Using a Tunable Ultraviolet Laser for the Inactivation of Water Pathogens

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Abstract

Ultraviolet (UV) radiation effectively inactivates common pathogens found in ground and surface waters such as *Cryptosporidium*, *Giardia*, and most bacterial pathogens (e.g. *E. coli*). Water treatment facilities are now using UV radiation for disinfection of drinking water, supplementing standard chemical treatment. This paper will give an overview of the National Institute of Standards and Technology's (NIST) involvement in research concerning the disinfection of drinking water with UV light. A measurement method with improved accuracy and simplified spectral analysis for water pathogen inactivation using narrowband UV (laser) light over broadband UV sources and bandpass filters will be presented.

A transportable tunable UV laser system for providing a known irradiance ($\mu W/cm^2$) or dose (mJ/cm^2) suitable for irradiating water samples in Petri dishes over the wavelength range of 210 nm to 300 nm will be described. This is not the first time NIST has been involved with the UV disinfection of drinking water. Previously, NIST participated in research to calibrate and characterize UV sensors used in water disinfection facilities.

A tunable laser provides advantages over the traditional use of bandpass filters with spectrally broad UV sources. The most apparent advantage of the laser is it provides light at a single wavelength that is fully tunable, allowing any wavelength to be selected, and not relying on the particular bandpass filters available. This also reduces the complexity of weighting the spectral source with the filter bandpass function. Another advantage of a laser is that it can, in theory, produce higher power levels than a traditional laboratory UV source, especially below 250 nm. In practice, a tunable UV laser is more complex than traditional UV sources and requires certain design considerations to provide the desired spectral purity and spatial uniformity.

The NIST tunable UV laser irradiance facility design and key components will be discussed, including modifications in the field to provide the required irradiance levels. The unique challenges encountered in deploying a tunable laser to a "remote" laboratory location are discussed. For example, the extensive use of the laser at wavelengths of interest led to a degradation of the laser output power due to damage to the laser's internal optics. This damage was unavoidable and led to unanticipated maintenance during the project.

The NIST standard detector used for this project and its calibration will be described. Measurements to characterize the spectral and spatial qualities of the UV beam will be presented along with sample results. Finally, the irradiance ($\mu W/cm^2$) and dose (mJ/cm^2) levels produced by the tunable UV laser during the project will be presented.

The results demonstrated that the NIST transportable laser system provided unique capabilities for use with water pathogens and has potential application for other biological experiments as

well. Future plans include additional automation, improved optics, and implementation of a different detector measurement method that facilitates the use of higher irradiance levels.

Previous NIST Research with UV Water Disinfection

NIST participated in a project with the American Water Works Association Research Foundation (AwwaRF) to develop new guidelines for ultraviolet (UV) sensor characteristics to monitor the performance of UV water disinfection plants [1]. Photographs of several of the UV sensors measured are shown in Fig. 1.

NIST tested several UV sensors (reference and duty sensors) used to monitor UV reaction chambers in water treatment facilities for the following characteristics:

- Absolute irradiance calibration at 254 nm
- Relative spectral responsivity, 200 nm to 400 nm
- Linearity of response
- Temperature dependence
- Angular responsivity

The relative spectral responsivity for six of the UV sensors measured is shown in Fig. 2 along with the microbicidal action spectrum, $s_{\mathrm{mik,rel}}(\lambda)$. Some issues were identified concerning the absolute calibration of these UV sensors. One important issue is that the large differences in spectral responsivity mean that errors can occur with medium-pressure (MP) lamp measurements. The results were published in "Design and Performance Guidelines for UV Sensor Systems" available from the Water Research Foundation [2].

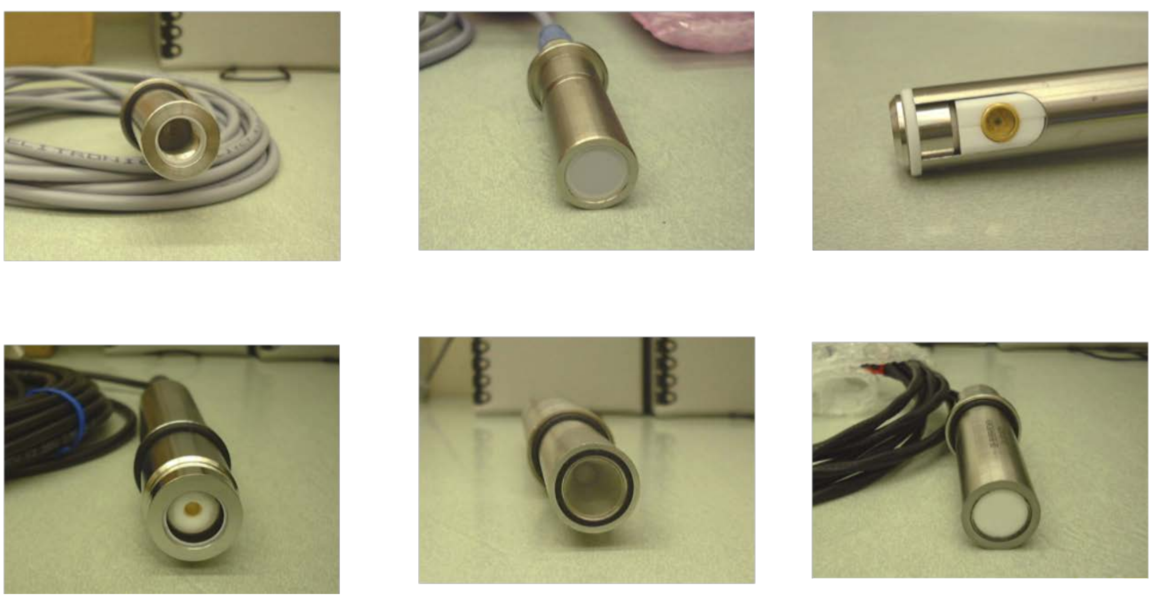


Figure 1. Photos of example UV sensors used in water disinfection.

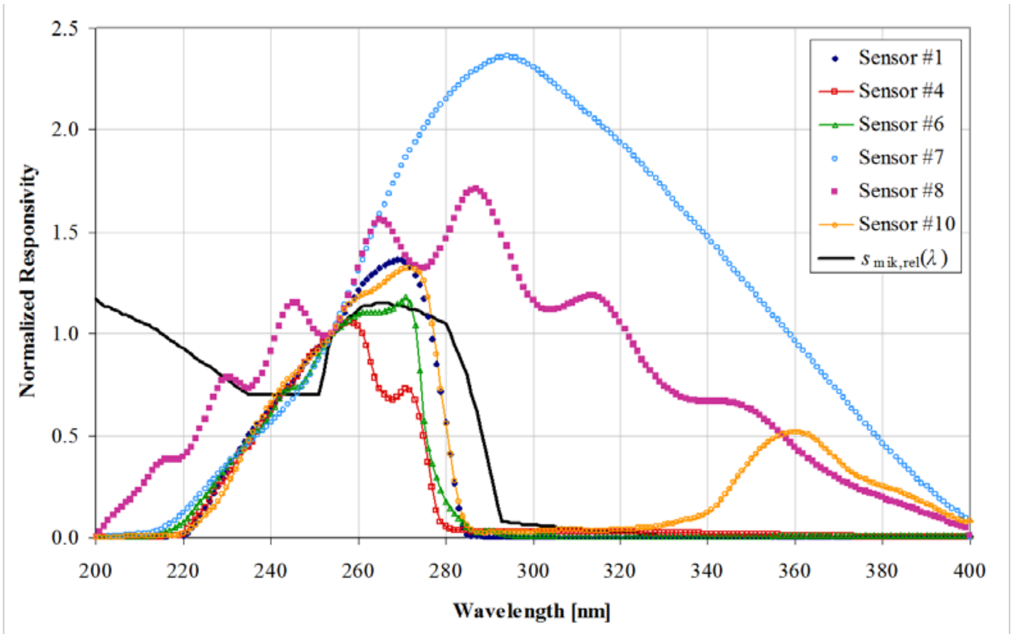


Figure 2. Plot of the relative spectral responsivities of six of the UV sensors measured. Also shown is the microbicidal action spectrum, $s_{\mathrm{mik,rel}}(\lambda)$.

NIST Tunable UV Laser Irradiance Facility

NIST has developed tunable laser facilities for research and calibration over the past decade [3], [4]. More recently, a transportable tunable laser system which covers the UV to near IR spectral range has been demonstrated [5]. In 2011, NIST was asked if they could provide the UV irradiation of water samples as part of a Water Research Foundation Project [6]. This led to the development of the transportable tunable UV laser system described here for providing a known irradiance (μW/cm^2) or dose (mJ/cm^2) to water samples in Petri dishes over the wavelength range of 210 nm to 300 nm.

A tunable laser provides advantages over the traditional use of bandpass filters with spectrally broad UV sources. The most apparent advantage of the laser is that it provides light at a single wavelength that is fully tunable, allowing any wavelength to be selected, and not relying on the particular bandpass filters available. This also reduces the complexity of weighting the spectral source with the filter bandpass function. Another advantage of a laser is that it can, in theory, produce higher power levels than a traditional laboratory UV source, especially below 250 nm. In practice, a tunable UV laser is more complex than traditional UV sources and requires certain design considerations to provide the desired spectral purity and spatial uniformity.

The fundamental component of the NIST tunable UV irradiance laser system is an EKSPLA [7] NT242-SH/SFG 1 kHz pulsed laser[1], tunable over 210 nm to 2600 nm. The optical layout of the EKSPLA NT242 laser is shown in Fig. 3.

[1] Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

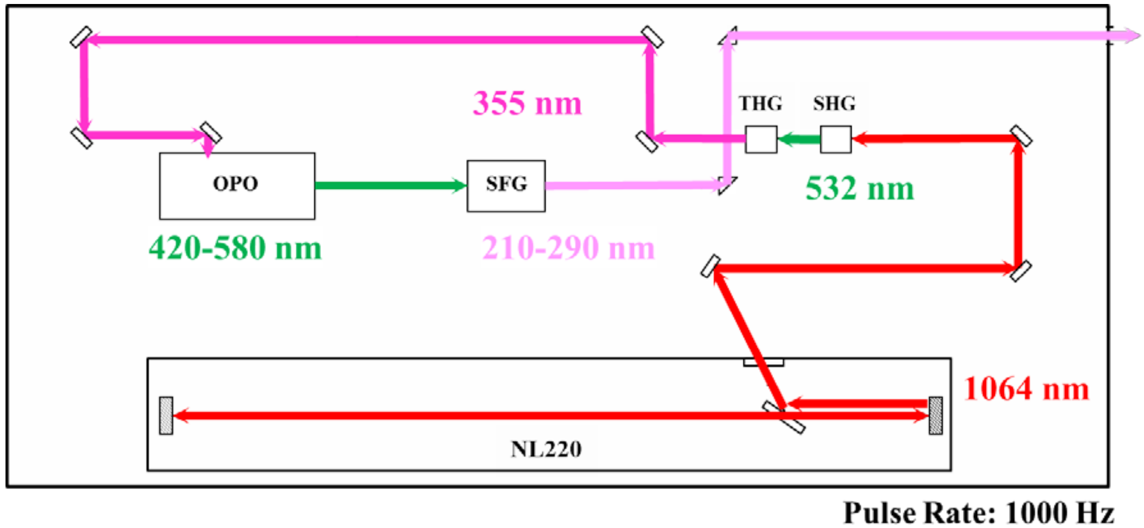


Figure 3. Schematic diagram showing the optical layout of the EKSPLA NT242 tunable laser. See Ref [8] for detailed information about the optical components.

The NIST tunable UV irradiance laser system optical configuration is shown in Fig. 4. Once the laser beam leaves the EKSPLA laser it enters a light-tight enclosure through a computer controlled shutter that determines the dose to the water samples. The beam is then reflected off two dielectric mirrors which filter out visible light co-aligned with the laser beam when the laser is set to wavelengths shorter than 300 nm. The beam then travels through a beam splitter and an etched fused silica diffuser [9]. The beam splitter sends a small portion of the UV light to a silicon photodiode which was used monitor the irradiance level during sample exposure. The diffuser was another critical component of the laser system. Unlike typical optical diffusers this diffuser is specifically engineered to modify the laser beam from a collimated oval shape (1.5 mm by 10 mm) to a uniform diverging beam (10° half-angle) to irradiate the water samples. Neutral density filters could be added to the optical path between the shutter and first mirror to reduce the irradiance level at the water sample. The UV standard detector is placed at the water sample location to measure the irradiance at each wavelength of interest.

A dielectric mirror is made of multiple thin layers of dielectric material deposited on an optical substrate. By designing the type and thickness of the layers a specific spectral band of light can be reflected. The dielectric mirrors, labeled M_1 and M_2 in Fig. 4, were highly reflective from 240 nm to 300 nm. For 210 nm to 230 nm the dielectric mirrors were replaced by aluminum mirrors and a fused silica prism and slit were added to filter out the visible light. This configuration is shown in Fig. 5. A second set of dielectric mirrors were procured partway through the project that worked well for 220 nm and 230 nm, but the prism and slit were still required at 210 nm due to the low reflectance from the dielectric mirrors.

The spectral filtering of the laser beam by the dielectric mirrors or prism and slit was required because of the visible light co-aligned with the laser beam but at double the selected UV laser wavelength (see for example the 420 nm peak in Fig. 6). An Instrument Systems [10] CAS 140CT array spectrometer was used to measure the laser system spectra at the water sample position from 200 nm to 600 nm. Fig. 6 shows the calibrated counts normalized to the peak wavelength for the laser system spectra at each of the UV wavelengths of interest confirming the visible light in the beam was reduced to an acceptable level.

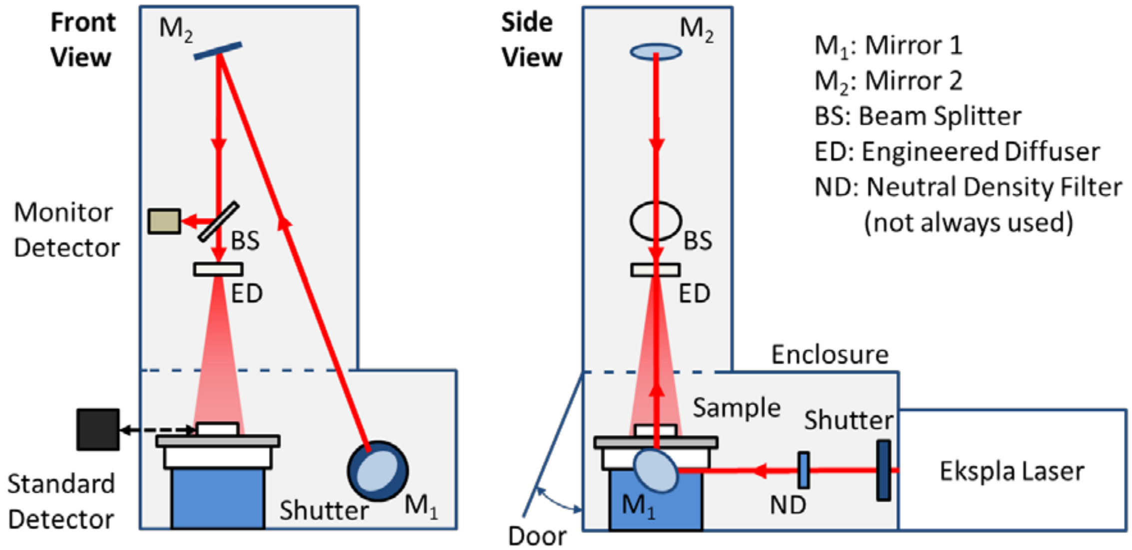


Figure 4. The NIST tunable UV irradiance laser system optical configuration. The Standard Detector is substituted for the Sample to measure the irradiance at each wavelength of interest.

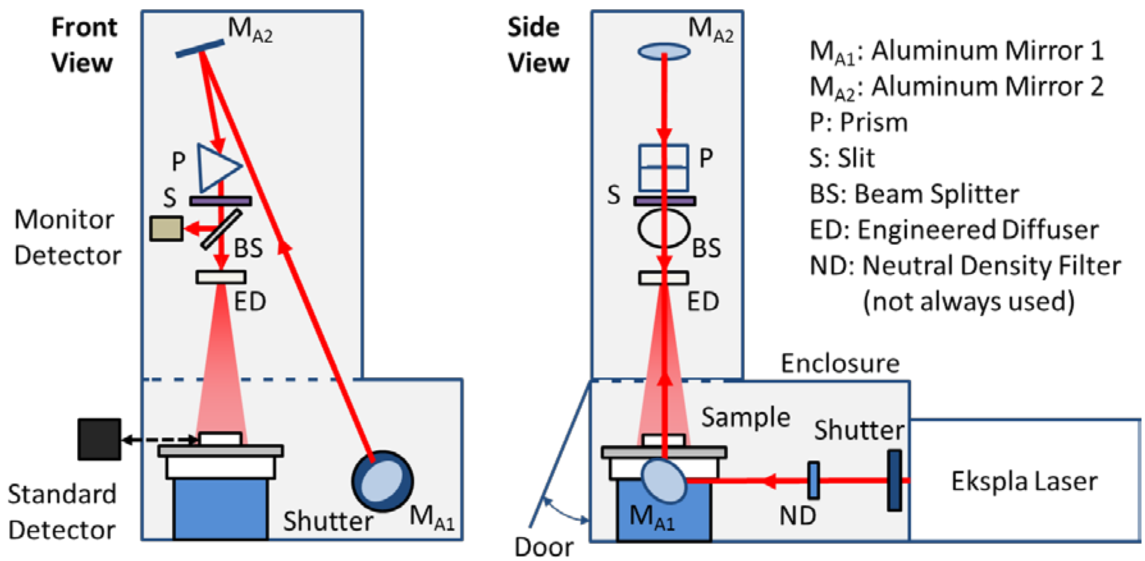


Figure 5. The NIST tunable UV irradiance laser system optical configuration for 210 nm to 230 nm. The dielectric mirrors are replaced by aluminum mirrors and a prism and slit are added.

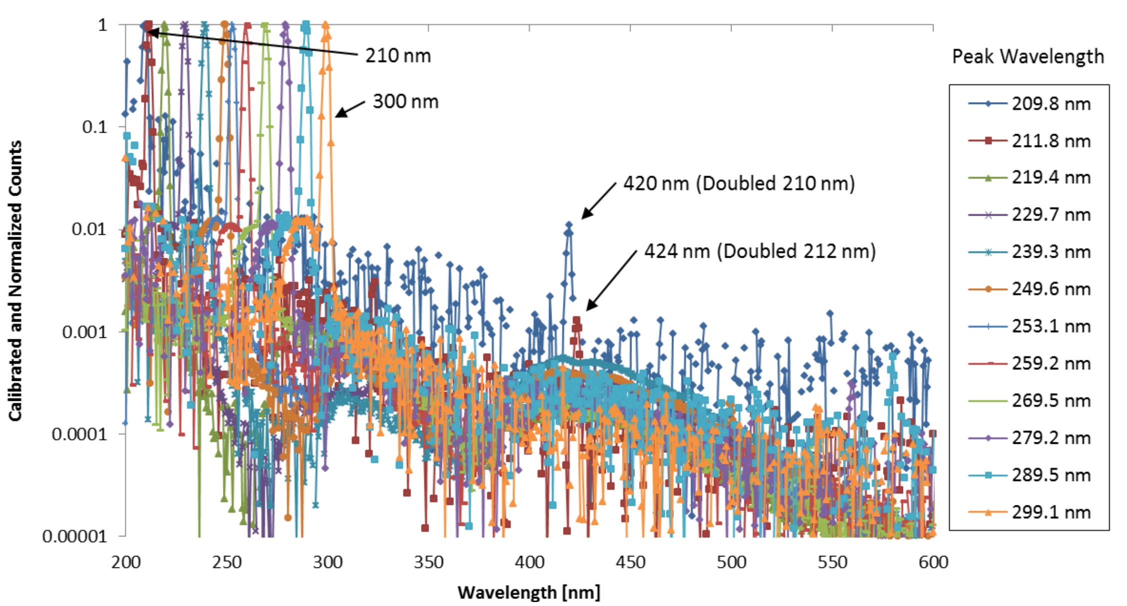
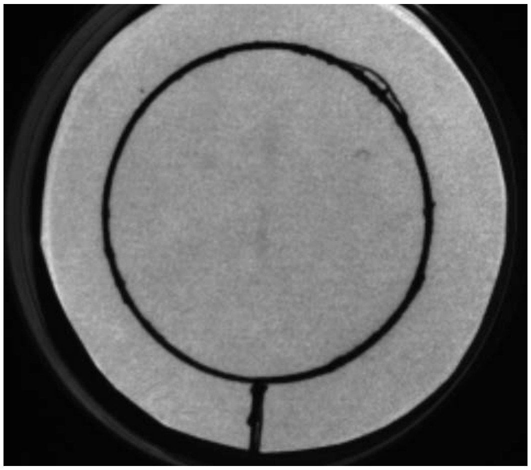


Figure 6. Plot of the calibrated counts normalized to the peak wavelength for the laser system spectra at the water sample position from 200 nm to 600 nm for each of the UV wavelengths of interest. The visible light has been reduced to an acceptable level.

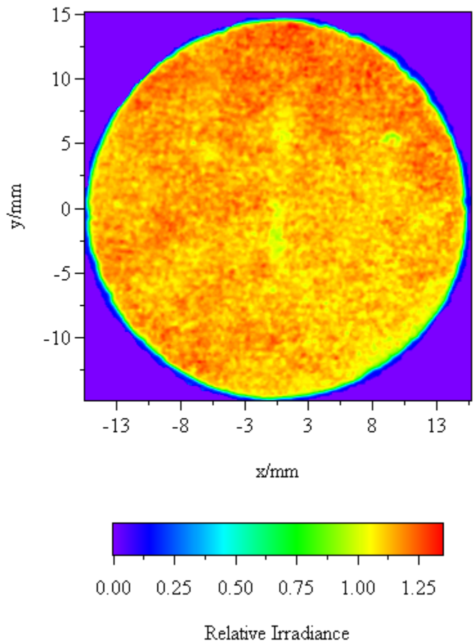
The NIST tunable UV irradiance laser system described above was not the first design. The first configuration deployed to the field was a more complicated design where the visible light was removed first by a prism after the shutter then the UV light was reflected off an aluminum mirror through a focusing lens and into a pure silica core multimode fiber optic cable. The light exiting the fiber optic cable then passed through the engineered diffuser and was imaged onto the water sample with a lens. This design was not practical in the field because of the time required to manually change the wavelength with the aluminum mirror and increasing losses in the fiber optic cable due to “solarization” (UV damage). The design described above was derived to simplify the optics to be aligned and reduce the number of optical components contributing to the power losses in the UV.

The UV standard detector consisted of an International Radiation Detectors (now Opto Diode Corp.) SXUV100 silicon photodiode, known to be stable with UV exposure [11], and a precision 8 mm diameter electroformed aperture in a cylindrical aluminum housing. The photodiode output was measured with a Keithley 6517 electrometer. The spectral irradiance responsivity [A/(μW/cm^2)] of the UV standard detector was calibrated at NIST in the UV Spectral Responsivity Facility [12].

The uniformity of the UV irradiance at the water sample was measured separately by the project collaborators who calculated the ratio of the average of the incident irradiance over the area of the Petri dish to the irradiance at the center of the dish, or Petri Factor [13], at each wavelength before exposing any water samples. But a more real time test, a method using digital images and the fluorescence from typical card stock paper, was explored by NIST. Time constraints kept this from being studied further during this project. An example of a digital image from a camera mounted above the water sample and irradiance uniformity normalized to the beam center at 253.7 nm is shown in Fig. 7.



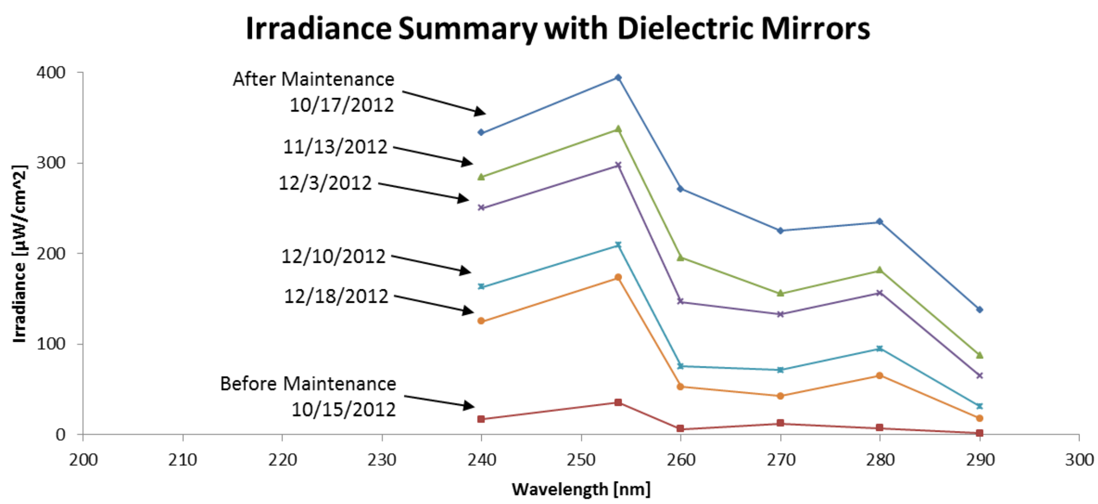
(a)



(b)

Figure 7. Example of the irradiance uniformity at 253.7 nm. A photograph (a) from the camera mounted above the water sample imaging the fluorescence from typical card stock paper. The dark circle marks the area of the water sample Petri dish. A plot (b) of the relative irradiance uniformity normalized to the beam center.

The irradiance levels consistently decreased over time due to unavoidable UV damage to some of the optical components in the EKSPLA laser. Midway through the project some of the damaged EKSPLA laser optical components were replaced which increased the irradiance levels at the UV wavelengths of interest. But the UV damage continued to decrease the irradiance levels that could be provided. This did affect the scheduling of which microbes where exposed and the sequential order of wavelengths. Fig. 8 shows the decrease in irradiance ($\mu W/cm^2$) over time and the improvements when some of the damaged EKSPLA laser optical components were replaced. The range of fluence or dose (mJ/cm^2) by wavelength used during this project is shown in Fig. 9.



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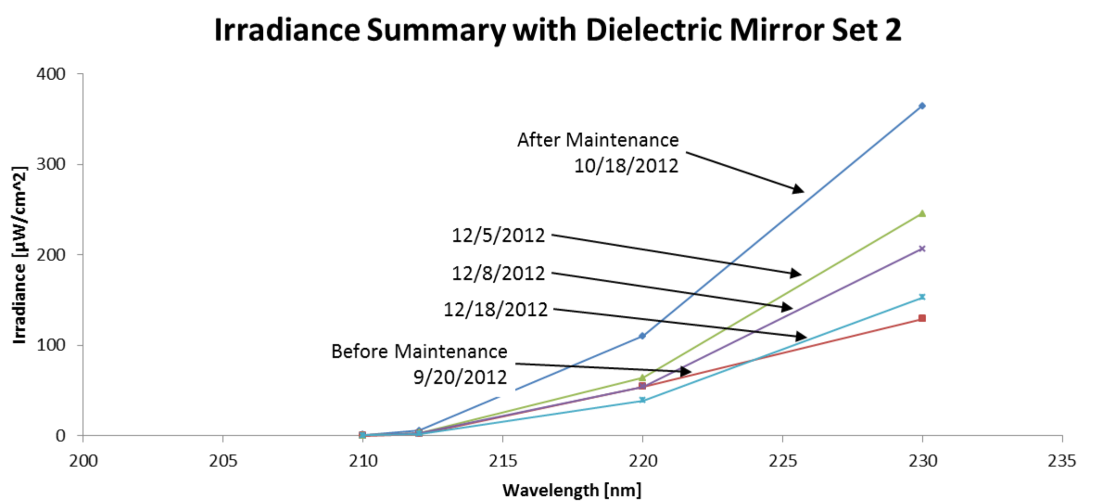

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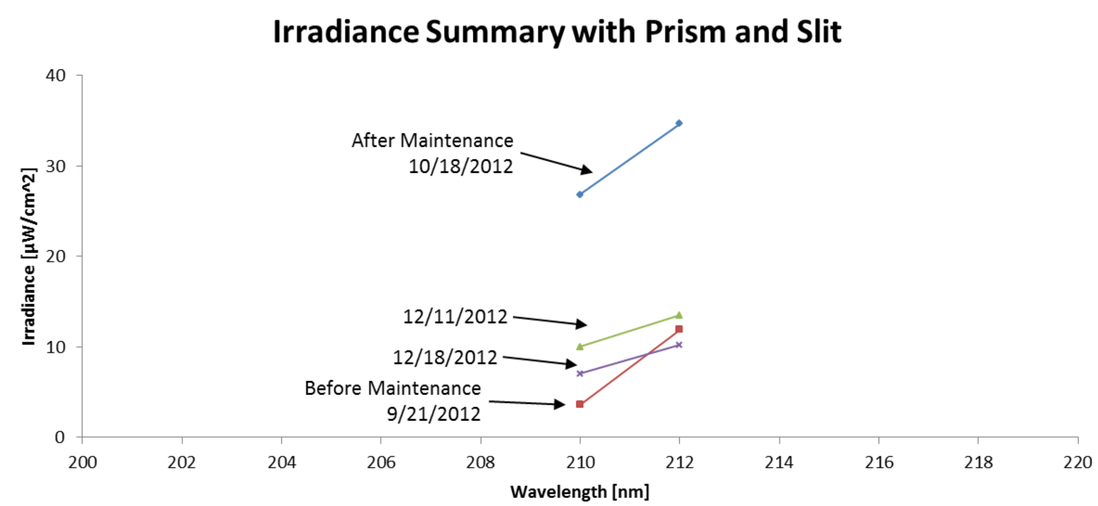

(c)

Figure 8. Graphs showing the decrease in irradiance (µW/cm^2) over time and the improvement when some of the damaged EKSPLA laser optical components were replaced.

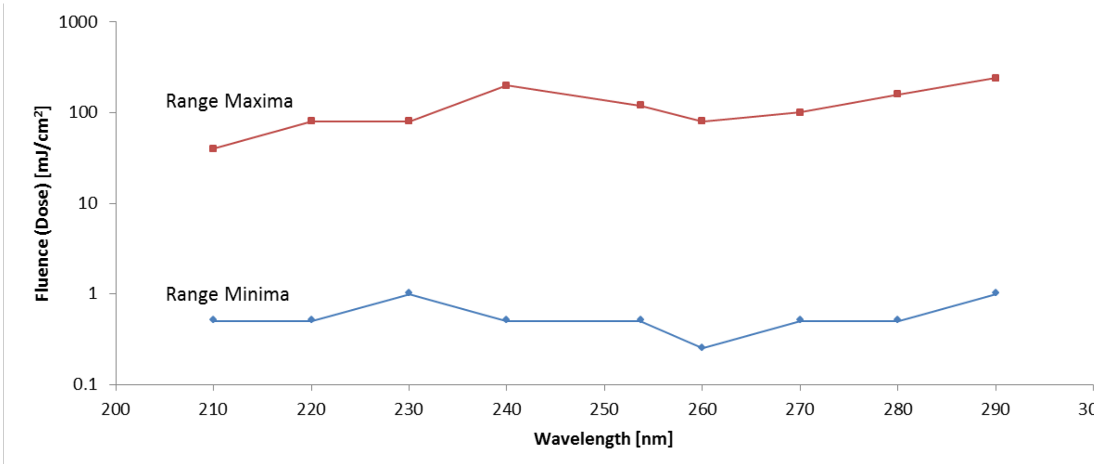

Figure 9. Graph of the range of fluence or dose (mJ/cm^2) by wavelength used during this project.

Future Plans

There are several improvements planned for the NIST tunable UV irradiance laser system. The simplest is computer control of the laser wavelength. Another improvement would be to acquire dielectric mirrors that can work at 210 nm. This would likely require some collaboration with the manufacturer to verify the mirror performance. The switching between mirror types could be automated. Additional improvements in the irradiance measurement could be made by using a calibrated detector to monitor the irradiance during the water sample exposure. Refinements to the detector signal measurement method could be explored. And finally, as mentioned above, using a camera to analyze the irradiance uniformity and calculate the Petri Factor in real time. This would reduce the time needed to measure the Petri factor and provide irradiance uniformity information for the entire beam and not just along two orthogonal axes through the beam center.

Conclusions

The NIST transportable tunable UV irradiance laser system has demonstrated its ability to be deployed to a field site and to provide irradiance at narrowband, and accurately defined, UV wavelengths at levels of 10 µW/cm^2 to > 100 µW/cm^2 and fluences (or doses) from < 1 mJ/cm^2 to > 100 mJ/cm^2 from 210 nm to 300 nm. This is a significant advance over present systems that rely on lamp sources that are broadband and spectrally non-uniform within that band. Such complications can degrade the resulting measurement accuracy and certainly complicate the measurement analysis process. The results published elsewhere on the inactivation of waterborne microorganisms to specific UV light wavelengths as part of WaterRF Project 4376 [6] demonstrate the unique capabilities of the NIST tunable UV irradiance laser system and its potential application for other biological experiments.

Acknowledgements

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