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# ***BASF Accelerated Acid Test***

BASF Corp. and Q-Lab Corporation®

“A 400 Hour of Laboratory Test Simulates 14 Weeks  
Outdoor Exposure in Jacksonville”

Bridget Murray, Q-Lab Corporation



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# *History and Background Information*



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## *History/Background*

Acid rain damage became a problem in the 1980's with the use of automotive clearcoats





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## *History/Background*

Blount Island (Jacksonville, Florida) is the key location for etch testing for Automotive companies and their Coating suppliers

### **WHY?**

**1. It is the busiest vehicle handling port in the United States.**





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## *Neighbors and Environment of Blount Island*

coal and oil fired  
electrical power  
plants,  
  
the salt  
atmosphere of the  
seaport,  
  
and the heat and  
humidity  
  
**.... Acid Etch**





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## *History/Background*

Panels, hoods, and fascia are tested on Blount Island each and every summer for 14-16 weeks

The panels are visually evaluated for etch damage using standard panels as a guide



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## *Other Accelerated Tests*

- Many laboratory techniques exist to predict etch resistance of Automotive clearcoats
  - Gradient Bar
  - Acid Spot
  - Palm and Carlsson paper (JCT – Vol 74 2002) and Schulz /Trubiroha/Schernau/Baumgart paper (Progress in Organic Coatings – Vol 40 2000) started exploration in the use of multiple field components in efforts to develop tests using acid rain



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## *Other Accelerated Tests*

- Some of these do not correlate because they lack critical field components such as humidity or UV light
- Others do not correlate because they lack horizontal panel orientation which produces a different panel appearance
- When all the components are taken into effect, panels can be generated with the same visual appearance as field etch panels



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## *Development of Current Test Procedure*

- Charge from BASF's marketing group to create a realistic laboratory etch test
- Development of the Q-Sun® Xenon Test Chamber which tests parts and panels in a near horizontal orientation
- Idea to use the Q-Sun® to duplicate field etch conditions
- Joint development agreement with Q-Lab® in April 2002 to specifically develop a realistic acid etch test



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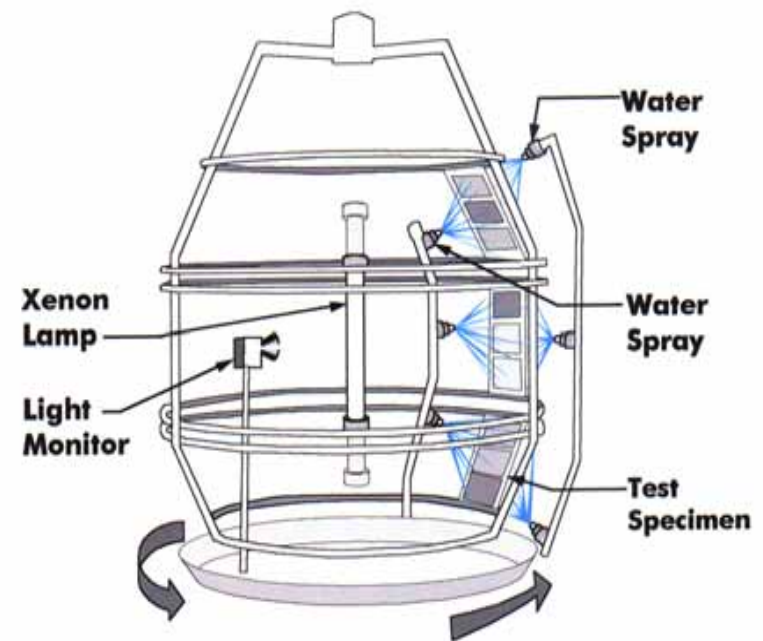
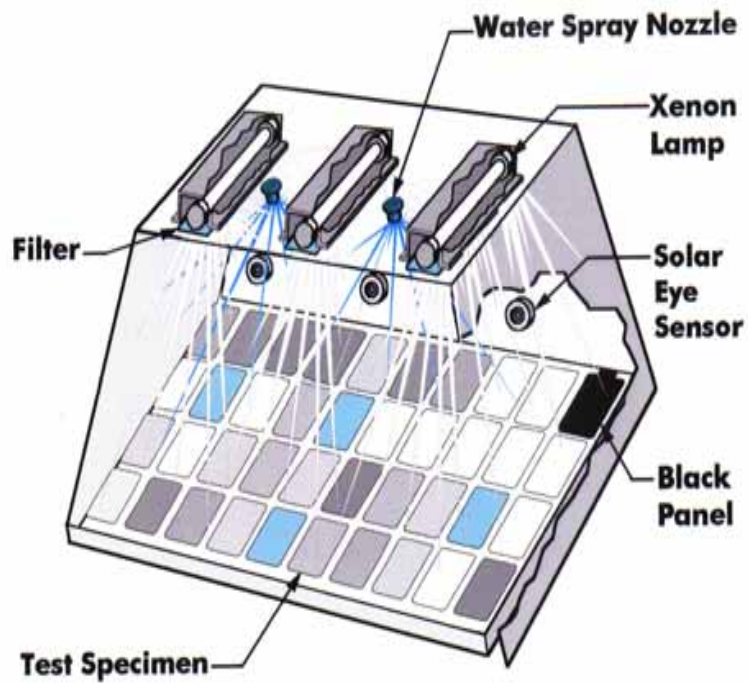
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# *The Q-Sun® Xe-3*



## Flat Array Q-Sun

## Rotating Drum Style Tester



## Conditions Considered in Acid Etch Test Development



- Temperature – match the maximum temperature as measured on parts/panels in field etch testing

- Rainfall – identify and use a rainfall of a specific pH and chemical content that is known to produce etch



**(RH)**

- Humidity – maintain a percent relative humidity that is consistent with the testing environment (a slow dry off condition)



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## *Conditions Considered in Acid Etch Test Development*



Orientation – use a test angle that is similar to the most severe field exposure condition



UV light – introduce UV light similar to the distribution and intensity seen in the field testing



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## *Jacksonville Specimen Temperatures*

Maximum Temperature	Pyrometer 2002 (ambient 31 C)	Thermocouple 1993 (ambient 36 C)
Steel Hood	72 C	80 C
Steel Panel	63 C	74 C
Rim panel/fascia	58 C	69 C

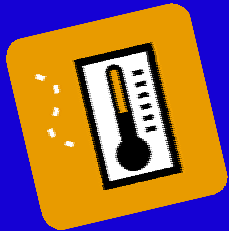


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## ***Test Conditions: Laboratory Acid Etch Test Temperature***



Based on the Blount Island  
(Jacksonville, FL) data, 80°C was  
chosen as the black panel  
temperature in the Q-Sun test  
chamber



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## *Rainfall*

- It is believed that trace afternoon rainfalls (less than 0.1 inch) with low cloud cover are the most responsible for producing etch
- Lower pH rainfalls are the most responsible for producing etch, such as the 3.49 pH rain collected in Jacksonville in 1989
- Weather data from Jacksonville indicates an average of 10-15 days of this type in June-August each year
- The pH and composition of the acid rain used is based from analysis of an actual Jacksonville rain



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## *Days with <0.1 inch rain 11am-4pm*

Year/month	June	July	August	Total
2002	5	3	2	10
2001	7	4	2	13
2000	9	3	3	15

The current test cycle uses approx. 13 acid rain spray steps



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## ***Test Conditions: Laboratory Acid Etch Test Rainfall***

The pH and composition of the acid rain used in the Q-Sun test chamber is based on analysis of actual Blount Island rainfall

The final simulated rain used in the Q-Sun test chamber will have a pH of 3.4.

The current test cycle uses approx. 13 acid rain spray steps

Recipe Contains:

- Calcium Chloride
- Potassium Chloride
- Sodium Hydroxide
- Nitric Acid
- Sulphuric Acid
- De-ionized Water





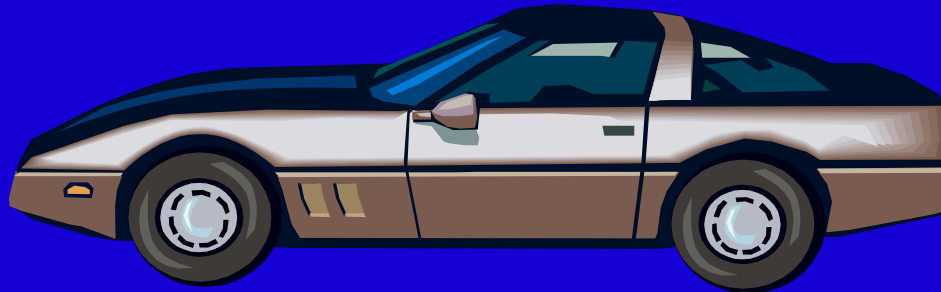
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## *Humidity and Orientation*

- In most summer days in Florida, dew forms on parts/panels that can last for many hours, and the relative humidity can average around 80% for the summer months (June – Aug)
- Test panels and parts are typically placed at 0-5 degrees for etch testing similar to the angle of exposure on an automobile. The Q-Sun was modified for the test panels to be at a 0° orientation.





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## *Test Conditions: Laboratory Acid Etch Test Humidity and Orientation*

**(RH)**

High humidity (80%) is maintained throughout the test cycle in the Q-Sun test chamber to simulate the summer environment in Blount Island



Q-Sun® was modified to position the test panels at a 0° orientation



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***Q-Sun  
Xe-3  
HDS***



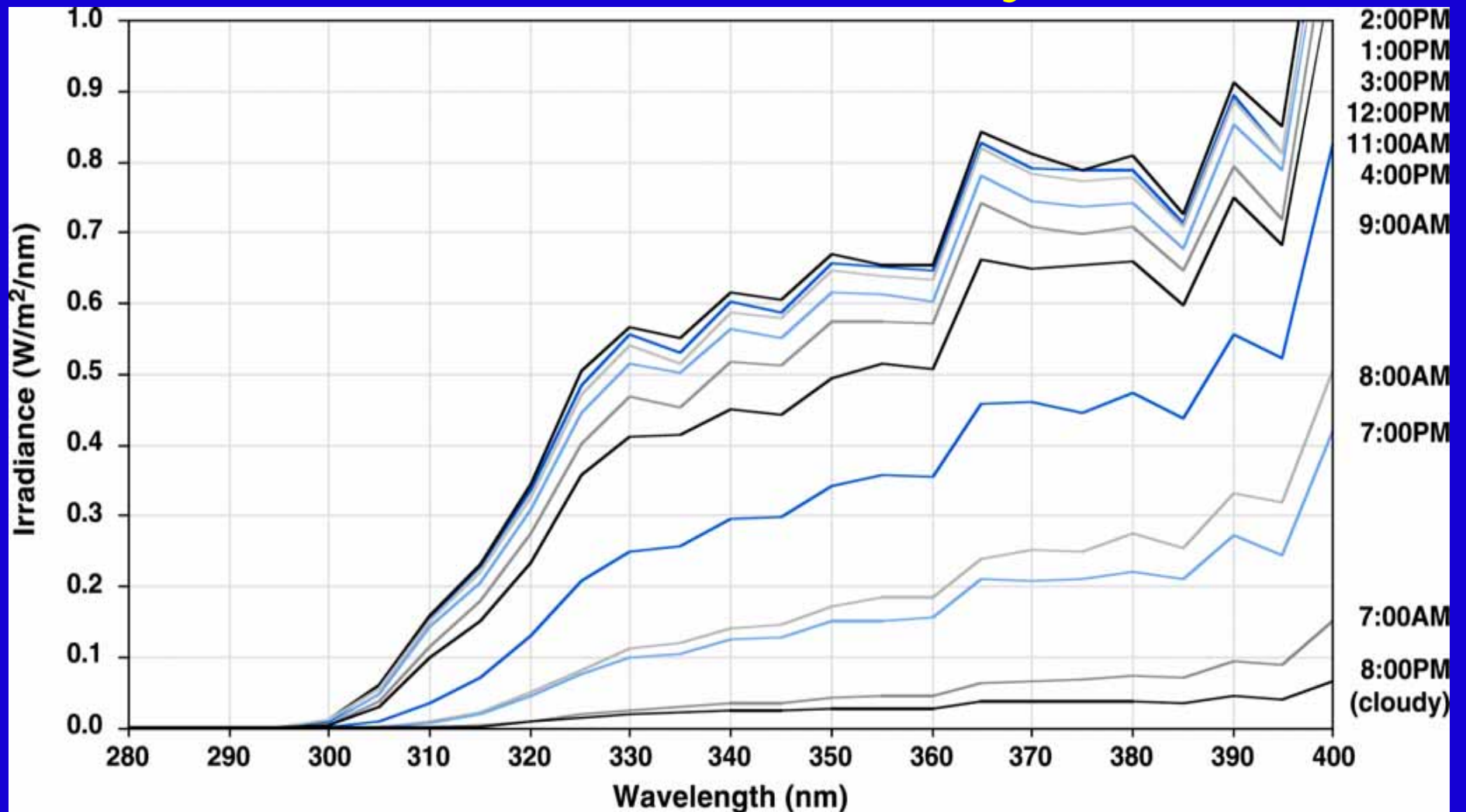


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# Solar SPD – All Day



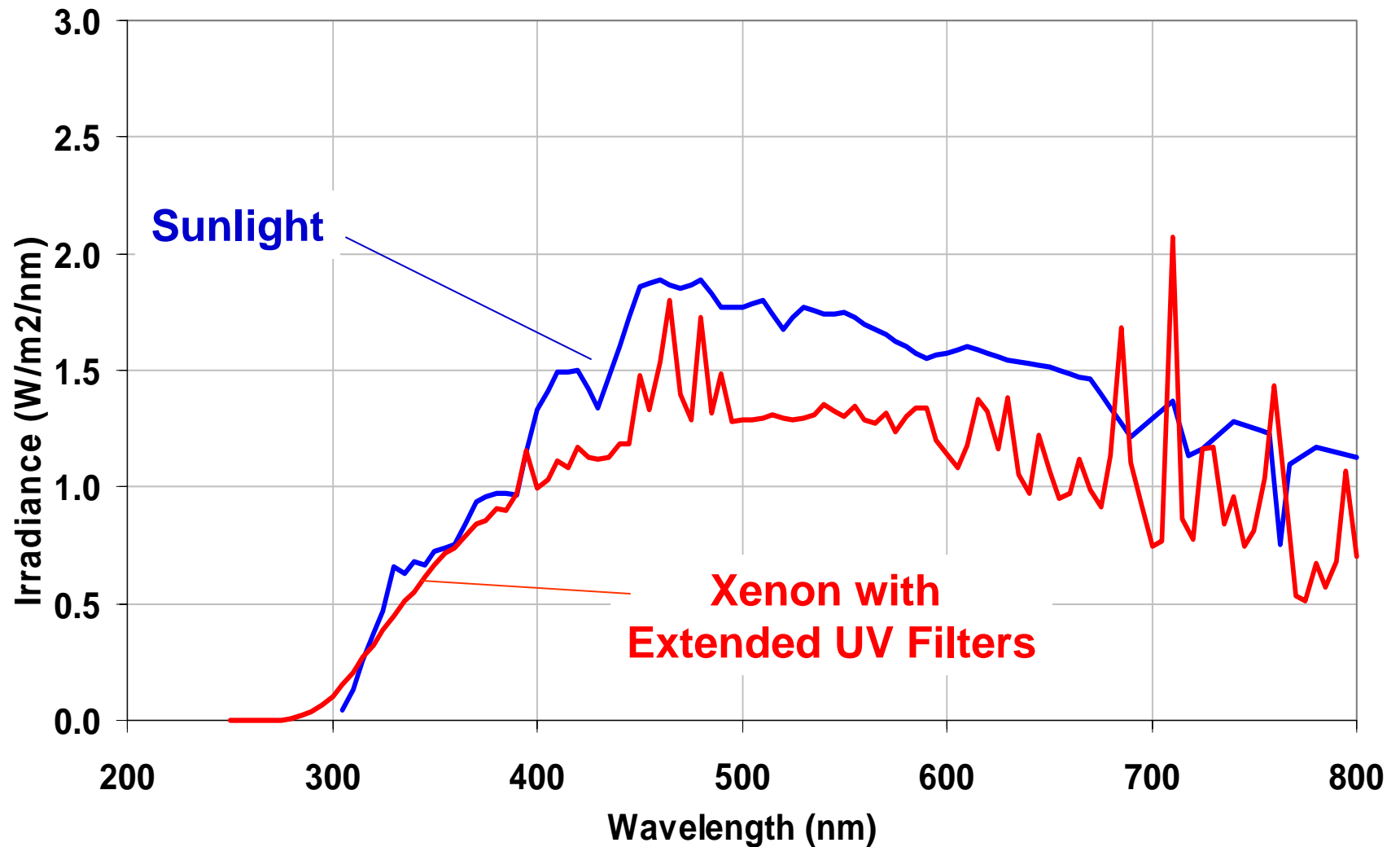


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# Xenon with Extended UV Filters



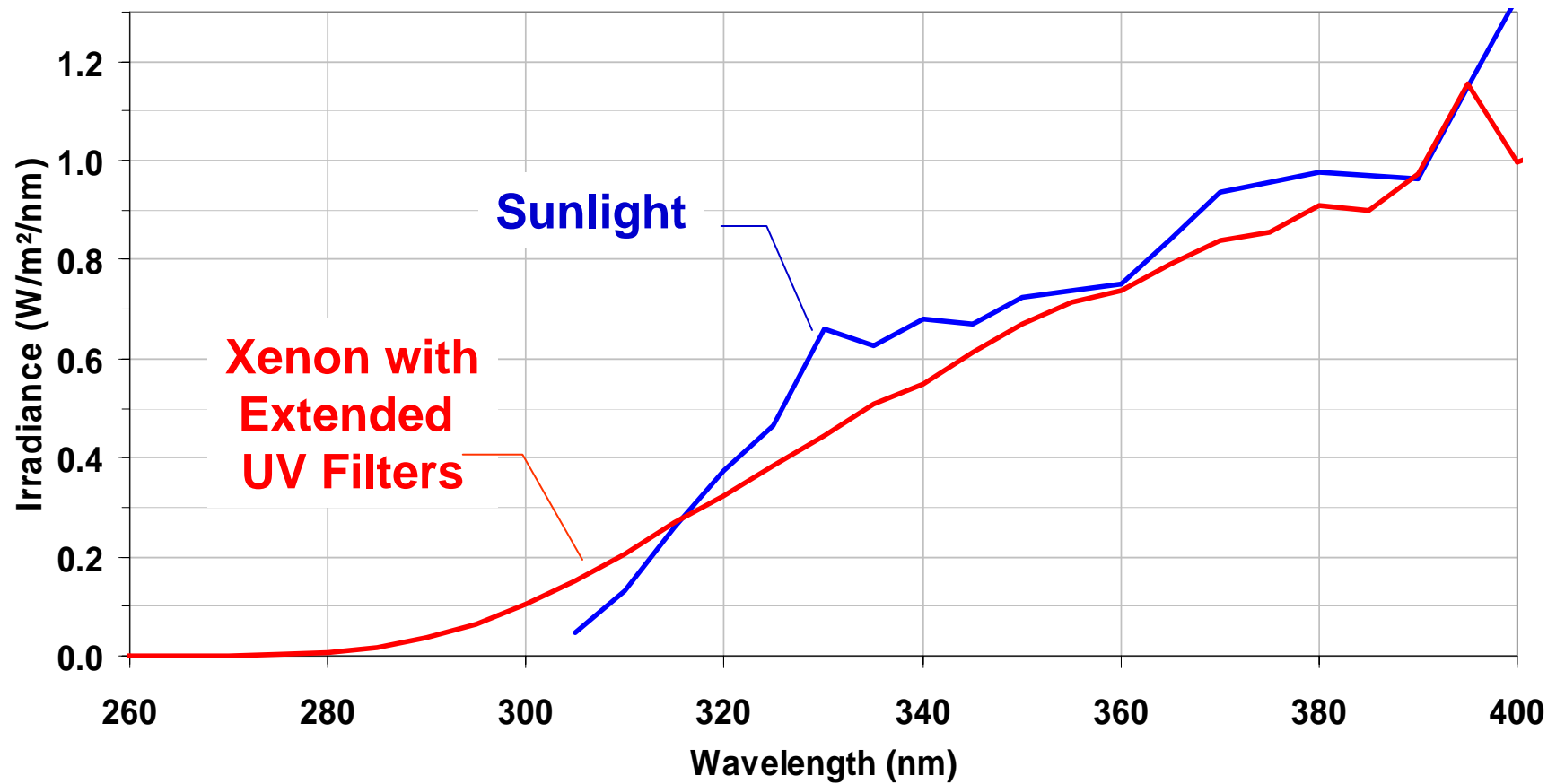


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# Xenon with Extended UV Filters



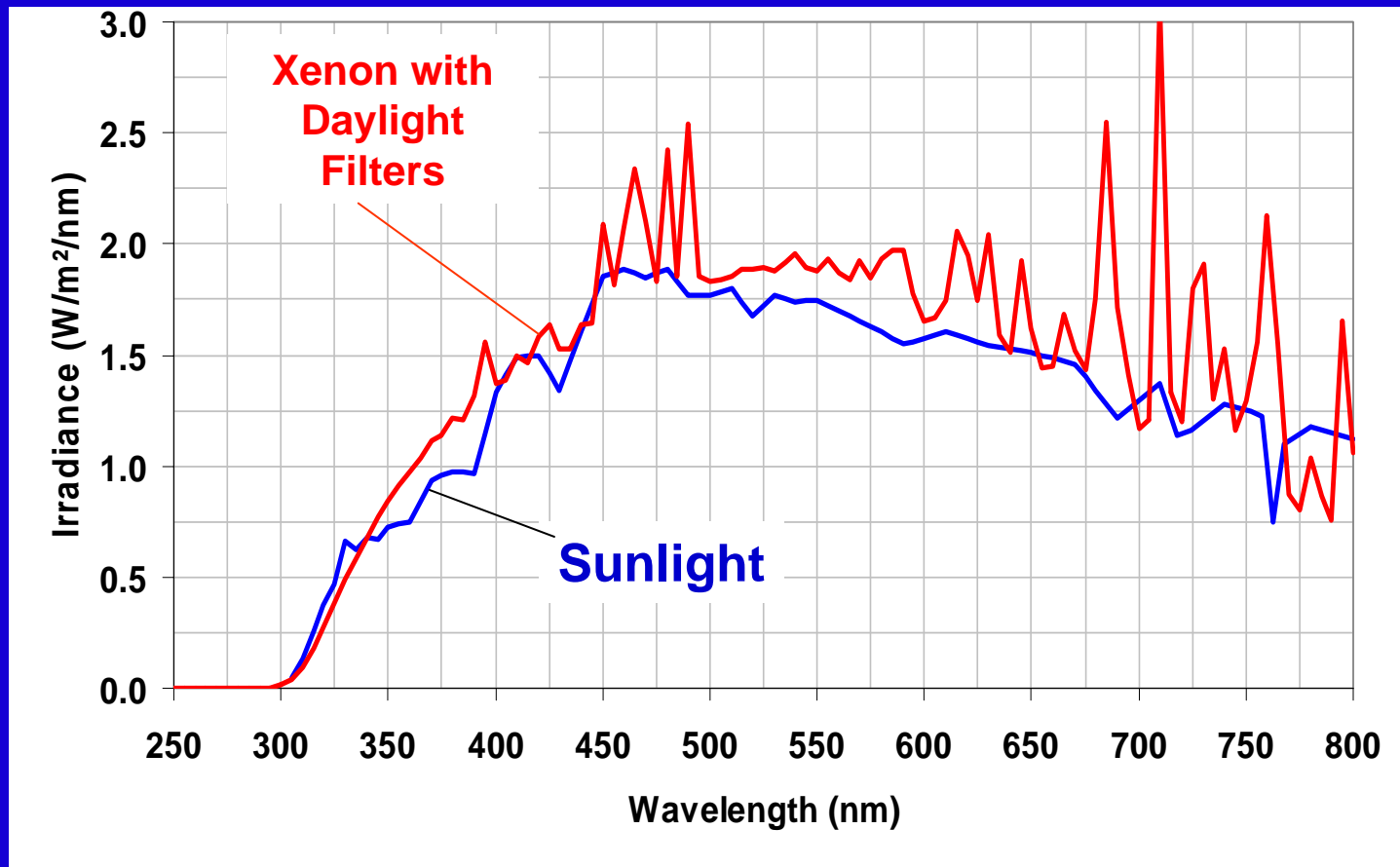


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# Test Conditions: Laboratory Acid Etch Test UV Light



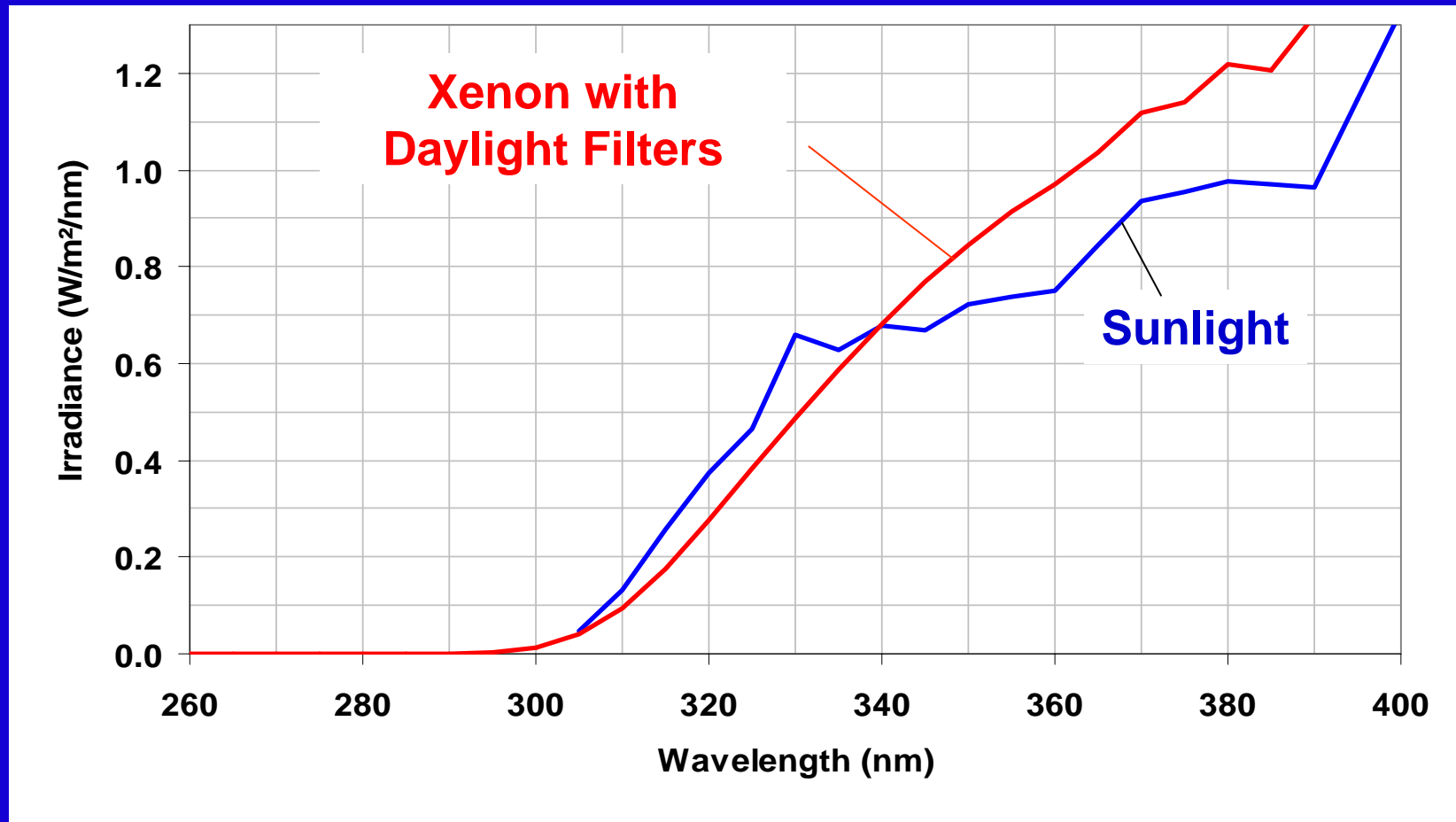


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# Test Conditions: Laboratory Acid Etch Test UV Light





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## Factors

Properties	Service Environment	Q-Sun® Conditions
Temperature	70-80 C	80 C
Rainfall	10-15 real events, lowest pH 3.5	13 acid sprays, pH 3.4
Dew	Evening Dew	Dark + Spray
Humidity	Typically 80% or greater RH	Maintain 80% RH throughout test
Orientation	Zero to five degrees	Zero degrees
UV Light Spectrum	Variable Spectrum	Noon summer sunlight
UV Light Intensity	0 – 0.68 W/m <sup>2</sup> at 340 nm (changing)	0.55 W/m <sup>2</sup> at 340 nm (constant)



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## ***BASF Accelerated Acid Etch Test Cycle***

Step 1	1 minute	Dark Exposure with Acid Rain Spray
Step 2	3 hours 50 minutes	Dark Exposure, 38 C panel and air temp, 80% RH
Step 3	12 hours	Light exposure 0.55 W/m <sup>2</sup> /nm at 340nm, Daylight filters, 80 C black panel temp., 55 C chamber air temp., 80% RH
Step 4	27 minutes	Dark Exposure, 38 C panel and air temp, 80% RH
Step 5	1 minute	Dark exposure with pure DI water spray
Step 6	3 hours 50 minutes	Dark Exposure, 38 C panel and air temp, 80% RH
Step 7	1 minute	Dark exposure with pure DI water spray
Step 8	3 hours 50 minutes	Dark Exposure, 38 C panel and air temp, 80% RH
Step 9	Repeat	Step #1



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## Acid Etch Visual Evaluation Procedure

Etch Rating (0 to 10 scale)	Description
0 to 3	The Etch is slight and only noticed by a trained observer (0-1 micron deep, 0-1% area damaged)
4 to 6	The etch would be slight to moderate and in some cases would be noticed by an untrained observer, such as a vehicle owner (1-3 micron deep, 1-3% area damaged)
7 to 10	The etch is severe enough where many vehicle owners would notice and potentially file warranty complaints(3-6 microns deep,3-6% area damaged)

At times, rating numbers over 10 are used for finer discrimination

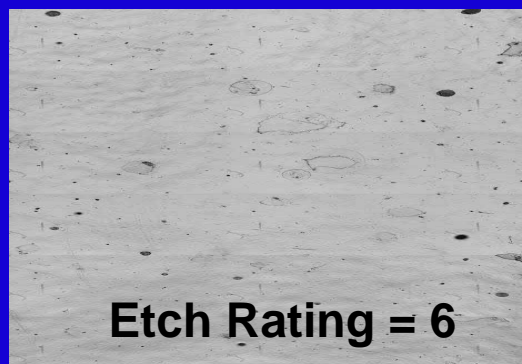


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## *Visual Rating Illustrations*



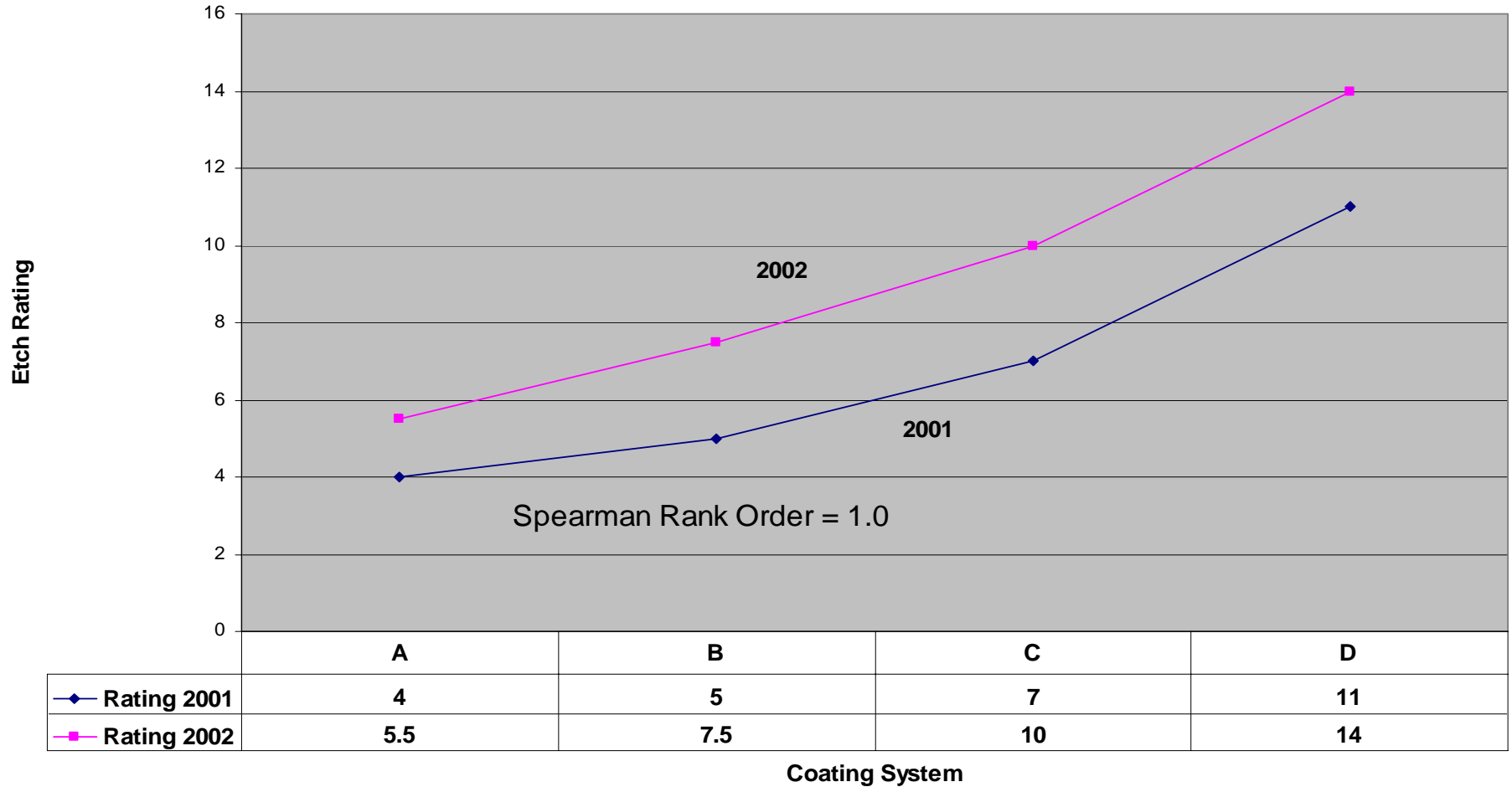


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# Jacksonville 2001 vs. 2002 field results



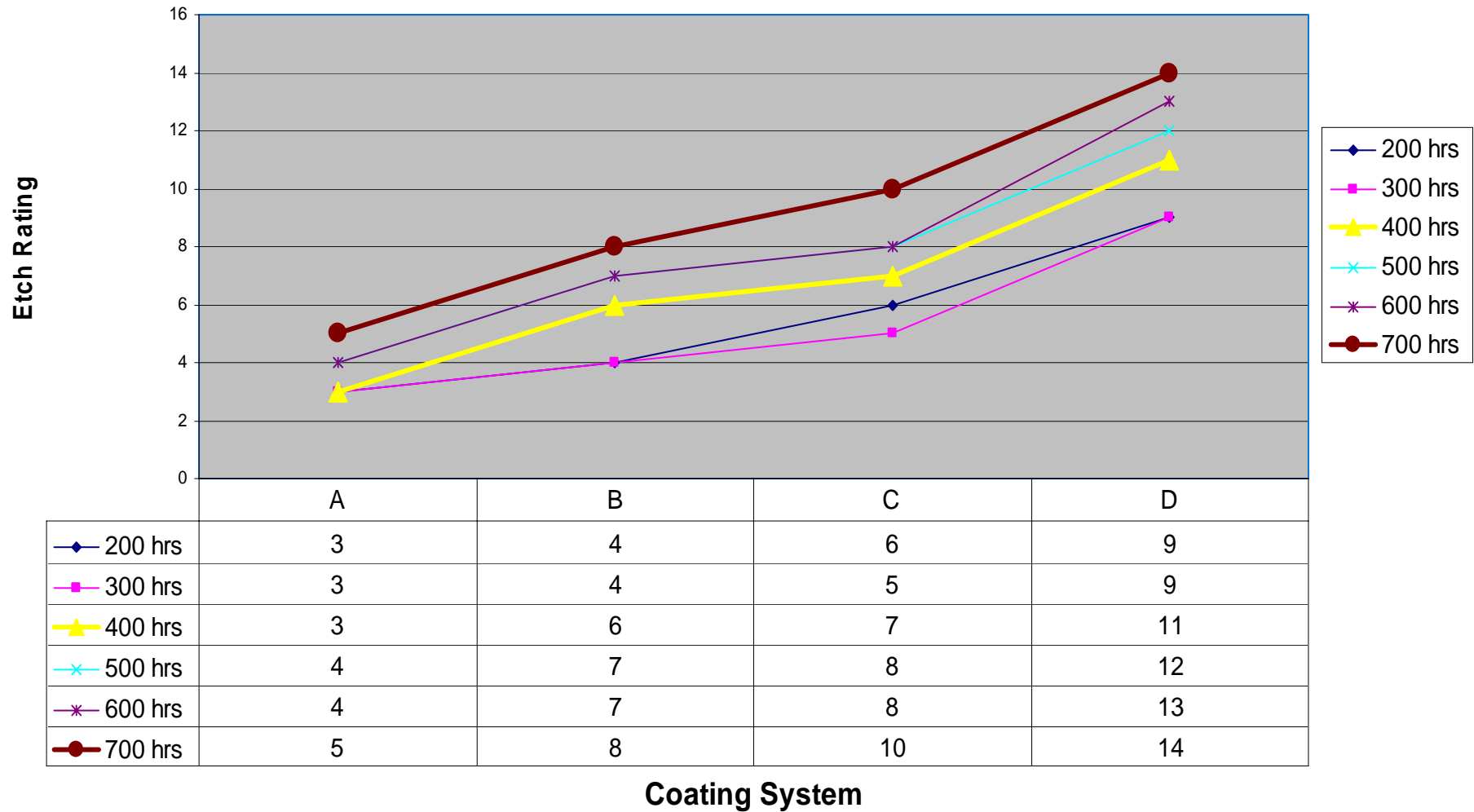


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# BASF/Q-Sun Results – Ratings



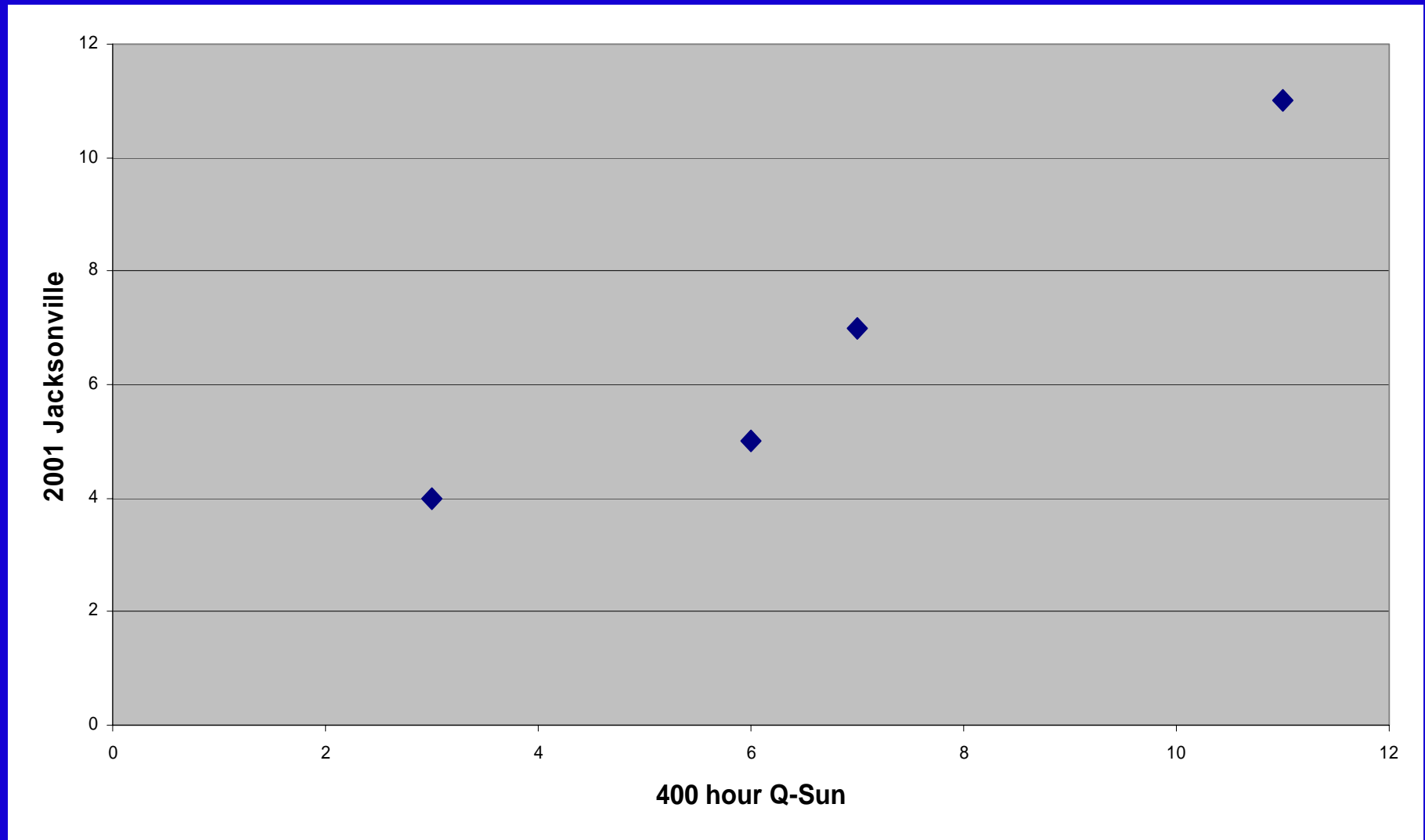


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## 2001 Jacksonville vs BASF/Q-Sun 400 hours – Ratings



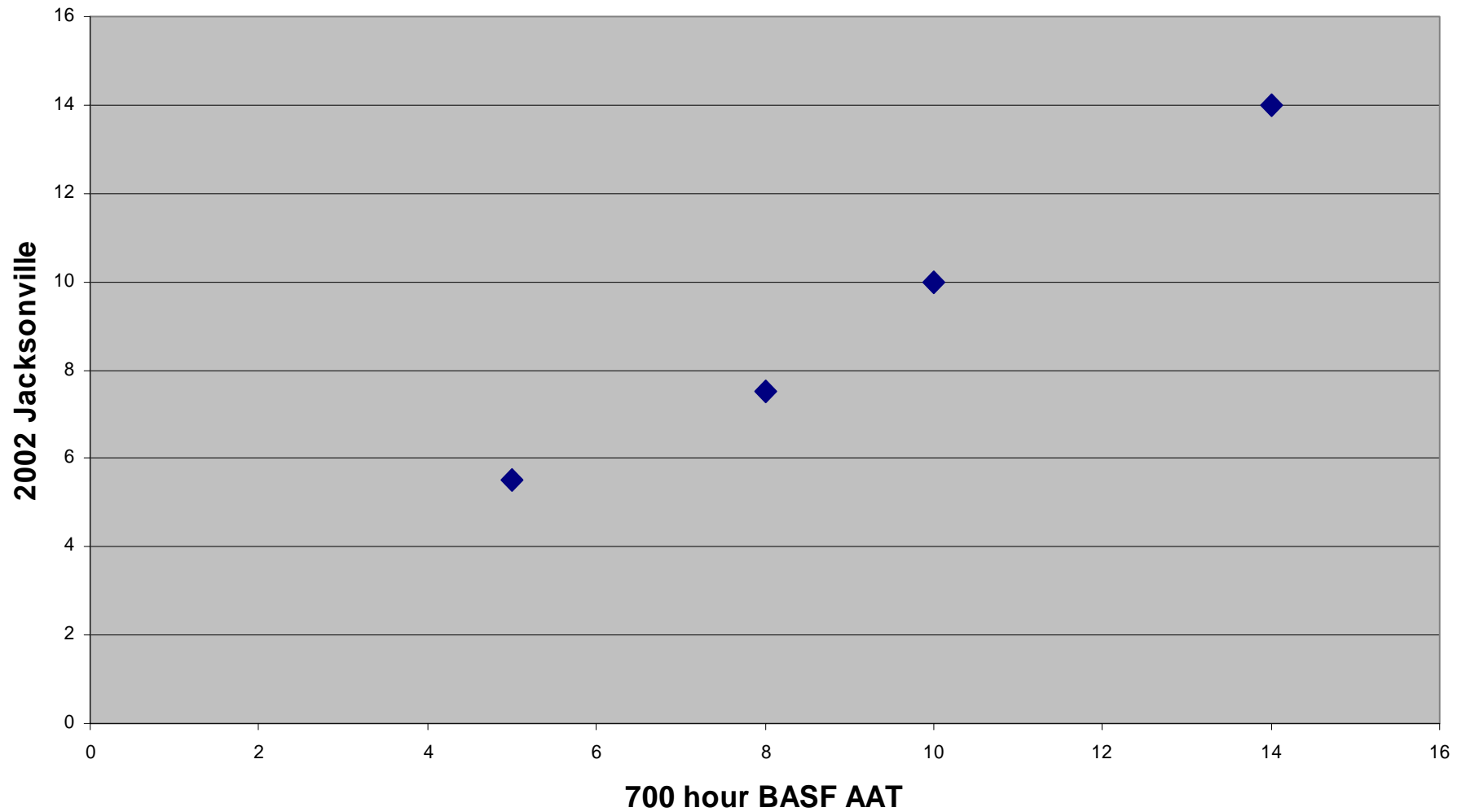


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## 2002 Jacksonville vs. BASF/Q-Sun 700 hours – Ratings





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## *Expanded Panel Testing*

- Based on the good correlations of 400 and 700 hour results to 2001 and 2002 Jacksonville, additional technologies were chosen to expand the scope of the test
- The testing was expanded to include a total of 9 technologies that had controlled field etch data from 2001 and 2002
- A 400 hour test cycle was run to try to repeat the earlier correlation to the 2001 results



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## *Jacksonville Results vs 400 hr Q-Sun® Results – Visual Ratings*

Coating System	400 hr. Q-Sun	2001 Jacksonville	2002 Jacksonville
1	5	5	5.5
2	5	6	4.5
3	6	4	4.5
4	6	5	7.5
5	6	6	7
6	8	6	5.5
7	10	9	10
8	10	10	10
9	10	10	10



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## Correlation Comparison

Q-Sun Test = 400 hour	Pearson Coefficient	Spearman rho
Jacksonville 2001 vs 2002	0.88	0.72
Q-Sun vs Jacksonville 2001	0.90	0.80
Q-Sun vs Jacksonville 2002	0.86	0.71
Q-Sun vs Jacksonville 2001- 2002 avg.	0.93	0.80



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## *Test Modifications*

- In order to optimize the correlation to field results, additional variations of the process were evaluated
- These variations included temperature, UV, process time, and humidity modifications
- The modifications were done with a set of controlled panels against field panels and hoods processed at the same time



## Experimental Data – Test Modifications

	Control	65C	95% RH	+ 75% Light	Site 1	Site 2
<u>CC</u>	<u>400 hr</u>	<u>400 hr</u>	<u>400 hr</u>	<u>400 hr</u>	<u>2003</u>	<u>2003</u>
A	7	6	9	8	4	5
B	8	7	8	8	5	7
C	6	6	9	10	4	7
D	14	10	10	14	8	10
Sum	35	29	36	40	21	29
Pearson	0.866	0.918	0.594	0.915	0.918	1.000
Spearman	0.718	0.937	0.789	0.839	0.937	1.000

- 400 hour data at 65 C represent a closer match to weather conditions and does as well as Site 1 does at matching the Site 2 results.



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## Experimental Data – Test Modifications

	Control	65C	95% RH	+ 75% Light	Site 1	Site 2
<u>CC</u>	<u>400 hr</u>	<u>400 hr</u>	<u>400 hr</u>	<u>400 hr</u>	<u>2003</u>	<u>2003</u>
A	7	6	9	8	4	5
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C	6	6	9	10	4	7
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# *Research Update*

## Accelerated Acid Etch Part II



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## *Expanded Testing*

- Exposure Site
- Exposure Angle
- Specimen Type
- Test Duration
- Evaluation Methods

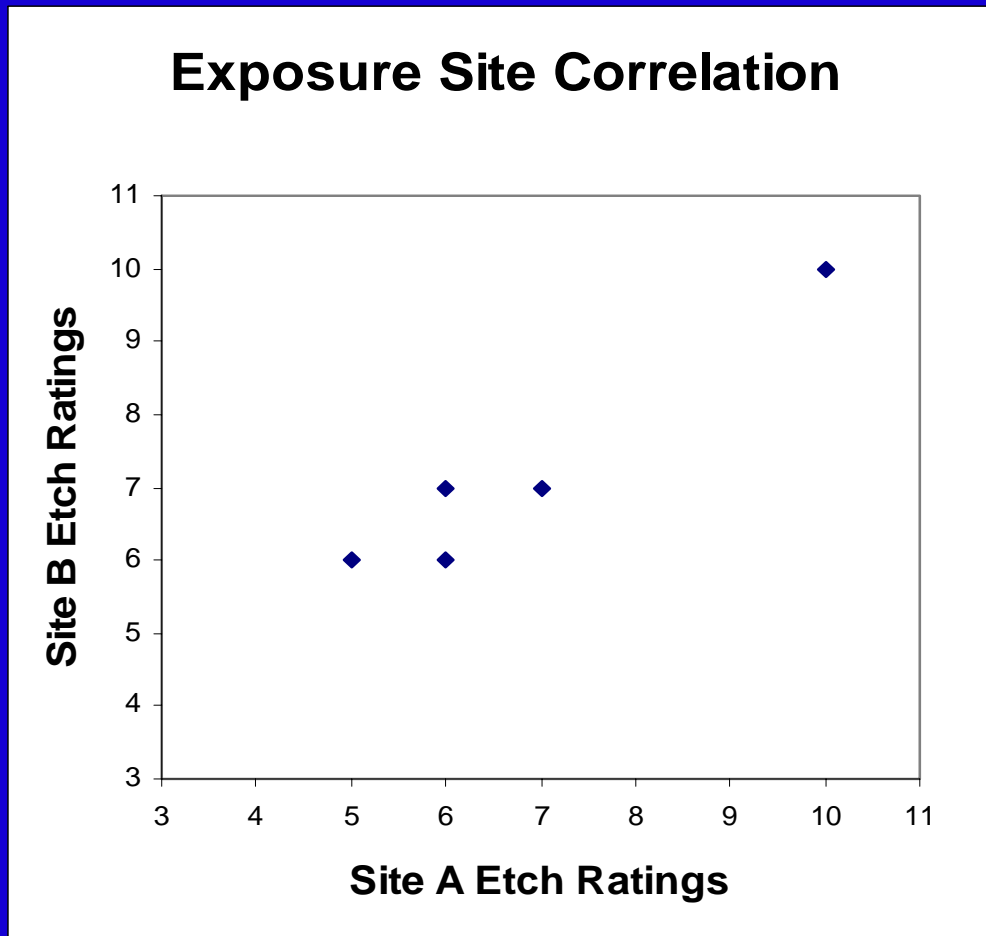


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# Effect of Exposure Site





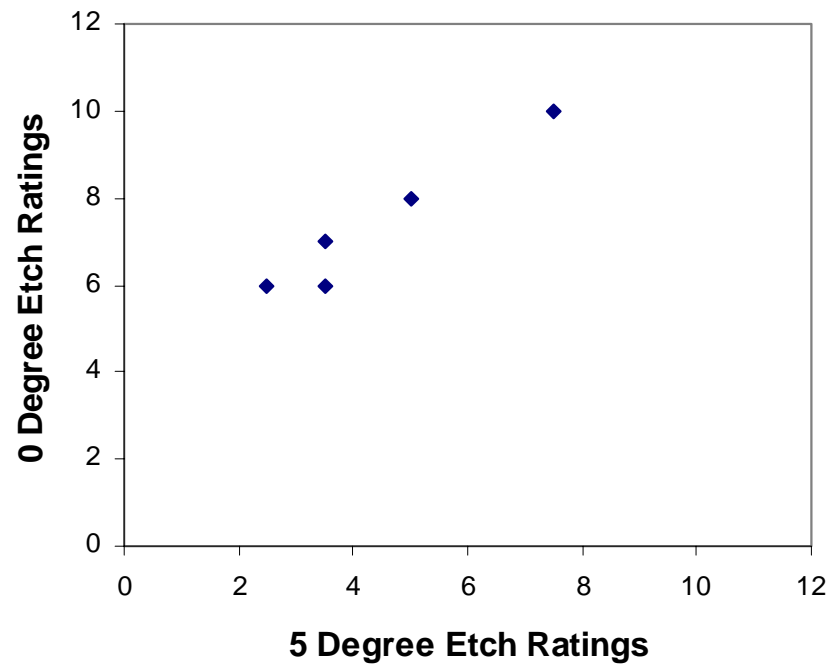
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# Effect of Exposure Angle

The Impact of Orientation Angle on Etch Ratings



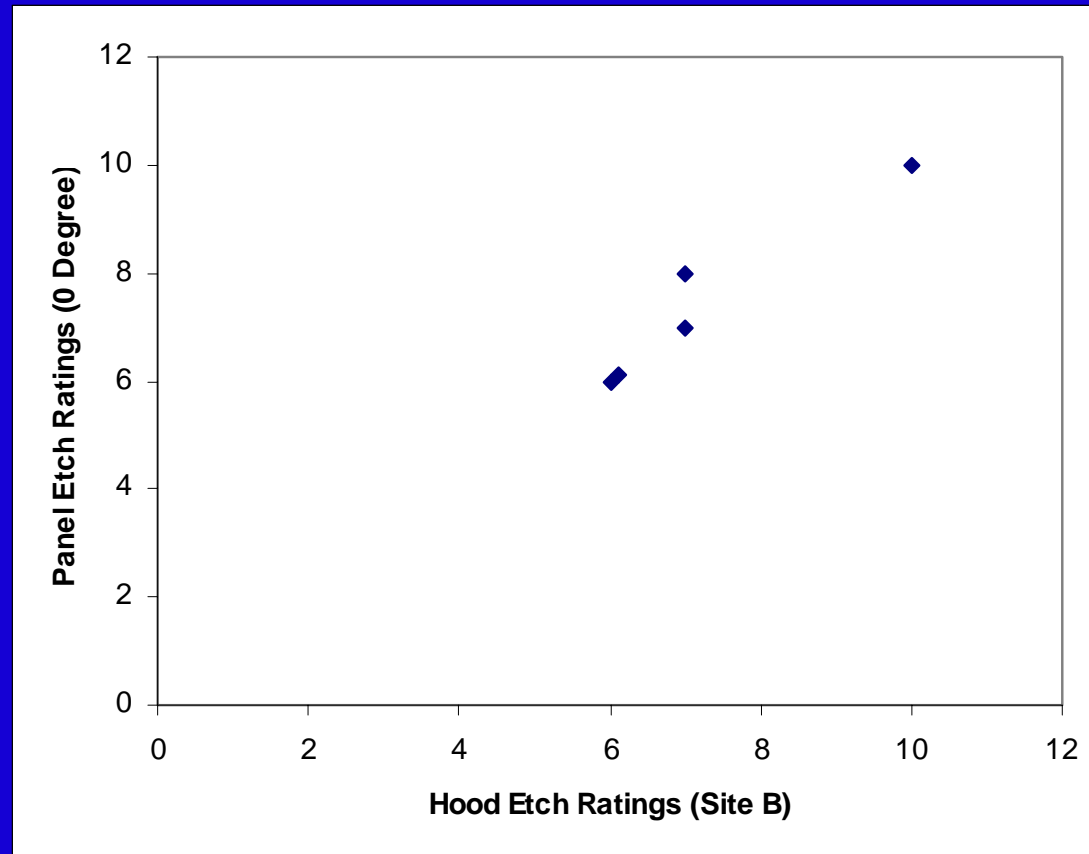


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# *Effect of Specimen Type*





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## *Modified Test Cycle*

- Temperature increased in light and dark cycles
  - Increase practicality between labs
- Test duration of 700 hours will be used
  - More severe than 400 hours
  - Faster results



## *Modified Procedure*

- Dark Cycle- Black panel temperature changed to 40°C  
Chamber air temperature changed to 40°C
- Light Cycle- Black Panel temperature changed to 65°C  
Chamber Air temperature changed to 50°C

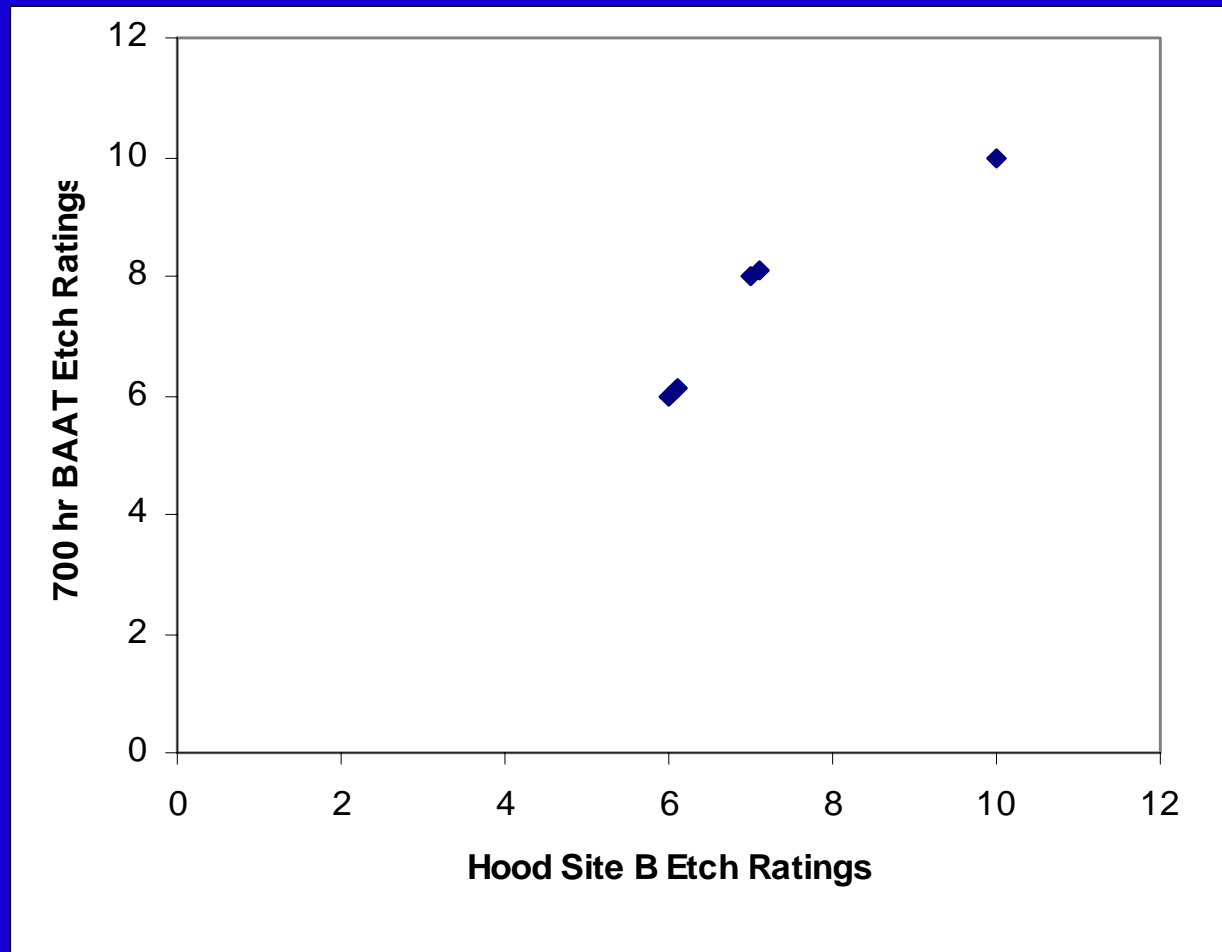


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## *Effect of Modified Cycle*





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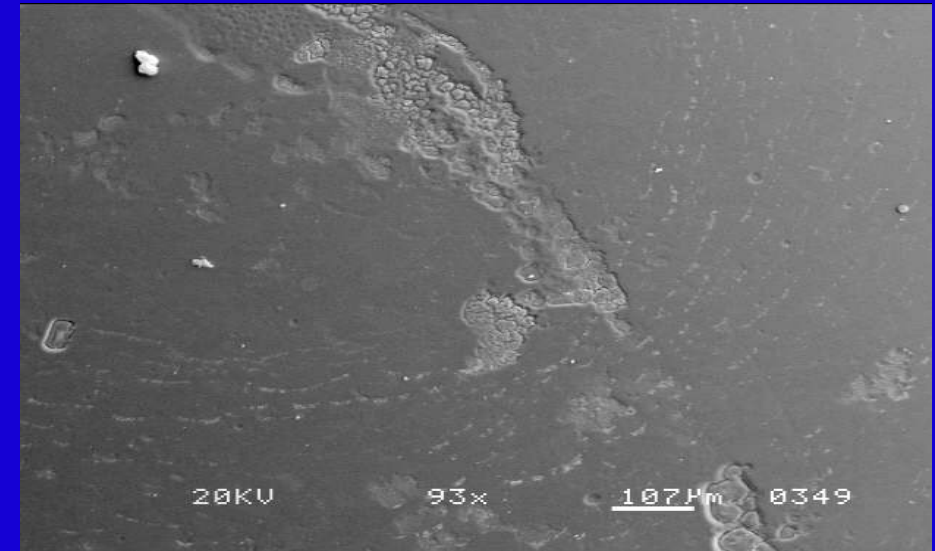
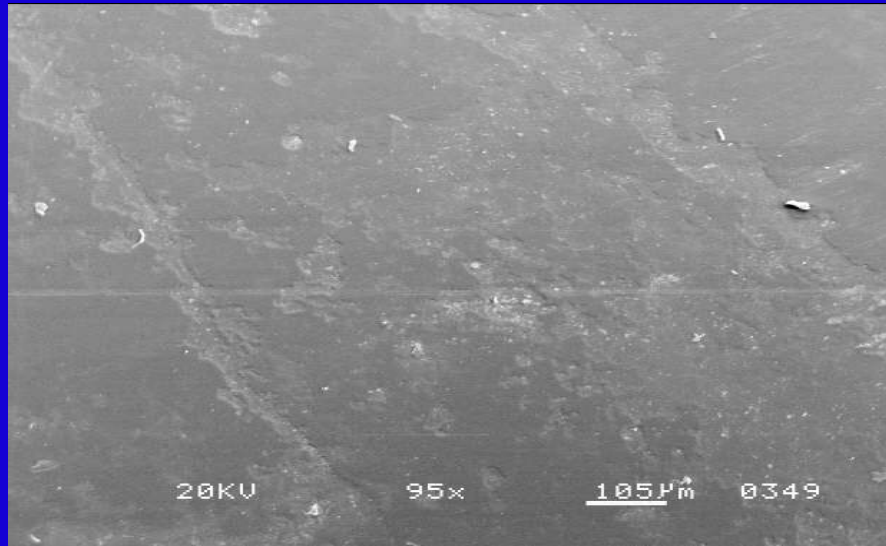
## *Enhanced Evaluation Techniques*

- Scanning Electron Microscope (SEM)
- Energy Dispersive X-Ray Spectroscopy (EDXS)
- Profilometer

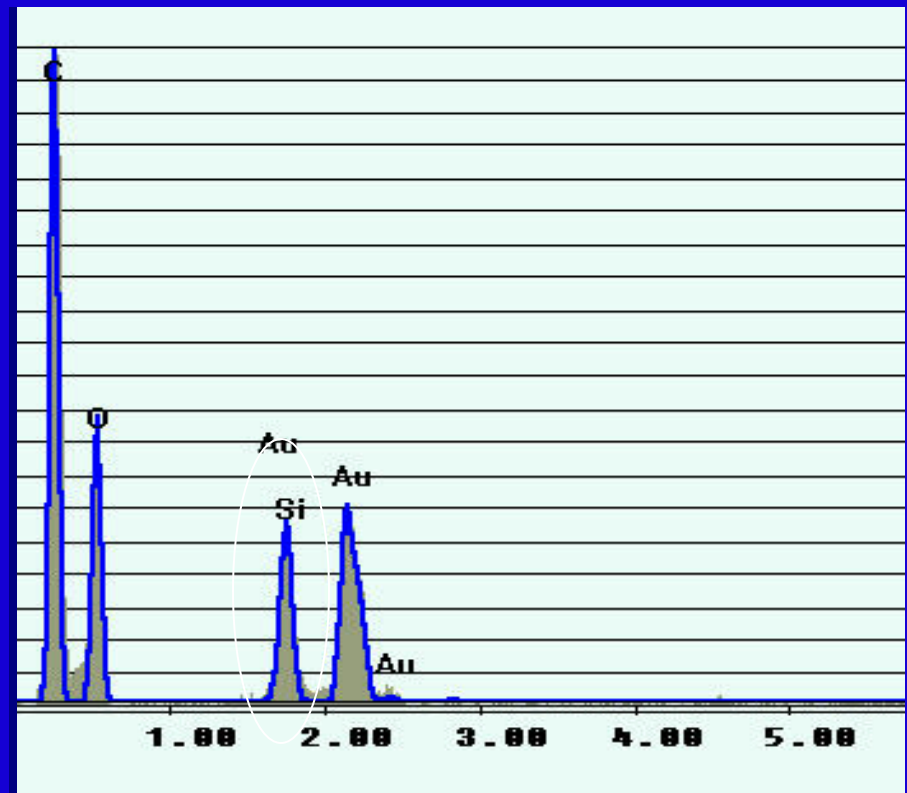
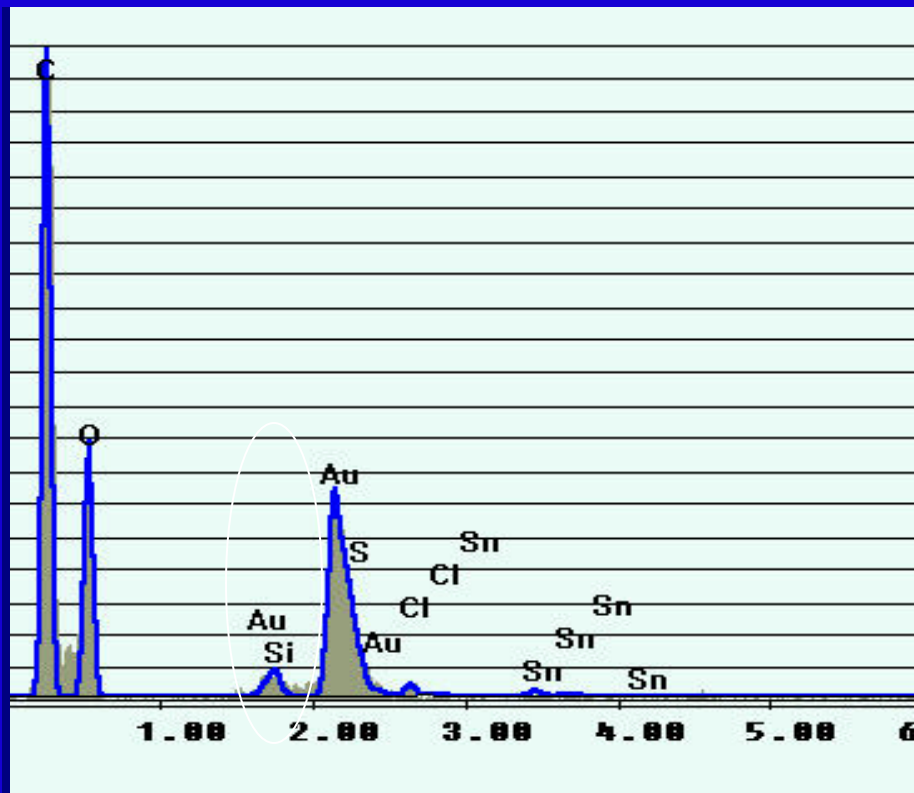
# Severity of Etch Damage SEM

○ Field Test

○ BAAT



# Severity of Etch Damage EDXS





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## *Test Benefits*

- Faster development of etch resistant coatings
- 20 iterations per year of formulate/test/reformulate vs. current 1 iteration per year in Jacksonville
- Can simulate other acid rain environments, where the rain chemistry differs from that in Jacksonville
- In the long run, fewer acid etch failures in service
- Instrumental analysis techniques allow better understanding of etch damage
- Data supports the use of steel panel substrates



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*Questions ?*