Calibration & Analysis of
Ultraviolet Radiochromic Strips

Report By:
Aguirre, F.M.
Funches, T.C.
Kuhns, D.W.
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1. Abstract

High intensity ultraviolet (UV) lamps are commonly used for solventless curing. Understanding lamp status during production is critical for maintaining UV process windows and to minimize out-of-specification product. Monitoring the energy density and lamp irradiance can be done at-line with various vendor devices. However, flat-web radiometers cannot pass through a nip or through complicated web paths, since they would fall off the web or be crushed. As an alternative, UV Process Supply sells the Rad Check™ system for checking UV-A energy density in such processes. The system consists of a thin polyester strip with a UV-sensitive coating and a reader. The optical density of the coating decreases under UV-A exposure. This decrease (extinction decrement) is measured using the reader and can be correlated to energy density measurements via radiometer calibration. This report examines the Rad Check™ 01 test strips for error, variability, and sensitivity on the Fusion UV Systems F600 D-bulb and H-bulb. Calibration charts were generated for the strips and show linearity of energy density to extinction decrement up to 500- and ~250-mJ/cm^2 (EIT UV-A) for each bulb respectively. Error analysis (95% confidence) was used to generate confidence bands on the calibration charts. The bands provide confidence ranges for predicted process energy density. The energy density confidence range was approximately +/-40-mJ/cm^2 and +/-20-mJ/cm^2 on the Fusion D-bulb and H-bulb, respectively. If product properties were insensitive to the magnitude of the energy density range, then the test strips would be useful for determining and maintaining a UV-A energy density process window.

Table of Contents

2. Objective

To evaluate UV Process Supply, Inc. CON-TROL-CURE® Rad Check™ 01 UV energy density test strips with regards to error, variability, and sensitivity. To calibrate the strips using ultraviolet radiometers from EIT—the UV PowerMAP™ and UV Power Puck™. To provide EIT UV-A energy density calibration charts for Fusion UV Systems F600 D-bulb and H-bulb.

3. Introduction

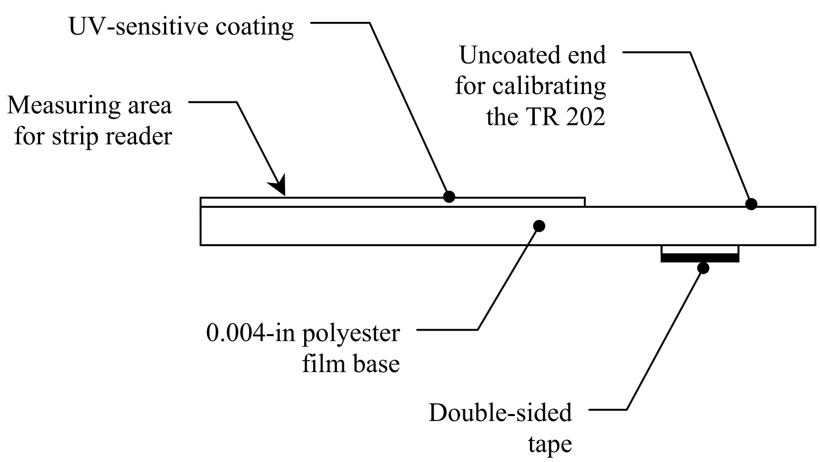
This report summarizes the error, variability, and sensitivity of UV Process Supply CON-TROL-CURE ® Rad Check™ 01 UV energy density test strips. The report is divided into seven major sections, which describe the Rad Check™ system and calibration, equipment, experimental procedure, results, conclusions, suggestions for future work, and relevant appendices.

The Background section describes the use of UV-curing and why monitoring lamp output is important for process and product understanding. This section also describes the Rad Check™ system, its advantages versus conventional radiometers, and how test strips can be calibrated for energy density measurements. The Equipment section is a summarized breakdown of the instruments and equipment used to perform the calibration and validation experiments. The next section, Experimental Procedure, describes how the calibration charts were generated and validated; how to store and handle the test strips; and how to calibrate the test strip reader and take extinction measurements. The Results section first explores the lot-to-lot test strip variation. Then, calibration charts for the Fusion F600 D-bulb and F600 H-bulb are presented, which depict the experimental data. The Results section also discusses performance limitations; calibration validation; error analysis and confidence bands; how to use the calibration charts; important considerations for using the test strips and charts; and a short evaluation of two different radiometers. The Conclusions section highlights important points from each of the previous sections. The next section, Suggestions for Future Work, describes appropriate research for a complete analysis of the Rad Check™ system. Finally, the Appendices section contains detailed specifications for the instruments and equipment used in the experiments.

4. Background

High intensity ultraviolet lamps are commonly used for solventless curing. Understanding lamp status during production is critical for maintaining good UV process windows and to minimize out-of-specification product. Monitoring the energy density and irradiance of the lamps can be done at-line with various vendor devices. At-line lamp monitoring devices include, but are not limited to, conventional flat-web UV radiometers from vendors such as International Light and EIT. Flat-web radiometers must be placed on a web or adhered to it some fashion. Consequently, the radiometers cannot be run through a nip or through complicated web paths, since they would fall off the web or be crushed.

UV Process Supply (UVPS) sells an alternative measurement system for complicated web paths. The system consists of a thin strip of polyester film with a UV-sensitive coating (see Figure 1) and a test strip reader (see Figure 2). The coating is sensitive to UV-A radiation (320- to 380-nm). When a test strip is exposed to radiation in this region, the optical density decreases. The optical density reduction (extinction) is measured using the test strip reader and correlated to UV-A energy density using a calibration chart.



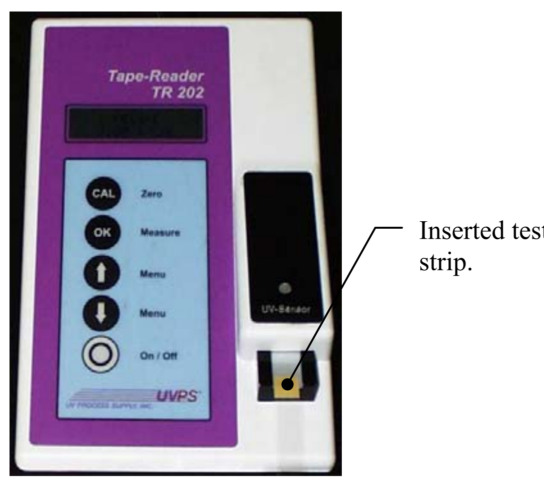


Figure 1: Diagram of Rad Check test strip.

Figure 2: TR 202 test strip reader.

At the time of this report, UVPS sells three types of test strips that can measure different levels of UV-A energy density—see List 1 below.

- Rad Check™ 01
- Rad Check™ 01/D
- Rad Check™ 01/L

List 1: Test strips available from UV Process Supply, Inc.

Rad Check™ 01 test strips are sensitive to energy density levels ranging from 0- to ~300-mJ/cm^2. If higher levels are present, Rad Check™ 01/D strips can measure beyond 300-mJ/cm^2. For low radiation processes, the Rad Check™ 01/L strips have improved accuracy with a sensitivity range of 0- to 200-mJ/cm^2.

The advantage of the test strips is that lamps can be characterized for UV-A energy density in processes unsuitable for conventional radiometers. This means test strip calibration is usually performed on a separate, flat-web lamp system. To provide exact energy density values for exposed test strips, the calibration lamp system should be as similar as possible to the lamp system in the process, i.e., same bulb type, same manufacturer, similar lamp focus, same reflector type, cleanliness, etc. It is important to realize that different UV bulb types have different spectral outputs. For example, a specific test strip extinction value will correspond to a different UV-A energy density on a Fusion F600 D-bulb versus a Fusion F600 H-bulb. Therefore, a calibration chart is needed for each bulb type.

To correlate extinction with an energy density measurement, a radiometer is passed under the lamp (of the calibration system) at the same time as a test strip. This is best accomplished by adhering the strip to the radiometer so that the measuring spot of the strip reader (~11-mm from the coated end of the test strip) aligns with the UV-A filter in the radiometer. Positioning the test strip in this manner ensures the radiation incident on the detector in the radiometer will be the same radiation incident on the test strip—see Figure 3 for positioning the test strip onto the EIT UV PowerMAP™. A second piece of double-sided tape helps secure the loose end of the strip to the radiometer. The test strip is positioned similarly for the EIT UV Power Puck™, which has the same UV filter configuration—see Figure 4.

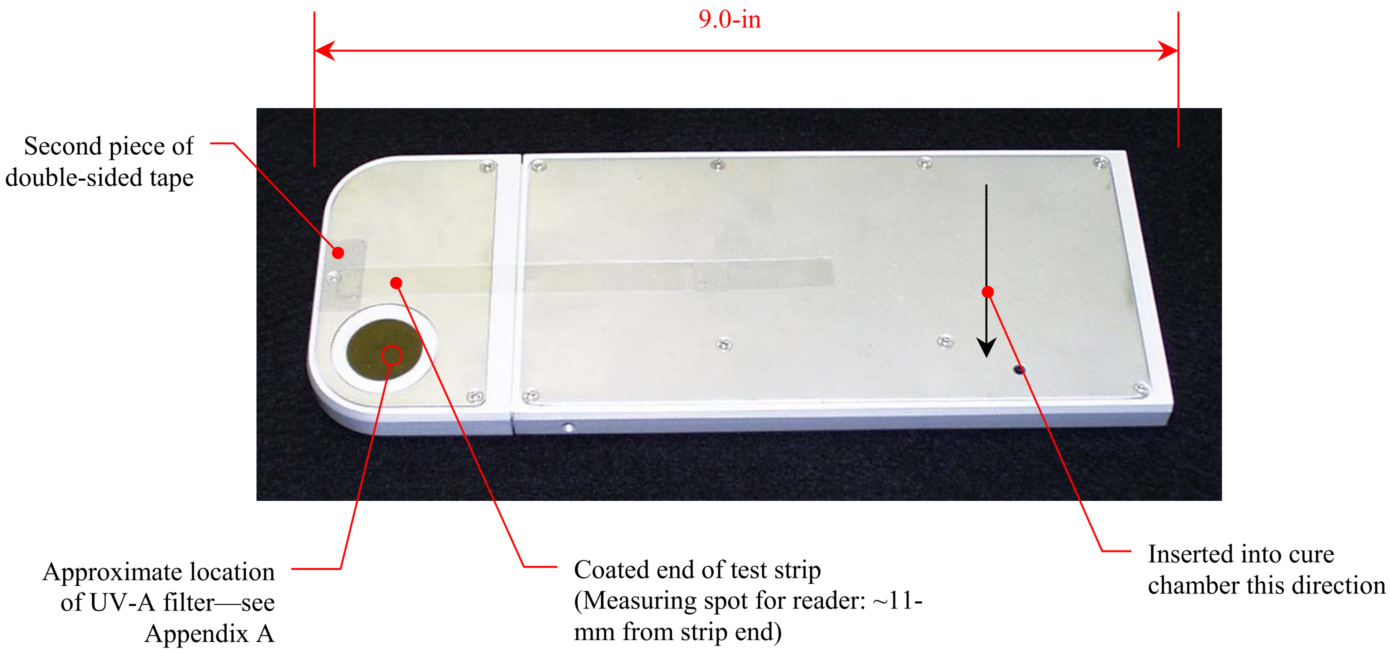


Figure 3: Positioning of Rad Check™ 01 test strip on EIT UV PowerMAP™ radiometer.

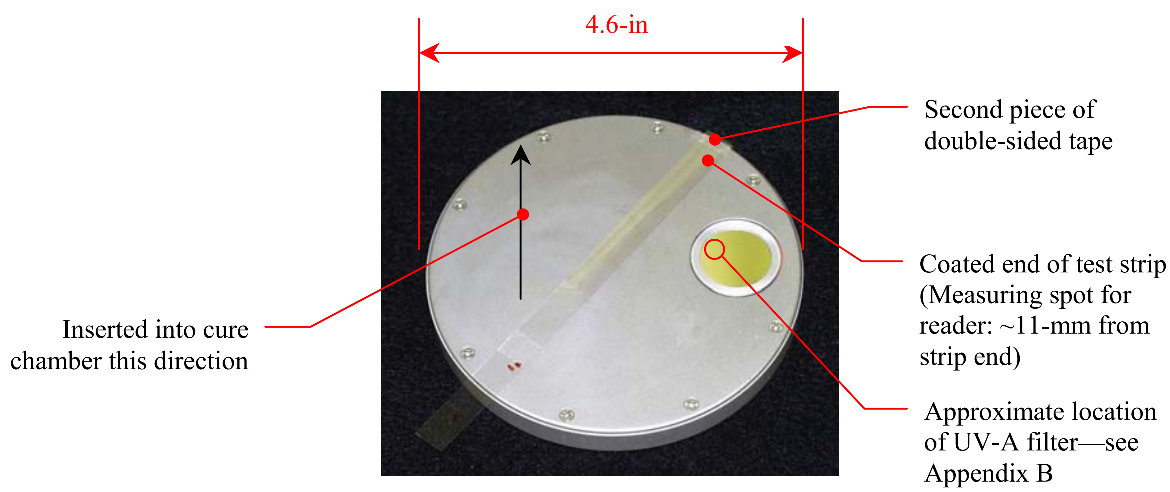


Figure 4: Positioning of Rad Check™ 01 test strip on EIT UV Power Puck™ radiometer.

The UV-A energy density measurement is plotted versus the extinction value to create a calibration data point. Lamp power and line speed determines the amount of radiation "seen" by both the radiometer and the test strip. By varying the lamp power and line speed, a scatterplot (calibration chart) of UV-A energy density versus extinction is created.

Thickness and chemical composition variability of the UV-sensitive coating results in exposed and unexposed extinction variability. To account for strip-to-strip variability, we can use the concept of extinction decrement. Extinction decrement is simply the normalized extinction decrease of an exposed test strip. Extinction decrement is hereby defined as the unexposed extinction, minus the exposed extinction, divided by the unexposed extinction. In the form of an equation,

$$DEC = \frac{U - E}{U}, \qquad \textbf{EQ 1}$$

where *DEC* is extinction decrement, *U* is the unexposed extinction, and *E* is the exposed extinction. Equation 1 can be simplified to

$$DEC = 1 - \frac{E}{U}. \qquad \textbf{EQ 2}$$

In addition to normalization, UVPS recommends at least three strips be exposed and averaged to reduce measurement error. More specifically, a single test strip should be adhered to the radiometer and passed under the calibration lamp. Thus, each strip has its own corresponding energy density value. Averaging the energy density measurements and corresponding extinction decrements helps minimize random error. Coupling averaged extinction decrements with averaged energy density measurements creates a linear calibration chart of EIT UV-A energy density as a function of *DEC*.

5. Equipment

5.A Cure Chamber & Lamp System: See Figure 5

1. Fusion UV Systems Corporation, UV Cure Chamber model DRW-120QNH:

   Cure chamber through which EIT radiometers and Rad Check™ test strips were passed. A Miltec 12-in wide UV-resistant conveyor belt carried the radiometers and test strips through the chamber.

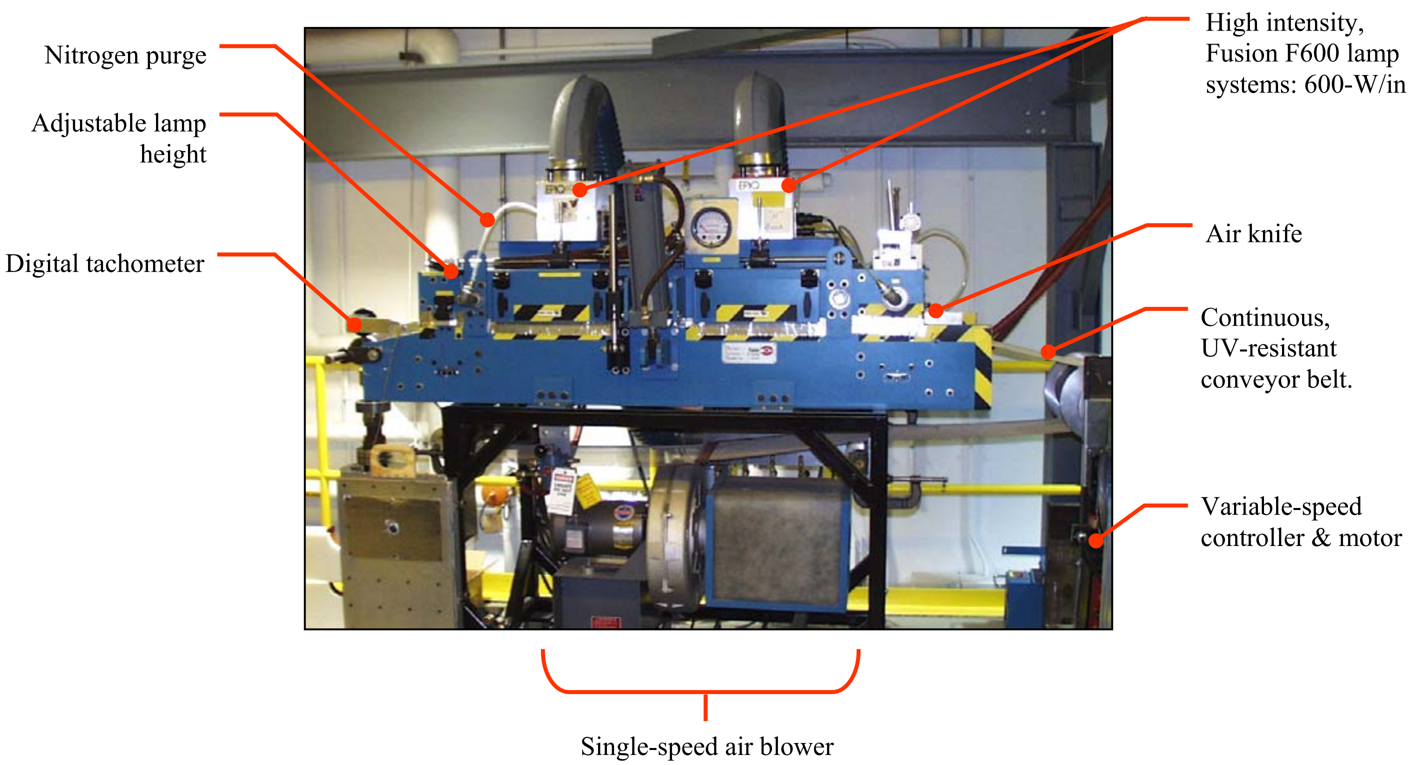


Figure 5: Fusion Systems UV cure chamber.

2. Fusion UV Systems Corporation, Irradiator model EPIQ 6000™:

   Irradiated via microwaves a single, 10-in wide F600, 600-W/in electrodeless UV bulb housed within the unit. Standard aluminum reflectors used. A Fusion D-bulb (mercury doped with iron) and Fusion H-bulb (mercury) were examined for this report.

3. Fusion Systems Corporation, Variable Power Supply model VPS-6:

   Provided power to Fusion lamps. Dial control for power settings ranging from 25% to 100% in 5% increments.

4. Cincinnati Fan & Ventilation Co., Inc., fan model PB-144 with a Baldor High Efficiency Electric Motor model M3613T.

   Single-speed blower, which supplied ambient air to cool the lamps.

5.B Line Speed Control

1. Durant Digital Instruments, Shaft Encoder model 39700-060 (60 pulses/rev) with

Dynapar Corporation, Measuring Wheel model 83-12X.5:

Mounted on an idler roll to measure line speed and provide signal to a digital display.

2. Red Lion Controls, 6-Digit Apollo Time Base Rate Indicator model APLR:

   Displayed line speed in feet per minute.

3. Dayton Electric Manufacturing Company, SCR Controller (model number not available):

   Controller has dials for adjusting torque and line speed signals sent to gearmotor.

4. Dayton Electric Manufacturing Company, Permanent Magnet DC Gearmotor model 4Z136A:

   Used to rotate a shaft with a 7-in diameter core, which moved the Miltec conveyor belt through the cure chamber.

5.C Energy Density Measurement

1. EIT, Inc., UV Radiometer model UV PowerMAP™:

   High power (200-mW/cm^2 to 20-W/cm^2) UV radiometer used to measure UV-A energy density from 320- to 390-nm on the F600 D- and H-bulb. The UV PowerMAP™ is a UV irradiance data logger, which downloads energy density and irradiance values to a PC via a serial connection. Sample rate of 512 samples/sec selected with threshold 'OFF'. See Appendix A for detailed specifications.

2. EIT, Inc., UV Radiometer model UV Power Puck™:

   UV radiometer (5-mW/cm^2 to 5-W/cm^2) used to measure UV-A energy density from 320- to 390-nm on the F600 H-bulb. The UV Power Puck™ measures and displays the total energy density and peak irradiance seen by the detector. The data is displayed on 4-digit LCD screen on the radiometer. See Appendix B for detailed specifications.

5.D CON-TROL-CURE® Rad Check™ UV Measurement System

1. Radiochromic UV energy density strips, Rad Check™ type 01:

   Test strips adhered to the EIT radiometers and passed underneath calibration lamps. Exposure to UV-A radiation reduces the optical density (extinction) of the UV-sensitive coating, which is measured using the TR 202 test strip reader. See Appendix C for detailed specifications.

2. Extinction measurement instrument model Tape-Reader TR 202:

Used to measure the test strip extinction values. See Appendix C for detailed specifications.

5.E HP Spectrophotometer

1. Hewlett-Packard UV-Vis Diode-Array Spectrophotometer model HP 8452A:

   This instrument was used to evaluate the lot-to-lot UV-A absorbance variability for exposed and unexposed test strips. See Appendix D for detailed specifications.

6. Experimental Procedure

6.A Generating a Calibration Chart

To generate a calibration chart, a sufficient number of test strips required exposure to different levels of UV-A energy density. The different UV-A energy density levels were attained by varying the VPS-6 power supply and the line speed controller.

A total of 100 test strips in sample sets of five were used to generate a calibration chart. Power was set to 40, 60, 80, and 100%. At each power level, line speed was set to 70-, 80-, 90-, 100-, and 110-ft/min. Five test strips were run at each line speed for a specific power setting. When five strips were exposed, the line speed was increased to the next setting. After test strips were run at 110-ft/min, the power was increased to the next setting and the line speed was reset to 70-ft/min. All 100 test strips were run under the same point of the lamp—approximately 2-in from the operator side of the lamp. The lamp was allowed to stabilize for 10 minutes on start-up and when lamp power was changed. The lamp was 0.5-in out of focus for all the experiments.

6.B Validating a Calibration Chart

A total of 60 test strips in sample sets of five were used to validate a calibration chart. Power was set to 60, 40, 100, and 80%. At each power level, line speed was set to 110-, 70-, and 90-ft/min. Five test strips were run at each line speed for a specific power setting. When five strips were exposed, the line speed was increased or decreased to the next setting. After test strips were run at 90-ft/min, the power was set to the next level and the line speed was reset to 110-ft/min. Each sample set was placed randomly (cross web) on the belt such that all test strips in a sample set were run under the same point of the lamp. Again, the lamp was allowed to stabilize for 10 minutes at start-up and after changing the power setting.

6.C Test Strip Storage & Handling

Storage and handling of the test strips are important for obtaining accurate extinction measurements. Since the coating on the polyester film is UV-sensitive, test strips should not be exposed to sunlight, mercury lamps, or fluorescent lamps. The coating also has a certain amount of thermal sensitivity so the strips should be stored in a refrigerator between 40 and 50°F (4 - 10°C). Limited transportation at room temperature is okay.

The strips are available from UVPS in packets of 50, which are airtight and protect the strips from UV exposure. UVPS recommends exposing a test strip to UV radiation as soon as possible after removing it from its packet. However, the unexposed extinction, U, appears stable for at least a few weeks after a strip is removed from its packet, measured, returned to its packet, and placed back into refrigeration. The exposed extinction also appears stable.

It is recommended to handle the strips using lint-free gloves such as Nitrile or latex. This will minimize dirt and oil deposits on the areas read by the strip reader (approximately 11-mm from each end of the strip).

6.D Calibrating the TR 202 Test Strip Reader

Once test strips were received from UVPS, they were refrigerated (40-50°F). Calibration of the strip reader was done in an area that had filters that blocked UV radiation from the fluorescent lamps.

When the strip reader is first turned on, it must be calibrated to account for inherent optical density and thickness variation of the polyester base film. Calibration is performed by inserting the uncoated end of the test strip into the reader and pressing 'Cal'. The zero point is stored in the memory of the instrument. UVPS states that the zero point is valid for about ten extinction measurements, after which the reader should be re-calibrated using the next test strip. The coated side of the strip can face either up or down for calibration.

6.E Measuring the Unexposed Extinction

After calibration, the message 'TR 202 Ready to measure' is displayed on the LCD screen. The coated end of each strip was inserted into the reader to measure the unexposed extinction, U. The measurements were performed in the same area where the strip reader was calibrated. After the *U* extinction was measured, each sample set of five was placed in an envelope and refrigerated until they were run through the cure chamber the following day.

The coated side of the strip can face either up or down in the strip reader when measuring the *U* extinction. However, it is recommended to follow the same measurement procedure to minimize operator-to-operator variability. For the experiments described in this report, the coated side of each test strip faced up.

6.F Adhering the Test Strip to the Radiometer

Using the double-sided tape on the test strip, a single strip was adhered to the radiometer so that the coated side faced the lamp. Since each test strip possesses a small degree of curl, a second piece double-sided tape was used to keep the strip flat when run under the lamp—see Figures 3 and 4. The coated side of the test strip must face the UV source.

6.G Measuring & Evaluating the Exposed Test Strip

After a strip was exposed to the UV lamp, the coated end of the strip was placed into the strip reader and the exposed extinction, *E*, was measured and recorded. This was done in an area that had filters that blocked the UV from the fluorescent lamps. The extinction decrement, *DEC*, was calculated for each test strip. The corresponding UV-A energy density from the UV radiometer was also recorded. The extinction decrements and energy density measurements were averaged for each sample set of five. The standard deviation was also calculated for error analysis, which is discussed in a later section of this report. Line speed, power setting, extinction measurements, and energy density measurements were entered into Microsoft® Excel 98. Excel also calculated the averages, standard deviations, error, and plotted the calibration charts.

The coated side of the strip can face either up or down in the strip reader when measuring the *E* extinction. However, it is recommended to follow the same measurement procedure to minimize operator-to-operator variability. For the experiments described in this report, the coated side of each test strip faced up.

7. Results

7.A Test Strip Variation

Some trial runs were performed using test strip samples from lots 160298 and 310399. After measuring the extinctions with the strip reader, a large degree of variability was found for sample sets that contained strips from both lots. Error analysis was done using the root-sum-squares (RSS) method with partial differentials and the Student t-test with 95% confidence. Thompson's Tau Rejection Criteria identified strips in 4 to 1 lot mixtures as outliers, i.e., the data from the differing strip was eliminated. For sample sets that had 3 to 2 mixtures, Thompson's Rejection Criteria could not identify any strips as outliers.

When test strip variance was suspected, 23 test strips from each lot (160298 and 310399) were evaluated using the HP UV-Vis spectrophotometer to determine their sensitivity to UV-A radiation. (The spectrophotometer uses very low intensity UV radiation, which does not affect the optical densities of the test strips). Chart 1 below shows typical UV-A absorbance curves for test strips from both lots.

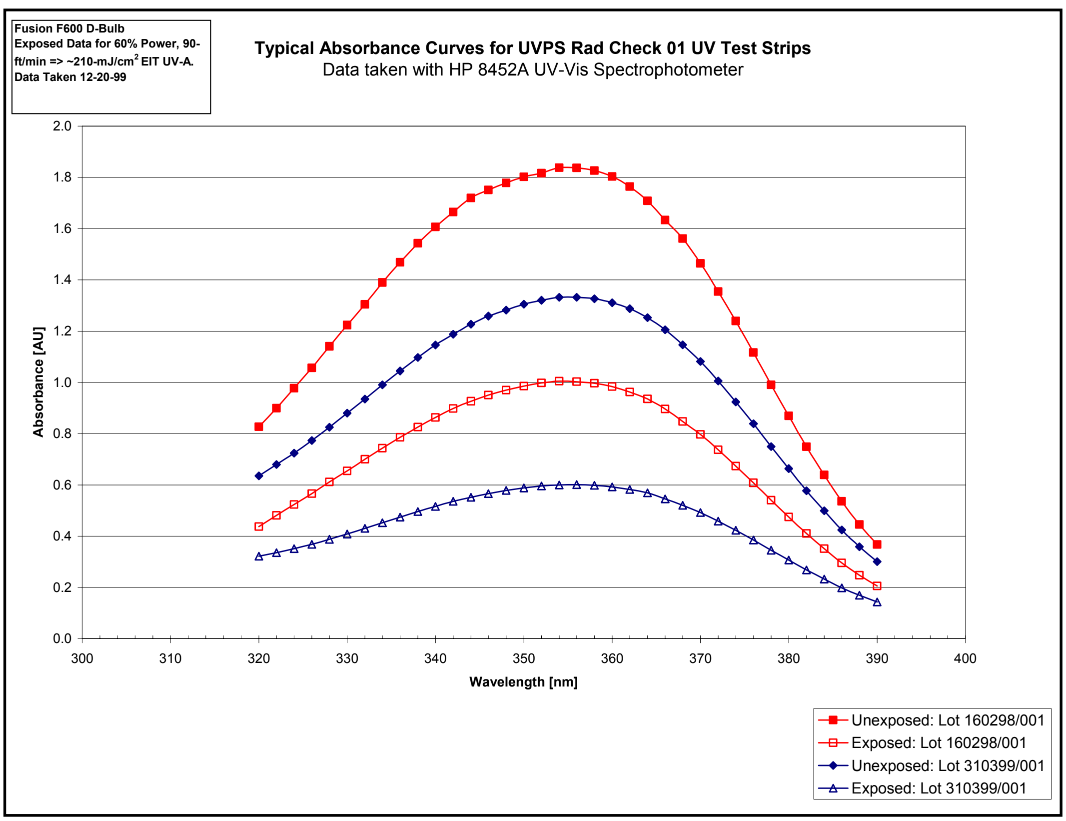


Chart 1: Typical absorbance curves for Rad Check 01 test strips from two separate lots.

It is evident from Chart 1 that test strips from differing lots do not have the same sensitivity to UV-A. The extinction variance was further explored for the unexposed absorbance.

The unexposed absorbance intensities were integrated from 320- to 390-nm for the 23 test strips from each lot. The average, standard deviation, and error were calculated for the integrated intensities. Twenty-three test strips were used to determine the lot average and standard deviation to minimize bias from a small sample size. A theoretical sample size of five was used to calculate random error using the Student t-distribution with 95% confidence. Calculations were done for the combined lots and as separate lots. Tables 1, 2, and 3 show the results of the calculations.

UV Process Dose Tape Lot 160298/001							
						For n = 5	
Strip	Unexposed Absorbance, U (AU)	Avg U	1 * s_U	2 * s_U	3 * s_U	ΔU (+/-)	Relative U Error (%)
1	98.369679	95.21570687	5.89	11.77	17.66	7	8
2	99.692723						
3	80.325976						
4	99.20964						
5	99.24306						
6	98.130379						
7	95.534464						
8	98.098744						
9	99.17281						
10	96.569903						
11	99.011391						
12	94.625888						
13	97.132421						
14	94.236834						
15	99.233046						
16	95.403145						
17	98.44478						
18	95.092485						
19	99.616392						
20	89.889073						
21	76.754789						
22	92.847122						
23	93.326514						

Table 1: Variation results for lot 160298.

UV Process Dose Tape Lot 310399/001							
						For n = 5	
Strip	Unexposed Absorbance, U (AU)	Avg U	1 * s_U	2 * s_U	3 * s_U	ΔU (+/-)	Relative U Error (%)
1	70.215624	69.04951104	3.04	6.08	9.12	4	5
2	70.106595						
3	70.536936						
4	67.088319						
5	62.954498						
6	63.027839						
7	67.732743						
8	74.639169						
9	65.450393						
10	71.064317						
11	72.596506						
12	71.907821						
13	65.523252						
14	72.373673						
15	67.282866						
16	69.276961						
17	70.211628						
18	68.241687						
19	67.295714						
20	69.381484						
21	73.375143						
22	68.929419						
23	68.926167						

Table 2: Variation results for lot 310399.

Combined UV Process Dose Tape Lots 310399/001 & 160298/001									
								For n = 5	
Lot 310xxx Strips	Unexposed Absorbance, U (AU)	Lot 160xxx Strips	Unexposed Absorbance, U (AU)	Avg U	1 * σ_U	2 * σ_U	3 * σ_U	Δ U (+/-)	Relative U Error (%)
1	98.369679	24	70.215624	82.132609	13.86	27.72	41.59	17	21
2	99.692723	25	70.106595						
3	80.325976	26	70.536936						
4	99.20964	27	67.088319						
5	99.24306	28	62.954498						
6	98.130379	29	63.027839						
7	95.534464	30	67.732743						
8	98.098744	31	74.639169						
9	99.17281	32	65.450393						
10	96.569903	33	71.064317						
11	99.011391	34	72.596506						
12	94.625888	35	71.907821						
13	97.132421	36	65.523252						
14	94.236834	37	72.373673						
15	99.233046	38	67.282866						
16	95.403145	39	69.276961						
17	98.44478	40	70.211628						
18	95.092485	41	68.241687						
19	99.616392	42	67.295714						
20	89.889073	43	69.381484						
21	76.754789	44	73.375143						
22	92.847122	45	68.929419						
23	93.326514	46	68.926167						

Table 3: Variation results for combined lots 160298 & 310399.

Combining lots increased the unexposed extinction error by 2.5 to 4 times as measured by the spectrophotometer. Measurements that are more accurate can be attained by simply using test strips from the same lot for calibration and for evaluating the process UV bulbs.

7.B Fusion F600 D-Bulb

7.B.i Data

The F600 D-bulb was evaluated by two operators on two different dates using the EIT UV PowerMAP™. The data is depicted below in Chart 2.

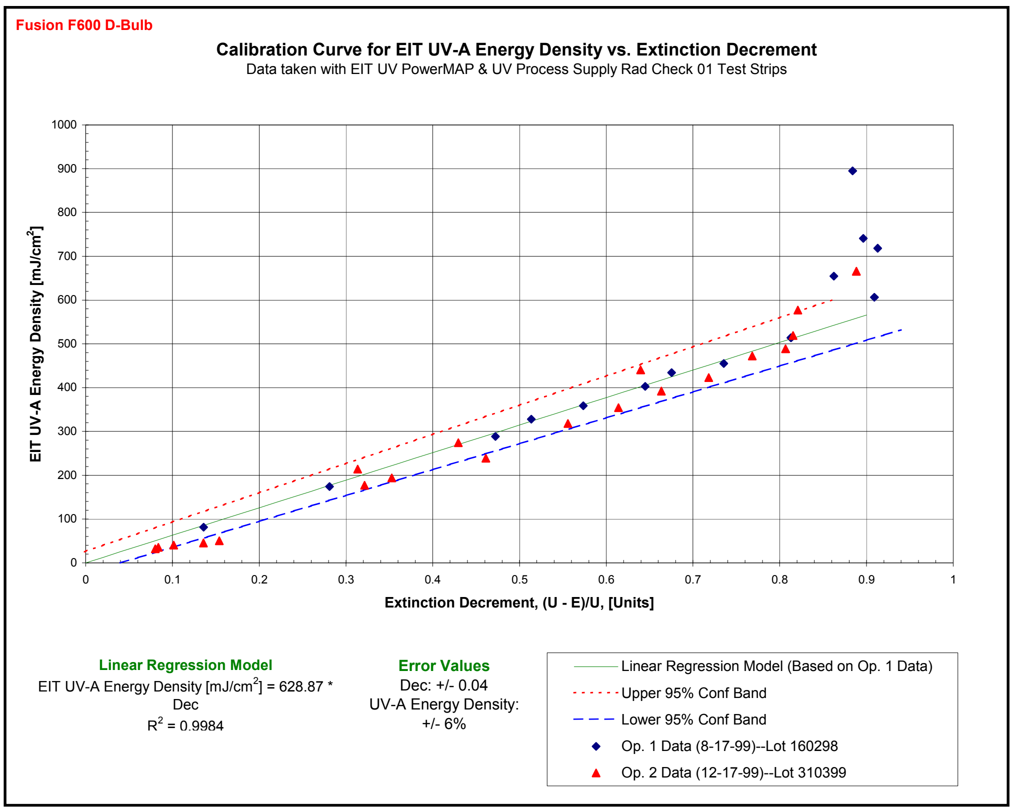


Chart 2: Test strip extinction decrement data and model for Fusion F600 D-bulb.

The centerline in Chart 2 is the linear regression model based on Operator 1's data taken on 8-17-1999. The model does not consider data beyond *DEC* = 0.80 because of non-linearity beyond that point. Operator 2's data taken on 12-17-1999 was an attempt to validate the model. Operator 1 used test strips from lot 160298. Operator 2 used test strips from lot 310399.

7.B.ii Performance Limitations

After the extinction decrement reaches ~0.80, the test strips indicate a non-linear response to increasing energy density levels. The non-linear response is probably due to saturation of the coating. At energy density levels higher than 500-mJ/cm^2 UV-A on the Fusion D-bulb, the coating started to blister. UVPS reports the maximum value of 300-mJ/cm^2. However, the response of the test strip to energy density is clearly linear up to *DEC* = 0.80 and 500-mJ/cm^2 UV-A energy density.

7.B.iii Validation

Operator 2's data falls mostly within the confidence bands, but the overall trend is lower than the model. The cause of the lower trend is unknown, but the only difference between the Operator 1 and 2's data is the test strip lot. The different slope suggests lot 310399 had a different response to the UV-A energy density. Therefore, a calibration chart should be generated for each lot, and test strips from that same lot should be used for energy density measurements on the process UV bulb.

7.B.iv Error Analysis & Confidence Bands

Instrument and random error was calculated for the UV PowerMAP™ using RSS and the Student-t test with a 95% confidence level. The typical error was +/-6% of the measured UV-A energy density. The extinction decrement error was calculated using RSS with partial differentials and the Student-t test. The typical error was +/-0.04 in arbitrary units. The reported errors for the UV PowerMAP™ and test strips are independent of the energy density exposure up to *DEC* = 0.80.

The upper and lower 95% confidence bands were generated using the worst-case error scenarios for the UV PowerMAP™ and extinction decrement. The upper band was generated using +6% energy density with –0.04 *DEC*. The lower band was generated using –6% energy density with +0.04 *DEC*. The concept of the bands is that we are 95% confident that the true process energy density lies within the upper and lower limits for a predicted energy density value obtained using an averaged extinction decrement.

The bands assume that the test strips are from the same lot and that the sample size used to measure a process bulb is equal to the sample size used to generate the calibration chart. For the experiments described in this report, the sample size was five. If an operator were to use a smaller sample size for measuring the process UV bulbs, then random error would likely increase for both energy density and extinction decrement. Consequently, the increased error will expand the confidence bands. If a larger sample set were used, the random error would likely reduce the random error and tighten the confidence bands. For example, if an operator evaluates the process UV bulbs using three strips and calculates the average extinction decrement, he or she would need to consult a calibration chart generated from sample sets of three. Similarly, if an operator evaluates the process UV bulbs using ten strips and calculates the average extinction decrement, he or she would need to consult a calibration chart generated from sample sets of ten.

7.C. Fusion F600 H-Bulb

7.C.i Data

The F600 H-bulb was evaluated by two operators on three different dates using the UV PowerMAP™ and UV Power Puck™. The data is depicted below in Chart 3.

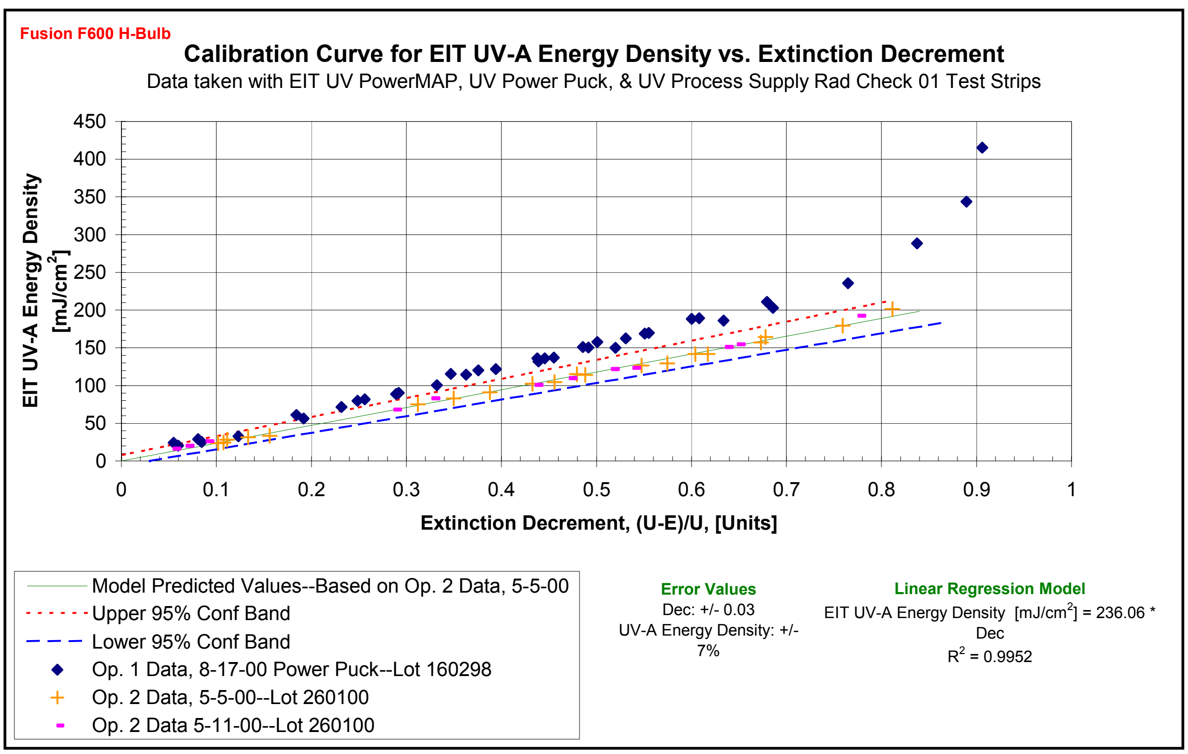


Chart 3: Test strip extinction decrement data and model for Fusion F600 H-bulb.

The centerline in Chart 3 is the linear regression model based on Operator 2's data taken on 5-5-2000. Operator 2's data taken on 5-11-2000 was an attempt to validate the model. The data represented by Operator 1 was overlaid in an attempt validate the model as well. Operator 1 used test strips from lot 160248. Operator 2 used test strips from lot 260100.

7.C.ii Performance Limitations

Operator 1's data showed a similar non-linear response to that observed on the Fusion D-bulb when *DEC* exceeded ~0.80. Operator 2 did not have data sufficiently past *DEC* = 0.80 to suggest the same response. The non-linear response appears to be due to saturation when the extinction decrement reaches 0.80. Physical degradation (blistering) was not evident on any of the test strips. The maximum UV-A energy density for the test strips on the Fusion H-bulb was 200- to 275-mJ/cm^2 depending on the test strip lot. In comparison, UVPS states the maximum energy density value of 300-mJ/cm^2 for the Rad Check™ 01 test strips.

7.C.iii Validation

Operator 1's data does not fall within the confidence bands of the model. However, Operator 2's data taken on 5-11-2000 follows the centerline closely—validating the model for in-lot test strips. Again, the overall linear trend is verified, but the model does not hold for test strips from the different lot. Since the model does not hold for test strips for the different lot, this suggests that a calibration chart should be generated for each lot. To evaluate the process UV bulbs, the test strips must belong to the same lot used to generate the calibration chart.

7.C.iv Error Analysis & Confidence Bands

Instrument and random error was calculated for the UV PowerMAP™ and UV Power Puck™ using RSS and the Student-t test with a 95% confidence level. The typical error was +/-7% and 10%, respectively, of the measured UV-A energy density. The extinction decrement error was calculated using RSS with partial differentials and the Student-t test. The typical error was +/-0.03 in arbitrary units. The reported errors for the UV PowerMAP™ and UV Power Puck™ and test strips are independent of the energy density exposure up to *DEC* = 0.80.

The upper and lower 95% confidence bands were generated using the worst-case error scenarios for the UV PowerMAP™ and extinction decrement. The upper band was generated using +7% energy density with –0.03 *DEC*. The lower band was generated using –7% energy density with +0.03 *DEC*.

7.D Using the Calibration Charts

To use the model depicted in Chart 2 or 3, the user would expose five test strips under the same point of a process bulb, calculate the average extinction decrement, and record the predicted energy density value and range from the chart. The model only provides the predicted value for the true energy density in the process. The confidence band provides the predicted range for the true energy density with 95% confidence. For example, suppose the average *DEC* for five test strips was 0.50. Using Chart 2, the predicted process energy density would be ~315 +/- 45-mJ/cm^2 UV-A with 95% confidence. In other words, we would be 95% confident that the true process energy density is within +/- 45-mJ/cm^2 of 315-mJ/cm^2.

7.E Important Considerations

First, the most significant consideration when using the Rad Check™ 01 test strips is the width of the confidence band. The width of the band, i.e., the range for a predicted energy density, might be too large for determining the process window for a product. This is because properties of the product may change significantly within the magnitude of the range. For the D-bulb, the band width was approximately +/-30-mJ/cm^2 at low extinction decrement and approximately +/-50-mJ/cm^2 at high extinction decrement (see Chart 2). In comparison, the band width for the Fusion H-bulb was approximately +/-10-mJ/cm^2 at low extinction decrement and approximately +/-25-mJ/cm^2 at high extinction decrement (see Chart 3). If product properties were negligibly sensitive to the magnitude of the energy density range, then the test strips would be useful for determining and maintaining a UV-A energy density process window. If tighter process control were needed, then the Rad Check™ 01 test strips would not be sufficient.

Second, the test strips appear to saturate when *DEC* is approximately 0.80. If more than one lamp is being studied, the test strips could easily saturate if the maximum total energy density is exceeded. For the F600 D-bulb, the maximum energy density that corresponds to *DEC* = 0.80 is approximately 500-mJ/cm^2, whereas the maximum energy density corresponding to an F600 H-bulb ranges from 200- to 275-mJ/cm^2—depending on the test strip lot. If saturation is possible, multiple lamps should be studied individually.

Third, it may not be physically possible to adhere multiple test strips to a moving web if the line speed is too fast. By measuring lamps at the power setting used for the process with a slower line speed, the true process energy density can be estimated using the relation that twice the line speed is equal to half the energy density. In form of an equation,

$$\text{True Process Energy Density} = \text{Measured Energy Density} \times \left(\frac{\text{Measurement Line Speed}}{\text{Process Line Speed}} \right), \qquad \textbf{EQ 3}$$

where *Measured Energy Density* is obtained from the extinction decrement calibration chart, *Measurement Line Speed* is the line speed at which the test strips were run under the process lamp (usually slower or faster than the normal process line speed), and *Process Line Speed* is the line speed at which the process is normally run. The width of the energy density range can be estimated using the same equation.

Finally, test strips may saturate if the measurement line speed is too slow. If this is the case, measurements should be taken at a faster line speed such that the strips do not saturate, and then adjust for the process line speed using Equation 3—the lamp would remain at the power setting normally used for the process. This method is useful if the process has high energy density at slow line speeds.

7.F UV PowerMAP™ vs. UV Power Puck™

Since the calibration charts depend upon radiometer measurements, the effects of using the EIT UV PowerMAP™ versus the UV Power Puck™ were not known. A study was done to explore these effects, if any. A single lot was used for the experiments (lot 160298) on the Fusion H-bulb. The results are shown below in Chart 4.

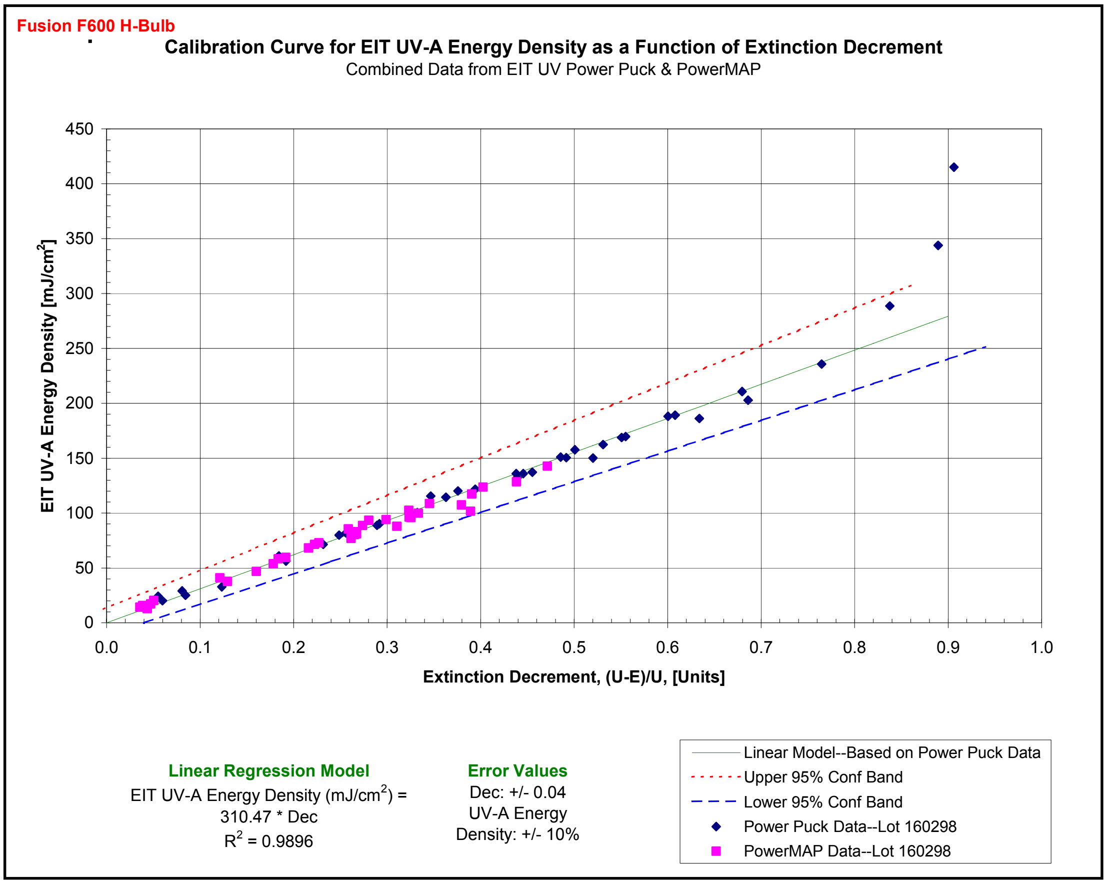


Chart 4: Comparison of UV PowerMAP™ and UV Power Puck™ for producing a calibration chart for the Fusion F600 H-bulb.

The data obtained in Chart 4 does not indicate a significant variance in energy density measurements for generating a calibration chart using either radiometer.

8. Conclusions

UVPS sells a measurement system for checking UV-A energy density in processes that have complicated web paths through which a conventional radiometer cannot pass. The system consists of a thin strip of polyester film with a UV-sensitive coating and a test strip reader. When a test strip is exposed to UV-A, the optical density of the coating is reduced. This reduction is measured as extinction by the test strip reader. To provide actual energy density measurements using the test strips, calibration is required on a separate, flat-web UV system that has a similar configuration (lamp type, focus distance, power supply, etc.) as the actual process system. By using a UV radiometer in conjunction with UVPS Rad Check™ 01 test strips, calibration charts can be generated for UV-A energy density as a function of extinction decrement.

Proper storage and handling of the test strips are necessary for obtaining reliable extinction measurements. The test strips, available in airtight packets of 50, should be stored in a refrigerator between 40 and 50°F. Limited transportation at room temperature does not affect the coating. When handling the strips, lint-free gloves such as latex or Nitrile should be worn. In addition, the test strips should not be exposed to stray UV light from fluorescent or mercury lamps. Exposure to sunlight should be avoided. UVPS recommends that when a strip is removed from is packet, it should be exposed to UV as soon as possible. However, the unexposed extinction, *U*, appears stable for at least a few weeks after a strip is removed from its packet, measured, returned to its packet, and placed back into refrigeration. The exposed extinction, *E*, appears stable at room temperature.

Lot-to-lot strip variation significantly increases the decrement error if lots are combined. Test strips from two lots were examined using an HP 8452A UV-Vis spectrophotometer, which revealed excessive variability for both the exposed and unexposed extinction values. Since energy density predictions rely on accurate extinction decrements, improved energy density predictions can be obtained by simply using test strips from the same lot for generating the calibration chart and for process measurements.

The relationship of energy density to extinction decrement is linear up to $DEC = 0.80$, after which the test strips display a non-linear response to increasing energy density levels. It is suspected that the test strips saturate when the extinction decrement reaches this level. UVPS states the maximum UV-A energy density for the Rad Check™ 01 test strips as 300-mJ/cm^2. However, Chart 2 suggests an actual maximum of 500-mJ/cm^2 for the F600 D-bulb. Chart 3 suggests an actual maximum ranging from 200- to 275-mJ/cm^2 for the F600 H-bulb—depending on the test strip lot. If multiple bulbs are to be measured, the test strips could easily saturate. Therefore, multiple bulbs should be examined individually.

Charts 2 and 3 also show that the linear regression models do not hold for out-of-lot test strips. Calibration charts should be generated using test strips from the same lot. For the models to be valid for in-lot test strips used to evaluate process UV bulbs, the average extinction decrement should be calculated using the same sample size as that used for the calibration chart.

Error analysis for the extinction decrement used RSS in conjunction with partial differentials and the Student t-distribution using a 95% confidence level. Error analysis for energy density measurement used RSS in conjunction with the Student t-distribution using a 95% confidence level. Outliers were eliminated using Thompson's Tau Rejection Criteria. The error values for each bulb are summarized in Table 4 below.

Bulb	Radiometer for Calibration	ΔDEC (+/- AU)	ΔEnergy Density (+/- $mJ/cm^2)_{EIT\ UV\text{-}A}$
Fusion F600 D-Bulb	EIT UV PowerMAP™	0.04	7%
Fusion F600 H-Bulb	EIT UV Power Puck™	0.03	10%

Table 4: Summary of error analysis for extinction decrement and UV-A energy density.

The maximum and minimum error values were used to create the 95% confidence bands for the linear regression models. The models themselves predict the true process energy densities. The confidence bands provide the predicted ranges for true energy densities with 95% confidence, i.e., the 95% probability that the true value lies within the upper and lower limits for a predicted energy density.

The energy density range using the 95% confidence band might be too large to determine a UV process window. This is because properties of the product could change significantly within the magnitude of the range. For the D-bulb, the band width was approximately +/-30-mJ/cm^2 at low extinction decrement and approximately +/-50-mJ/cm^2 at high extinction decrement. In comparison, the band width for the Fusion H-bulb was approximately +/-10-mJ/cm^2 at low extinction decrement and approximately +/-25-mJ/cm^2 at high extinction decrement. If product properties were negligibly sensitive to the magnitude of the energy density range, then the test strips would be useful for determining and maintaining a UV-A energy density process window. If tighter process control were needed, then the Rad Check™ 01 test strips would not be sufficient.

9. Suggestions for Future Work

At the time of this report, UVPS sells three types of Rad Check™ test strips for measuring different levels of UV-A energy density. Rad Check™ 01 test strips (0 – 300-mJ/cm^2) were evaluated for this report. To complete the test strip analysis, the Rad Check™ 01/D and 01/L test strips should be evaluated. The effect of multiple bulbs on the test strips was not studied for this report. Operator-to-operator variability (the procedure used to expose and measure the test strips) was not quantified for this report.

Appendix A: Technical Data for UV PowerMAP™

This appendix contains technical data regarding the EIT, Inc. UV PowerMAP™ radiometer.

SOURCE: EIT, Inc. (1998). UV PowerMAP and UV MAP Plus with PowerView Operator's Manual. Sterling, VA: Author.

12. Specifications

Electrical Specifications	
Configuration	2 part: Detachable Optics Head and UV Data Collector (UDC)
	Optics Head: Supports optics to measure 4 spectral regions
	UDC: 256 bytes non-volatile memory
UV Ranges	High Power: UVA, B, V- 200mW/cm^2 to 20W/cm^2; UVC- 20mW/cm^2 to 2W/cm^2
	Low Power: UVA, B, V- 2mW/cm^2 to 200mW/cm^2; UVC- 1mW/cm^2 to 100mW/cm^2
Spectral Response	UVA (320-390nm), UVB (280-320nm), UVC (250-260nm), UVV (395-445nm)
UV Accuracy	+/-5% typical, +/- 10% maximum
Temperature Measurement	Type J; Input Range: 500°C Maximum (Thermocouple range determined by thermocouple wire used. 250°C thermocouple wire supplied with unit.); Sample rate: 32 samples per second
UV Sample Rates	User-settable: 128, 256, 512, 1024, or 2048 samples per second
UV Sample Period	Maximum of 1 hour, determined by configuration
Operating Temperature Range	0-70°C; overtemperature alarm settable from 0-65°C; default setting is 65°C
Unit Operation	One Push Button Switch
Indicators	One Single Tone Audible Indicator
	Dual-Color LED (Red/Green)
Battery	Nickel Metal Hydride (NiMH)
Battery Cycles	500 typical
Charging Period	1 hour quick charge at temperatures below 35°C
Charging Adapter	AC input: 100-130VAC, 50/60Hz or 200-240VAC, 50/60Hz DC output: 12 VDC @ 250 mA
Operating Time	Determined by configuration. Guideline: four channels on @ 512 Samples/ second for a 2-minute sample period yields 30+ readings on one charge.
Communication to PC	
Format	RS232 Serial Port
Speed	User-settable: 9600, 19200, 38400, 57600, or 115200 baud
PowerView Software	
Minimum Computer Requirements	Pentium 60MHz, 16MB RAM, one serial port, one parallel port; 20MB space available on hard drive; CD-ROM drive or 3.5" HD floppy disc drive; Windows 95 or Windows 98 operating system
Interface	Windows-based fully graphical interface
Mechanical Specifications	
Unit Dimensions	3.50"W X 9.0"L X 0.5"D (8.89cm X 22.86cm X 1.27cm)
Weight	20.2 ounces (570 grams)
Materials	Aluminum chassis with stainless steel covers

Specifications are subject to change.

Table 2. Electrical and mechanical specifications

100200.PWR Rev. A 11/98 30

Spectral Response Curves

The Spectral Response Curves for the four UV channels are shown in Figure 22 below. The UV PowerMAP has all four channels, the UV MAP Plus has only one channel.

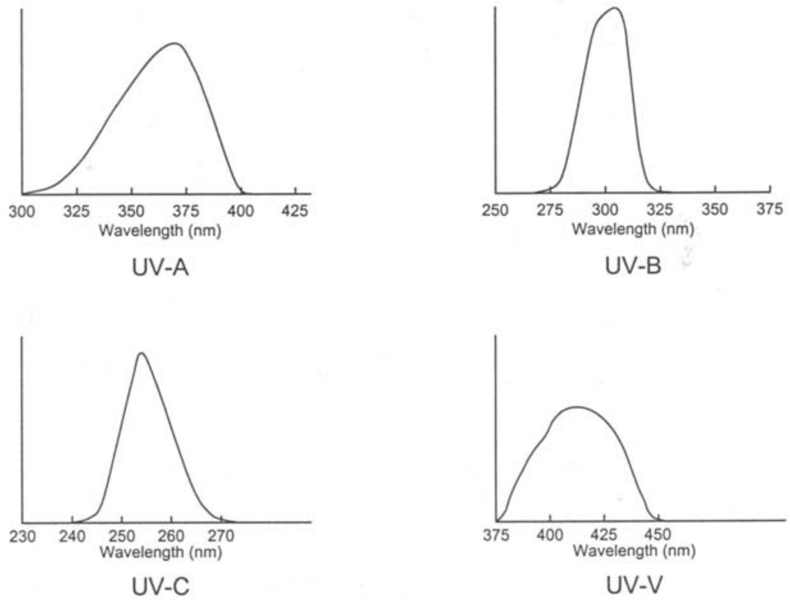


Figure 22. Spectral response curves

Optics Locations

The locations of the optics for each UV channel are shown in Figure 23. The UV PowerMAP has all four channels, the UV MAP Plus has only one channel.

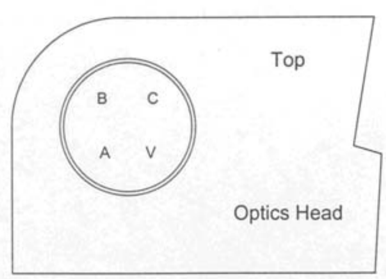


Figure 23. UV channel locations

Appendix B: Technical Data for UV Power Puck™

This appendix contains technical data regarding the EIT, Inc. UV Power Puck™ radiometer.

SOURCE: EIT, Inc. (1994). UVICURE Plus & UV Power Puck User's Manual. Sterling, VA: Author.

Hardware

Specifications *

Display:
4-digit LCD.

Range:
5 mW/cm² to 5W/cm².

Accuracy:
± 5% typical; ± 10% guaranteed or 10mW, whichever is greater.

Spectral Response:

UV Power Puck: Four channels continuously monitored during operation.

UVA	320-390nm
UVB	280-320nm
UVC	250-260nm
UVV	395-445nm

UVICURE Plus: One channel continuously monitored during operation.
(Example: UVA 320-390nm)

Spatial Response:
Approximately cosine.

Operating Temperature Range:
0-75 ° C internal temperature; tolerates much higher external temperatures for short periods; audible alarm indicates when temperature is too high. **CAUTION:** The maximum internal temperature is 80° C. If internal temperature exceeds this, the warranty is voided.

8

Time-out Period:
4 minutes RUN mode (no energy observed); 2 minutes DISPLAY mode (no key activity).

Batteries:
Two user-replaceable lithium cells, Duracell DL2450, Sanyo CR2450 or equivalent.

Battery Life:
1500 readings with typical use.

Dimensions:
4.60 x .50 inches; 117 x 12.7 millimeters (D x H).

Weight:
11.75 ounces (333.1 grams).

Package Material:
Aluminum, stainless steel.

Carrying Case Material:
Cut polyurethane interior to accommodate radiometer. Scuff-resistant nylon exterior cover.

Carrying Case Weight:
1 pound (453.6 grams).

Carrying Case Dimensions:
12 x 4.7 x 8.25 inches ; 304.8 x 119.4 x 209.6 mm (WxHxD).

* Specifications subject to change without notice.

9

Physical Features

The main physical features of the UV Power Puck and UVICURE Plus are shown in Figure 3.

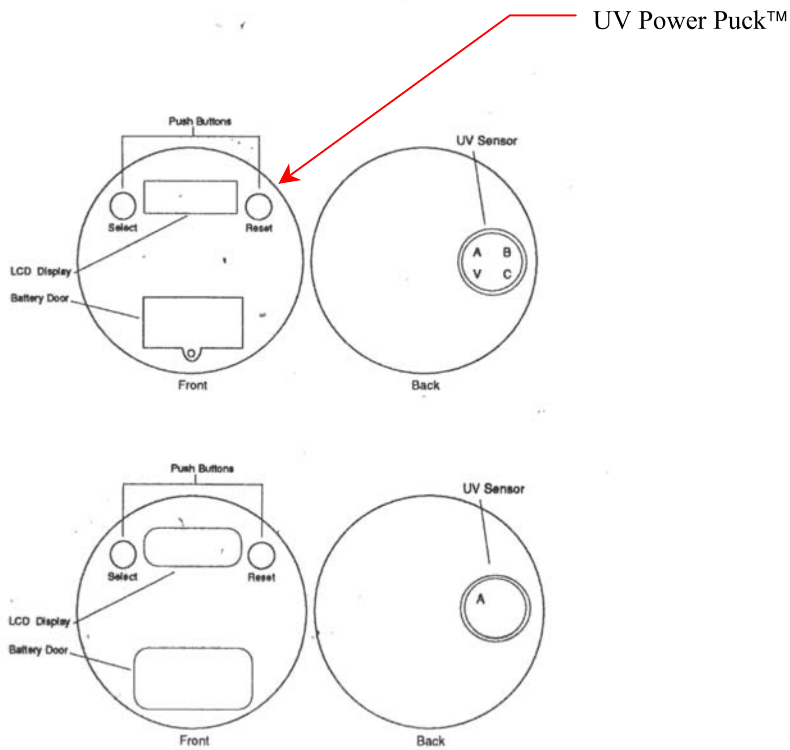


Figure 3 - UV Power Puck and UVICURE Plus Physical Features

10

Appendix C: Technical Data for Test Strips & Reader

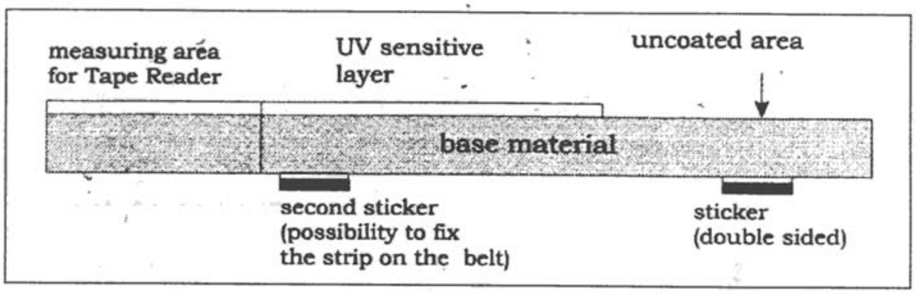
This appendix contains technical data regarding the UV Process Supply, Inc. CON-TROL-CURE® Rad Check™ test strips and reader.

SOURCE: UV Process Supply, Inc. (n.d.). CON-TROL-CURE® Rad Check System Instruction Manual. Chicago, IL: Author.

UV PROCESS SUPPLY, INC.

CON-TROL-CURE® RAD CHECK SYSTEM INSTRUCTION MANUAL

PART # M007-078

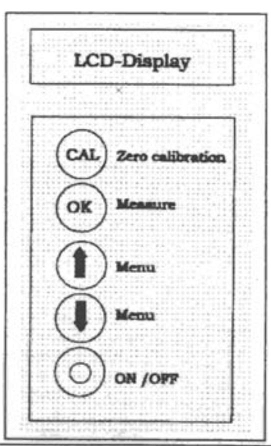


After the tape has passed through the radiation source, the optical density, or extinction, should be measured immediately.

Measurement of UV Intensity

Structure and Functioning of the Dosimeter Measuring Instrument (Brief Description)

Measuring principle:	Photometric transmitted-light measurement of test strips sensitive to UV radiation
Light source:	12v, 16 W commercial halogen lamp
Sensor:	UV-selective photodiode max. rel. sensitivity: 320 nm 50% rel. sensitivity: 300 - 360 nm
Measuring transformer:	Aluminium block to support the halogen lamp; photodiode, and guidance for measuring tape with end stop.
Power:	110/220v



View to the surface of Dosimeter

UV Process Supply, Inc.
1229 W. Cortland St.
Chicago, IL 60614-4805

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FAX 773-880-6647 • 800-99FAXUV™
UV FAXTS...Service® 773 880-6649

UV PROCESS SUPPLY, INC.

CON-TROL-CURE® RAD CHECK SYSTEM INSTRUCTION MANUAL

PART # M007-078

4. time	as under 3.
5. K-factor	provides a correction of deviations in different test strip batches.
6. autom. OFF	time for the automatic switch-off after the measurement has been completed.
7. system check	check of the electronic equipment.

Using the K factor:
The first batch of test strips has a density of 1.60, the second batch a density of 1.73. Adjust the K factor to 0.95 to obtain a recalibrated optical density of 1.62. This will hold the values within acceptable error limits.

3. Reproducibility and Measuring Accuracy

When establishing System repeatability, we determined mean values, double standard deviation, and error percentage. The error percentage is approximately 2% for test strips not exposed to radiation. This increases slightly following exposure, and varies slightly depending on dose strength.

To obtain a reliable result, perform at least three measuring sequences for each determination. This method minimizes possible deviations, and permits clear determination of the correct value. Direct comparison between exposed and non-exposed test strips is favorable for a more accurate evaluation of radiation tests.

Exposed and non-exposed tapes are measured one after the other, and the quotient EIE_0 is formed. This method recognizes and minimizes errors relating to layer thickness. The calibration curves (provided with each test strip batch) indicate the dependence of the extinction E as well as the dependence of the quotient E/Eo.

Test Strip Specifications:
- Dose Levels: Test Strip 01: 0-300 mJ/cm2;
- Range: 320-380nm
- Durability: Approx. 6 months
- Storage: Store in dark, cool environment, preferably refrigerated at 4-10°C
- Packaging: Quantities of 100

Dosimeter Specifications:
- Light source: 12V/6w commercial halogen lamp
- Sensor: UV-selective photodiode; max. rel. sensitivity: 320nm; 50% rel. sensitivity: 300-360nm

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Appendix D: Technical Data for HP Spectrophotometer

This appendix contains technical data regarding the HP 8452A Diode-Array Spectrophotometer.

SOURCE: Hewlett-Packard Company. (1990). HP 8452A Diode-Array Spectrophotometer Handbook. Federal Republic of Germany: Author.

Performance Specifications

Item	UV/VIS Instrument	UV Instrument	Extended Visible Instrument
Full Spectrum Scan	0.1 s	0.1 s	0.1 s
Time Until Next Scan			
Up to 35 wavelengths (no variance/0.1 s integration)	0.1 s	0.1 s	0.1 s
Full spectrum scan (no variance/0.1 s integration)	0.6 s	0.6 s	0.6 s
Full spectrum scan (with variance/0.5 s integration)	1.8 s	1.8 s	1.8 s
Time To Display Full Spectrum (HP 9000 Series 300 & MS-DOS UV/VIS software)	6 s	6 s	6 s
Wavelength Range	190-820 nm	190-510 nm	470-1100 nm
Wavelength Accuracy	±2 nm	±1 nm (190-400 nm)	±2 nm
Wavelength Reproducibility (typical with constant conditions)	±0.05 nm	±0.05	±0.05 nm
Spectral Bandwidth	2 nm	1 nm (190-400 nm)	2 nm
Photometric Range			
(0.5 s measurements at 350 nm)	0.0022-3.3 AU	0.0022-3.3 AU	—
Full dynamic range, dA/A 50% (0.5 s measurements at 600 nm)	—	—	0.0011-2.8 AU
Photometric Accuracy			
(at 1 AU with NBS 930D solid filter at 440 nm)	±0.005 AU	±0.005 AU	—
(with NBS 930D solid filter at 635 nm)	—	—	±0.005 AU
Baseline Flatness (0.5 s blank followed by 0.5 s measurement)	<0.001 AU rms	<0.001 AU rms	±0.0005 AU rms

General Information 1-3

Performance Specifications (continued)

Item	UV/VIS Instrument	UV Instrument	Extended Visible Instrument
Noise			
(0 AU, 60 × 0.5 s measurements)	<0.0002 AU rms (500 nm)	<0.0002 AU rms (340 nm)	<0.0001 AU rms (780 nm)
Stability			
(0 AU, constant ambient conditions)			
Measured over 1 hour, every 5 s without internal referencing	<0.002 AU (340 nm)	<0.002 AU (340 nm)	<0.001 AU (620 nm)
with internal referencing at 400 nm	<0.001 AU	<0.001 AU	—
Measured over 60 s period, every 5 s	<0.001 AU	<0.001 AU	—
Stray Light			
(measured with Hoya 056 at 220 nm and 340 nm)	<0.05%	<0.05%	—
(measured with Schott RG850 filter at 530 nm and 650 nm)	—	—	<0.05%

All specifications are measured after one hour from cold start or from lamp turn-on, with no cell or filter unless specified.

1-4 General Information

Physical Specifications

Dimensions	60.5 × 18.5 × 42.4 cm
	(23.8 × 7.3 × 16.7 inches)
	width × height × depth
Weight	15.1 kg (33.2 pounds)
Line Voltage (selectable)	100-120 or 220-240 VAC
Frequency	47 to 66 Hz
Power	100 watts maximum

Environmental Specifications

Ambient Temperature	
Operating	0 to 40°C (32 to 104°F)
Non-operating	−40 to 65°C (−40 to 149°F)
Maximum rate of change	10°C/hour (18°F/hour)
Humidity (25-40°C, non-condensing)	5 to 85%
Altitude	
Operating	4,600 m (14,950 ft)
Non-operating	15,300 m (49,700 ft)