Progress Report: Development of a Visible-NIR Photoluminescence Microspectrometer

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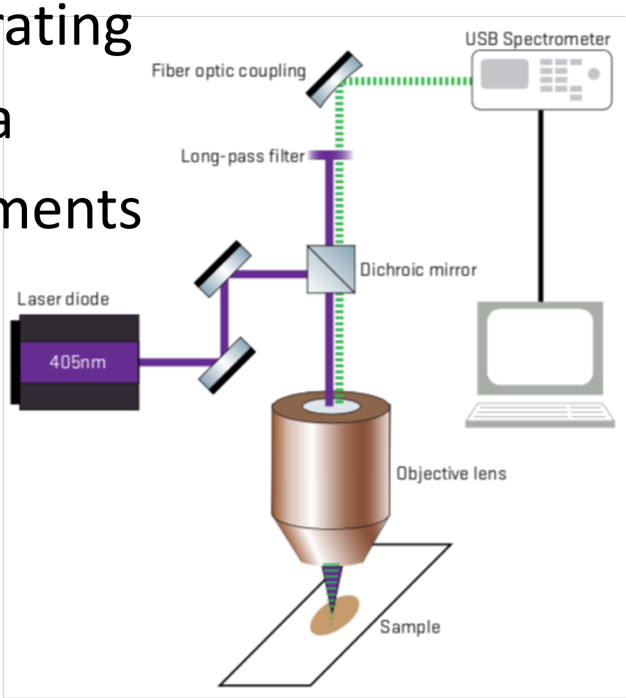
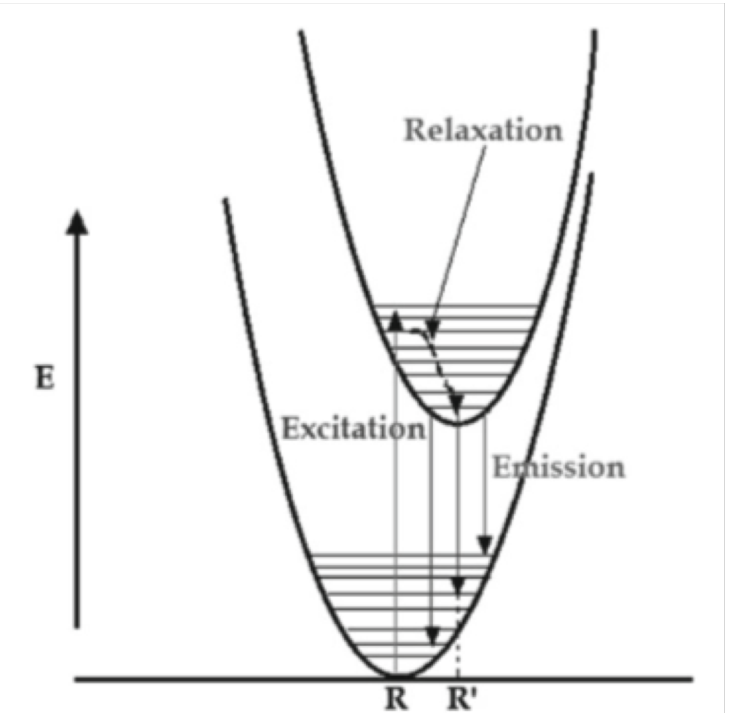
Goals

- Couple laser light source into microscope
- Couple PL light into Ocean Optics with fiber
- Measure visible PL, compare to other data
- Couple PL light into InGaAs CCD with grating
- Measure NIR PL, compare to other data
- Write detailed SOP for taking measurements
- *(Optional)* Measure new materials



Photoluminescence

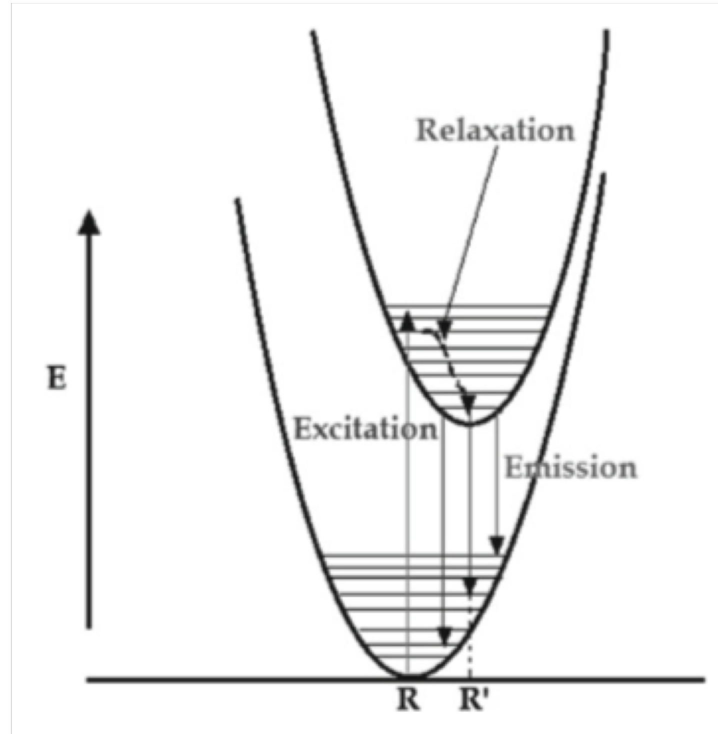
- Excitation (Single wavelength)
  - Absorb photon
  - Electron to "excited state" ("bound state"?)
- Relaxation
  - Lose energy to vibration
  - Electron to slightly lower "vibronic state"



Shinde, K. N., et al (2012). Basic Mechanisms of Photoluminescence. *Phosphate Phosphors for Solid-State Lighting* (pp.41-59). Berlin: Springer.

Photoluminescence

- Emission (Spectral)
  - “Radiative relaxation”
  - Lose energy to photon emission
  - Electron back to ground state
- Measurement
  - Spectrum of emission
  - Intensity of emission



Detectors

- Not universally sensitive to all wavelengths
- Visible Range (400-700 nm)
  - Si sensors commonly used
  - Easy application: Ocean Optics spectrometer
- Near-Infrared (NIR) Range (800-2500 nm)
  - InGaAs sensors commonly used
  - Challenge: Grating, linear CCD

Configuration for Visible PL

- Completed summer 2018
- Excites with 405nm laser diode
- Fiber-coupled Ocean Optics spectrometer
- Early results compared to measurements on existing system look good

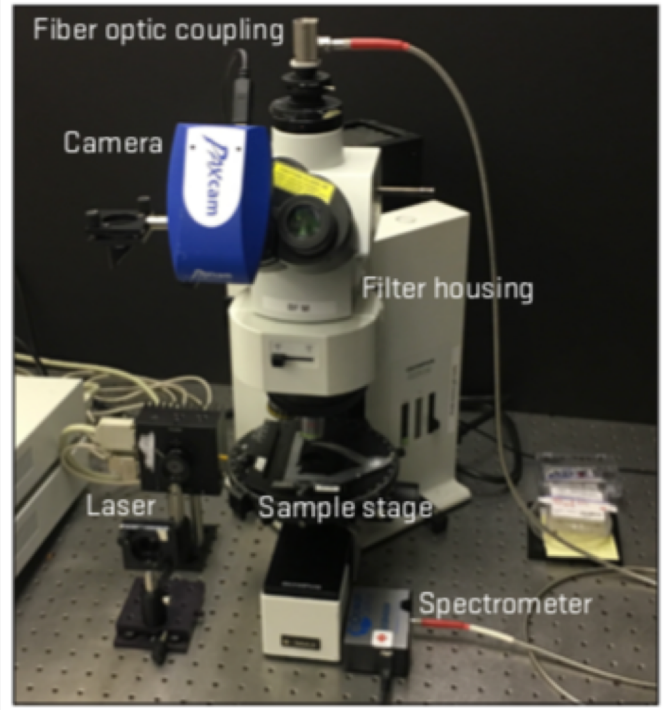
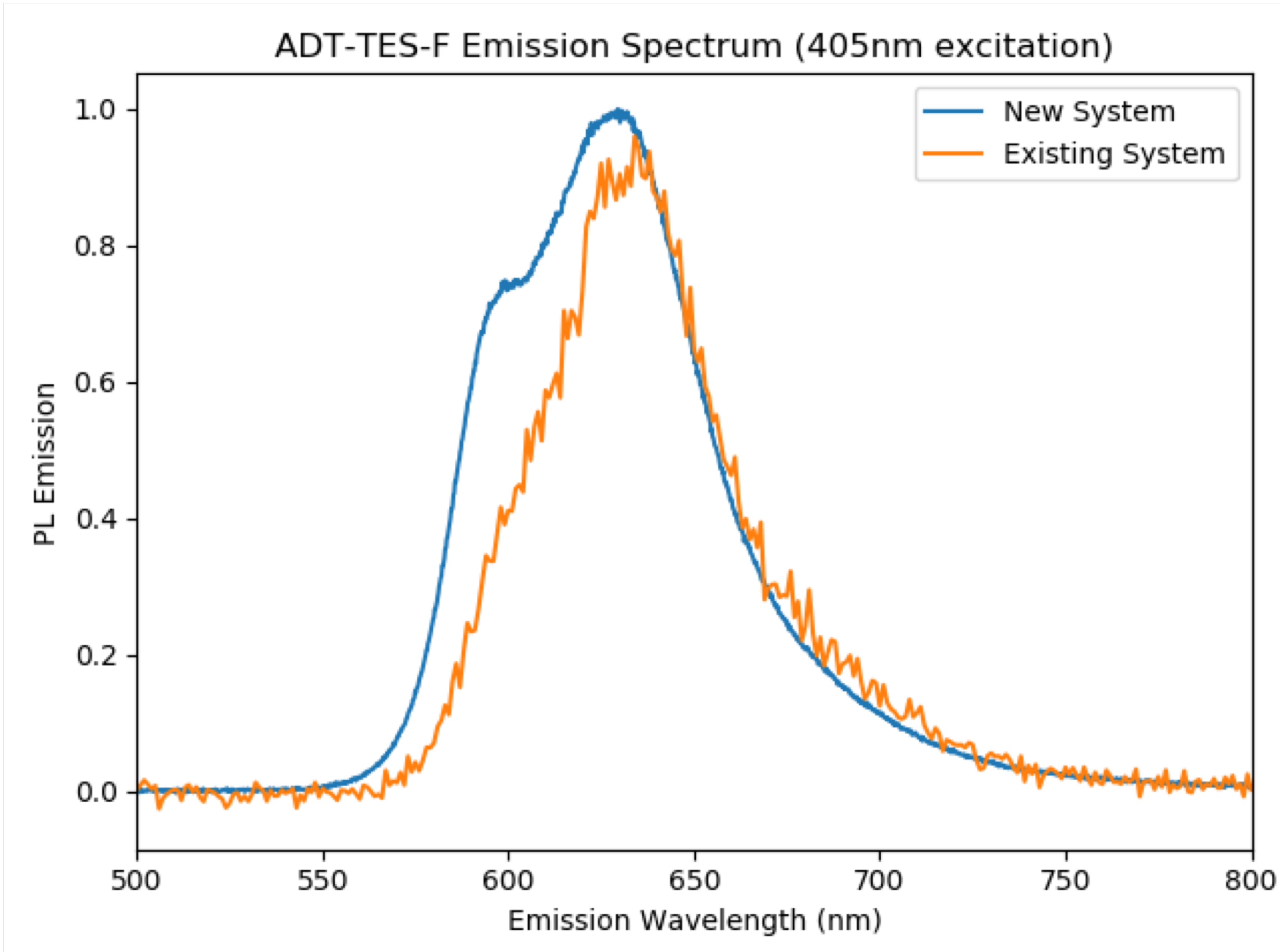
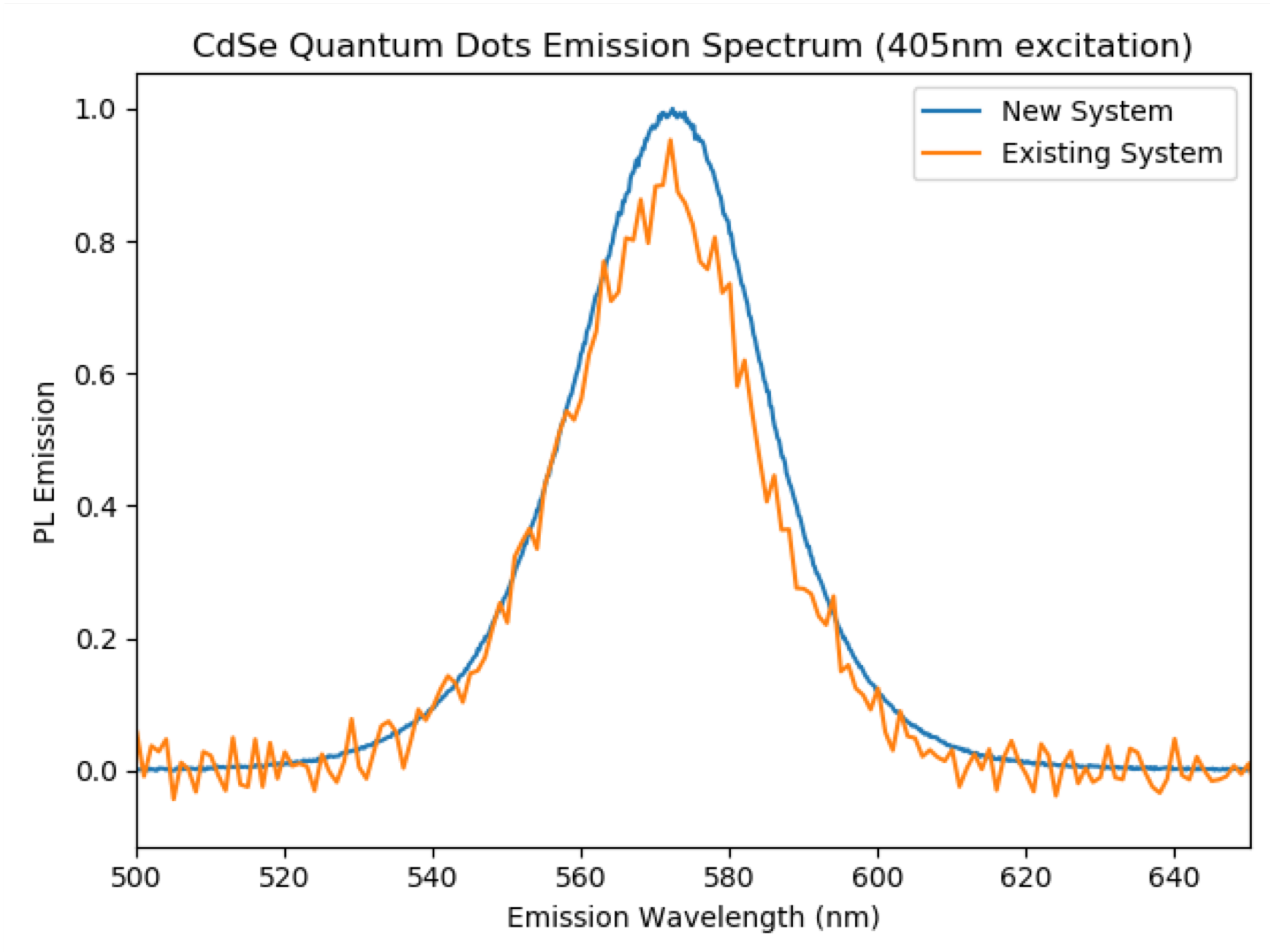


Figure 4: Experimental setup. The laser light source, fiber optic spectrometer, camera, and sample stage are shown. Laser light enters the housing for the filter cube on the left side of the system.

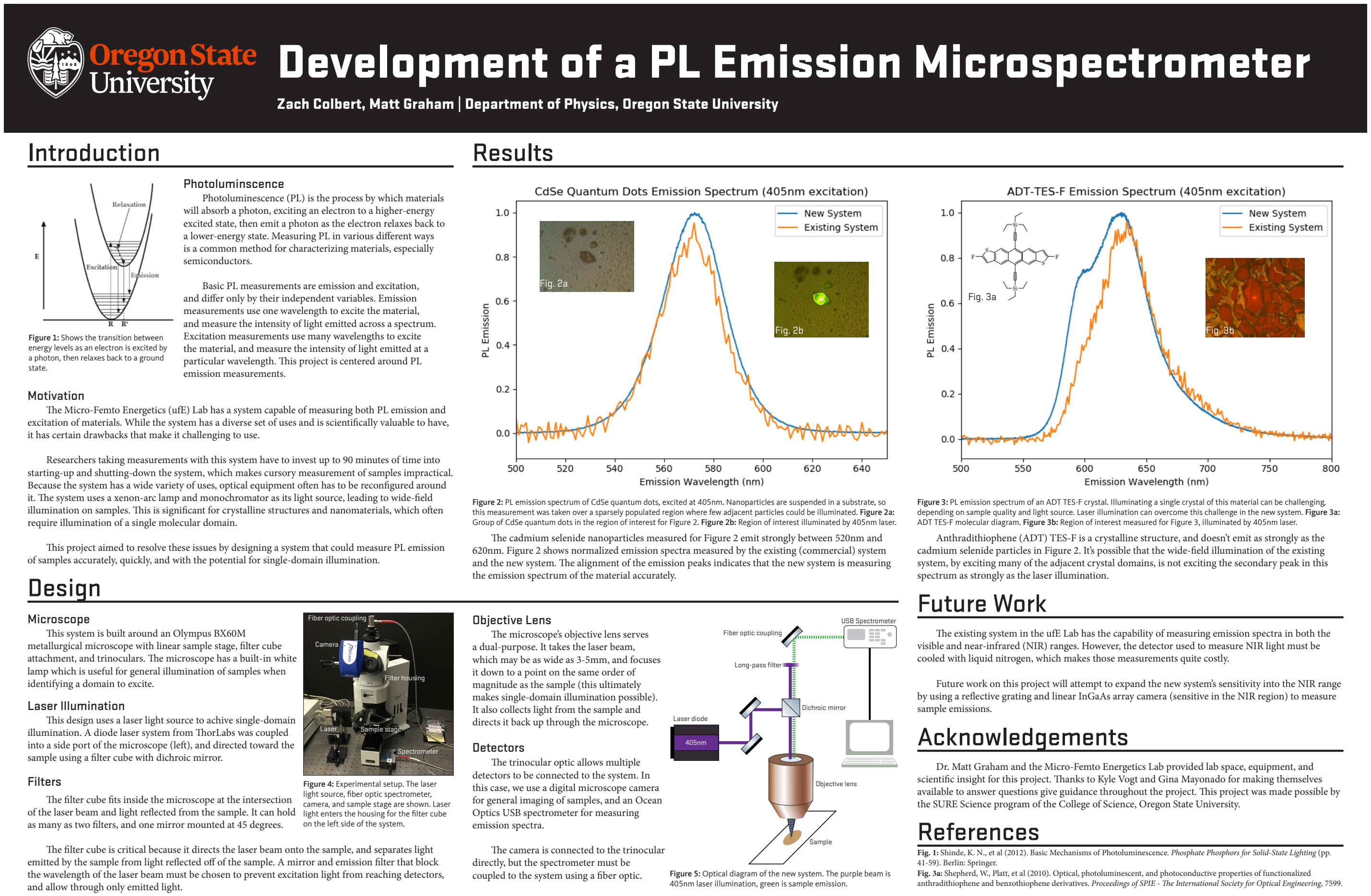
Configuration for Visible PL



Configuration for Visible PL



Poster



Development of a PL Emission Microspectrometer

Oregon State University

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Introduction

Photoluminscence

Photoluminescence (PL) is the process by which materials will absorb a photon, exciting an electron to a higher-energy excited state, then emit a photon as the electron relaxes back to a lower-energy state. Measuring PL in various different ways is a common method for characterizing materials, especially semiconductors.

Basic PL measurements are emission and excitation, and differ only by their independent variables. Emission measurements use one wavelength to excite the material, and measure the intensity of light emitted across a spectrum. Excitation measurements use many wavelengths to excite the material, and measure the intensity of light emitted at a particular wavelength. This project is centered around PL emission measurements.

Figure 1: Shows the transition between energy levels as an electron is excited by a photon, then relaxes back to a ground state.

Motivation

The Micro-Femto Energetics (ufE) Lab has a system capable of measuring both PL emission and excitation of materials. While the system has a diverse set of uses and is scientifically valuable to have, it has certain drawbacks that make it challenging to use.

Researchers taking measurements with this system have to invest up to 90 minutes of time into starting-up and shutting-down the system, which makes cursory measurement of samples impractical. Because the system has a wide variety of uses, optical equipment often has to be reconfigured around it. The system uses a xenon-arc lamp and monochromator as its light source, leading to wide-field illumination on samples. This is significant for crystalline structures and nanomaterials, which often require illumination of a single molecular domain.

This project aimed to resolve these issues by designing a system that could measure PL emission of samples accurately, quickly, and with the potential for single-domain illumination.

Design

Microscope

This system is built around an Olympus BX60M metallurgical microscope with linear sample stage, filter cube attachment, and trinoculars. The microscope has a built-in white lamp which is useful for general illumination of samples when identifying a domain to excite.

Laser Illumination

This design uses a laser light source to achive single-domain illumination. A diode laser system from ThorLabs was coupled into a side port of the microscope (left), and directed toward the sample using a filter cube with dichroic mirror.

Filters

The filter cube fits inside the microscope at the intersection of the laser beam and light reflected from the sample. It can hold as many as two filters, and one mirror mounted at 45 degrees.

The filter cube is critical because it directs the laser beam onto the sample, and separates light emitted by the sample from light reflected off of the sample. A mirror and emission filter that block the wavelength of the laser beam must be chosen to prevent excitation light from reaching detectors, and allow through only emitted light.

Figure 4: Experimental setup. The laser light source, fiber optic spectrometer, camera, and sample stage are shown. Laser light enters the housing for the filter cube on the left side of the system.

Objective Lens

The microscope's objective lens serves a dual-purpose. It takes the laser beam, which may be as wide as 3-5mm, and focuses it down to a point on the same order of magnitude as the sample (this ultimately makes single-domain illumination possible). It also collects light from the sample and directs it back up through the microscope.

Detectors

The trinocular optic allows multiple detectors to be connected to the system. In this case, we use a digital microscope camera for general imaging of samples, and an Ocean Optics USB spectrometer for measuring emission spectra.

The camera is connected to the trinocular directly, but the spectrometer must be coupled to the system using a fiber optic.

Figure 5: Optical diagram of the new system. The purple beam is 405nm laser illumination, green is sample emission.

Results

Figure 2: PL emission spectrum of CdSe quantum dots, excited at 405nm. Nanoparticles are suspended in a substrate, so this measurement was taken over a sparsely populated region where few adjacent particles could be illuminated. **Figure 2a:** Group of CdSe quantum dots in the region of interest for Figure 2. **Figure 2b:** Region of interest illuminated by 405nm laser.

The cadmium selenide nanoparticles measured for Figure 2 emit strongly between 520nm and 620nm. Figure 2 shows normalized emission spectra measured by the existing (commercial) system and the new system. The alignment of the emission peaks indicates that the new system is measuring the emission spectrum of the material accurately.

Figure 3: PL emission spectrum of an ADT TES-F crystal. Illuminating a single crystal of this material can be challenging, depending on sample quality and light source. Laser illumination can overcome this challenge in the new system. **Figure 3a:** ADT TES-F molecular diagram. **Figure 3b:** Region of interest measured for Figure 3, illuminated by 405nm laser.

Anthradithiophene (ADT) TES-F is a crystalline structure, and doesn't emit as strongly as the cadmium selenide particles in Figure 2. It's possible that the wide-field illumination of the existing system, by exciting many of the adjacent crystal domains, is not exciting the secondary peak in this spectrum as strongly as the laser illumination.

Future Work

The existing system in the ufE Lab has the capability of measuring emission spectra in both the visible and near-infrared (NIR) ranges. However, the detector used to measure NIR light must be cooled with liquid nitrogen, which makes those measurements quite costly.

Future work on this project will attempt to expand the new system's sensitivity into the NIR range by using a reflective grating and linear InGaAs array camera (sensitive in the NIR region) to measure sample emissions.

Acknowledgements

Dr. Matt Graham and the Micro-Femto Energetics Lab provided lab space, equipment, and scientific insight for this project. Thanks to Kyle Vogt and Gina Mayonado for making themselves available to answer questions give guidance throughout the project. This project was made possible by the SURE Science program of the College of Science, Oregon State University.

References

Fig. 1: Shinde, K. N., et al (2012). Basic Mechanisms of Photoluminescence. *Phosphate Phosphors for Solid-State Lighting* (pp. 41-59). Berlin: Springer.

Fig. 3a: Shepherd, W., Platt, et al (2010). Optical, photoluminescent, and photoconductive properties of functionalized anthradithiophene and benzothiophene derivatives. *Proceedings of SPIE - The International Society for Optical Engineering*, 7599.

Next Steps

- Literature Review – Winter Break
  - Optics: How do reflective gratings work?
  - Optics: How to rigorously align light from grating to InGaAs array CCD?
  - Works using similar systems
- Optical Design and Setup – End of Break
- NIR Measurements – Winter Break
- Comparison and Analysis – Winter Break