Ultraviolet degradation study of photomultiplier tubes at SURF III

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ABSTRACT

Photomultiplier tubes (PMTs) are used in biological detection systems in order to detect the presence of biological warfare agents. To ensure proper operation of these biological detection systems, the performance of PMTs must be characterized in terms of their responsivity and long-term stability. We report a technique for PMT calibration at the Synchrotron Ultraviolet Radiation Facility (SURF III) at the National Institute of Standards and Technology (NIST). SURF III provides synchrotron radiation with a smooth and continuous spectrum covering the entire UV range for accurate PMT measurements. By taking advantage of the ten decade variability in the flux of the synchrotron radiation, we studied properties of commercial PMTs such as the linearity, spatial uniformity, and spectral responsivity. We demonstrate the degradation of PMTs by comparing new PMTs with PMTs that were used and operated in a biological detection system for a long period of time. The observed degradation is discussed.

Keywords: photomultiplier tubes, ultraviolet, degradation, synchrotron radiation, calibration

1. INTRODUCTION

The threat of biological attacks on the United States homeland and military forces overseas continues to expand. As a result, the Department of Defense (DoD) has a growing need for accurate and reliable biological detection systems [1] to counter the threat and ensure the safety and mission effectiveness of the warfighters. Biological point detection systems provide commanders with information in order to take protective actions and limit biological warfare agent exposure to the warfighters. These systems incorporate photodetectors, such as photomultiplier tubes (PMTs), to detect the presence or absence of biological warfare agents in the air.

Biological detection systems that use an ultraviolet (UV) laser-induced fluorescence detection approach take advantage of the fact that the cross sections for particles in the 1-10 μm range are large enough to make single particle interrogation feasible [2]. These detection systems draw outside air into the system and then aerosolized particles are illuminated by a UV laser with the purpose of exciting fluorescence due to the presence of common constituents in biological matter. This is achieved when the exciting laser, operating at a specific wavelength, propagates perpendicular to the sample air flow. Particles illuminated by the laser beam emit both elastically scattered radiation and fluorescence radiation [3]. The elastically scattered radiation has the same wavelength as the laser radiation while the fluorescence radiation covers a spectral band consisting of longer wavelengths. The emitted radiation from particles is detected and monitored with PMTs through the use of dichroic mirrors and glass filters to separate the different wavelength detection regions. The PMT output signal is then processed through a computational algorithm [3] to identify if a biological warfare agent is present by comparing the spectral signature of the sample against the known signatures of particular pathogens. Fig. 1 shows the general course of events starting with the laser beam.

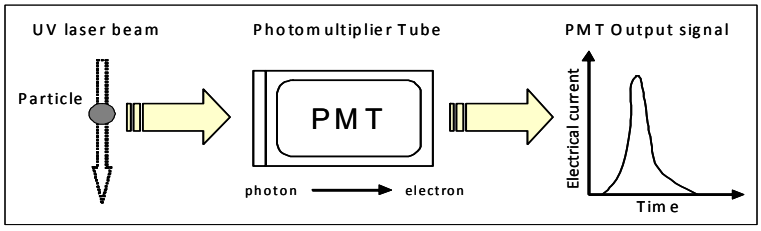

Fig. 1. Particles in the UV laser beam path emit radiation that is detected by PMTs. Photons incident on the PMTs are converted to electrons and then a charged pulse is produced as the PMTs output signal.

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The PMT that detects and monitors the elastically scattered radiation, as opposed to the fluorescence radiation, will be more closely examined in this study with respect to its degradation characteristics since it is continuously exposed to the shortest wavelength of radiation. The elastically scattered PMT plays a center role in the detection of biological warfare agents within biological detection systems. To assure proper detection of biological agents, PMT behavior, especially long-term stability, must be carefully studied. While PMTs are widely used for low-light detection [4], reports on the characterization of PMTs remain scarce.

For this work, the measurement of PMTs was performed at beamline 4 of the Synchrotron Ultraviolet Radiation Facility (SURF III) at the National Institute of Standards and Technology (NIST). SURF III provides synchrotron radiation with a smooth and continuous spectrum covering the entire UV range [5] [6]. Beamline 4 monochromatizes the radiation from SURF III with a 2-m monochromator and uses the resulting UV radiation for radiometric measurements of detectors [7]. In the past, beamline 4 was used primarily for the calibration of solid state UV detectors with its primary scale derived from a cryogenic radiometer [8]. Typical power of light used at beamline 4 to probe detectors is on the order of 1 μW, which, while suitable for most solid state detectors, can easily saturate low-light detectors like PMTs. To be able to operate beamline 4 at a much lower power level while maintaining its scale traceable to a cryogenic radiometer, we make use of the fact that synchrotron radiation is directly proportional to the electron beam current in the storage ring. Thus, the primary scale at beamline 4 can be transferred to low-light level if the electron current in the storage is known. Given that the electron current of SURF III can be varied more than 10 orders of magnitude (from about 100 mA to 10 pA) [5] [6] the power range suitable for PMTs can be readily achieved. With this technique, we can perform radiometric measurements on PMTs such as the absolute calibration of linearity, spatial uniformity, and spectral responsivity. Comparison of new and used PMTs reveal the degradation of PMTs after prolonged exposure to radiation.

2. EXPERIMENTAL

To demonstrate our PMT calibration technique, we performed calibration on four commercially available UV PMTs. All four PMTs are of the same type, head-on with a bialkali photocathode and a 10-dynode linear-focused structure [4]. The active area of the PMTs is 18 mm x 18 mm and the spectral range is from 185 nm to 650 nm [4]. Of the four PMTs, two were newly acquired from the manufacturer and the other two PMTs were used in an actual biological detection system with logged operating time of 549 hours and 1699 hours before our measurements. The PMT exposure hours are recorded on the PMTs' housing sub-system and are assumed to be accurate based on the assumption that the PMTs were not replaced at any given time. The operating voltage for all PMTs was set at 765 V. The goal of our measurements is to compare these PMTs and to see if we can gain insight into the degradation of PMTs subjected to long exposures of UV radiation. While the exact dose of UV radiation to these PMTs is unknown, our measurement results illustrate our calibration capacity and the severity of the degradation process as a guide for more quantitative studies of PMT degradation in the future.

The experimental setup for the PMTs under study is illustrated in Fig. 2 where the test detector, a PMT, is placed inside a detector chamber and mounted on an x-y translation stage. The linear motions were used to raster scan the detector for spatial response mapping and to position the monochromatized UV beam for calibration at any point of interest on the surface of the PMT. The monochromatized UV beam is from the 2-m monochromator and an imaging system which images the exit slit of the monochromator onto the detector surface with a dimension of 1 mm x 2 mm. An external high voltage power supply provides the operating voltage to the test PMT. Current from the output of the test PMT was measured by a picoammeter which in turn sends the digitized data to a computer. The computer also collects and records values of SURF electron beam current which are measured and broadcasted in real time over the internet by SURF control system. The spectral range for PMT calibration was from 200 nm to 500 nm. For this work, all PMTs were measured in current mode instead of photon counting mode.

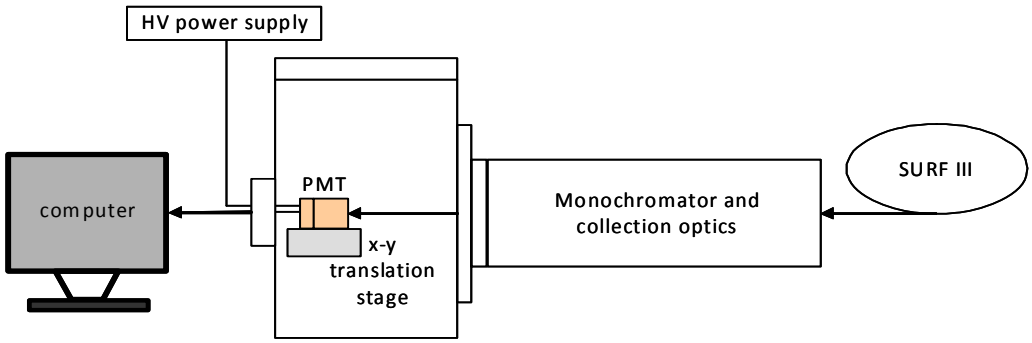

Fig. 2. Experimental setup for the calibration of PMTs using SURF III beamline 4. The synchrotron radiation from SURF III passes through a set of collection optics and a 2-m monochromator before reaching the detector chamber where the PMT resides.

To establish the scale for PMT calibration, we used a NIST calibrated silicon photodiode to establish the relationship between the power of the UV beam and the SURF electron beam current prior to PMT calibration. For this measurement, we positioned the silicon photodiode inside the detector chamber and let the UV beam irradiate the photodiode. Measurement of the photocurrent from the silicon photodiode started with SURF current at several tens of milliamperes (mA) and continued while the SURF current was gradually dropped to several mA to a point where the signal to noise ratio from the silicon photodiode became unacceptable because of the low-light level of the UV beam. After this measurement, the PMT under test was moved into the UV beam path with the SURF beam current dropped below one mA to perform PMT calibration with varying input power levels of UV radiation.

3. RESULTS

We calibrated four PMTs, two new ones from the manufacturer and two used ones from a biological detection system, and measured their linearity, spatial uniformity, and spectral responsivity. For all measurements, the anode current of the PMT was normalized by the SURF beam current in real time. For spectral responsivity measurements, the normalized signal from the calibrated photodiode was used to derive the absolute responsivity of the PMT in A/W.

3.1 Linearity of PMT

Linearity of the PMT was measured at a fixed position on the PMT active area. The measurement was performed with SURF current that spanned four orders of magnitude from about 1 mA to 0.1 μA. At the highest SURF current of 1mA, the corresponding PMT output current was about 20 μA, a value that is higher than the manufacturer's recommended operating value and is known to saturate the PMT. On the other hand, at the lowest SURF current of 0.1 μA, the corresponding PMT output current was about 2 nA, where the signal-to-noise ratio decreased significantly and the measured relative standard deviation of PMT current was just below 5%. Measurement of linearity was performed at several wavelengths from 240 nm to 320 nm.

Shown in Fig. 3 is the linearity of one of the newly acquired PMTs. For all wavelengths, the response of the PMT shows a similar decreasing trend with decreasing flux of incident light. This behavior is particularly pronounced at high SURF current between 1 mA and 0.1 mA. In this region where PMTs saturate, this trend is opposite to the saturation trend of most photodetectors that show a reduced response with increasing flux of incident light. However, the measured saturation effect in Fig. 3 is consistent with previous observations [4] and was attributed to a change in voltage distribution across the anode voltage-divider resistors. With higher flux of incident light, higher current flow through the resistors actually increased the voltage at the first few stages of anode and resulted in higher gain.

Outside the saturation region, we still observed a deviation of about 3% from linearity for this type of PMT. For precision measurements, this is an important factor that has to be accounted for.

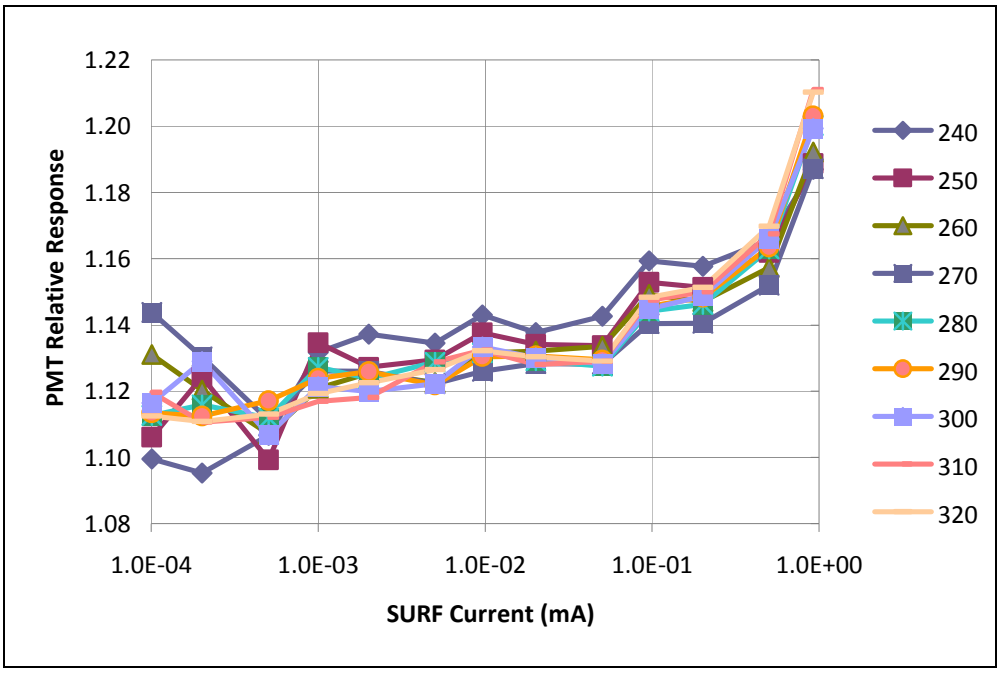

Fig. 3. Linearity of a new PMT at several different wavelengths.

3.2 Uniformity and spectral responsivity of a new PMT

In the responsivity calibration of any photodetector, measurement of uniformity across the active area of the photodetector is required because spatial uniformity of a photodetector directly impacts the reproducibility in responsivity calibration. Uniformity of a photodetector is a major factor in estimating the calibration uncertainty. For our PMT calibration, we started by measuring the uniformity of the PMT and using the uniformity map as a guide to select positions for later spectral response calibration.

Of all the newly acquired PMTs, we found they exhibit a very similar degree of spatial uniformity. Fig. 4 shows the uniformity of one of the new PMTs. The uniformity scan was performed at a fixed wavelength of 270 nm. The SURF beam current was about 10 μA and the PMT current was approximately 200 nA. The PMT supply voltage was held at a constant 765 V. As can be seen from Fig. 4, the type of PMT we studied has a very poor uniformity map. There is as much as 50% variation from the highest response to the lowest response even in the central region of the active area. Such a large variation in response poses a serious challenge for the power response calibration of this type of PMT. Any meaningful calibration requires knowledge of where on the PMT active area the calibration was performed. For this work, we chose two positions that had close to the maximum and minimum response as indicated in the bottom right uniformity contour plot in Fig.4. The two positions resided at a local maximum and a local minimum position such that the calibration was less sensitive to the positioning of the light beam. Shown in the bottom left plot of Fig. 4 is the spectral responsivity of the PMT measured at these two positions. While the responsivity at the two positions show a large difference, the spectral shape of the two responsivity curves are very similar.

The typical gain from the anode is approximately 10^6 for the type of the PMT under study [4]. From Fig. 4, the estimated cathode responsivity is on the order of 100 mA/W. We are planning to perform more PMT measurements to determine the cathode responsivity and thus derive a more accurate PMT gain factor.

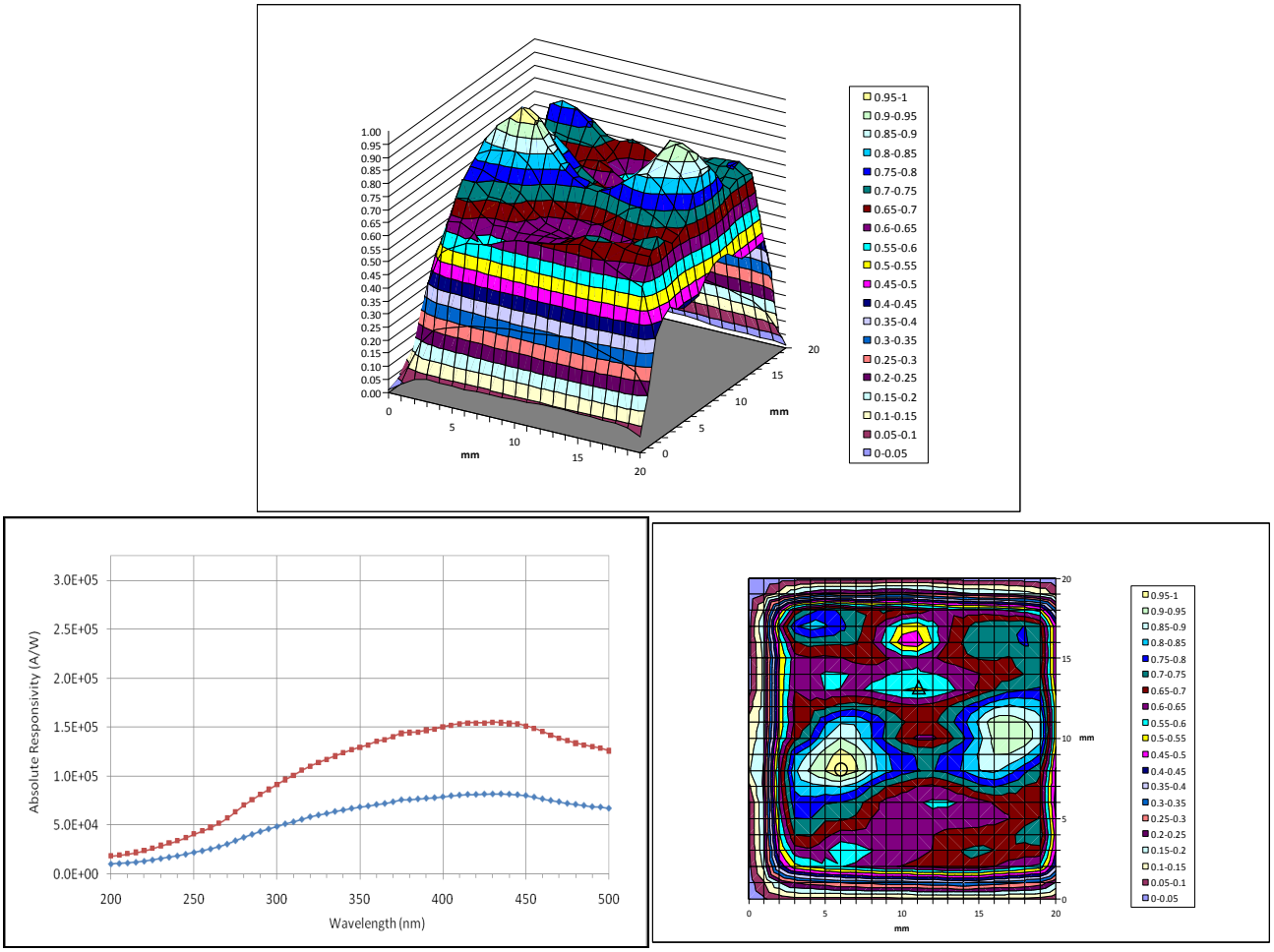

Fig. 4. Uniformity and spectral responsivity data of a newly acquired PMT. Top: spatial uniformity graph. Bottom-right: spatial uniformity contour graph with the two positions marked by a circle and a triangle. Bottom-left: PMT spectral responsivity. Top and bottom curve measured at the position marked by circle and triangle, respectively, on the contour graph to the right.

3.3 Uniformity and spectral responsivity of used PMTs

Two used PMTs, logged 549 hours and 1699 hours of operating time on a biological detection system, were measured for spatial uniformity and spectral responsivity in the same manner as the new PMT which is discussed previously. The measurements were performed under the same testing conditions and parameters.

Fig. 5 shows the spatial uniformity and spectral responsivity of the 549 hour PMT. This PMT exhibits a very similar degree of uniformity to the newly acquired PMT shown in Fig. 4. The shape of the spectral responsivity curve is also very similar to the new PMT and the variation in the absolute values is in line with the difference among new PMTs.

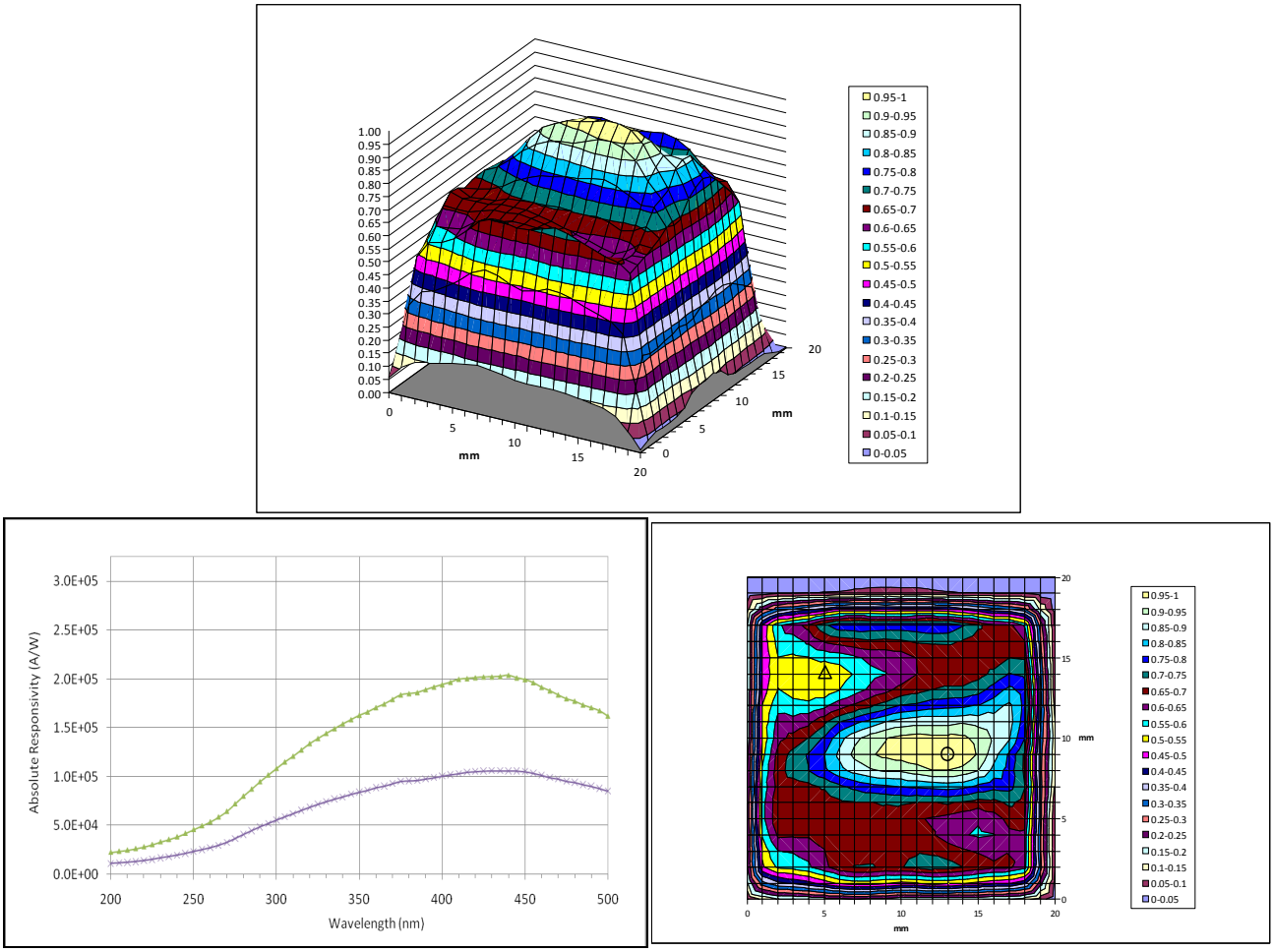

Fig. 5. Uniformity and spectral responsivity data of the 549 hour PMT. Top: spatial uniformity graph. Bottom-right: spatial uniformity contour graph with the two positions marked by a circle and a triangle. Bottom-left: PMT Spectral responsivity. Top and bottom curve measured at the position marked by circle and triangle, respectively, on the contour graph to the right.

On the other hand, the spatial uniformity and spectral responsivity of the 1699 hour PMT shows a dramatic difference compared to the other PMTs as shown in Fig. 6. In the uniformity graph, the central region of the PMT shows a large area with a very depressed response and a rim around this depressed region. Spectral responsivity performed at the rim of the PMT (top curve of the bottom-left graph in Fig. 6) indicates responsivity in line with all of our measurements with other PMTs. However, the spectral responsivity measured in the central region of the PMT is approximately a factor of 10 less than the measurement in the rim region. Such a reduction in responsivity is clearly due to the aging of the PMT. The shape of the uniformity map can also be explained by the fact that the central region of the PMT received most of the UV irradiation from the biological detection system while the rim around the border of the active area was shadowed by baffles and apertures of the biological detection system and therefore received a much smaller UV dose than the central region.

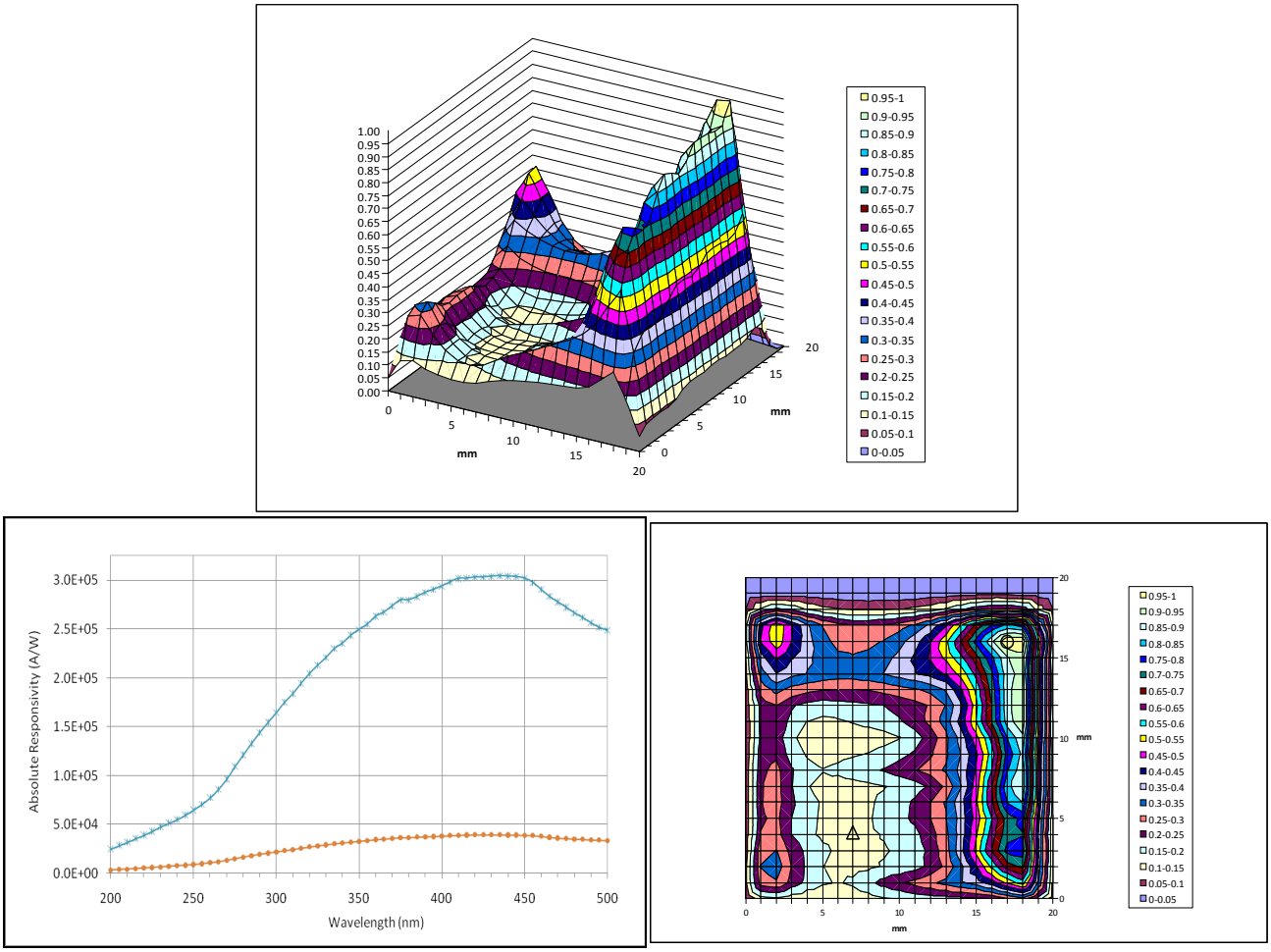

Fig. 6. Uniformity and spectral responsivity of the 1699 hour PMT. Top: spatial uniformity graph. Bottom-right: spatial uniformity contour graph with the two positions marked by a circle and a triangle. Bottom-left: PMT Spectral responsivity. Top and bottom curve measured at the position marked by circle and triangle, respectively, on the contour graph to the right.

4. CONCLUSION

We have shown that synchrotron radiation can be used to characterize and calibrate PMTs. The primary scale for such calibration is traceable to a cryogenic radiometer and derived using the linearity of the synchrotron radiation on the electron beam current in the storage ring. The capability to characterize and quantify PMT degradation using SURF III at NIST has been demonstrated and proven through the measurements of PMT linearity, spatial uniformity, and spectral responsivity. We observed significant degradation with the PMT that had been exposed to UV radiation in a biological detection system. We plan to conduct more measurements with a variety of PMTs. Future studies will also aim towards the goal of establishing a PMT degradation rate for PMTs exposed to UV radiation and eventually a degradation interval. This study, along with future UV degradation studies, will ultimately reduce biological detection systems' calibration and troubleshooting costs. Furthermore, PMT measurement support will reduce the rate of false-positives and false-negatives. Measurement support for the PMTs will increase the biological detection systems' measurement repeatability and accuracy which will ultimately help prevent warfighter casualties.

5. ACKNOWLEDGEMENTS

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