Calibration and Standardization Issues of UV Sensors for Water Disinfection

Speaker: Thomas Larason Optical Technology Division, Physics Laboratory National Institute of Standards and Technology 100 Bureau Drive, Stop 8441 Gaithersburg, MD 20899-8441, USA Phone: 301-975-2334; Fax: 301-869-5700 Email: thomas.larason@nist.gov

Authors: Thomas Larason and Yoshi Ohno Optical Technology Division, Physics Laboratory National Institute of Standards and Technology, USA

Abstract

The National Institute of Standards and Technology, USA (NIST) studied the current ultraviolet (UV) water disinfection standards, ÖNORM M5873-1 and M5873-2 (Austria), and DVGW W294-3 (Germany), on the requirements for UV sensors for low-pressure mercury (LMP) and medium-pressure mercury (MPM) lamp systems. Pertinent to the study, NIST is measuring and analyzing the characteristics of various types of UV sensors. This information will aid in the development of new guidelines to address issues such as sensor requirements, calibration methods, uncertainty, and traceability.

The results of the NIST relative irradiance responsivity measurements of 10 sensors from several different commercial vendors are summarized. Practical problems were found in the calibration methods and evaluation of spectral responsivity requirements for sensors designed for MPM lamp systems. To solve the problems, NIST is proposing an alternate sensor calibration method for MPM lamp systems. A description is given of a future calibration service intended for low and medium-pressure mercury lamp systems used in water disinfection applications.

1. Current Requirements

Ultraviolet radiation effectively inactivates most bacterial pathogens, e.g., Cryptosporidium, Giardia, etc., commonly found in ground and surface waters. Water treatment facilities recently started using ultraviolet radiation for disinfection of drinking water, replacing standard chemical treatments. Typically, LPM and MPM lamps are used in the UV reactors at the facilities. In these reactors, water flows at a given rate past the UV lamps to receive an appropriate total UV dose. Sensors mounted on the wall of the reactor or inserted into the water flow monitor the dose level by measuring the UV irradiance from the lamps. The UV sensors currently in use have a variety of designs and performance characteristics. Austria and Germany have developed or are developing standards for the sensor design and performance. These two standards differ in their requirements and do not address many of the problems associated with the UV monitors. Furthermore, there are already many water plants in the U.S. employing UV sensor systems

consistent with one or the other standard. To resolve this confusion, American Water Works Association Research Foundation (AwwaRF) is developing new guidelines for UV monitors. NIST is participating in this project, with funding from AwwaRF, in collaboration with Carollo Engineers (Boise, ID), Camp Dresser and McKee (Denver, CO), and the University of Veterinary Medicine (Vienna, Austria).

The physical quantity to be measured is the microbicidal irradiance, defined as the total irradiance (W/m²) weighted by the microbicidal action spectrum $s_{\text{mik,rel}}(\lambda)$ (Fig. 1). According to ÖNORM M5873-1, M5873-2, and DVGW W294-3, UV sensors for both LPM and MPM systems are calibrated for irradiance responsivity against an LPM lamp. Since the value of $s_{\text{mik,rel}}(\lambda)$ is unity at 254 nm, and LPM lamps only have significant flux at 254 nm, the measured irradiance from an LPM lamp is equal to the microbicidal irradiance. Instruments can be calibrated for microbicidal irradiance responsivity using an LPM lamp, regardless of the sensor's spectral responsivity. This method works perfectly for LMP lamp systems. However, there is a problem for MPM lamp systems. The spectral output from MPM lamps differs significantly from LPM lamps. In addition, real UV sensors never have spectral responsivities perfectly matched to $s_{\text{mik rel}}(\lambda)$. In fact, many of the sensors used for MPM lamp systems have fairly large deviations from $s_{\text{mik,rel}}(\lambda)$. As a consequence of the differences between LPM and MPM spectral distributions and differences between the sensor spectral responsivities and $s_{\text{mik,rel}}(\lambda)$, measurement errors of the microbicidal irradiance occur. This source of measurement error, called a spectral mismatch error, is well known in other applications, e.g., photometry, where a detector's responsivity is tuned to match the action spectrum, $V(\lambda)$. Note that if a UV sensor had a spectral responsivity perfectly matched to $s_{\text{mik,rel}}(\lambda)$, there would be no problem, that is, the measured irradiance value would be equal to the microbicidal irradiance.



Figure 1. The microbicidal action spectrum, $s_{mik,rel}(\lambda)$; the spectrum of an LPM lamp and a MPM lamp; and the spectral responsivity of a UV sensor.

To ensure that such errors will not be significant, the ÖNORM and DVGW standards specify requirements for the relative spectral responsivity of sensors used for MPM lamp systems. DVGW W294-3 requires that a term $f_{1,z}$ be calculated from the relative spectral responsivity of the sensor, and the sensors must meet $f_{1,z} \le 0.25$ (reference sensors) or $f_{1,z} \le 0.40$ (duty sensors).

ÖNORM M5873-2 does not require the relative spectral responsivity of the sensors. However, it requires measurement with two specified cutoff filters and a MPM lamp that is to be calibrated with a spectroradiometer. The D value (relative difference between the microbicidal irradiance measured by the sensor and spectroradiometer) is calculated from these results. The sensors must meet D < 0.2 for both filters. The evaluation of relative spectral responsivity is critical but not easy in either standard. In addition, NIST found that many of the currently used commercial sensors do not meet these requirements. Even if they meet the requirements (reference sensors), errors can still be as much as ± 20 %.

2. NIST Relative Spectral Responsivity Measurement Results

NIST normally uses a monochromator-based spectral responsivity measurement facility (Spectral Comparator Facility (SCF) [1]) to calibrate photodiodes and other detectors. This facility covers the wavelength range from 200 nm to 1.8 μ m, and provides calibrations with expanded uncertainties of 1 % to 2 % (k = 2) in the UV region (200 nm to 400 nm). This system was designed for spectral power responsivity measurements. The output beam (≈ 1.5 mm diameter) underfills the detector surface. The facility also has the capability to measure a detector's irradiance responsivity. The absolute irradiance responsivity in V/(W/m²) or A/(W/m²) is measured by spatially scanning the beam across the detector entrance aperture in very small distance intervals using an X-Y stage [2]. In this fashion, NIST was able to measure the relative spectral irradiance responsivity of 10 sensors; results are shown in Fig. 2. This irradiance responsivity calibration, however, is a time-consuming measurement. In addition, the incident flux in this facility is fairly low, on the order of 1 μ W, while these sensors are designed for very high irradiance levels (up to 2000 W/m²). Some of the results did not seem to be reliable due to insufficient signal-to-noise ratio.

The relative spectral responsivity was also measured in another NIST facility capable of generating higher UV flux. This facility, the Spectral Irradiance and Radiance Responsivity Calibrations using Uniform Sources facility (SIRCUS) [3], can generate monochromatic beams with up to ~100 mW of power. This facility's wavelength coverage has recently been extended to the UV region to cover the 200 nm to 400 nm region using the frequency doubled, tripled and quadrupled output from pulsed Ti-Sapphire laser, which has a quasi-CW emission (pulses at very high frequency, 76 MHz). To measure the irradiance responsivity of the sensors, a frosted quartz diffuser plate was placed in front of the detectors to generate a quasi-uniform irradiance field at the detector reference plane. The sensors were placed at about 8 cm from the diffuser. The diffuser plate was a temporary set up to increase the irradiance levels for the measurement of these UV sensors. The responsivity was compared with a reference irradiance standard detector (precision aperture and silicon detectors arranged in a trap configuration). The irradiance levels ranged from approximately 2 W/m² to 20 W/m² at 254 nm, with a minimum of 0.8 % of full scale (for sensor #9).

The measurements with the SIRCUS facility were performed successfully for 8 of the sensors, but a part of the wavelength region (320 nm to 350 nm) was not measured due to a present limitation of the lasers at the facility. The results are shown in Fig. 2, where the relative spectral responsivity curves (normalized to the peak) are shown in comparison with the results from the SCF facility.



Figure 2. Relative spectral responsivities of the 10 sensors measured with the SCF facility. The data from SIRCUS facility are also plotted for comparison.

3. Discussion of Measurement Results

Measurements on the SCF were made at very low levels of the sensors' output signals. Even though the dark signal was measured and subtracted for all measurements, the noise was significant for some of the sensors. The signal-to-noise ratios of the data for these sensors were, therefore, very poor. The SCF results for some of the sensors (#4, #5, and #6) show noisy structure in the spectral responsivity and are not considered reliable. The SIRCUS data are more accurate than SCF data for these sensors. Particularly, sensor #4 shows significant discrepancy of the shape of the curve. This is possibly caused by part of the dark signal being truncated by the electronics, and thus the lower part of the curve measured by the SCF was cut off. Again, the signal level was very low (only 0.02 % of full scale), but this will not cause a problem for normal measurements.

The SIRCUS facility currently has a gap in a spectral region necessary to fully calibration the UV water sensors. In addition, the measured responsivities occasionally exhibited instabilities. The uncertainty for the SIRCUS measurements was estimated to be as large as ≈ 20 %, two orders of magnitude larger than the typical uncertainties in the SIRCUS facility (≈ 0.15 % (k = 2)). The increase in the SIRCUS uncertainty is considered to be primarily due to the occasional instability of the system, the non-uniformity of the beam coming from the frosted quartz diffuser plate, and a large uncertainty in the reference plane position of the sensors. The SIRCUS facility is being improved by solving these problems for UV irradiance responsivity calibrations. The SCF data seem to be more accurate for sensors #1, #2, #3, #7, and #9 (relative expanded uncertainty ≈ 5 % (k = 2) around the peak of each curve).

The preliminary results shown in Figure 2 indicate a large variation in the spectral responsivities of the commercial sensors. The spectral mismatch of these sensors (deviation of the relative spectral responsivity curve from the microbicidal action spectrum $s_{mik,rel}(\lambda)$) causes significant errors in the measured microbicidal irradiance as large as 160 % (excluding sensor #7 – designed for LMP lamp systems). Many of the sensors did not meet ÖNORM M5873-2 and DVGW W294-3 requirements for relative spectral sensitivities of the sensors.

4. Proposed Alternate Calibration Method

As reported above, the spectral mismatch errors of many of the sensors currently used are unacceptably large for the measurement of MPM lamp systems. Further, testing sensors for DVGW or ÖNORM requirements is not easy. To solve these problems with the MPM sensor calibration and bring a much higher accuracy in MPM lamp measurements, NIST is proposing an alternate calibration method. The root of the problem is that the MPM lamp has a very different (multi-line) spectrum than the LPM calibration lamp (a single emission line at 254 nm). The proposed method is to use a MPM lamp as a calibration source for the sensors used to measure MPM lamp systems. This approach is based on the well-established principle that errors are minimized in any measurement system when the standard and test sample are of the same type (strict substitution). In strict substitution, many of the measurement error components are cancelled out. The idea of using the substitution method for general UV sensors was previously proposed [4]. If the UV sensor is calibrated using an MPM lamp, and subsequently measures MPM lamps having the same spectral distributions, the error will be zero, theoretically, regardless of the spectral responsivity of the sensor. In real cases, there are variations in the spectra of MPM lamps, so the errors will not be zero, but errors will be significantly reduced even with sensors having a large deviation from $s_{\text{mik,rel}}(\lambda)$.

The actual calibration of sensors with this method can be done simply. It only requires a MPM lamp and a reference standard sensor calibrated for MPM microbicidal irradiance responsivity. Then, the UV sensor under test can be calibrated simply by comparison to the reference standard sensor under illumination by a MPM lamp. To ensure consistency in the calibration approach, a reference MPM lamp spectrum may need to be defined; ÖNORM M5873-2 already lists a nominal MPM lamp spectrum. A similar example exists in photometry with the CIE standard source, Illuminant A. The source has a standardized, defined spectral distribution, an incandescent source (lamp) approximating a blackbody at a temperature of 2856 K. All photometers are calibrated using a standard incandescent lamp having a spectrum close to the CIE standard Illuminant A. With this approach, photometric calibration results are universal [4].

5. Future Work

The spectral responsivity of the 10 sensors will be measured again by NIST after UV exposure testing by the Institute of Medical Physics and Biostatistics at the University of Veterinary Medicine, Vienna, Austria. Publication of the final NIST results, including sensor characterizations of linearity, absolute responsivity at 254 nm, temperature dependence, and angular responsivity, is planned for next year.

The development of a facility at NIST dedicated to the calibration of UV water disinfection sensors is planned. The facility will consist of a set of reference standard UV sensors (and/or a reference spectroradiometer), a set of stable LPM and MPM lamps, a variable attenuator that allows decadal changes in the irradiance level at the reference plane, a filter wheel to simulate typical spectral absorption by water, an automatic shutter to minimize exposure (and damage) of the sensors by high UV fluence, and a radiometric bench on where the distance between the test sensor and the lamp can be variably set, all of which will be enclosed in a light-tight housing.

With such a facility, reference standard UV sensors can be calibrated traceable to the national scale for microbicidal irradiance responsivity for the MPM lamp spectrum (as well as for LPM lamp spectrum) at the irradiance levels the sensors are used. The target calibration uncertainty using the proposed new method and facility is $\approx 5 \%$ (k = 2). This facility and calibration service will establish traceability of the UV sensors for the water treatment community.

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