Radiometric Methods for UV Process Design and Process Monitoring

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[Presented at RadTech North America 2002, Indianapolis, IN; April 9-12, 2002]
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ABSTRACT

A wide variety of radiometric instruments are now available for measuring the radiant characteristics of industrial and laboratory UV lamps. Relating these characteristics to the performance of a UV-cured product depends on how well the selected parameters match the critical factors of the cure process. Further, the distinction between process *design* and process *monitoring* is significant, especially in the characteristics to be measured.

The difference in instruments and the reasons for these differences is followed by suggestions of how to avoid some common errors and to report data more completely. Optical terminology, specifications, methods, and how radiometric measures are used in system design and in production monitoring are presented.

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Introduction

The most important principle of effective radiometry is that the measurements must be relevant to the process or, in other words, must be related to the development of the physical properties of the final product. By thoroughly understanding the lamp-chemistry-application interactions, more precise and useful specifications can be determined for what to measure in the design of a process and for the establishment of meaningful limits that can be applied to process monitoring. In addition, data from radiometers need to be communicated in a consistent and uniform way. This facilitates the duplication of the UV exposure conditions that produce the desired curing result, and is also important in the event that problem-solving communication with suppliers is necessary.

This discussion of radiometry in UV will touch on

- the use of radiometry for process DESIGN
- the use of radiometry for process CONTROL or MONITORING
- key optical factors to measure
- types of radiometers
- some sources of error in radiometric measurement
- recommended methods of reporting data

Process Design versus Process Control

Process design is the optimization of the UV exposure conditions based on the requirements of an ink, coating, or adhesive. A typical laboratory method is to vary formulation ingredients and/or UV exposure, and evaluate the resulting cured material. Generally, the material's spectral absorption, optical thickness, and photoinitiator types and concentration determine what exposure is most effective. Attaining a specific set of physical properties in the cured film on an end-product is ultimately what is important, and design information is critical to achieving an efficient process with an adequate operating range, or "window."[(1)]

The process design phase determines *exposure* requirements such as peak and focus, irradiance profile, spectral distribution, power level, peak-to-energy ratio, temperature, and time. Radiometric

measurements are useful in quantifying the successful exposure parameters, so the process can be reliably duplicated. The function of radiometry is to provide *quantitative* information about the critical

Process Design	**Process Monitoring**
Objectives: ***Optimize*** the cured properties of the end product. ***Quantify*** the key exposure conditions: • Effective irradiance or Profile • Spectral Distribution • Time or speed (determines energy) • Infrared or temperature ***Transfer*** the process to production	*Objectives:* ***Verify*** that the key optical conditions remain within specified limits ("process window") ***Interpret*** changes in the exposure conditions to maintain control

requirements of the process and to establish the *limits* within which the process is successful.

Process control maintains the process within the "window" of operating limits. The primary purpose of process monitoring is to know when something has changed *before* it threatens the process. If the radiometric data collected doesn't relate to the process "window," it doesn't mean much. For example, if the chemistry requires UV wavelengths in the 250 nm region, measurement in the 365 nm region would not be relevant. Also, if lamp design characteristics such as irradiance profile or spectral radiance have been determined in the design of the system, it is not necessary to repeatedly measure these for process control -- unless they are expected to change. Proper measurements can be valuable to determine when replacement or maintenance is required as lamps age or become dirty.

Irradiance, Spectral Distribution and Energy

There are four key factors (outside of the formulation itself) which affect the curing and the consequent performance of the UV curable material. These factors are the UV *exposure* conditions, which are a consequence of the optical characteristics of the curing system. Simply stated, these are the exposure parameters that are sufficient to define the process:

- **irradiance** -- either peak or profile, measured in W/cm² or mW/cm²;
- **spectral distribution** – emitted radiant power versus wavelength in nanometers (nm);
- **time** (or 'speed') – energy is the time-integral of irradiance measured in J/cm² or mJ/cm², and
- **infrared** (IR) or heat – usually observed by the temperature rise of the substrate.

Irradiance data must <u>always</u> include identification of the <u>wavelength range</u> to which it applies. This is one of the most common omissions in radiometry. When irradiance is measured in any specific range of wavelengths, it is called "*effective irradiance*."[(2)] When this wavelength range is clearly

understood, the term "*irradiance*" is sufficient. ("Intensity" is not a technically defined term but is commonly, although improperly, used to mean irradiance). Peak irradiance has a distinct effect and benefit on speed and depth of cure.[3]

UV Effective Energy Density is the UV energy to which a surface is exposed as it travels past a lamp or a sequence of lamps. It is sometimes loosely (if not incorrectly) referred to as "dose." For an exposure in which irradiance is not constant, such as rising then falling, energy is the time-integral of irradiance. Effective energy density incorporates irradiance profile, the wavelength range of interest ($\lambda_1 \div \lambda_2$) and time:

$$E_{(\lambda_1 \to \lambda_2)} = \int_{t_0}^{t_1} I_{(\lambda_1 \to \lambda_2)}\, dt$$

As with irradiance, when the wavelength range is clearly stated, and it is clear that the meaning is "per unit area," this term can be simply abbreviated as "energy."

Infrared (IR) energy is emitted primarily by the quartz envelope of the UV source. Because commercial UV radiometers do not measure IR irradiance, measurement of surface temperature is the usual method of determining the effect of IR. The heat may be a benefit or a nuisance, but is an inseparable factor in the curing process. A non-contacting optical pyrometer[4] is recommended for surface temperature measurement.

Information about the peak of irradiance, or of the entire exposure *profile* is important to the design. Figure 1 illustrates several irradiance profiles, all of which exhibit approximately the same energy content, but distinctly different profiles -- duration and peak. The fact that these exposures produce different physical properties in most UV-curable materials is the reason that profile information is needed in the process design stage.

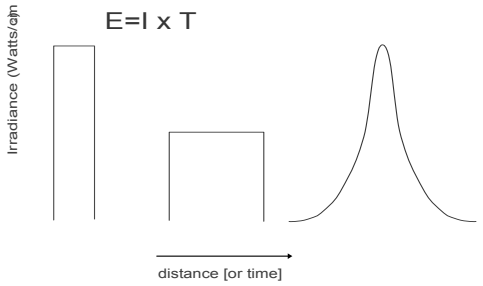

Figure 1 Irradiance Profiles

The exposure *profile* is characteristic of any lamp design. The peak of the exposure profile is the peak irradiance; the area under the exposure curve is proportional to total energy. The relationship of the peak to energy/time is important to cure efficiency. It is not possible to increase the peak-to-energy/time ratio of a lamp -- this is determined by its physical design -- it is a characteristic of the irradiance profile of the lamp. In use, the profile of a lamp can deteriorate if the bulb sags out of the focused position, or if the reflector has been deformed.

A measurement of total UV energy ("dose") is a composite of irradiance profile and velocity, but information about neither irradiance nor time can be extracted from it. Consequently, data on energy alone is less important to design, but more useful in monitoring or control.

Radiometric Instruments and Devices

In selecting radiometric instruments, the desire is to find one instrument that 'does it all' and provides universally understood information. Unfortunately, no one method gets a perfect score.

Radiometers measure *irradiance* (usually watts/cm²) at a point, but over a uniquely defined wavelength band. Differences in detectors, filters, construction, and principles of operation result in the fact that different narrow-band radiometers give different results when measuring broad-band sources (see Figure 2). A radiometer from one manufacturer can report UV data significantly different from another instrument from a different manufacturer. This is because instruments have different *responsivity*, or wavelength sensitivity. Further, instruments differ in their spatial sensitivity (angle of view), although most have diffusers to give them a *cosine* response. As a practical matter, many users prefer to compare data from instruments only of the same type.

In addition, integrating radiometers electronically calculate energy.

Dosimeters measure accumulated energy at a surface (watt-seconds/cm² or joules/cm²), also over some uniquely defined wavelength band. There are electronic and chemical types. Because this is the only measurement that incorporates time of exposure, it tends to be commonly used.

Spectroradiometers are very narrow-band instruments, essentially responding to spectral irradiance, and are highly wavelength-specific -- some with resolution as fine as ½ nanometer. These instruments -- actually miniature monochromators -- can be valuable when there is a need to evaluate irradiance in a selected wavelength band of interest, but they don't measure time-integrated energy. Recent developments in these instruments include the ability to *select* a specific wavelength band for easier evaluation of the spectral distribution of a lamp output or spectral irradiance.(5)

Radiochromic dosimeters are tabs or films that attach to a test surface and respond to total time-integrated energy by changing color or by changing optical density. Depending on the chemistry of the detector, it can change permanently or only temporarily. These photochromic detectors typically respond to a wide range of UV wavelengths.

Responsivity

The amplitude of response of a detector to different wavelengths is referred to as *responsivity*. The design of the instrument, cell type and filter results in a singular response curve. The net response curve, in percent of maximum response, is called its *relative spectral responsivity*. Examples of response of selected wavelength bands of two commercial instruments are shown in Figure 2.

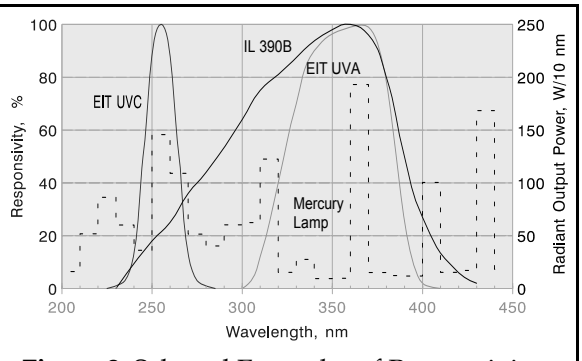

Figure 2 Selected Examples of Responsivity and Mercury Lamp UV Emission

Spectral responsivity is the characteristic that differentiates wide-band radiometers from narrow-band radiometers. It can be easily seen, from Figure 2, that any of these yield very different measurements when exposed to the same lamp!

The manufacturer's designations for the band of the examples illustrated are:

EIT, Inc.	EIT UVC[6]	240-260 nm	(50%)
International Light	IL 390C[7]	250-400 nm	(10%)
EIT, Inc.	EIT UVA[6]	320-390 nm	(50%)

All instrument manufacturers provide the responsivity data for their instruments. Note that the wavelength response band of an instrument may be specified at the 10% response wavelengths, or at the 50% response wavelengths (compare figure 2).

A Note About Wavelength Designation, "UVA," "UVB," "UVC"

It is interesting that the *language* of UV wavelength designations has no equivalent terms such as "red," "yellow," "green" or "blue." Historically, the technology was forced to adopt terms from the field of physiology, where "UVA," "UVB," and "UVC" are generally accepted designations for distinct UV ranges, differentiated by effects on biological tissue, primarily by sunlight. UV curing technology, having no designations of its own, adopted these. In UV processing, "UVA," "UVB" and "UVC" are only loosely defined, having been modified slightly to represent dominant emission ranges in the emission of mercury (Hg) vapor plasmas.

Similar designation are used in various technologies involving UV light.[8] Typically, the generally accepted range designations are:

UVC 200-280 nm

UVB 280-315 nm

UVA 315-400 nm

Some Limitations

Few commercial radiometers accurately respond in the 200-240 nm range. This is primarily due to limitations in filter materials used with photo-detectors, and to internal scattering effects in spectroradiometers. Radiochromic detectors are very responsive to short-wavelength UV, but are rarely calibrated for responsivity in any wavelength band -- they typically require correlation to a radiometer.

Sources of Error

Of the many types of instruments available, all have some characteristic element, which can result in errors in their readings. The purpose here is not to evaluate these sources of error in any specific instruments, but to illustrate them so that the user can be aware of them, and know what information is

available from instrument manufacturers.

Figure 3 illustrates the typical action of a sampling radiometer as it passes the irradiance profile of a lamp.

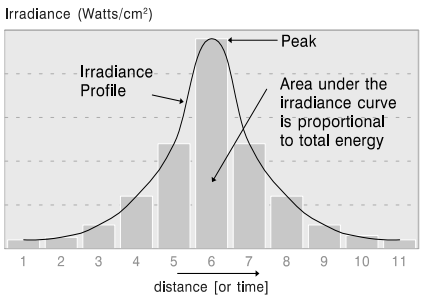

Figure 3 Typical Sampling Radiometer

Over-Range Error

The dynamic range of the instrument must be adequate for the irradiance to which it is exposed. Radiometers may have a limit to the "intensity" that they can tolerate, or yield accurate measurement. All radiometer manufacturers provide dynamic range information on their instruments, and electronic instruments should have an over-range indicator. The peak irradiance of a high-power, finely focused lamp can exceed 10-15 watts/cm².

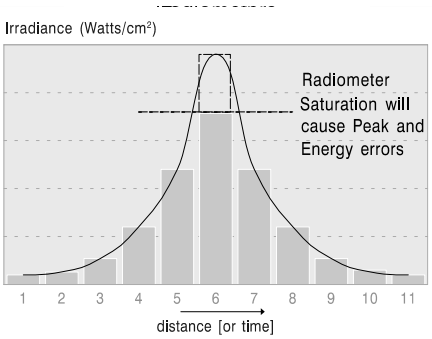

Figure 4 Over-Range Error

The effect of over-ranging, is illustrated in Figure 4. The irradiance information that is missed causes an obvious error in its peak reading, and if the instrument sums these samples to calculate energy, that will contain a hidden error, also. Averaging several measurements does not correct this error.

Sample Rate Error

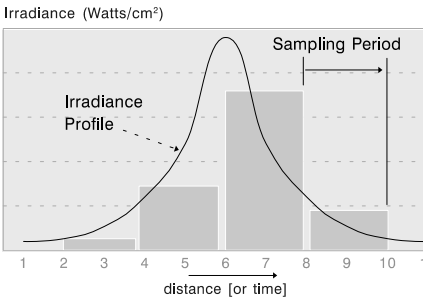

Figure 5 Sample Rate Error

Radiometers that display irradiance or calculate effective energy density ("dose") do so by accumulating a sequence of samples, under control of an internal clock, which determines the sampling frequency. For example, a radiometer with a sampling rate of 25 samples/second[9] would take a sample at one-inch intervals when traveling at a speed of 25 inches/second (48 m/min). This could result in serious error if the irradiance pattern has a peak width of ½ inch (13 mm). In this example, illustrated in Figure 5, the test speed is too fast for the lamp/radiometer combination.

Because sampling error causes erratic measurements, there is an inclination to take several measurements and average them. When sampling error occurs, taking multiple measurements does not eliminate the error -- it simply incorporates it in the average. This problem can be avoided by making measurements at a lower speed, and calculating energy for higher speeds.

Avoiding Speed Errors

It's not necessary to repeatedly run a laboratory radiometer under a lamp at different speeds to

evaluate exposure conditions. First, the irradiance peak and profile *DO NOT CHANGE* with speed. Secondly, energy varies strictly and inversely with speed and can be accurately and quickly calculated, but errors can be introduced at higher speed, depending on the instrument.

The energy-speed relationship is simply an inverse relationship for any given configuration and power of lamp. In other words, *the energy at any speed can be calculated from the energy at any other speed*. This is a useful point, because it allows radiometric measurements to be made in speed ranges where measurement error is a minimum. The *recommended method* for energy measurement is to select a speed, v_o, at which speed errors are a minimum, record several measurements, E_o, and speed -- then *calculate* energy, E_x , for any other speed, v_x.

$$\text{Since} \quad E_x v_x = E_o v_o , \quad \text{then} \quad E_x = E_o \cdot v_o / v_x$$

To calculate energy at any speed, simply multiply an error-free energy measurement by its speed and divide by the desired speed.

A *recommended test* for determining when sampling error occurs in measurements under any given lamp relies on the fact that energy is strictly an inverse relationship to speed. By making test measurements at high speeds and comparing them to the calculated value, the speed at which these begin to deviate is evident. This can also be observed in the peak reading.

Fast Sampling Error

In order to overcome the problem of low-sampling-rate errors, some sampling radiometers are designed for extremely fast rates -- one or two thousand samples per second. In this case, a different kind of error can occur. All lamps exhibit fast fluctuations, caused by the fact that they are powered by 50 or 60 Hz AC voltage or by pulsating DC voltage. The plasma within the UV bulb responds instantly to these fluctuations, and a fast-response instrument faithfully records instantaneous peaks that occur throughout the exposure profile, rather than an average. This results in a higher-than-average peak irradiance reading. It does not cause an error in time-integrated energy.

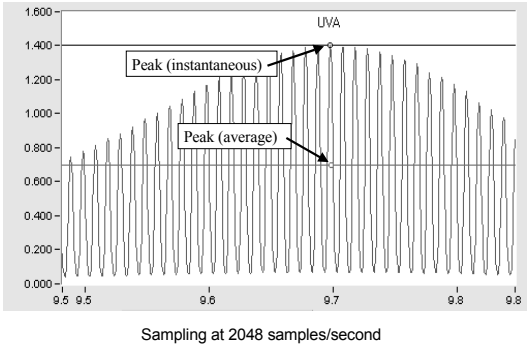

Figure 6 Interpretation of Peak for Fast Sampling

Figure 6 shows the irradiance profile of a typical mercury arc lamp, sampled at the rate of 2048 samples per second.[10] Here, the difference between the *absolute* peak (1.4 mJ/cm^2) and the *average* peak (~.7 mJ/cm^2) is clear. The difference in "peak" reading can cause confusion.

Unless there is a reason to evaluate the *instantaneous* radiance of the lamp, a sample rate of

approximately 128 samples per second is adequately fast to record the peak of any lamp and avoid sampling errors, and slow enough to integrate the radiant fluctuations of the plasma. The slower rate has the benefit of reducing the electronic file size required to store the data. If a fast sampling rate is used,

What you should know about your Radiometer

- Responsivity (Wavelength Range, λ_2-λ_1)
- Dynamic Range (Saturation, W/cm²)
- Sampling Rate (Samples per Second)
- Spatial Response (Cosine or other)
- Orientation Preferred (Radial Symmetry)
- Threshhold Response (mW/cm²)
- Instantaneous or Average Peak
- Temperature Tolerance

then electronic "smoothing"[10] should be applied to the data before reporting the peak.

Practical Issues of Measurement

By the way they are applied, UV curing radiometers might be divided roughly into two groups: *static* and *dynamic*.

Dynamic measurements are made by devices, which move or are moved with respect to the lamps. The object is to emulate the surface to be cured, and the devices record the conditions of exposure they observe. They may be attached directly to the surface or, as in the laboratory, be passed under a lamp or lamps at controlled speeds in order to duplicate the exposure conditions.

Static measurements are made with the instrument positioned at a single location. Typically, the purpose of static measurement is to provide long-term information about the condition of a lamp -- any changes that may occur in the overall radiant power output, focus conditions, or changes in bulb or reflector. If the radiant energy detected is altered, then, by inference, it is altered at the work surface. Static measurement methods also include instruments with hand-held probes, which are inserted into the field of exposure. Static measurements can record irradiance at a location, and spectral distribution, but not energy, because there is no relationship to an exposure time interval.

"Mapping" Radiometers

Some of the most dramatic adaptations of radiometers for UV processing are sampling radiometers with on-board memory. After a test exposure, the instrument is connected to an external device -- either a computer or a dedicated processor -- to display the entire exposure profile. These instruments can also calculate peak irradiance and energy. Single-band and multiple-band instruments are available.[11] Since these record the "history" of a pass under lamps, they can provide data on the

irradiance profile of each lamp in rows of lamps. Relating the time scale to distance requires only the knowledge of the precise speed of the measurement. A profile of a two row lamp system is illustrated in Figure 7.

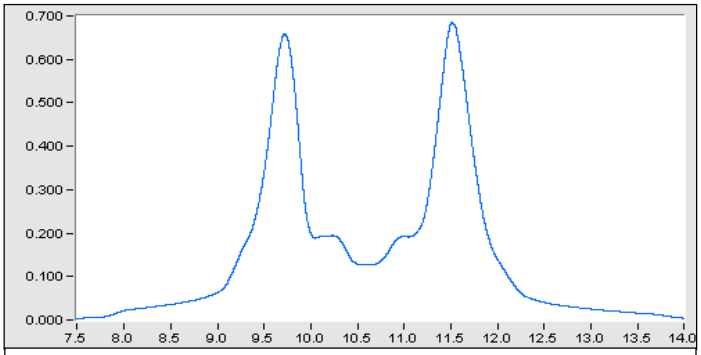

Figure 7 Irradiance Profile, Two Lamps in Sequence

Radiochromic Films

Radiochromic dosimeters are found in the form of tabs or strips that attach to a test surface, respond to total time-integrated energy ("dose") by changing color[12] or by changing optical density.[13][14] They can be very handy, especially for 3-D objects, as a number of them can be placed about the object to measure and compare the energy delivered to any part of the surface. Tabs and strips have the obvious advantage that they can be attached to a flat web or sheet and passed through the UV Curing system. They can survive transit through nips, rollers, and the like, without damage. They are inexpensive and easy to apply.

A drawback to radiochromic films is that they generally respond to and record accumulated energy only. In a multiple lamp system, they cannot distinguish the individual exposures of successive lamps. Commercial radiochromic films are not wavelength-specific. In fact, very little *spectral responsivity* data is available. Radiochromic chemistries tend to respond to short UV wavelengths, typically from 200 up to 300 or 350 nm.

Some preparation has to be done in order to correlate the results of these films with either radiometer measurements,[15] or physical properties, or both. Figure 7 illustrates the correlation of tabs whose optical density (at 510 nm) has been correlated specifically to an EIT PowerPuck® radiometer. This type of correlation must be done for each specific exposure -- type of bulb and spectral distribution. Once done, the

Figure 8 Correlation of O.D. of Film with Radiometer Exposure to "D" bulb followed by "H" Bulb

correlation can make quick work of multiple measurements.

This suggests that these can be very effective for use in process monitoring or in evaluation of configurations in process design. Radiochromic films can be helpful in the design of a system in the specific task of physical arrangement of lamps in, for example, surface curing of 3D objects.

With more development in the area of spectral calibration, radiochromic coatings and films could become a significant process control tool.

"On-Line" Monitors

On-line monitoring of the production process can use relatively simple detectors or more sophisticated instruments that record a number of key variables and compare them to pre-set limits. Situated in a fixed location, on-line monitors view the lamp and possibly the reflector, recording any change that may occur. The purpose for these fixed detectors may range from a simple comparison over time (used to determine, for example, when to change a bulb) to multiple-wavelength detectors and even spectroradiometers, located carefully to detect changes in reflectors or bulbs.

The disadvantage of "on-line" monitors is that they are not at the substrate, and therefore do not actually measure the process. Because they monitor a lamp system at discreet points, system monitoring may require a large number of locations to be inspected and, as a result, a large number of instruments or elaborate methods of re-positioning detectors.

The advantage of on-line monitoring is that it is simple and automatic. It is not necessary to include a large number of parameters--in fact, one or two is usually adequate. This type of monitoring is best used to detect serious changes in key exposure parameters, and to indicate the need for maintenance before the process is affected. Periodic correlation with a calibrated radiometer is recommended.

Recommended Practice for Reporting Data

A *recommended practice* is to accompany any reported measurement with either an indication of the wavelength range for that measurement or, at least, the identification of the instrument used. All radiometer manufacturers provide information on the wavelength band response of their instruments, and instrument-to-instrument agreement within the same model is generally good. It is essential that this information be included in the descriptive text, if not on the axis labels of charts and graphs. When this information is not reported, the data cannot be duplicated, verified, or transferred to production specifications.

A *suggested form* of the reported numerical data might be, for example, to include an indication of the instrument or wavelength band. These examples show different ways of communicating the radiometer information: (a) by manufacturer's designated band, (b) by wavelength-defined band, or (c) by identifying the specific instrument used. Whatever form or format is used to report radiometer data, it must include adequate information about the method in order for it to be repeated or to be used as a basis for process

(a) _____ $mW/cm^2|_{EIT\ UVA}$ - Identify response range by the manufacturer's designation, or

(b) _____ $mW/cm^2|_{320\text{-}390}$ - Identify the wavelength range (note: 10% or 50%)

and for energy, the same designations above, plus speed or time

(c) _____ $mJ/cm^2|_{IL390B@\_\_ft/min}$ - Identify response by manufacturer's designations and speed

monitoring.

When *energy* data is recorded or reported, a precise indication of the speed at which the measurement is made is essential. Further, this permits the correlation of energy to other speeds.

Conclusion

Radiometry is a powerful analytical tool for UV curing process design and invaluable as a QC tool for process monitoring. It is important to identify the key exposure parameters that have the most significant effect on the performance of the end product.

For process *design*, it may be necessary to evaluate a significant number of parameters and variables in order to optimize a process and assure a wide operating "process window." Process optimization means matching the lamp system and the required exposure variable to the ink, coating, adhesive and its application, and matching the UV curable chemistry to the job to be done. In order to evaluate the effects on the physical properties of the final cured product, correlation with exposure variables is essential. These variables can be expressed in terms of irradiance profile, spectral distribution, total energy, and infrared energy (or temperature). Multi-band radiometers, mapping radiometers (to evaluate profile), and spectrophotometers can record information on a significant number of these parameters. In addition to facilitating the optimization process, these measurements are used to determine the operating limits for production process control.

Once designed and optimized, monitoring the process in production may be limited to "surveillance" on only a few key parameters -- those which, when out of pre-determined limits, affect the result. From process design, these critical parameters were identified. Relatively inexpensive, simple and rugged tools and methods can be used in production monitoring. These may be on-line monitors, dosimeter tabs, single band radiometers, and the like. Ultimately, these measurements must be related to target properties to be achieved and must be correlated with measurements of the optimized process from the process design parameters.

Selecting a method of measurement or a particular radiometer should be based on the specific process and the identification of the key variables that have the greatest effect on the process.

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References

1. Stowe, R. W.; "Practical Relationships Between UV Lamps and the UV Curing Process *Window*;" Proceedings, RadTech North America; 1994
2. Kostkowski, Henry J., Reliable Spectroradiometry, Spectroradiometry Consulting, 1997, ISBN 0-9657713-0-X
3. Stowe, R. W.; "Practical Aspects of Irradiance and Energy Density in UV Curing;" Proceedings, RadTech North America; 2000.
4. Raytek, Inc.; Santa Cruz, CA (www.raytek.com)
5. Solatell Instruments, 4D Controls Ltd, Cornwall, UK (www.solatell.com)
6. EIT Instruments, Sterling, VA (www.eitinc.com)
7. International Light, Newburyport, MA, (www.intl-light.com)
8. Bolton, James R., Ultraviolet Applications Handbook, Bolton Photosciences, Inc., Ontario, Canada, 1999; ISBN 0-9685432-0-0
9. EIT PowerPuck®, EIT Instruments, Sterling, VA (www.eitinc.com)
10. EIT PowerMap®, EIT Instruments, Sterling, VA (www.eitinc.com)
11. Eit UviMap® and PowerMap®, EIT Instruments, Sterling, VA (www.eitinc.com)
12. UV/EB Labels; UV Process Supply, Inc. Chicago, IL; (www.uvprocess.com)
13. Rad Check™ Dosimeter; UV Process Supply, Chicago, IL; (www.uvprocess.com)
14. Far West Technology, Inc., Goleta, CA; (www.fwt.com)
15. L'Abbe, P. and Diehl, D.; "Measuring UV Cure: A Simple Method for Difficult Applications;" FSCT Coating Conference, Dallas, TX; 1999.