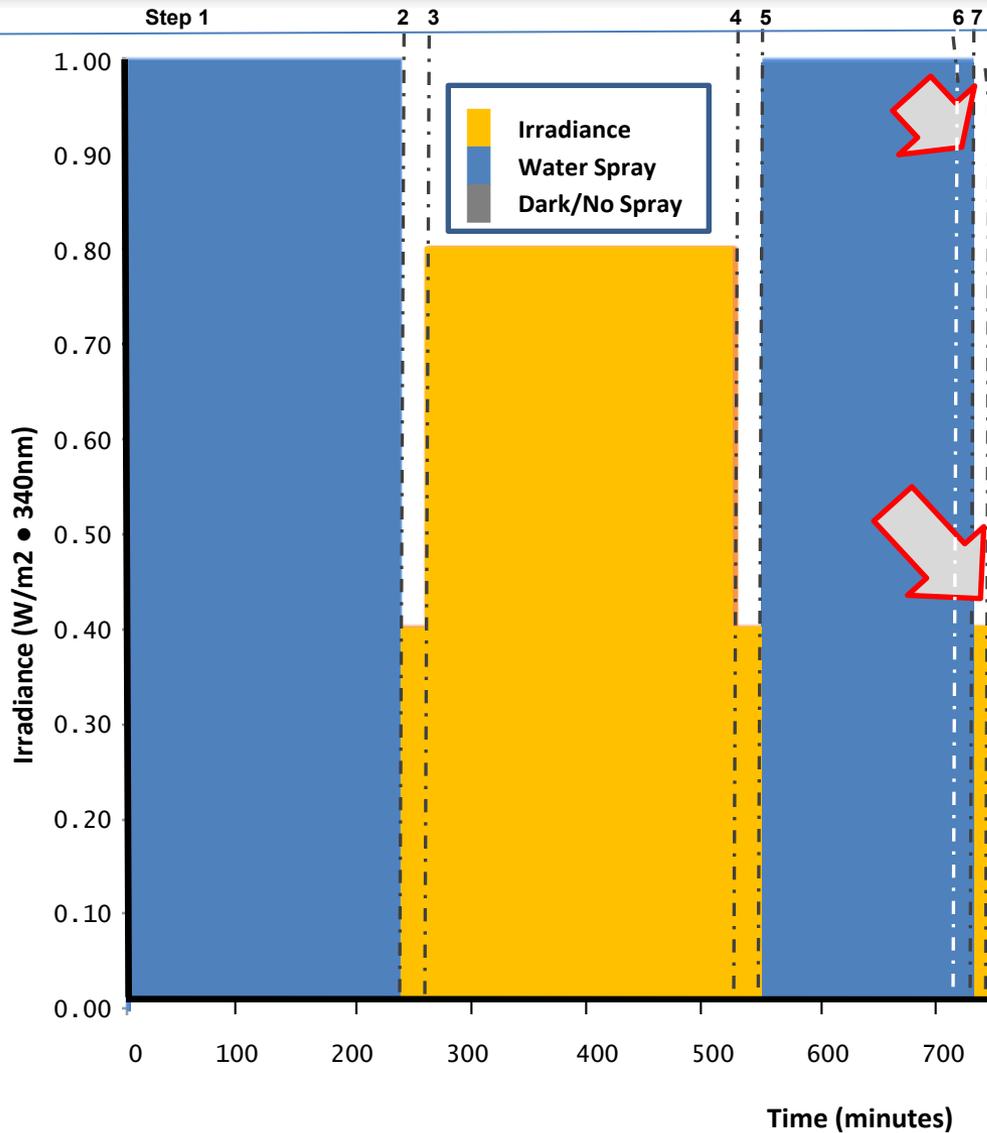
Step 5 – Water (Again)



**150 min, Dark + Spray, BPT 40°C,
CHT 40°C, 95% RH**

- **Purpose** - to produce significant water uptake within the coating, but at somewhat less than the maximum uptake observed
- The temperatures and humidities in Step 5 are the same as in Step 1, for the same reasons.
- **The data has shown that the maximum water uptake does not occur every day in Florida. This step is intended to simulate those days of less than maximum uptake.**

Steps 6 & 7 Rain Event and Controlled Dry Out



30 min, Dark + Spray, BPT 40°C, CHT 40°C, 95% RH

- **Purpose** – to simulate a very short time water event, such as a night where little condensation occurs, **or a very short rain event**

20 min, Light, 0.40 W/m² @340nm, BPT 50°C, CHT 42°C, RH 50%

- **Purpose** – to **remove the water** from the coating at a controlled rate

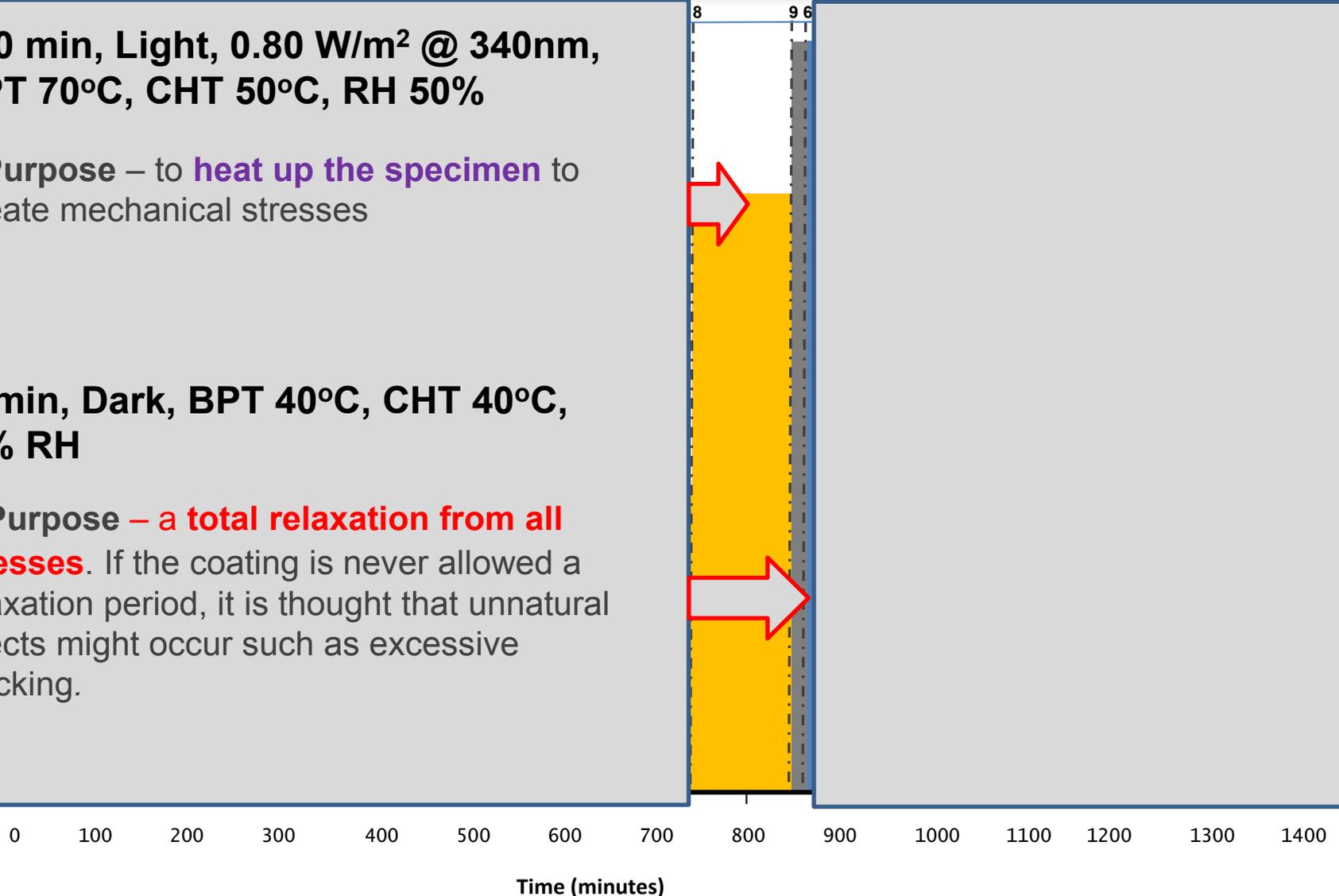
Steps 8 & 9 – Heat/Mechanical Stress & Relaxation

**120 min, Light, 0.80 W/m² @ 340nm,
BPT 70°C, CHT 50°C, RH 50%**

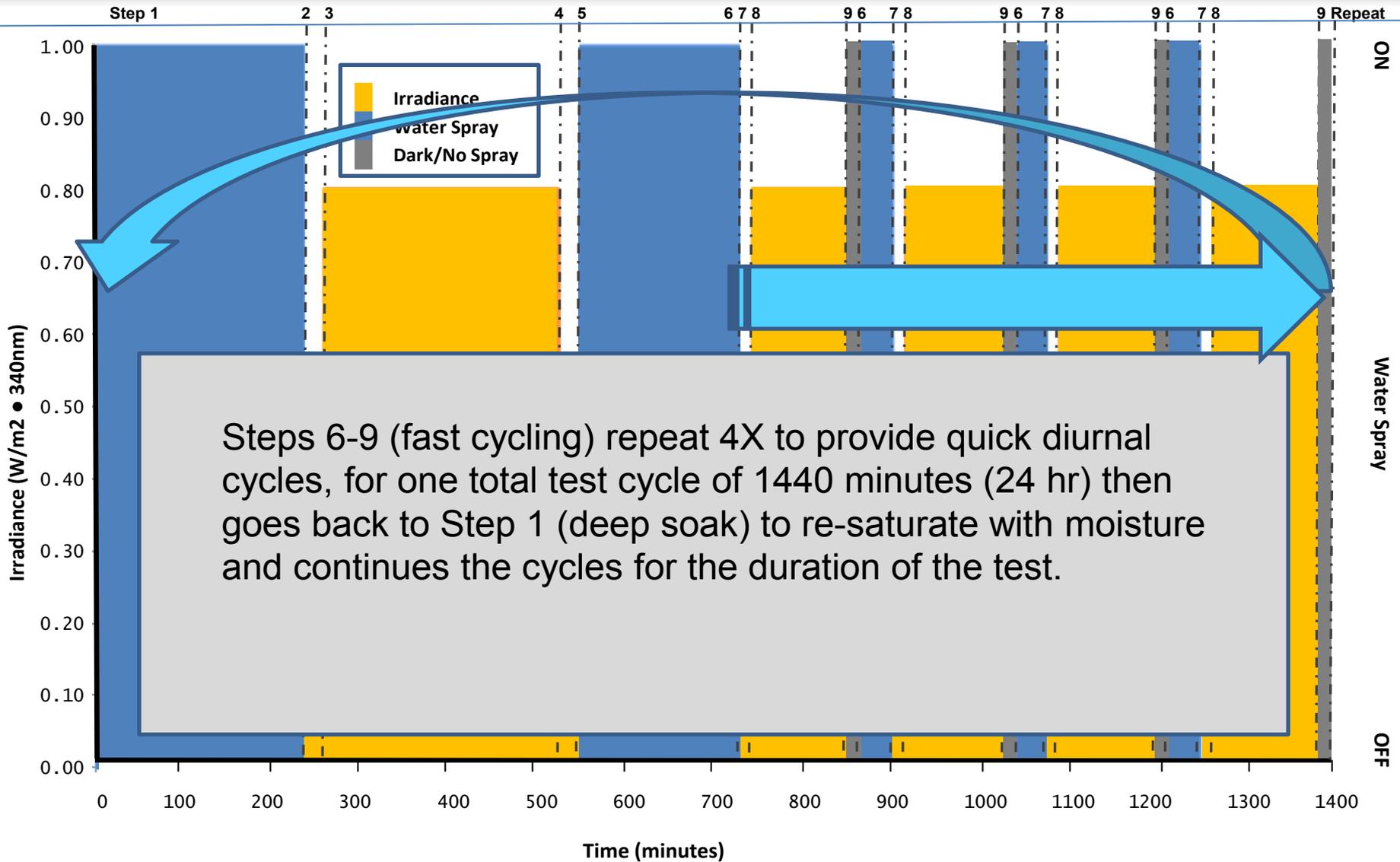
- Purpose – to **heat up the specimen** to create mechanical stresses

**10 min, Dark, BPT 40°C, CHT 40°C,
50% RH**

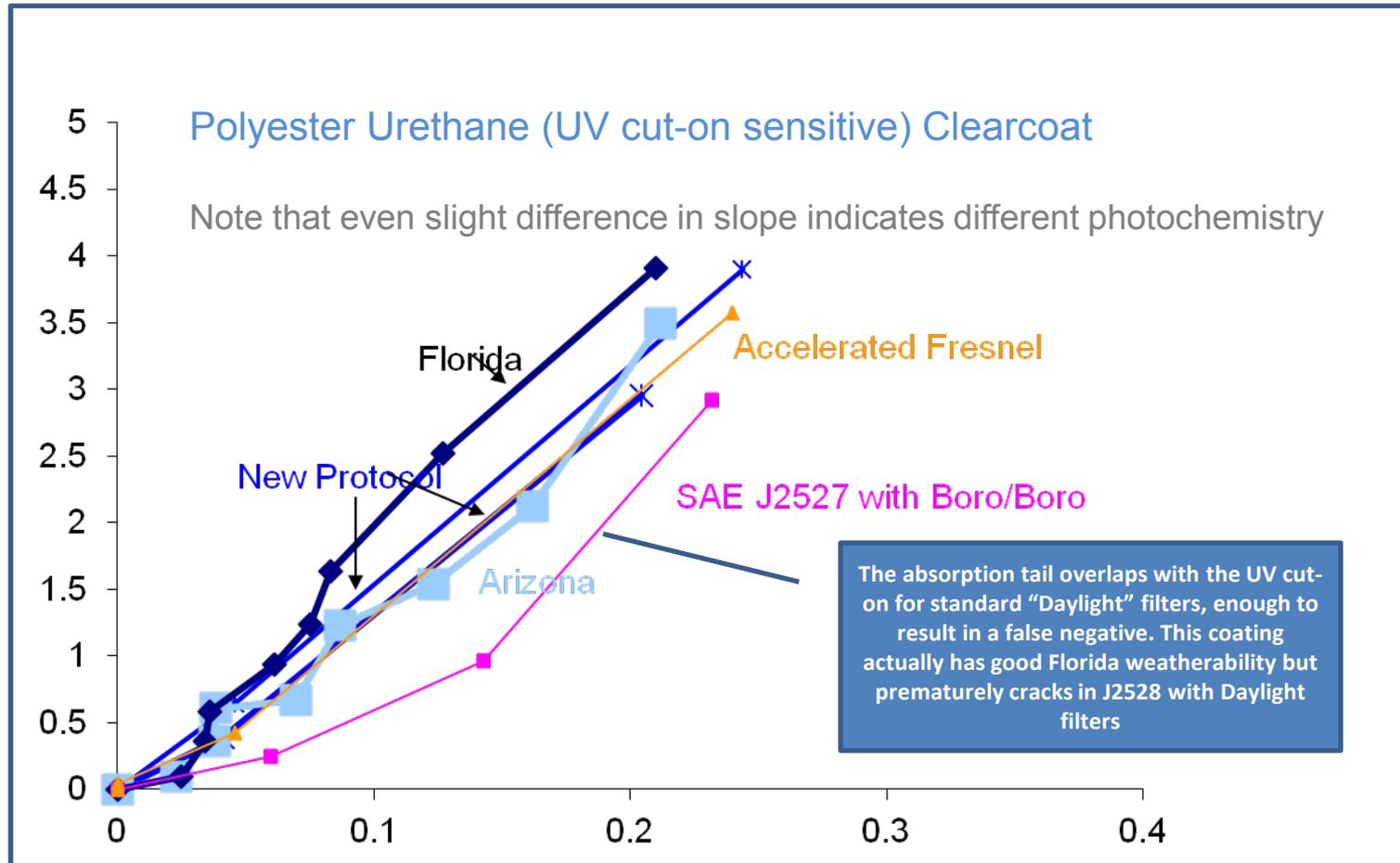
- Purpose – **a total relaxation from all stresses**. If the coating is never allowed a relaxation period, it is thought that unnatural effects might occur such as excessive cracking.



Final Iteration ASTM D7869 Test Cycle

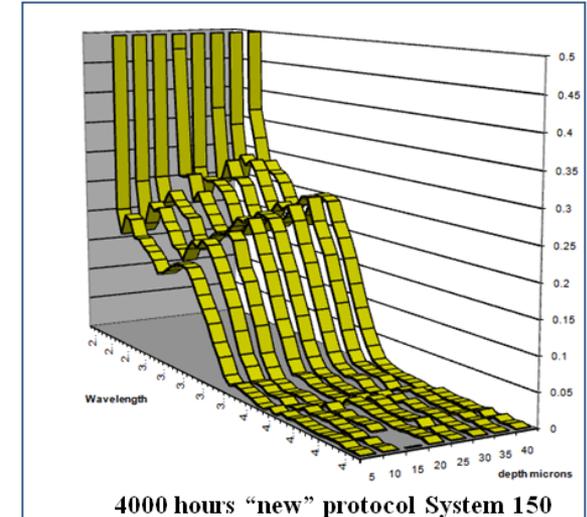
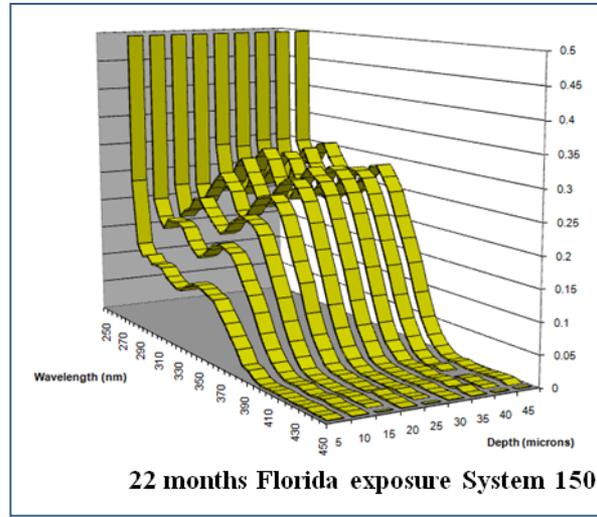
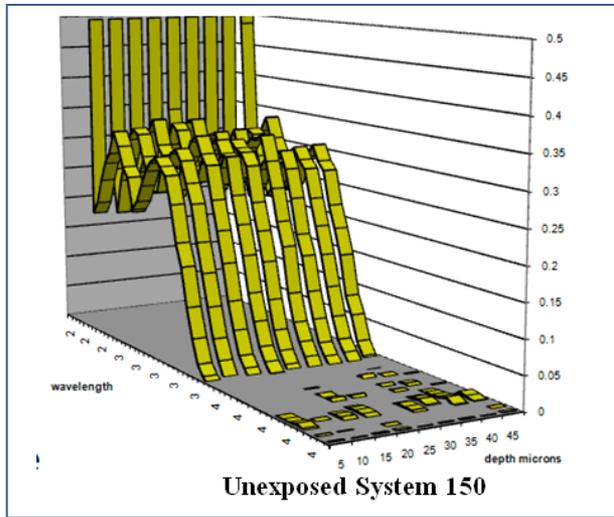


But Does It Work?

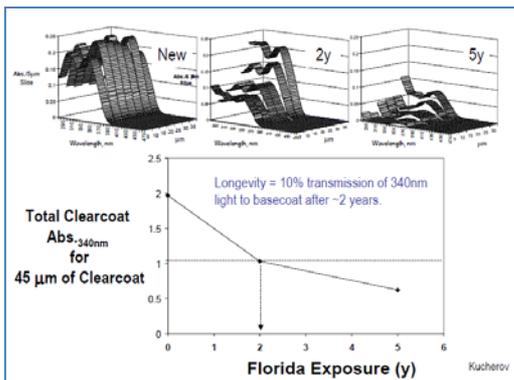


But does it work?

Gradient loss of UV absorber (from free radical attack) Carbamate SBBC



Source: M.Nichols, *et al*, "Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results.", 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011



Loss of UV absorber in CC consistent with Florida results

← Note failed system allowed 10% UV transmission in 2 yrs FL

Source: M.Nichols, *et al*, "Accelerated Weathering of Automotive Coatings: Exposure Conditions and Analysis Methods", Atlas Technical Conference on Ageing in the Environment, Oxford, UK, September, 2008.

But did it work?

System 97 2K Polyurethane WBBC on blue basecoat

Florida Exposure

J2527

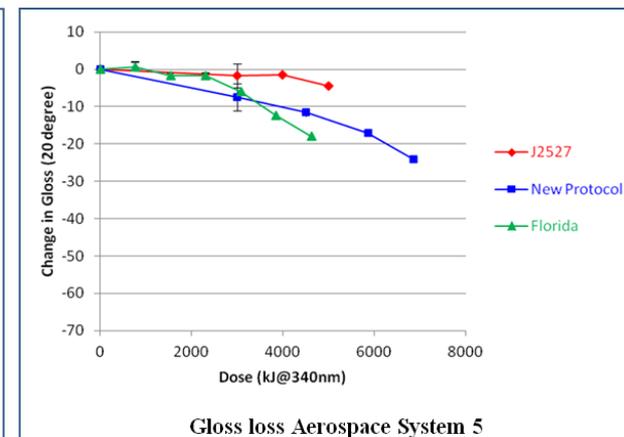
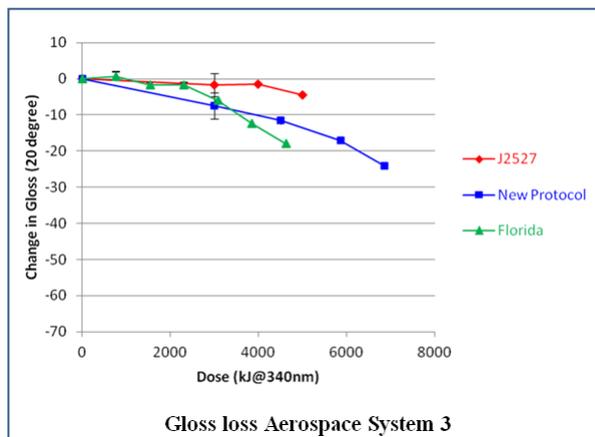
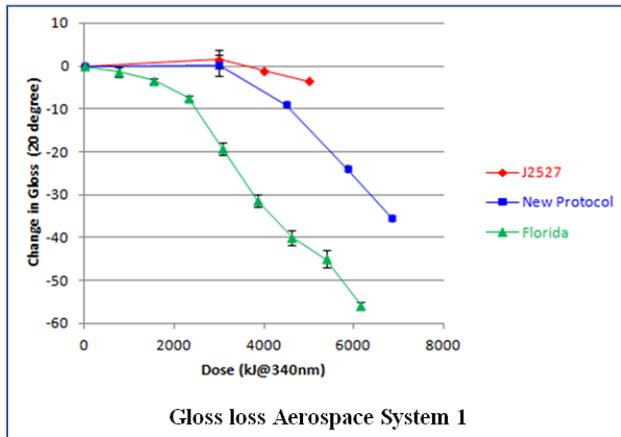
New Protocol



System 97 failed two years Florida exposure by delamination of the clearcoat from the basecoat, and also had severe blistering of the clearcoat. The same system in the new protocol also exhibited clearcoat delamination and blistering while SAE J2527 showed neither delamination failure nor blistering.

But did it work?

Comparison of monocoat aircraft coatings for gloss loss – 3 different paint systems



■ Florida ■ J2527 ■ New Protocol

The inconsistent correlation of accelerated weathering results with South Florida performance has been a hindrance to aerospace coatings development. Coatings that perform well in accelerated testing have failed prematurely in service, while others, whose performance was differentiated in accelerated testing, performed equivalently in service.

These graphs illustrate this point where it is shown that SAE J2527 cannot discriminate between the performance of monocoat and basecoat/clearcoat systems while Florida shows significant differences.

Source: M.Nichols, *et al*, "Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results", 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011

Still work to do for aerospace – higher UV levels (*but not lower cut-on*), altitude freeze, O₂, other ????

It works – further improvements still to be made

New Protocol Results Compared to SAE J2527 and Florida – Automotive

- Physical failures correctly reproduced
- Degradation chemistry is correct
- Additive loss rate – better than J2527, slower than Florida???
- New protocol is as fast as J2527 on a dose basis
- New protocol is ~40% faster on a time basis due to increased irradiance level during light cycle and improved water distribution in paint system
- Acceleration Factor 7 → 10

Source: M.Nichols, *et al*, “Accelerated Weathering Testing: A New Approach to Anticipating Florida Exposure Results”, 2011 Coatings Science International, Noordwijk, Netherlands, June 30, 2011

- ✓ The new protocol is now required by Ford worldwide to qualify all new paint systems and external components (roof racks, side mirrors, bumper fascia, badges, etc).
- ✓ Honda Motor is rolling out the method as a requirement for their suppliers.
- ✓ Other OEMs and groups have expressed interest in the new method

Implications for PV materials and module testing

Key takeaways for PV:

Serves as a model for science-based test method development

- Weathering can be complex: both both chemical and physical
- Chemical - Full spectrum light source required - proper activation energies
- Can't ignore material-specific degradation mechanisms
- Physical – Full spectrum source for appropriate thermal heating effects
 - Stress material to material interfaces (no fluorescent UV testing) similar to nature
- Chemical - Get the Right Light for proper actinic reactions in critical, high energy UV cut-on region – don't alter the photochemistry for “speed”
- Physical / Chemical – Cycling is important - steady state isn't natural
 - Material to material interface (adhesion)
 - Promotes cracking, delamination, corrosion as seen in nature
 - Transitions are where much of the stress occurs
- Need to match service environment climate conditions and cycles for the test to be predictive – *implications for climate-based module durability ratings and suitability for use of specific materials*

Thank you!



Questions?

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