Supplemental Information for:

Standardization of a UV LED Peak Wavelength, Emission Spectrum, and Irradiance Measurement and Comparison Protocol

*Kari Sholtes, Ryan Keliher, and Karl G. Linden**

**Corresponding author: Karl.Linden@colorado.edu*

Department of Civil, Environmental, and Architectural Engineering, University of Colorado Boulder, Boulder CO 80309 USA

Introduction

This supplemental information (SI) contains the standardized protocol for both spectrometric and radiometric measurements of UV LEDs. In an effort to make the protocol more practical, it contains quality control and quality assurance steps which serve as a checklist to ensure all steps and comparisons were completed. The standard protocol provides guidelines to ensure that measurements are valid, particularly determining the distances for measuring the emission spectra and irradiance. For example, the 15 and 20 cm distances used in the study informing the protocol were determined by ensuring the peak wavelength as measured with a spectrometer was less than 85% of the saturation value for all UV LEDs. These values initially corresponded with the conventional wisdom regarding irradiance measured at a distance, but the 15 cm distance was not sufficient to meet new standards for irradiance measurements. The protocol details that all irradiance measurement need to be taken at >7x the largest dimension of the UV LED array, but it is not prescriptive about what the numeric distances should be. Also contained in this SI are general instructions for how to use the protocol and suggested documentation.

Quality Assurance and Quality Control (QA/QC) Spectrometer and Radiometer Measurements

In order to establish QA/QC for radiometer and spectrometer measurements, the following steps are recommended for each type of equipment and associated measurement:

Spectrometer QA/QC

1) Verify that the spectrometer (with either spectrometric or spectroradiometric calibration) has been calibrated over the applicable range by a qualified or accredited third party or using a standard method within one year

2) Test a UV plasma source with a well-defined wavelength emission profile (i.e., 253.7 nm low pressure mercury arc) near the range of the target UV LED wavelengths to be tested

    a) Verify ±1 nm difference from NIST atomic spectra database (“NIST: Atomic Spectra Database Lines Form” n.d.) expected value

3) Follow the protocol found in the SI, Emission Spectrum and Irradiance Measurement Protocol for UV LEDs

4) Take all measurements in duplicate

    a) Ensure measurements are within the reported uncertainty due to the calibration range as indicated on the calibration certificate

5) Repeat all measurements at a minimum of two different heights

    a) Ensure repeated measurements are within the reported uncertainty based on the calibration range

6a) Compare spectrometer results to UV LED manufacturer or device developer’s source spectrum data

    a) Calculate the mean wavelength of the replicate samples and the manufacturer’s data

    b) Verify all measurements are ±1 nm difference from the mean

OR

6b) Test with secondary spectrometer and compare with primary results (annual)

    a) Calculate the mean wavelength of the replicate samples from both spectrometers

b) Verify all measurements are ±1 nm difference from the mean calculated above

Radiometer QA/QC

1) Ensure that the radiometer is calibrated over the ultraviolet range of interest (typically every 2 nm) to accurately characterize the target UV LED spectrum (as opposed to calibration at a single wavelength) and verify that the sensor has a linear response (meaning that the error is uniform) across the applicable wavelength range

2) Verify that the radiometer and detector have been calibrated over the applicable range by an accredited third party using standards traceable to a national laboratory such as the National Institute of Standards and Technology (NIST) within one year

3) Follow the protocol found in SI, Emission Spectrum and Irradiance Measurement Protocol for UV LEDs

4) Take all measurements in duplicate

    a) Ensure repeat measurements are within the reported error or uncertainty as published on the calibration certificate by the radiometer manufacturer

5) Repeat all measurements at a minimum of two different distances between the emission source and the sensor plane of calibration

    a) Compare the radiometric data for various heights using the calculated radiant intensity from the inverse square law:

Equation S1: $E_1 d_1^2 = E_2 d_2^2 = I$

where E is the irradiance (Watts/cm^2), d is the distance (cm), and I is the radiant intensity (Watts/sr).

b) Calculate the mean of the radiant intensity, I, for all matched radiometric samples (various heights and duplicates)

c) Calculate the percent error from the mean for each measurement

Equation S2: Percent error from the mean = $\frac{(\lambda_{experimental} - \lambda_{expected})}{\lambda_{expected}} * 100$

d) All radiant intensities should vary by the less than the error as reported on the calibration certificate or ±5% error from the mean (whichever is larger) across various heights and duplicates using the same equipment operated under the same electrical conditions

6a) If possible, compare radiometer results to UV LED manufacturer or device developer's irradiance data (when new or otherwise comparable)

a) Calculate the mean of the radiant intensity, I, for all radiometric samples (various heights and duplicates)

b) Verify that radiant intensities vary by ±10% error from the mean

OR

6b) Test with secondary radiometer and compare with primary results (when new or otherwise comparable)

a) Calculate the mean of the radiant intensity, I, for all radiometric samples (various heights and duplicates)

b) Verify that radiant intensities vary by ±10% error from the mean

UV LED Source Emission Spectrum and Irradiance Measurement Protocol

General Instructions

1) Perform protocol testing in an area where stray light < 400 nm can be minimized
2) Take photos of the setup for reporting purposes
3) Use proper Personal Protective Equipment
    a) Properly fitted UV protective glasses and gloves
    b) Avoid unprotected skin
    c) No access to work area for those not protected by goggles and proper clothing
4) Verify that all equipment is in good condition prior to starting the protocol
    a) Tampering with measurement equipment can void calibrations and negate all measurements
5) Verify that all equipment has been calibrated over the applicable range within one year
    a) Spectrometers need to be calibrated by a qualified or accredited third party or using a standard method
    b) Radiometers with a detector traceable to a national laboratory such as NIST needs to be calibrated over the applicable range by an accredited third party
6) Identify the UV-C LED emission plane

a) The emission plane is the chip itself if no lens is present

b) The emission plan is the face of the lens if a lens is present

c) Mark the UV-C LED emission plane for repeatable measurements (if applicable)

7) Measure and document the largest dimension of the UV LED source

a) If one UV LED, measure the largest diagonal dimension of the chip

b) If multiple UV LEDs or chips in an array, measure the largest diagonal distance across all UV LEDs

c) Calculate 7x this largest dimension of the UV LED (s), this distance will mark the closest measurement distances allowable for the radiometric measurements

Spectrometer Protocol Initial Documentation

1) Document the spectrometer manufacturer, name/type of spectrometer and sensor

a) Document the size and type of integrating sphere (if applicable)

2) Document the temperature in the facility and time/location of testing

3) Ensure spectrometer is calibrated to the proper wavelength range

a) UV: 200-400 nm is recommended to minimize uncertainty error associated with the calibration range

-OR-

UV/Vis: 200-1000 nm if required, but the larger wavelength range has a larger uncertainty associated with it

    b) Document the calibration type (spectroradiometric or spectrometric), date, and range
    c) Document the error associated with the calibration range (per manufacturer)

4) If a lens was present during calibration, verify that the lens is present, intact, and the calibration is valid
    a) Document the surface area of your sensor and the type of lens if applicable

5) Ensure usage of proper settings for integration time, nonlinearity correction, scans to average, etc., depending on the operation of your UV LEDs and your data collection and experimentation goals
    a) Read the spectrometer manual to understand operation and proper settings
    b) Call the spectrometer manufacturer to discuss testing goals and suggested input values for the integration time, corrections, etc., if needed
    c) Integration time can be increased to improve the signal to noise ratio by the square root of the integration time, but this will increase the total acquisition time
        i) Ensure no pixels are saturated (limit maximum saturation to 85% of saturation)
    d) Spatial averaging is not recommended for UV LED spectra due to the flattening of the peak
        i) Applicable with a spectrum that is relatively flat
    e) Scans to average can be increased to increase the signal to noise ratio
        i) Total acquisition time is a product of the scans to average times the integration time

   f) Boxcars (0-5) can be increased to increase the signal to noise ratio but it will also flatten the peaks

   g) Nonlinearity correction should be used to correct for the non-linear response to sensor or cosine correctors

   h) Signal to noise ratio simply refers to the true signal of a spectrum relative to the unwanted signal (from numerous sources) in a spectrum

   i) Document all settings with an explanation/justification of their use

6) Ensure access to current and valid digital calibration data if using a spectrometer with radiometric calibration before testing

Spectrometer Protocol Experimental Set-up and Measurements

1) Locate the sensor plane on the spectrometer sensor from which to take measurements

   a) Mark the sensor plane if necessary for repeatable measurements

2) Adjust distance from the UV-C LED emission plane and the sensor plane to the experimental distance (s)

   a) If using a spectrometer with a radiometric calibration, ensure that the following conditions are met:

      i) the sensor is normal to the beam of light emitted from the UV source

      ii) the sensor is located at the centerline of the UV source

        iii) the sensor is located at a distance of greater than 7x the largest dimension of the UV LED light source

   b) If using a spectrometer with only a spectrometric calibration, simply ensure that you are capturing your light source without saturating the spectrometer

   c) If using an integrating sphere, follow integrating sphere instructions for set-up

3) Plug in and turn on the spectrometer and any associated equipment (i.e. laptops, etc.)

4) Store dark spectrum data according to manufacturer’s instructions

   a) Typically, the cap will be kept on for this reading then can be removed for UV measurements

5) Ensure that minimal measurable light < 400 nm is present in the vicinity (with the UV source OFF)

6) Take one ambient/background reading with the UV source OFF in the experimental set-up

   a) Save these data

7) Ensure PPE are adjusted properly

   a) UV protective goggles fit properly

   b) Skin which could be in contact with UV light is covered

   c) No access to work area for those not protected by goggles and proper clothing

8) Turn UV source ON

a) Collect UV spectrum at 5 and 10 minutes

b) Save data

9) Turn UV source OFF

10) Analyze the spectral data for the 5 and 10 minute time points:

a) Visualize the data and remove any negative values or anomalous data

b) Identify and document the maximum value of the spectrum

i) If data are noisy near the peak, a data smoothing technique may be required (i.e., splining)

c) Normalize all values to the peak value

d) Identify the thresholds on either tail where the curve nears approximately 0.5% of the value of the peak; this will serve as the data analysis cut off point (see Figure SI 1).

i) If the curve does not approach zero on both sides of the peak, first analyze the ambient/background light.

(1) Subtract the ambient/background light from the raw curve data to see if the curve approaches zero

(2) Try zeroing the device again or call the spectrometer manufacturer for troubleshooting

e) Find the area under the curve between the two 0.5% peak value thresholds of each tail

f) Document the wavelength range of curve between the 0.5% peak value thresholds

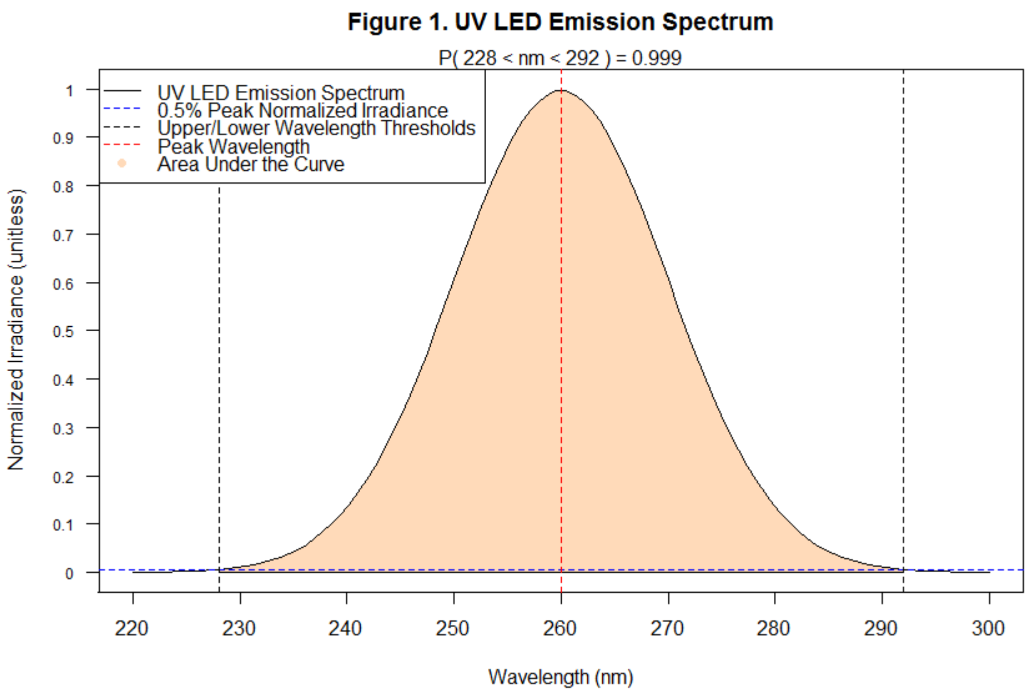

Figure **Error! No sequence specified.**: Normalized Emission Spectrum Area under the Curve (AUC) Quantification with Thresholds

g) Compare the visualized data, the peak, the range, and the area under the curve between the 5 and 10 min time points.

   i) Calculate the mean of the peak wavelength and the area under the curve

   ii) Calculate the percent error from the mean for the peak and the area under the curve for each measurement (Equation S2)

h) Verify the peak wavelengths vary by less than the reported spectrometer uncertainty (this is a function of the calibration range)

i) If radiometrically calibrated: verify that the areas under the curve at the same height are within ±5% error from the mean, if applicable

    j) If radiometrically calibrated: verify that the normalized areas under the curve are within ±1% error from the mean

11) Adjust the UV emission plane and the spectrometer sensor plane to the second experimental distance

    a) If there is no second experimental distance planned, simply develop an arbitrary one for QA/QC purposes

    b) If using an integrating sphere, simply run the experiment again after removing all sources from the integrating sphere for duplication purposes

    c) If using a spectrometer with a radiometric calibration, ensure that the following conditions are met:

        i) the sensor is normal to the beam of light emitted from the UV source

        ii) the sensor is located at the centerline of the UV source

        iii) the sensor is located at a distance of greater than 7x the largest dimension of the UV LED light source

    d) If using a spectrometer with only a spectrometric calibration, simply ensure that you are capturing your light source without saturating the spectrometer

12) Ensure that minimal measurable light < 400 nm is present in the vicinity (with the UV source OFF)

13) Take one ambient/background reading with the UV source OFF in the experimental set-up

a) Save these data

14) Ensure PPE are adjusted properly

a) UV protective goggles fit properly

b) Skin is covered which could be in contact with UV light

c) No access to work area for those not protected by goggles and proper clothing

15) Turn UV source ON

a) Collect UV spectrum at 5 or 10 minutes

b) Save data

16) Turn UV source OFF

17) Replace the cap to protect the sensor on the spectrometer

18) Repeat all of the above steps again

a) Save files as Trial 2 of each test

19) Analyze and compare the spectral data as described in Step 10 above

20) Verify that the peak wavelengths vary by less than the reported spectrometer uncertainty

21) If radiometrically calibrated: verify that the areas under the curve at the same height are within ±5% error from the mean, if applicable

22) If radiometrically calibrated: verify the normalized areas under the curve at all heights are within ±1% error from the mean, if applicable

23) Compare these data to either the manufacturer's data or to the same analysis using a different spectrometer

   a) Verify all peak wavelength data are within ±1 nm of the mean (even when produced by different spectrometers at different heights)

   b) If radiometrically calibrated: verify that the area under the curve for each height are within 10% error from the mean and from radiometer irradiance data at the same height, if applicable

   c) If radiometrically calibrated: verify all normalized areas under the curve are within ±5% error from the mean, if applicable

Radiometer Protocol Initial Documentation

1) Document the radiometer manufacturer, name/type of radiometer and sensor

2) Document the temperature in the facility and time/location of testing

3) Ensure radiometer and sensor is calibrated to the proper wavelength range and within calibration date by an accredited third party

   a) Document the calibration date and range

   b) Document the error associated with the calibration range

4) Avoid direct irradiation of the radiometer sensor entrance slit with either an integrating sphere or diffuser.

5) Ensure integrating sphere or diffuser lens is present (if applicable), intact, and the calibration is valid (i.e., that the sensor lens has not been removed or damaged)
    a) Document the surface area of sensor and the type of lens/diffusor (if applicable)
    b) Document the size and type of integrating sphere (if applicable)
6) Ensure usage of proper radiometer and sensor depending on the operation of your UV LEDs and your data collection and experimentation goals
    a) Call the radiometer manufacturer to discuss testing goals if needed
7) Ensure access to current and valid digital calibration data before testing

Radiometer Protocol Experimental Set-up and Measurements

1) Locate the sensor plane on the radiometer sensor from which to take your measurements
    a) This information is likely available in the manual
    b) Mark the sensor plane if necessary for repeatable measurements
2) Adjust and document the distance from the UV-C LED emission plane and the sensor plane to the experimental distance (s)
    a) Ensure that the sensor is normal to the beam of light emitted from the UV source
    b) Ensure that the radiometer sensor is located at the centerline of the UV source
    c) Ensure that the sensor plane is located at a distance of greater than 7x the largest dimension of the UV LED light source
    d) Or follow the integrating sphere instructions (as applicable)

3) Plug in and turn on the radiometer and any associated equipment (i.e. laptops, etc.)

4) Ensure that minimal measurable light < 400 nm is present in the vicinity (with the UV source OFF)

   a) Measurable light < 400 nm ideally should be less than 0.1% of the irradiance measurement at the peak wavelength

5) Use the average peak wavelength for the applicable UV-C LED (s) from the spectrometer analysis for all radiometric analysis

   a) Determine the calibration factor associated with the peak wavelength

   b) Apply that calibration factor to all applicable radiometric measurements – refer to the radiometer manual

6) Zero the device with a cover over the detector once the calibration factor is set

7) Take one ambient/background reading with the UV source OFF in the experimental set-up

   a) Save these data

8) Ensure PPE are adjusted properly

   a) UV protective goggles fit properly

   b) Skin which could be in contact with UV light is covered

   c) No access to work area for those not protected by goggles and proper clothing

9) Turn the radiometer data logger on (if applicable)

10) Turn UV source ON

   a) Collect radiometric data continually with a data logger or every 30 seconds until steady state has been achieved or the irradiance is changing by less than 1% over the time horizon of the planned experiments (if applicable)

   b) Record the time frame to achieve steady state

   c) Continue to take readings for another 5 minutes (in the case of a data logger) or take another reading in 5 minutes

   d) Save or record data

11) Turn UV source OFF

12) Calculate the error from the mean for the steady state experiments

   a) Verify that the steady state measurements are all within the radiometer uncertainty (if published) or ±1% error from the mean

13) Adjust and document the second experimental distance from the UV emission plane to the radiometer sensor plane

   a) If there is no second experimental distance, simply develop an arbitrary one for QA/QC purposes

   b) If using an integrating sphere, simply run the experiment again after removing and replacing the UV source

   c) Ensure that the sensor is normal to the beam of light emitted from the UV source

   d) Ensure that the radiometer sensor is located at the centerline of the UV source
   e) Ensure that the sensor plane is located at a distance of greater than 7x the largest dimension of the UV LED light source

14) Ensure PPE are adjusted properly
   a) UV protective goggles fit properly
   b) Skin which could be in contact with UV light is covered
   c) No access to work area for those not protected by goggles and proper clothing

15) Turn the radiometer data logger on (if applicable)

16) Turn UV source ON
   a) Collect radiometric data until steady state has been achieved or the irradiance is changing by less than 1% over the time horizon the planned experiments (if applicable)
   b) Record the time frame to achieve steady state
   c) Continue to take readings for another 5 minutes (in the case of a data logger) or take another reading in 5 minutes
   d) Save or record data

17) Turn UV source OFF

18) Replace the cap to protect the diffuser lens on the radiometer

19) Compare the radiometric data for various heights using the calculated radiant intensity from the inverse square law:

   a) The inverse square law of electromagnetic radiation explains the relationship between a source and measurement distance. Irradiance is proportional to the inverse of the squared distance (d in cm): $E = \frac{I}{d^2}$ where E is the irradiance (Watts/cm^2) and I is the radiant intensity (Watts/sr). In measuring the irradiance at two distances, the equation can be manipulated to Equation S1

   b) Calculate the mean of the radiant intensity, I, for all radiometric samples for each UV LED

   c) Calculate the percent error from the mean for each measurement

   d) The radiant intensity should vary by ±5% error from the mean across various heights and duplicates

   e) If data are not comparable

      i) Ensure no ambient/background irradiance

      ii) Retest with enhanced attention to the time required to achieve steady-state emission, measurement distances, testing along the centerlines of the UV LEDs and sensor, and ensuring that the sensor face is parallel to the face of the UV LEDs

      iii) Consider irregular angular emission and perhaps increase the distance between emission and sensor planes

20) Determine the average time to achieve steady state for these UV-C LEDs, and use this for future radiometric and/or spectrometric experiments

a) If this time to achieve steady state is significantly different from the time-frames tested in the spectrometer measurement section, go back to re-test the spectrometer to ensure that the emission spectrum does not change over time (especially if using a spectrometer with a radiometric calibration)

21) Repeat all of the above steps again for the pre-determined steady-state times only

a) Save files as Trial 2 of each test

22) Analyze and compare the irradiance data as described above

a) Ensure the steady-state irradiance of all duplicate UV-C LED data (same heights, same UV-C LEDs, same radiometer) are within the reported radiometer uncertainty or within ±1% distance from the mean

b) Ensure the steady-state irradiance of the same UV-C LEDs over various heights (but the same radiometer) are comparable: the radiant intensity (irradiance times the squared distance from the source emission face to the sensor plane) should vary by ±5% error from the mean across various heights and duplicates

23) Compare these data to either the manufacturer's data or to the same analysis using different radiometric or spectroradiometric data

a) Find the radiant intensity for all irradiance data

b) Average the radiant intensity for each UV-C LED at both (all) experimental heights and ensure that all measurements are within ±10% error from the mean

QA/QC Verification Summary

Spectrometer

- Data for one spectrometer, various heights and UV LED operation times
    - Verify that the average peak wavelengths vary by less than the reported spectrometer uncertainty
    - If radiometrically calibrated: verify that the areas under the curve at the same height are within ±5% error from the mean, if applicable
    - If radiometrically calibrated, verify that all normalized emission spectrum area under the curve varies by ±1% error from the mean
- Data for more than one spectrometer, various heights and UV LED operation times
    - Verify that the average peak wavelengths vary by ±1 nm from the average peak wavelength
    - If radiometrically calibrated: verify that the areas under the curve at the same height are within ±10% error from the mean, if applicable
    - If radiometrically calibrated: verify that the normalized emission spectrum area under the curves vary by ±5% error from the mean (can compare different heights together with normalized AUC)

Radiometer

- Data for one radiometer, one height, two steady-state UV LED operation times

    - Verify that the steady state irradiances vary by less than the reported radiometer uncertainty or ±1% error from the mean if no uncertainty or error is reported by the manufacturer

- Data for one radiometer, various heights and steady-state UV LED operation times
    - Verify that the radiant intensities vary by ±5% error from the mean
- Data for more than one radiometer (or spectrometer with radiometric calibration), various heights and steady-state UV LED operation times
    - Verify that the radiant intensities for all heights vary by ±10% error from the mean