Measurement Systems and Calibrations for UV Radiation

The pandemic situation caused by the COVID-19 virus has inspired many companies, research institutes and lighting designers to adopt UV radiation as a new tool in their projects and research. The promising germicidal effect of UV-C radiation, also on the coronavirus, raises the question of the reliable measurement of UV radiation. However, this complex task needs expertise and the appropriate equipment. Conventional radiometers are easy to use, but limited only to the very narrow spectral range, typically around one emission line.

The system best suited for UV measurements consists of a high-precision spectroradiometer with stray light correction and irradiance probes or PTFE-coated integrating spheres for total radiant flux measurements. However, the reliable and traceable calibration of the system is the challenging factor. So far, no national metrological institute has been able to offer a reference standard for the total radiant flux in the UV-B and UV-C spectral region. Therefore, we have realized traceable UV LED calibration standards that complete the measurement system for UV radiation presented here.

The UV LED calibration standards were developed for the typical peak wavelengths of 280 nm (UV-C), 305 nm (UV-B) and 365 nm (UV-A). The traceability of the radiant flux is ensured by the precise calibration of the spectroradiometer with the irradiance probe and a subsequent integrative measurement using a goniospectroradiometer. Such UV LED calibration standards can be used for monitoring and for absolute calibration of UV measurement equipment consisting of the stray light corrected spectroradiometer and the integrating sphere. The largest contribution to the measurement uncertainties of the systems containing integrating spheres is the fluorescence of the reflective material. A special manufacturing procedure with optically pure Polytetrafluorethylen (PTFE) enabled us to produce new integrating spheres with permanently low fluorescence.

Introduction

Ultraviolet (UV) radiation covers a wide wavelength range between 100 nm and 400 nm and is divided into three main areas according to the standard ISO 21348: UV-A between 315 nm and 400 nm, UV-B between 280 nm and 315 nm and UV-C between 100 nm and 280 nm. Typical applications in the UV-A range are UV curing or UV ink printing. Light sources in the UV-B range are mainly used in medical skin treatments. The main application in the UV-C range is water and surface disinfection, which is currently of great importance in the fight against pathogens such as the novel coronavirus COVID-19.

In the light source industry, there has been an increased demand for UV-C radiation sources since the outbreak of the SARS-CoV-2 pandemic. UV-C radiation between 255 nm and 265 nm has been recognized as a highly efficient method for inactivating the DNA or RNA of microorganisms such as the coronaviruses, effectively preventing their replication and ability to actively infect other cells.

Because of their specific areas of application, all UV radiation sources should be characterized very precisely with regard to their radiant flux and spectral distribution. This requires reliable and accurate UV measurement systems over the entire UV range. The use of radiometers, however, is restricted to a very narrow range of wavelengths, typically only around one spectral line. The system, which meets all requirements for UV measurements, consists of a high-precision spectroradiometer with stray light correction and either coupling optics for measurements of the irradiance [W/m^2] or integrating spheres made of polytetrafluoroethylene (PTFE) for radiant flux measurements [W].

However, a reliable and traceable calibration of the measuring system is a challenge. So far, no national metrological institute has been able to offer a reference standard for the radiant flux in the UV-B and UV-C spectral range. For this reason, we have developed traceable UV-LED calibration standards that complete the UV measurement system. The special test procedure for determining the radiant flux in the UV is accredited according to ISO 17025 (D-PL-19052-01-00).

Optimization of Measurement Systems for UV

The main limitation of the performance of an array spectroradiometer in photometry and radiometry is the occurrence of stray light in the instrument, especially in the sensitive UV range. This means that a particular element of the array detector registers radiation from a different spectral region than the designated one. The reason for the occurrence of stray light can be found in various mechanisms:

- scattered light from the diffraction grating due to manufacturing inaccuracies in

the shape and spacing of the lines, or roughness of the surface of the grating,
- higher diffraction orders, particularly for detectors with a wide spectral range,
- double diffraction of the light reflected back on the grating,
- inter-reflections between the detector and other optical components,
- reflection and scattering from surfaces, especially from the inner wall of the spectrograph,
- fluorescence of optical components,
- the way the light is coupled into the spectroradiometer.

Thus, the total amount of the measured radiant power contains a partial amount of incorrect radiation that causes an error in spectral power distribution. The main approach to improve the radiometric performance of the spectroradiometer is to avoid, or at least largely suppress, the stray light by design measures of the spectrograph. When further suppression is technically not possible, the residual stray light can be effectively corrected to a great extent, for example by applying the NIST method [1].

The main idea of this method is that monochromatic radiation can be attributed, for the most part, to a certain pixel of the CCD array detector. The entire radiation that is measured outside the bandpass function for this wavelength is the stray light contribution of this pixel that is seen from all other pixels in the detector. In the practical realization, the stray light correction is achieved with a tunable optical source that emits narrow (<1 nm) spectral lines over the entire spectral range. Excitation wavelengths are tuned within the measurement range of the spectroradiometer in 10 nm steps and a spectrum is recorded for each laser excitation wavelength. The set of all recorded spectra over all excitation wavelengths results in a device-specific matrix. If the band-pass function of the real signal is subtracted, one obtains a stray light distribution matrix. The inverse of the stray light matrix can be numerically multiplied with raw spectra in order to obtain stray light corrected spectra.

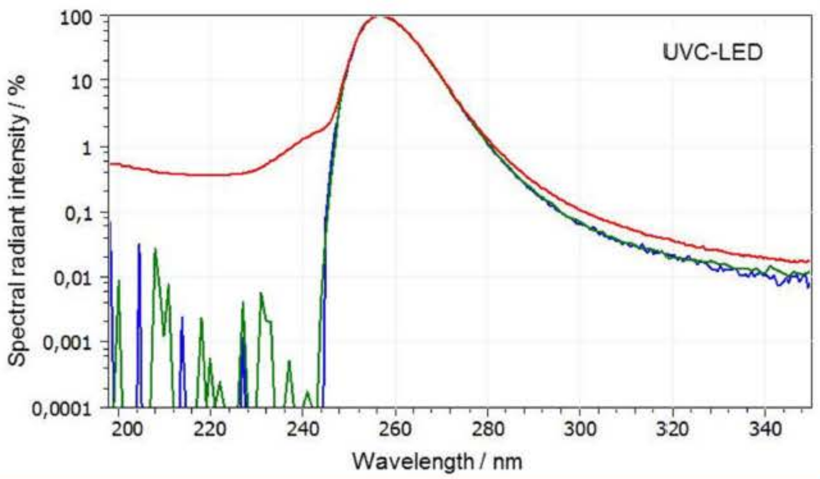

Figure 1: Logarithmic display of the spectra of a UVC LED with (blue) and without (red) stray light correction of an array spectroradiometer, measured with a double monochromator (green).

Figure 2: Integrating spheres made of optically pure polytetrafluoroethylene (PTFE) for low-fluorescence measurements of the radiant flux in the UV.

Figure 1 shows spectra of a UV-C LED with and without stray light correction in logarithmic presentation, as an example. The suppression of the stray light in the spectral curve by more than one order of magnitude in the UV range is clearly recognizable. The impact of the stray light correction on an array spectroradiometer is almost as high as the stray light level of a double monochromator, which is the best spectroradiometer worldwide, that can be reached. A direct benefit of the stray light correction is higher precision in radiometric evaluation, especially in the UV-C range.

For measurements of the irradiance [W/m^2], simple coupling optics can be used, which are connected to the spectroradiometer via a UV-optimized fiber bundle. A special diffuser element is used for UV applications, which ensures homogeneity in the detection of the radiation. The fiber bundle allows flexible handling in combination with a high throughput. The traceable calibration to the spectral irradiance [W/m^2/nm] is realized directly using a deuterium lamp as a reference standard.

Reliable Measurements of Radiant Flux

For measurements of the radiant flux [W] in the UV, integrating spheres made of polytetrafluoroethylene (PTFE), which is also highly reflective in the UV range, should be used (**Figure 2**). The reflection index of barium sulphate ($BaSO_4$) drops sharply towards the UV range so that integrating spheres made of barium sulphate can only be used to a limited extent in the UV range. Depending on the size of the source, spheres of different sizes can be used, but the lateral sphere opening should not exceed 1/3 of the sphere diameter. Since the sources themselves absorb part of the emitted radiation, depending on their size and body color, a so-called self-absorption correction should be carried out over the entire spectral range, e.g. with a combined deuterium-halogen lamp.

The greatest contribution to the measurement uncertainties of the measurement systems with integrating spheres is the fluorescence of the reflective material. The portion of the fluorescence in the spectrum can be made visible with a blue LED measurement. Much higher fluorescence is visible on the right flank of the blue LED for poor quality PTFE than for the PTFE material with optical quality (**Figure 3**). A special manufacturing process with optically pure polytetrafluoroethylene (PTFE) enabled us to manufacture new integrating spheres with permanently low fluorescence. These PTFE spheres can be audited and recalibrated with the newly developed UV-LED calibration standards.

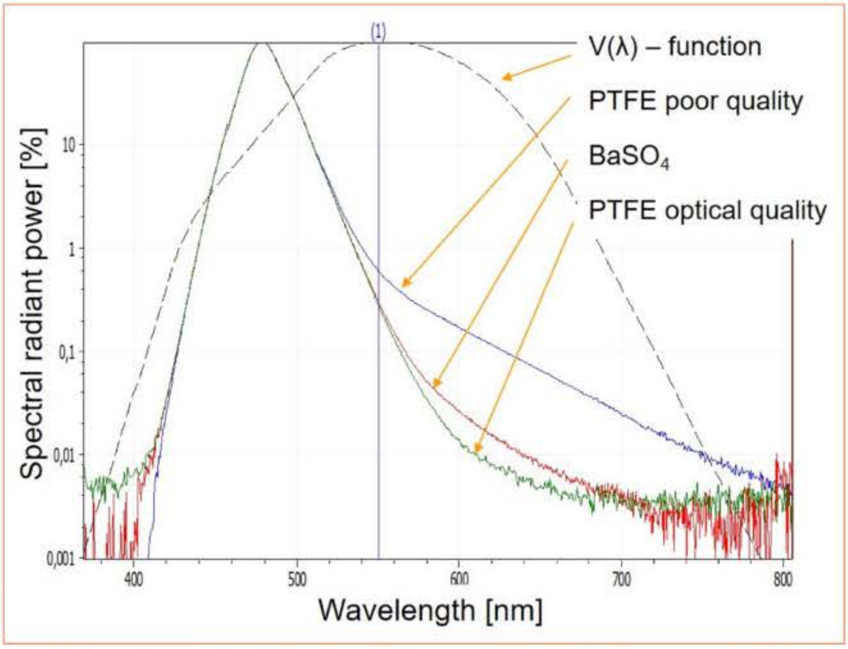

Figure 3: Blue LED measurements show higher fluorescence on the right flank for poor quality PTFE and lower fluorescence for the PTFE material with optical quality compared to barium sulfate ($BaSO_4$).

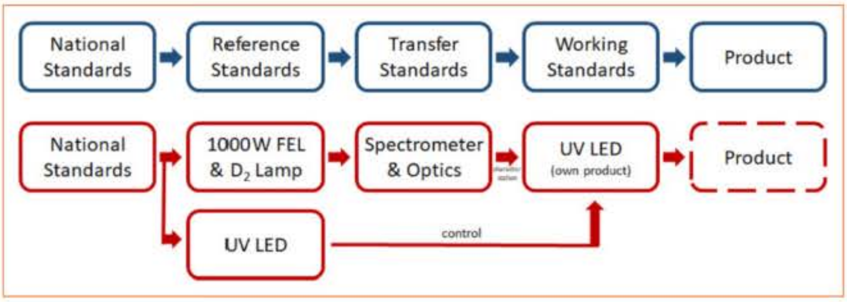

Figure 4: Calibration chain at national metrology institute (blue) and at Instrument Systems (red).

Calibration Chain

A defined calibration chain must be maintained for traceable spectral characterization and calibration, as shown in **Figure 4** top line (blue). National metrology institutes (NMIs) create the national standard of an SI unit, in this case the definition of the candela or a derived radiometric quantity. The standards of national institutes are compared globally on a regular basis. The NMIs create calibrated reference standards for interested companies or institutes that can use them to create calibrated transfer standards. The transfer standard is used to calibrate or characterize other light sources, the so-called working standards. These are then used for the factory calibration of measuring systems. Each of these steps brings a certain contribution of the measurement uncertainty into the budget.

Figure 4 lower line (red) shows the characterization chain at Instrument Systems. The NMI (e.g. Physikalisch-Technische Bundesanstalt in Germany) supplies a calibrated 1000 W FEL halogen lamp, a deuterium (D2) lamp and a UV LED. All standards are calibrated to irradiance E [W/m^2] at certain intervals. With the lamps FEL and D2, a spectroradiometer with the connected coupling optics is spectrally and absolutely calibrated and set as the transfer standard. This can be used to calibrate other light sources (e.g. UV LEDs) and define them as a new working standard. The third PTB-calibrated standard (UV-LED) is used as a control unit. This double control system guarantees the highest precision in the traceability of the standards, which in turn are used for further quality control and product characterization.

Realization of UV-LED Calibration Standards

The most important requirement for the UV-LED calibration standards is the audit of the radiant flux of UV measurement systems with integrating spheres and their characterization. At a certain distance, UV-LED standards can also be used to check the irradiance. So far, international metrology institutes such as the Physikalisch-Technische Bundesanstalt (PTB) and the National Institute for Standards and Technology (NIST) have not offered such a reference standard for the UV-B and UV-C spectral ranges.

As a result, Instrument Systems has developed its own UV calibration standards, namely the Advanced Calibration Standards of the ACS series (**Figure 5**, **Figure 6**). These are regulated and actively temperature-stabilized UV LEDs on a heat sink in an insulated housing. The UV-LED calibration standards were developed for the typical peak wavelengths of 280 nm (UV-C), 305 nm (UV-B) and 365 nm (UV-A). As for any type of calibration or characterization of light sources, essential requirements must be met: low drift of the wavelength, low drift of the optical power, stable mechanical interface and robustness against environmental influences. Internal qualification tests (e.g. long-term optical characterization, heat, humidity and mechanical tests) are required to ensure the quality of the sources. In addition to the light sources, measuring devices must also meet these requirements in order to achieve precise and reproducible results.

The traceability of the radiant flux of UV-LED calibration standards was achieved through a traceable factory calibration of the spectroradiometer with the coupling optics for irradiance and a subsequent integrative measurement with a goniospectroradiometer. The test object is rotated step by step in two mutually orthogonal angles: C in $[0, \Pi]$ and γ in $[-\Pi/2, \Pi/2]$. The measuring system measures an irradiance $E\,(r_0, C, \gamma)$ in [W/m^2] at a known distance r_0 at each specific angle. The radiation characteristics of the light source are recorded and the total radiant flux is determined through numerical integration of the measured irradiances at each angle. This procedure is accredited by the DAkkS (German accreditation body) according to ISO 17025 (D-PL-19052-01-00). Since goniopectroradiometric measurements take a little longer, the long-term stability of the optical characteristics is implemented in the basic requirements on the LED calibration standards (<0.2% in 12 h and <1% in 100 h).

A major aspect of the reliability of this system is the control point at $E\,(r_0, C = 0°, \gamma = 0°)$. Before a new UV LED is characterized, a PTB reference UV LED can be measured as a control unit. Its irradiance is precisely known and any major deviation would lead to an incorrect calibration of the transfer standard. Thus, a double check with differently calibrated standards is done.

Figure 5: Advanced Calibration Standard (ACS) on UV-LED basis.

Each measurement process contributes a measurement uncertainty to the total budget. The measurement uncertainty contributions of various influencing variables are based on statistics and are either determined by many measurements or simulated by the so-called Monte Carlo method. The overall measurement uncertainty of the three UV-LED calibration standards with different peak wavelengths results in these very low measurement uncertainty values ($k = 2$):

- UV-A (≈ 365 nm): 2.0%
- UV-B (≈ 305 nm): 3.5%
- UV-C (≈ 280 nm): 4.5%

Conclusions

Stray light corrected spectroradiometers with different coupling optics are the best suitable measurement systems for the entire UV range. When using PTFE integrating spheres, a low level of fluorescence should be ensured. Suitable self-absorption correction for the entire spectral range should be carried out with a combined deuterium / halogen lamp. UV-LED calibration standards can be used to check the radiant flux or the irradiance and, if necessary, for the absolute recalibration of the system.

The factory calibration of the UV standards, which is accredited according to ISO 17025, is a combination of several precision steps:

- All measurements are traceable to national standards.
- The light source itself is manufactured with the highest requirements in terms of optical properties, mechanical tests and thermal stability in order to guarantee a long calibration period.
- Spectral measurement setups (UV spectroradiometer, coupling optics and UV fiber bundles) are optimized in order to minimize the effects of fluorescence and stray light.
- The mechanical setup (goniospectroradiometer) enables not only the determination of the radiant flux of the test object, but also its radiation characteristics.
- When using a double test with separately calibrated light sources (both with devices that are traceable to national standards) a maximum of reliability and a minimum of uncertainty is achieved.

The very low measurement uncertainties ($k = 2$) of the UV-LED calibration standards are comparably low with those in the metrologically unproblematic visible range. Therefore, we are a global pioneer for radiant flux calibration in the UV-B and UV-C range. ■

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Denan Konjhodzic is a Product Manager at Instrument Systems GmbH, one of the world's leading manufacturers of solutions for spectral light measurement. He earned his doctorate at the Free University Berlin for his experimental work in the group "Optical Materials and Nanostructures" at the Max Planck Institute in Mülheim an der Ruhr, Germany. For many years he worked as an Application Engineer at Instrument Systems. His main topics nowadays are UV measurements, artifacts for calibrations in radiometry and photometry and solid state lighting measurements. He is a collaborator in the standardization bodies of CIE, DKE and DIN.

References

[1] Y. Zong S.W. Brown, B.C, Johnson, K.R. Lykke and Y. Ohno, "Simple spectral stray light correction method for array spectroradiometers", Applied Optics 45, 1111-1119 (2006).

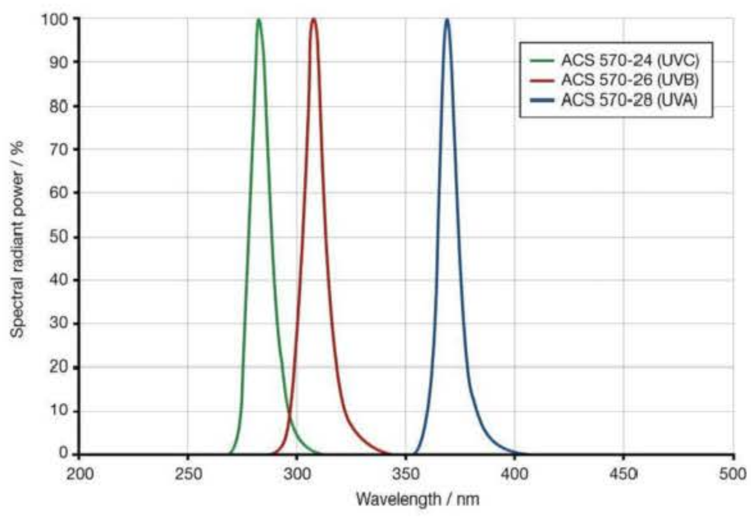

Figure 6: Typical spectra for UV-LED calibration standards.