Fade Resistance of Lithographic Inks: A New Path Forward——Part 1

by John Lind, Director of Research, GATF, John Stack, Chemist, National Institute for Occupational Safety and Health/National Personal Protective Technology Laboratory, and Eric T. Everett, Q-Panel Lab Products

Real-World Exposures in Florida & Arizona

How can you know whether an ink will remain fade resistant under the variety of lighting conditions that it may encounter during its service life? What is the cost of product failure? What is the price/performance trade-off between affordability and performance? Is there a quick and easy way to determine (or decide) which ink is best for your application?

This article is first in a three-part series that will answer these questions and provide a useful roadmap for assessing ink durability. Part 1 presents results from real-world sunlight through glass window exposures in Florida and Arizona. These internationally recognized test locations provide a "worst case" scenario by exposing inks to high UV, high temperatures, and high relative humidity.

Part 2 will show how accelerated lab exposures mimic real-world results. Part 3 will provide guidance and recommendations on how to determine which ink is best for your needs. This definitive study correlates real-world and accelerated laboratory test results for lithographic inks.

Q-Lab Weathering Research Service Test Facilities in Florida and Arizona performed fade resistance testing of lithographic inks in extreme environments.

Test Program

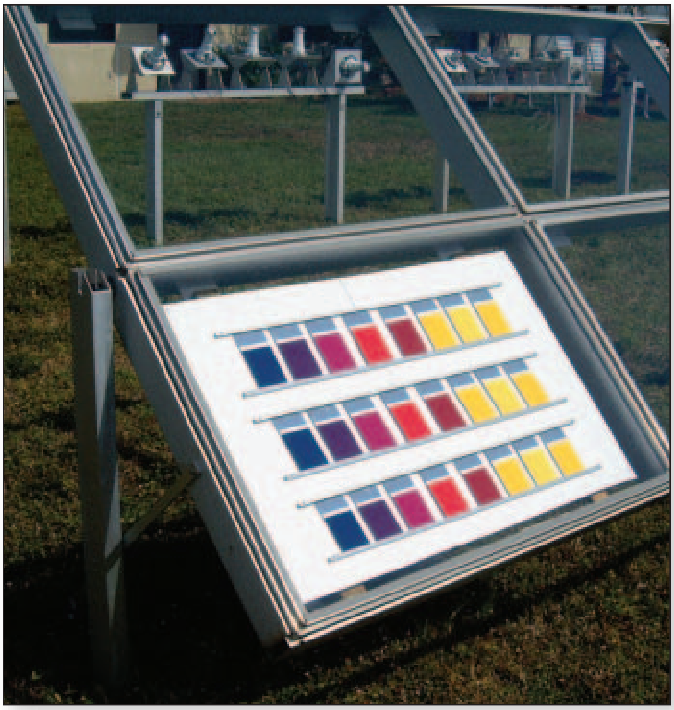
Sunlight contains short wavelength UV and visible and infrared energy. UV is the primary cause of degradation in inks, with temperature and moisture acting as secondary stressors that can accelerate the rate of degradation.

Lithographic inks may encounter intense sunlight by being placed near a window, or worse yet, placed on a car dashboard during the summer months, where they will likely encounter high UV, high temperatures, and high humidity. Florida and Arizona test locations were chosen for this study because they provide these extreme environments.

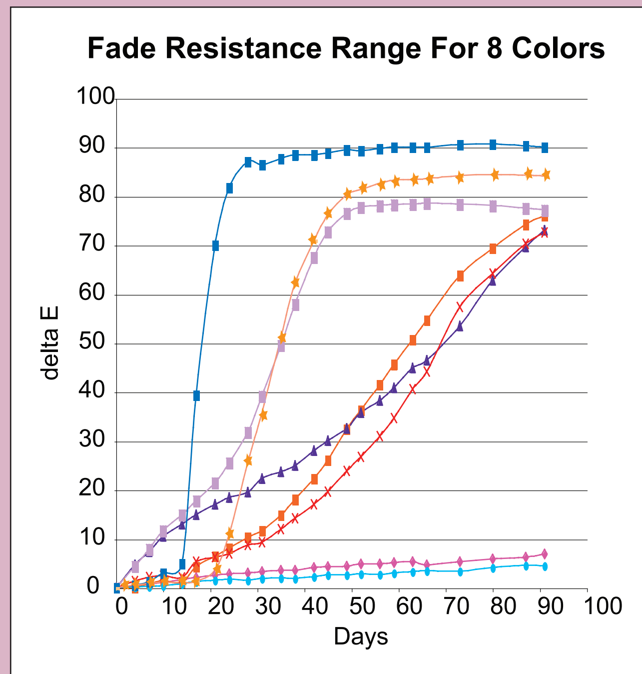
GATF selected eight widely-used lithographic ink colors: Yellow #1, Yellow #2, Yellow #3, Magenta, Violet, Orange, Red, and Purple. A Little Joe Proofing Press was used to make prints at typical offset film thicknesses. The inks were printed on a typical, coated, 70-pound paper substrate. Replicates of each ink color were printed for all of the exposures.

The ink test specimens were tested by Q-Lab Weathering Research Service in Florida and Arizona. They were placed in glass-covered cabinets, angled at 45 degrees south,

GATF technical staff selected eight representative lithographic ink colors printed on a standard substrate for fade resistance testing.



Ink specimens were placed in ASTM G24 glass-covered exposure racks in Florida and Arizona benchmark locations.



Graph 1 - Fade Resistance Range for Eight Colors.

to maximize exposure of sunlight filtered through window glass. The sunlight through window glass spectrum was chosen because it best simulates worst-case indoor lighting conditions.

The ink test specimens were measured for color change before, during, and after exposure. A spectrophotometer was used to take the color measurements in accordance to ASTM D2244. The total color change, expressed in delta E units, was recorded for each specimen. *Note: It is also possible to perform a visual color assessment of inks or to have instrumental color measurements performed by a qualified test lab.*

Florida and Arizona Exposures

Florida exposure tests were started at four seasonal intervals: Fall Equinox (9/21/02), Winter Solstice (12/21/02), Spring Equinox (3/21/03) and Summer Solstice (6/21/03). The Arizona exposure test was started in the fall (10/7/02). Table 1 shows the total light energy intensity (i.e. radiant dosage) expressed in megajoules/m^2 at the conclusion of the various outdoor exposure tests after ninety days in Florida and Arizona:

Graph 1 shows the fade resistance performance of the eight ink colors during the Florida Fall exposure.

As the graph illustrates, there is a wide range of durability in the inks. Some pigments had excellent fade resistance, while others had very poor fade resistance.

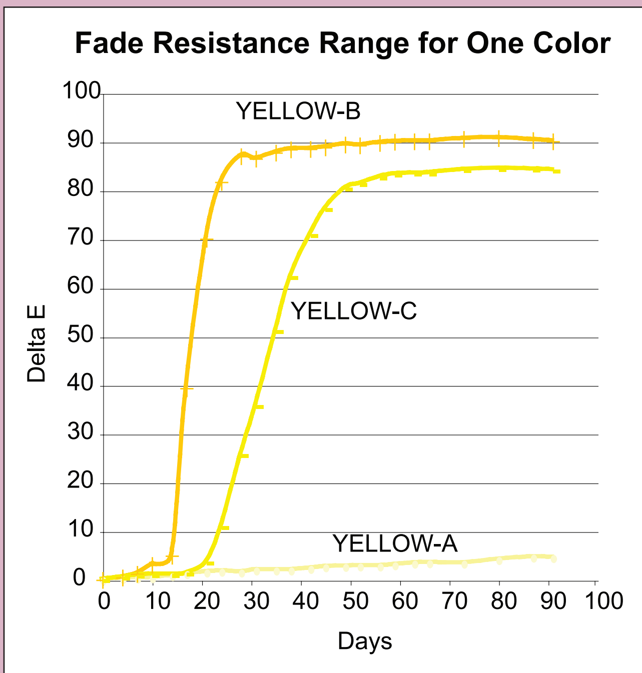
TABLE 1: Total Sunlight Outdoor Exposure Summary MJ/m^2 at 300–3000nm

Exposure	Days	MJ/m^2
Florida Fall	90 Days	1926.17
Florida Winter	90 Days	1541.13
Florida Spring	90 Days	1252.54
Florida Summer	90 Days	1081.73
Arizona Fall	90 Days	1611.20

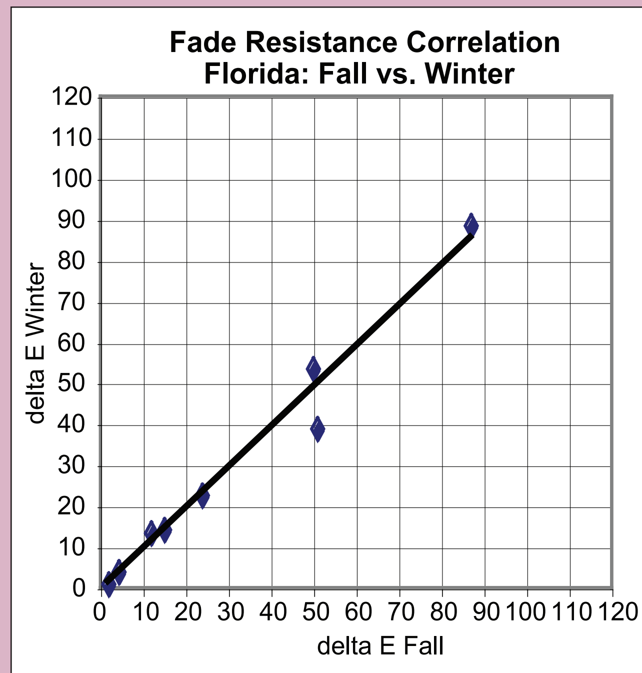
After ninety days, the majority of ink test specimens were severely faded and not useful for analysis. However, by thirty-five days, the inks exhibited a wide range of fade resistance, from excellent to poor. Therefore, thirty-five days was chosen to evaluate the performance of the inks in the various outdoor exposures.

Graph 2 shows the range of durability for the three yellow ink test specimens in the Florida Fall exposure.

This graph is a powerful example showing the wide range of durability between three different inks of the same color. Even though they all were the same color, they had very significant differences in their fade resistance. The Yellow A performed dramatically better than either Yellow B or Yellow C. In fact, Yellow A is a fade-resistant yellow, suitable for fine art reproductions or outdoor applications, while Yellow B and C are intended for general commercial printing.



Graph 2 - Fade Resistance Range for One Color



Graph 3 - Fade Resistance Correlation

Effect of Seasonal Variation

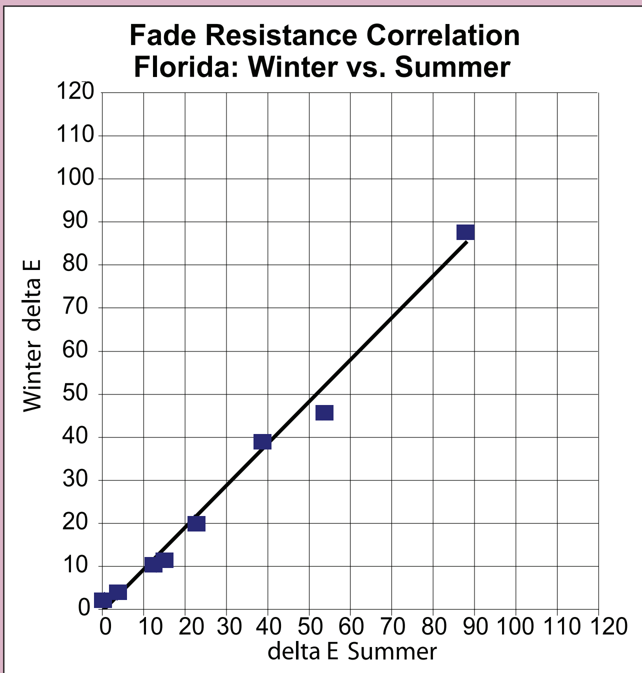
Because the natural exposures were so short-term (thirty-five days), the test was repeated at seasonal intervals to determine the effect of time of year on degradation. The results showed that the time of year did not affect the fade resistance rankings. All seasonal exposures correlated with each other in terms of rank order. However, there was a difference in the rate of degradation.

In order to quantify the difference in the seasonal rates of degradation, the average delta E (at 35 days) can be compared. For example, the average delta E value of all the inks in the Florida Winter exposure is 21, while it is 44 for the Florida Fall exposure. In this case, the Fall exposure was approximately 2-to-1 more severe than the Winter exposure. Because the rate varies by season, you should not compare absolute values of specimens exposed at different times of the year. Such inconsistency may not play a factor when using accelerated fade testing equipment. Table 2 shows the total delta E color change for all inks after 35 days for the various Florida seasonal exposures and Arizona exposure:

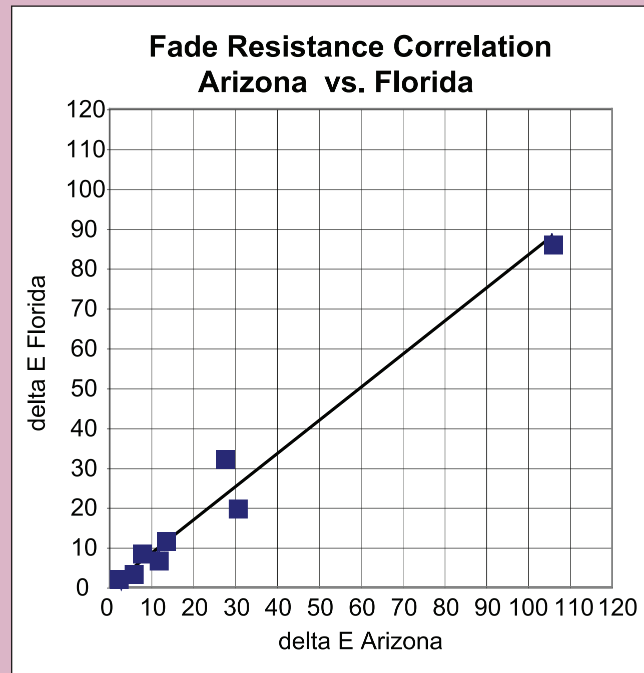
Graph 3 compares the Florida Fall (35 days) and Winter (45 days) exposures. There was perfect rank order* correlation between the two exposures.

Table 2, delta E Color Change of Inks in Various Outdoor Exposures

Specimen	FL Fall 35 days	FL Winter 35 days	FL Spring 35 days	FL Summer 35 days	AZ Fall 35 days	Avg. delta E
Yellow A	3.78	0.94	0.56	2.03	2.80	2.02
Yellow B	106.82	86.81	88.41	87.36	105.65	95.01
Yellow C	89.49	10.90	39.94	38.47	13.20	38.40
Magenta	5.68	3.29	3.71	3.52	5.82	4.40
Orange	23.23	8.19	11.79	11.02	8.37	12.52
Red	18.21	8.38	12.60	10.06	11.65	12.18
Violet	73.82	31.98	55.18	45.33	28.38	46.93
Purple	33.36	20.44	21.23	20.05	30.94	25.20
Avg delta E	44.30	21.37	29.18	27.23	25.85	29.58



Graph 4 - Fade Resistance Correlation



Graph 5 - Fade Resistance Correlation

Florida: Fall vs. Winter

Rank Order = 1.0. *Perfect rank order correlation is represented as a value of 1.0. Random rank order correlation is represented as a value of 0. Negative rank order correlation is represented as a value of –1.0.

Graph 4 compares Florida Winter (45 days) vs. Florida Summer (35 days). Again, there was excellent rank order (1.0) between the two seasonal exposures.

Florida: Winter vs. Summer

Rank Order = 1.0

Arizona Exposure

The Arizona exposure correlated well with the Florida exposures. The Arizona exposures indicate that fast test results were obtained in the same time period (thirty-five days) as the Florida exposures.

Graph 5 compares Arizona vs. Florida at 35 days:

Arizona vs. Florida

Rank Order = 0.96

As shown in Table 3, rank order values were all very good (0.90 and above) for all of the outdoor exposures.

Conclusions (Part 1)

1. Both Florida and Arizona, under glass exposures, provided an extreme environment and quickly separated pigments with excellent fade resistance from pigments with poor fade resistance.

Table 3, Rank Order Correlation Matrix

	FL Summer	FL Fall	FL Winter	FL Spring	AZ Fall
FL Summer	—	0.98	0.93	0.98	0.90
FL Fall	0.98	—	1.0	0.95	0.98
FL Winter	0.93	1.0	—	0.97	0.96
FL Spring	0.98	0.95	0.97	—	0.93
AZ Fall	0.90	0.98	0.96	0.93	—

2. All outdoor exposures correlated with each other.
3. All outdoor exposures were fast and effective. In thirty-five days, the relative durability of all ink specimens was determined.
4. In this series of tests, the time of year did not make a difference in ranking performance of inks. Seasonal variability only made a difference in the speed of degradation.
5. Outdoor exposures are highly useful for fade resistance testing of any ink. Because it has been reported (Tobias and Everett, 2002) that the substrate that an ink is printed on may also affect its stability, it is recommended that the ink be tested on a variety of substrates.

Part 2 test results will show how the Q-Sun Xenon Arc correlated with the outdoor exposures. Part 3 will offer recommendations on how to develop a quick and easy way to determine which ink is best for your needs.

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Further Reading

Lucas, Julie. "Keep Your True Colors: Lightfastness and Weathering Testing." *GATFWorld* May/June 2001.

ASTM D2244. "Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates."

ASTM G24. "Standard Practice for Conducting Exposures to Daylight Filtered Through Glass."

Tobias, Russell H. and Eric T. Everett. "Lightfastness Studies of Water-Based Inkjet Inks on Coated and Uncoated Papers." *IS&T Publications* 2002.

Q-Panel Lab Products is a global provider of light stability and weatherability products and services. For more info visit www.q-panel.com. Eric T. Everett is a standards administrator for Q-Panel Lab Products with ten years experience in standards development and has authored several articles and technical papers addressing light stability. He can be reached at 440-835-8700 or email eeverett@q-panel.co.

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Workshop Leader

Greg Workman is a multicolor press operator at GATF's state-of-the-art production facility. With over twenty years experience in the printing industry, he offers real-world knowledge as an instructor in a number of GATF workshops. Workman is also a certified press operator by the National Council for Skills Standard for web and sheetfed presses.

For additional information about this workshop or other GATF's technical workshops, visit http://www.gain.net/PIA_GATF/workshop.html or contact Sara Hantz at 412-741-6860 x113 or via e-mail at shantz@gatf.org.

Fade Resistance of Lithographic Inks: A New Path Forward—Part 2

by John Lind, Director of Research, GATF, John Stack, Research Lab Technician, National Institute for Occupational Safety and Health/National Personal Protective Technology Laboratory, and Eric T. Everett, Q-Panel Lab Products

The purpose of our overall study was to (1) quantify the fade resistance of typical lithographic inks in an environment which approaches "worst case" service conditions; (2) establish a reasonable benchmark for fade resistance testing of inks; and to (3) identify test methods which provide quick and easy ways to evaluate ink performance.

In Part 1 (*GATFWorld,* April 2004), we presented test results from actual "sunlight through the window" exposures in Florida and Arizona. Our primary reason for doing exposure tests in these extreme environments was to see if our lithographic inks would remain fade resistant in a very severe indoor environment.

In Part 2, we now present results of laboratory xenon arc exposures performed on an identical set of lithographic ink specimens. Our goals were two-fold. We wanted to determine (1) how well did the lab exposures mimic actual, real-world exposures with regard to their actual degradation mode and relative rank order, and (2) how much faster were the lab exposures compared to the natural exposures?

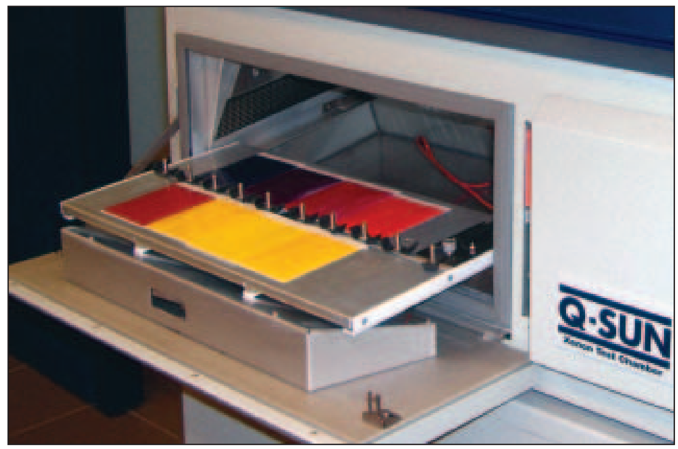
Why Xenon Arc Testing?

Historically, the ink industry has used accelerated laboratory tests to get fast results. Xenon arc testers are widely used because they provide fast results by accelerating critical environmental stresses such as light spectrum, light intensity, relative humidity (RH), and temperature.

For most indoor products, direct sunlight coming through the window is the most severe indoor lighting condition. A recent Kodak study concluded that even *indirect,* window-filtered daylight dominates the indoor lighting environment in homes (Bugner, LaBarca et. al, 2003). Consequently, this GATF/Q-Panel study used Q-Sun Xenon Testers equipped with "window glass filters"to achieve an appropriate spectrum.

The Test Program

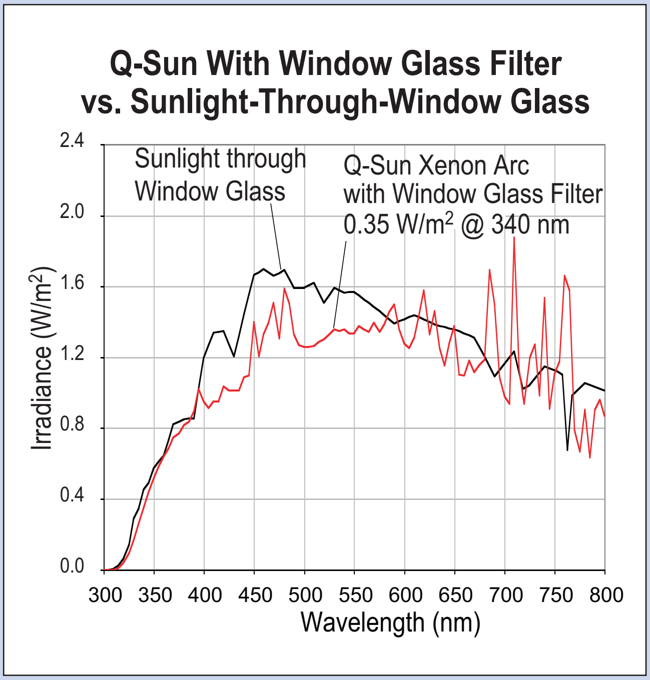
GATF and Q-Panel's Q-Lab Weathering Research Service tested ink specimens in Q-Sun Xenon Test Chambers. One xenon tester was a small, less-expensive "tabletop" unit without RH controls (Xe-1), while the other was a full-size, full-featured xenon tester with precise control of RH (Xe-3). We conducted exposures in different models to see if we could get the same results.



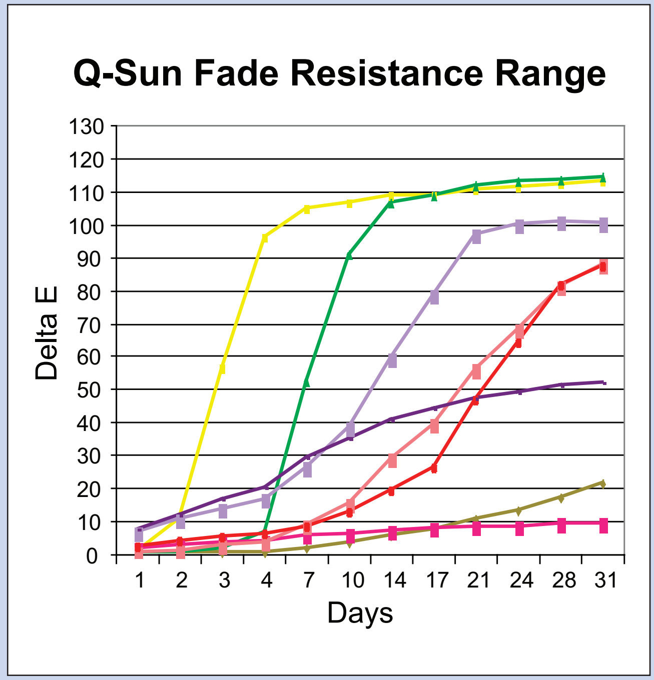
GATF and Q-Lab tested inks in Q-Sun Xenon test chambers.

Q-Sun testers are available in tabletop and full-sized models

GATF selected eight widely-used lithographic ink colors, including magenta, violet, orange, red, purple, and three different yellows. A Little Joe Proofing Press was used to produce prints at typical offset film thickness. The inks were printed on a typical, coated, 70-pound paper substrate. Identical replicates of each ink color were printed for all of the exposures.



Graph 1 - Q-Sun Xenon Arc with Window Glass Filter vs. Sunlight Through Window Glass



Graph 2 - Q-Sun Fade Resistance of Eight Litho Inks

Xenon Arc Exposure Test Conditions

Q-Sun Xenon (Xe-1 & Xe-3)
ASTM D3424, Method 3

Window Glass Filter

Irradiance Level: 0.55 $W/m^2/nm$ at 340 nm

Relative Humidity: Xe-1 Effective RH = 15%
Xe-3 RH = 50%

Exposure Cycle: Continuous Light at 63 ± 3˚C (145 ± 5˚F)

Test Duration: 31 Days

Total Radiant Exposure = 1473 kJ/m^2 at 340 nm

Florida and Arizona Summary

In Part 1, sunlight through window glass exposures provided a high-UV, high-temperature and high-relative humidity (RH) exposure environment for our inks. We exposed inks in the benchmark exposure locations of Florida and Arizona, which are widely used for testing paints, plastics, textiles, and a variety of other materials.

In about a month, these extreme, real-world test environments quickly separated inks with excellent fade resistance from those inks with poor fade resistance. Regardless of the season or location, the tests were fast and effective and the relative rankings of the inks were very similar. The only real differences among the natural exposures were in the speed of degradation.

Inks were measured for color before, during, and after exposure using a spectrophotometer in accordance to ASTM D2244. Delta E (total color change) was recorded for each specimen.

Q-Sun Test Results

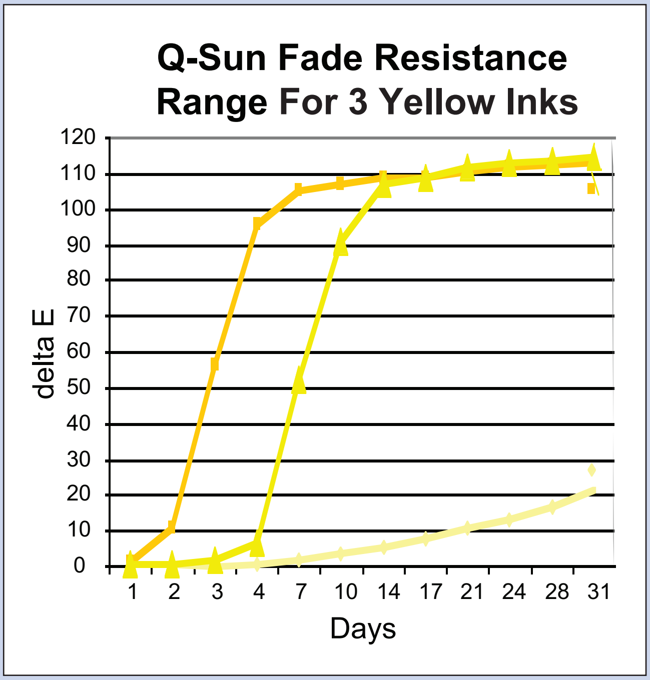
After ten days, both models of Q-Sun tester discriminated the good performing inks from bad performing inks. Graph 2 shows the fade resistance performance of the eight ink colors in a Q-Sun Xe-1.

Both xenon arc exposures were also able to sort out the yellow inks with excellent fade resistance from those with poor fade resistance. Graph 3 shows the range of durability for the three yellow ink test specimens in Q-Sun Xe-1.

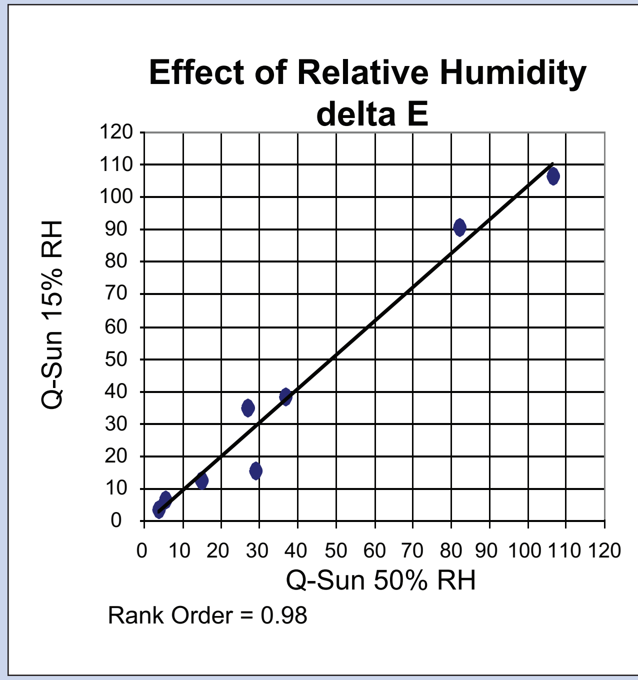
Although their initial appearance was similar, one of the yellow inks performed much better than the others. This shows the range of durability possible in various ink formulations and illustrates the value of testing. As it happens, the yellow ink with excellent fade resistance is designed for fine art reproductions or outdoor applications, while the others are designed for general commercial printing.

Relative Humidity

To determine the effect of RH, the inks were exposed in a Q-Sun which controlled RH at 50% (Xe-3-H) and another where the effective relative humidity was approximately 15% (Xe-1). Graph 4 compares the exposures after ten days.



Graph 3 - Q-Sun Fade Resistance for 3 Yellow Inks



Graph 4 - Relative humidity had no noticeable effect on fade resistance

There was excellent correlation. RH made very little difference for these particular inks.

Q-Sun Compared to Florida

In Part 1, we established a benchmark for fade resistance testing of inks in the extreme environments of Florida and Arizona. For Part 2, we wanted to compare benchmark data with the Q-Sun results. Graph 5 compares the Q-Sun exposure at 10 days to the Florida Under-Glass exposure at thirty-five days.

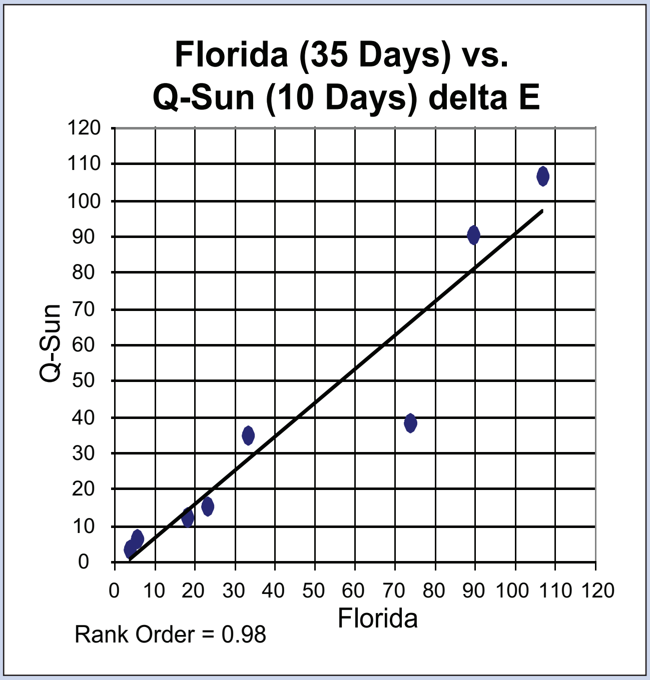
Although the numerical results were not identical, the results show perfect rank order* correlation between the two exposures. In this comparison, the Q-Sun at ten days was approximately equivalent to thirty-five days in Florida. For this particular set of test specimens, the Q-Sun provided an acceleration factor of almost four-to-one.

As shown in Table 1, rank order values for the Q-Sun and outdoor exposures were all very good.

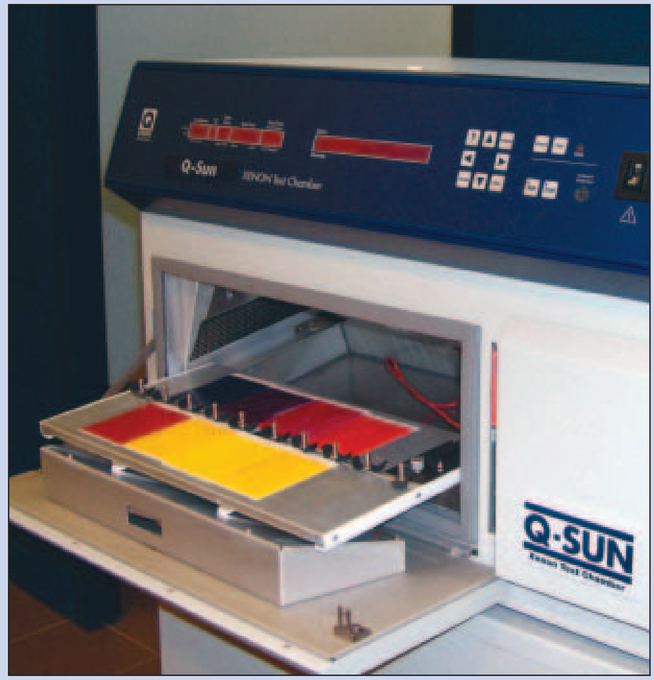
* Perfect rank order agreement is represented as a value of 1.0. Random rank order correlation is represented as a value of zero. Negative rank order correlation is represented as a value of –1.0.

Table 1. Rank Order Correlation Matrix

	Q-Sun Xenon	FL Summer 35 Days	FL Fall 35 Days	FL Winter 35 Days	FL Spring 35 Days	AZ Fall 35 Days
Q-Sun Xenon	—	0.98	0.97	0.83	0.95	0.88
FL Summer	0.98	—	0.98	0.93	0.98	0.90
FL Fall	0.97	0.98	—	1.0	0.95	0.98
FL Winter	0.83	0.93	1.0	—	0.97	0.96
FL Spring	0.95	0.98	0.95	0.97	—	0.93
AZ Fall	0.88	0.90	0.98	0.96	0.93	—



Graph 5 - The Q-Sun showed a very high correlation with Florida



Even the smallest lab can afford a tabletop tester like the Q-Sun.

Conclusions

Part 1 established real world benchmark data for the fade resistance of these particular litho inks. Regardless of the season, the Florida and Arizona under-glass exposures quickly (thirty-five days) sorted out inks with good fade resistance from inks with poor fade resistance. In Part 2, we can draw the following conclusions:

1. Just like the Florida and Arizona exposures, the Q-Sun Xenon separated the good inks from the bad inks. Although no accelerated lab test can replace actual real-world exposures, the Q-Sun exposures correlated very well with the established outdoor exposure benchmark data.

2. The Q-Sun Xe-1 tabletop xenon tester gave the same test results as the more expensive Q-Sun Xe-3 xenon tester. For this particular study, RH did not make a noticeable difference in the test results. The Q-Sun Xe-1 is easy to use.

3. The Q-Sun was fast. In only ten days, the Q-Sun reproduced thirty-five days of Florida under glass exposure. Depending upon the time of the year, the acceleration factor ranged from about four-to-one to almost seven-to-one. However, a word of caution. These acceleration factors may not be valid for other sets of inks.

Further Reading

Bugner, Douglas, et. al, Research & Development Laboratories, Eastman Kodak Company, "Survey of Environmental Conditions Relative to Display of Photographs in Consumer Homes," IS&T Publications 2003.

Lucas, Julie, "Keep Your True Colors: Lightfastness and Weathering Testing," *GATFWorld,* May/June 2001

Tobias, Russell H. and Eric T. Everett, "Lightfastness Studies of Water-Based Inkjet Inks on Coated and Uncoated Papers," IS&T Publications 2002.

ASTM D3424, Standard Test Methods for Evaluating the Relative Lightfastness and Weatherability of Printed Matter

ASTM G151, Standard Practice for Exposing Nonmetallic Materials in Accelerated Test Devices That Use Laboratory Light Sources

ASTM G155, Standard Practice for Operating Xenon Arc Light Apparatus for Exposure of Nonmetallic Materials

Fade Resistance of Lithographic Inks—A New Path Forward—Part 3

by John Lind, Director of Research, PIA/GATF; John Stack, Research Lab Technician, National Institute for Occupational Safety and Health/National Personal Protective Technology Laboratory; and Eric T. Everett, Q-Panel Lab Products

This article is the final installment of our three-part series on fade resistance of lithographic inks. Our goals were to (1) test the fade resistance of typical lithographic inks under "worst-case," real-world service conditions; (2) establish a reasonable exposure benchmark for fade resistance testing of inks, and (3) use standardized test methods for a quick and easy means to evaluate ink performance.

In Part 1, we presented test results from "sunlight through window glass" exposures in the extreme environments of Florida and Arizona. This was done to determine if our lithographic inks would remain fade resistant in a worst case indoor scenario.

In Part 2, we presented test results from laboratory xenon arc exposures were performed on an identical set of lithographic ink specimens to find out (1) how well the lab xenon exposures mimicked Florida and Arizona exposures in terms of actual degradation and relative rank order and (2) how much faster the lab exposures were to the natural exposures.

In Part 3, we conclude our definitive study by providing guidance and recommendations on how to establish an appropriate testing protocol to help you determine which ink is best for your needs.

Developing a Meaningful Test Program

In order to design an appropriate test program and achieve meaningful results, you must first define your testing goals. That is, are you testing your ink to:

- Avoid product failure
- Verify supplier claims about an ink's performance
- Match the ink's performance to its intended application

To get meaningful answers to these questions, you need to have a firm understanding of your product by defining these four criteria:

1. Application. What are the intended uses of your ink? (e.g., general applications, packaging, archival)

2. Service Environment. Will the ink be used indoors or outdoors?

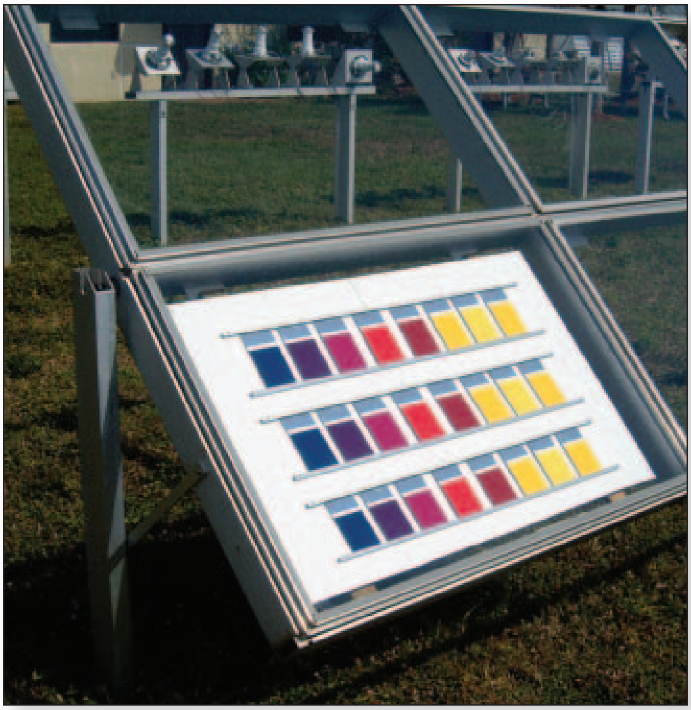
3. Failure Mode. What constitutes product failure? (e.g., fading, color change, delamination)

Outdoor testing is very affordable and provides real world exposure data.

4. Endpoint. How can you quantify whether a given formulation is acceptable?

Once you have defined these criteria for your particular product, you should understand certain general assumptions about testing. Since you cannot simulate every possible environment that your product may encounter, you should design a test that captures the "worst-case" environment. Choose extreme service environments to establish benchmarks to evaluate how your product will perform. By doing this, you will gain confidence in how your product will perform in a variety of end-use applications.

A word of caution: Each end-use environment produces a different rate of degradation. Therefore, service life predictions are *dangerous* and *unreliable*. There are a myriad of other factors that can cause product degradation besides UV light: temperature, moisture, humidity, and ozone. These environmental factors can work independently or in tandem with UV light. Because of these factors, you cannot simply input data in the form of absolute values into a mathematical equation to generate accurate lifetime predictions. If you really want service life predictions, then you must test in the product's *actual* service environment.



An ideal test program includes natural outdoor exposures in Florida or Arizona benchmark locations and accelerated lab testing in a Q-Sun Xenon Test Chamber.

Putting It All Together

After you have defined your testing goals and general assumptions about testing, you are ready to begin testing.

As shown in Part 1, select internationally recognized benchmark locations, like Florida and Arizona, to test your product in extreme, yet real world environments. This testing will provide you with benchmark durability data.

Once you have (1) determined the performance of your product in benchmark locations, and (2) confirmed your product failure modes, you now have the confidence to do accelerated testing to get even faster test results. In other words, "Simulate then Accelerate."

Accelerated lab testing for light stability is best accomplished by using a tester like the Q-Sun Xenon Test Chamber. A properly filtered xenon arc provides an excellent simulation of the sunlight through window glass spectrum. This spectrum is typically the most severe indoor lighting condition. A xenon arc tester also provides fast test results by accelerating critical environmental stresses, such as light intensity, RH, and temperature.

After doing multiple tests in a xenon arc tester to generate durability data, you can evaluate the data using statistical methods, like rank order, to assess relative performance (e.g., "A is better than B for a certain set of conditions").

At this point, you can determine the agreement between the natural and lab results. Once correlation is established, you can use accelerated data to begin assessing product performance in the lab. This information allows you to choose between suppliers or formulations. It is important to periodically reconfirm your lab tests with more natural exposures.

Conclusions and Recommendations

The ideal test program includes:

1. Outdoor Weathering. Test in Florida and Arizona benchmark locations.

2. Accelerated Lab Testing. Test in a Q-Sun Xenon Test Chamber to provide the best simulation of sunlight through window glass exposure.

3. Standardized Test Procedures. Use standardized testing procedures, such as ASTM or ISO, proven methods for product evaluation. These documents provide credibility between vendors and suppliers.

For example, there is a standardized test procedure for light stability testing of litho inks (ASTM D3424). It specifies both outdoor under glass exposures and accelerated laboratory xenon arc exposures for ink performance evaluation.

4. Test, Evaluate, and Test Again. You should always perform both natural and accelerated tests. The natural tests provide real world data, while the accelerated tests can give you fast answers.

Q-Panel Lab Products is a global provider of light stability and weatherability products and services. For more info visit www.q-panel.com.