Using Electroabsorption to Measure Changes in Exciton Binding Energy in Organic and Hybrid Organic-Inorganic Perovskite Materials

Amelia Poulin

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John Colton, Advisor

Department of Physics and Astronomy

Brigham Young University

ABSTRACT

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Amelia Poulin
Department of Physics and Astronomy, BYU
Bachelor of Science

In the search for efficient semiconductors, perovskite materials have been shown to have a steep increase in efficiency over time compared to silicon semiconductors in solar cell and fiber-optic applications. We are studying hybrid organic-inorganic perovskites and organic semiconductors. The perovskite materials we are studying are in the form phenylethylammonium-x tin iodide (x = 3-F, 4-F, 2-CF_3, and 2-methyl). To increase perovskite semiconductor efficiency in various applications, we need to find a way to synthesize the material to get specific exciton binding energies. Exciton binding energy directly affects the efficiency of charge carrier separation and transport, so knowing how to tune exciton binding energy will allow us to improve perovskite semiconductor efficiency. To find the exciton binding energy of these perovskite materials, we use electroabsorption techniques. We are trying to determine how and why exciton binding energy changes in these 2D perovskite materials. It seems that the position of the modifier (x = 3-F, 4-F, 2-CF_3, and 2-methyl) on the carbon ring of phenylethylammonium-x tin iodide effects exciton binding energy much more than the modifier itself. More investigation is needed to conclude this with greater certainty. We are also studying organic solar cells, specifically Y6 as the acceptor and PM6 as the donor, to see if it has a charge transfer state, which is where electrons are moved to another molecule causing electron density to redistribute. Previous electroreflectance studies have shown that there is an intermolecular charge transfer, but through our studies we have not seen any evidence of a charge transfer state, meaning that there isn't a charge transfer state, or that electroabsorption won't help us see it. There is also a list included in Appendix A of important terms that can be helpful in gaining a better understanding of electroabsortion.

Keywords: Electroabsorption, Perovskite, Organic Semiconductor, Octahedral Tilt

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Contents

Chapter 1

Introduction

Finding efficient renewable energy is important in the USA today with our increasing need for more power. Semiconductors are an important thing to understand in renewable energy applications because they are useful in solar cells and optoelectronics (like LEDs). Optoelectronics are significantly used in renewable energy and fiber-optic communications.

A semiconductor has conductivity between a conductor and insulator. Semiconductor properties that are important for understanding efficiency are bandgap, excitons, and exciton binding energy.

Silicon is often used in semiconductor technology because it is a common resource, but it has an indirect bandgap which can limit it in some applications because indirect bandgaps can have phonon interaction for electron transitions. [1] Perovskite semiconductors are important to understand because they have been shown to have an steeper increase in efficiency over time compared to silicon semiconductors (see Figure 1.1). They are also "promising candidates for next-generation photodetectors due to their unique optoelectronic properties and flexible synthesis routes." [2] Perovskites also have "possible future development of...devices such as solar cells, photodetectors, and light-emitting diodes." [3]

In collaboration with the University of Kentucky, we are studying perovskites to try to find out what factors contribute most to bandgap energy, which can increase perovskite semiconductor efficiency because exciton binding energy directly affects the efficiency of charge carrier separation and transport.

A charge transfer state is an excited state in which electrons are moved from one molecular unit to another, either partially or completely, resulting in the redistribution of electron density.

We will discuss how altering the perovskite's chemical composition controls exciton binding energy in hybrid organic-inorganic perovskite materials and in organic perovskite materials. For organic-inorganic perovskite materials, we will discuss how altering the position of the modifier on the phenylethylammonium ring changes properties like exciton binding energy of the material. For organic perovskite materials, we will discuss how we have seen that electroabsorption doesn't show if the material has a charge transfer state. [4]

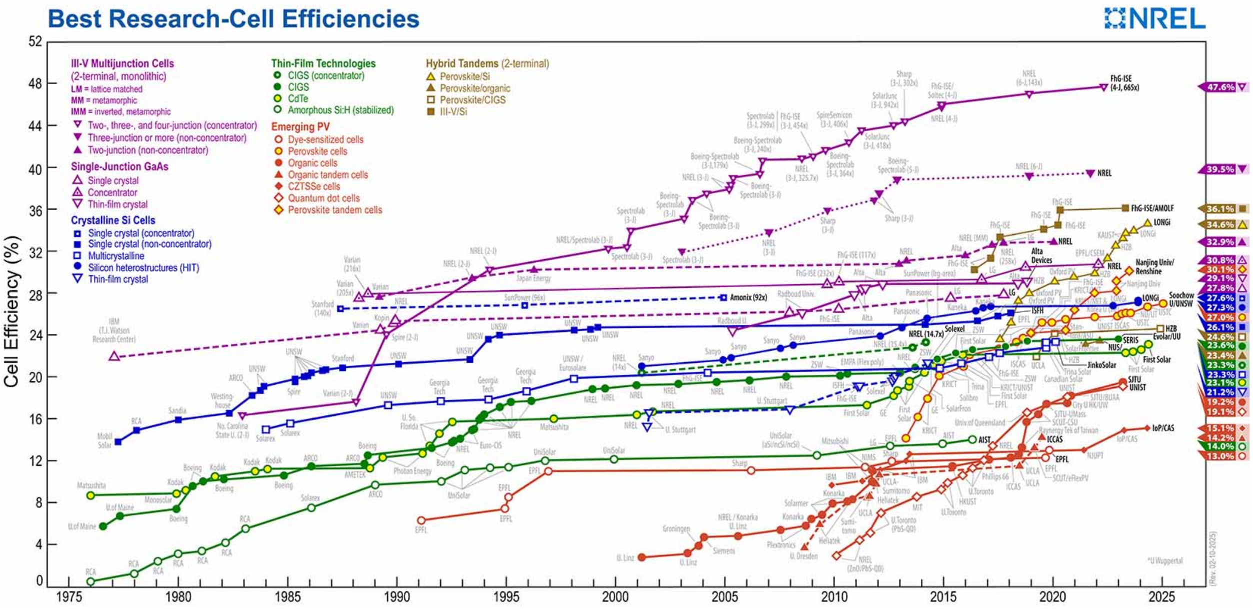


Figure 1.1 Research cell efficiencies over time. Perovskites (gold and orange lines) have a steeper increase in efficiency over time compared to silicon (blue line) semiconductors. [5]

1.1 Excitons

Excitons are quasiparticles made of bound electron and hole pairs. [6] An exciton is created when a photon excites an electron from the valence band to the conduction band, and the exciton is the coupling of the electron in the conduction band to the hole in the valence band (see Figure 1.2).

Exciton binding energy is the energy required to dissociate an exciton into its electron and hole charge carriers (see Figure 1.3). Exciton binding energy is influenced by properties like the material's dielectric constant and the effective mass of the electron and hole. [6]

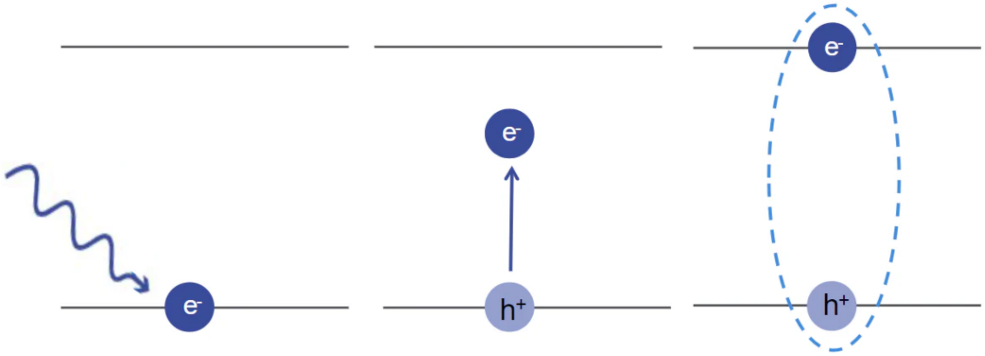


Figure 1.2 An exciton is created when a photon excites an electron from the valence band to the conduction band. The exciton is the coupling of the electron in the conduction band and the hole in the valence band. [6]

1.2 Organic Semiconductors

Organic semiconductors can be flexible with coating plastic sheets as opposed to fixed substrates. An organic semiconductor is composed of carbon and hydrogen atoms and has two components: an electron donor which supplies a photoexcited electron, and an acceptor, which accepts the electron or equivalently supplies a hole. When the hole and electron are linked, it is called a charge transfer exciton since the photoexcited electron goes to a different molecule.

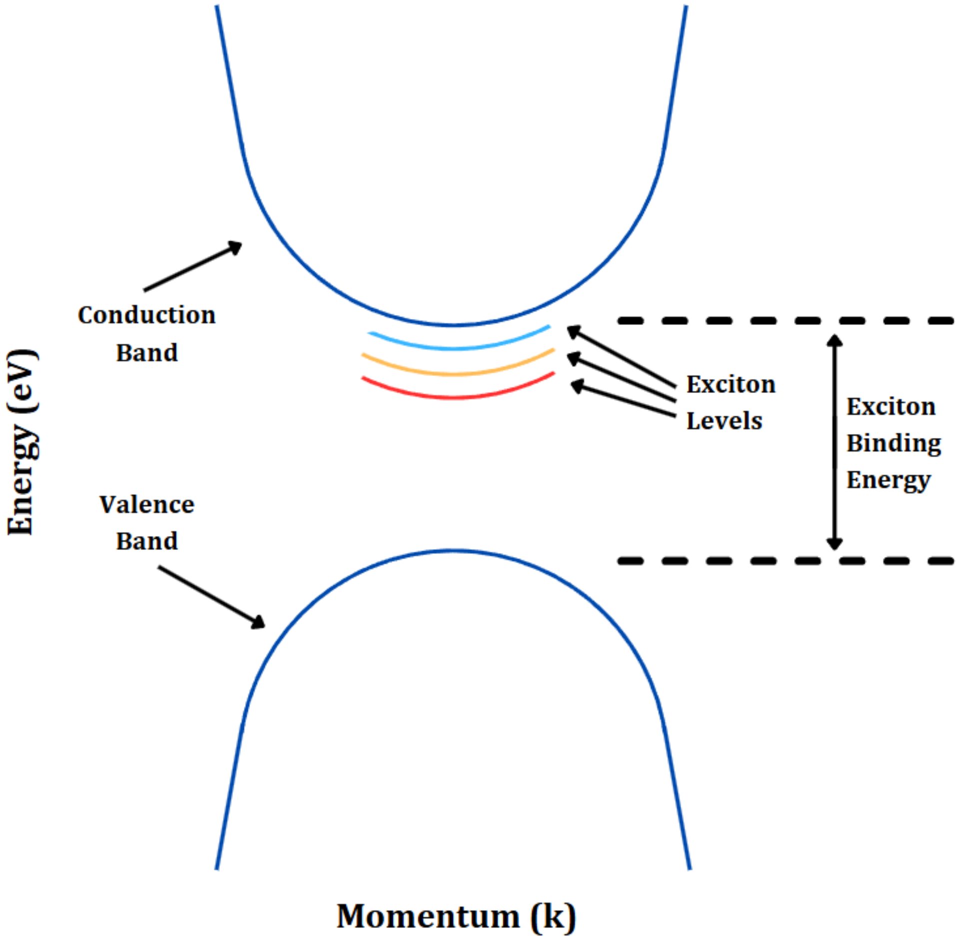


Figure 1.3 The band gap energy (E_g) is the energy difference between the valence band and the conduction band in a semiconductor. Excitons can be formed at energies less than E_g due to the binding energy between electron and hole. The lowest energy exciton peak, corresponding to the 1s exciton level, is depicted in red. [6]

We are studying the formation of the charge transfer exciton, with different concentrations of PM6 and Y6 where one material has a high concentration of holes, and the other with electrons. For organic solar cells, the charge generation mechanism and the recombination loss are heavily linked with charge transfer states.

Organic solar cells have tightly bound excitons because the dielectric constant is low (lower than perovskites), which creates a higher exciton binding energy. The organic semionductor is made of the polymer or acceptor (Y6) and the donor (PM6), along with an additive chloronaphthalene, or fluoronaphthalene. The organic solar cells is spin coated to maximize the area of the interfaces that the parts are overlapping. Based on Wan's research [7] using electroreflectance to observe an intermolecular charge transfer, we hoped to see direct excitations from LUMO (lowest unoccupied molecular orbital) of donor to HOMO (highest occupied molecular orbital) of the acceptor (intermolecular charge transfer) from EA. However, we did not observe any evidence of the charge transfer state using our EA technique.

1.3 Hybrid Organic-Inorganic Perovskites

A perovskite, another material we are working with, is a type of crystal structure with chemical formula ABX_3. The specific perovskite materials we are studying are phenylethylammonium-x tin iodide (x = 2-F, 3-F, 4-F, 2-CF_3, and 2-methyl; see Figure 1.4). We used electroabsorption (EA) to find the bandgap energies of each of the samples (phenylethylammonium-3-F tin iodide, phenylethylammonium-4-F tin iodide, phenylethylammonium-2-CF_3 tin iodide, etc.).

Perovskite materials that include tin are generally more unstable than lead, and don't have as much known about how the bandgap energy changes with different materials. Lead iodide perovskites have been studied in some detail, but tin iodide are much more unstable [8]. Studying different materials of perovskites has helped us see significant improvement in light emitting, detection performance and device design, especially operating in the visible and near-infrared regions. [3]

1.4 Electroabsorption

To find the bandgap energies of these materials, we use electroabsorption (EA). EA is used to measure changes in absorption with and without an applied electric field to determine exciton

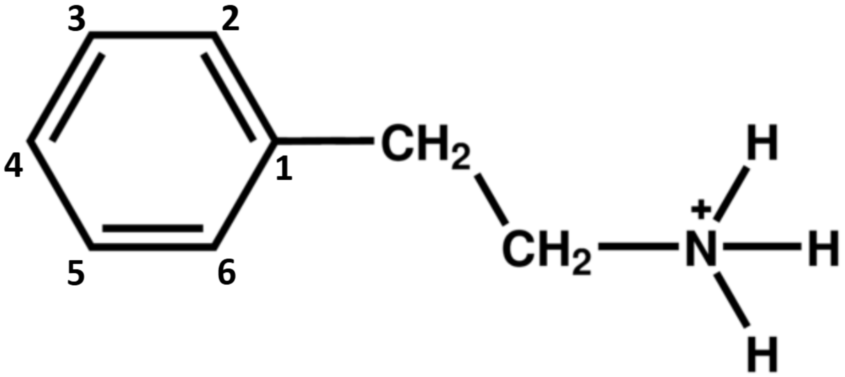


Figure 1.4 Phenylethylammonium molecule structure; The positions of the modifier on the ring are labeled, where 4-F PESI would indicate an F attached to the 4th position on the ring.

binding energies. A red-shift is observed in the exciton absorption peak with electric field, coming from the Stark effect where energy levels shift in response to an applied electric field, producing an oscillation in the EA signal as a function of photon energy. The Franz-Keldysh (FK) effect changes the bandgap absorption when an electric field is applied, producing additional oscillations in the EA signal. The bandgap energy can then be determined by the first zero crossing of these FK oscillations. [9]

1.5 Results Overview

As we tried to find what contributes most to the bandgap energies of different materials, I was most involved in the process of taking EA scans, and plotting the results found from those scans to find the exciton binding energies. In this thesis I will describe the method of electroabsorption (EA) that we used to find the exciton binding energies of the hybrid organic-inorganic perovskites and organic semiconductors. I will also discuss the results and implications of the EA data we collected. For the hybrid organic-inorganic perovskites, we observed that the position of the modifier (x = 3-F, 4-F, 2-CF_3, and 2-methyl) on the phenylethylammonium ring seems to effect exciton binding energy

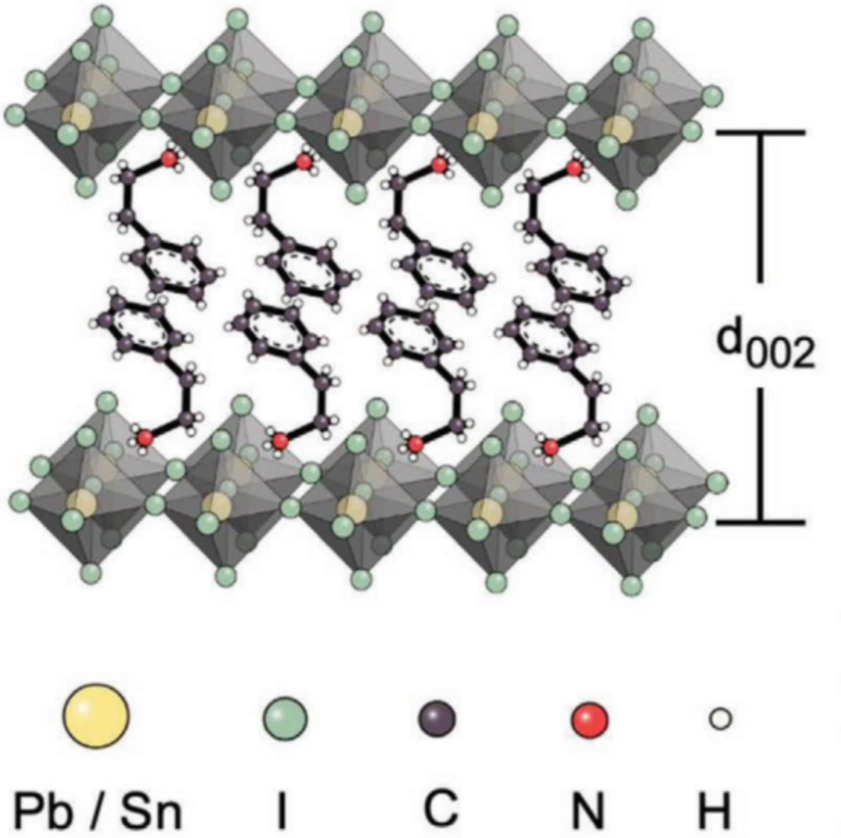


Figure 1.5 A representation of the PEA_2PbI_4 and PEA_2SnI_4 (PESI) crystal perovskite structure with alternating organic–inorganic layers. The inorganic metal-based materials (tin) are the gray octahedra surrounding the organic carbon based materials in-between (also shown in Figure 1.4). The elements listed at the bottom correspond with the colors indicated above them and also correspond with the circles in the PESI structure representation. d002 indicates the spacing in between crystallographic planes. [8]

much more than the modifier itself. For organic semiconductors, EA didn't seem to indicate that there is a charge transfer state.

Chapter 2

Methods

2.1 Electroabsorption Process

Electroabsorption (EA) is used to measure changes in absorption with and without an applied electric field to determine exciton binding energies. When electric field is applied, a red-shift in the ground (1s) exciton absorption peak is observed. This effect is called the Stark effect—where energy levels shift in response to an applied electric field, producing an oscillation in the EA signal as a function of photon energy. The Franz-Keldysh (FK) effect is also observed in EA as seen through changes in the bandgap absorption when an electric field is applied, producing additional oscillations in the EA signal. The bandgap energy can then be determined by the first zero crossing of these FK oscillations after the exciton energy.

To setup EA, we used an optical setup as pictured in Figure 2.1. The spectrometer separates spectral components of light and the lock-in amplifier is used to locate a signal from noise. The cryostat maintains a low temperature for the samples by using a vacuum insulated device (uses a roughing pump, turbo pump, and cryo pump) to keep the samples at very low temperatures. See Appendix A for a list of terms.

2.2 Organic Semiconductors

Based on Wan's research with electroreflectance, we hoped to observe intermolecular charge transfer using electroabsorption (EA) rather than electroreflectance. Electroreflectance is the change of reflectivity of a solid with and without an applied electric field. Wan's research used electroreflectance and observed an intermolecular charge transfer—direct excitations from the LUMO of the donor to the HOMO of the donor—from an energy feature that is lower than normal exciton binding energy in the electroreflectance, which is characteristic of a charge transfer state. [7] We hoped to observe intermolecular charge transfer using electroabsorption (EA) rather than electroreflectance and to study the effects of different ratios of donor vs. acceptor molecules, as well as the effects of different additives.

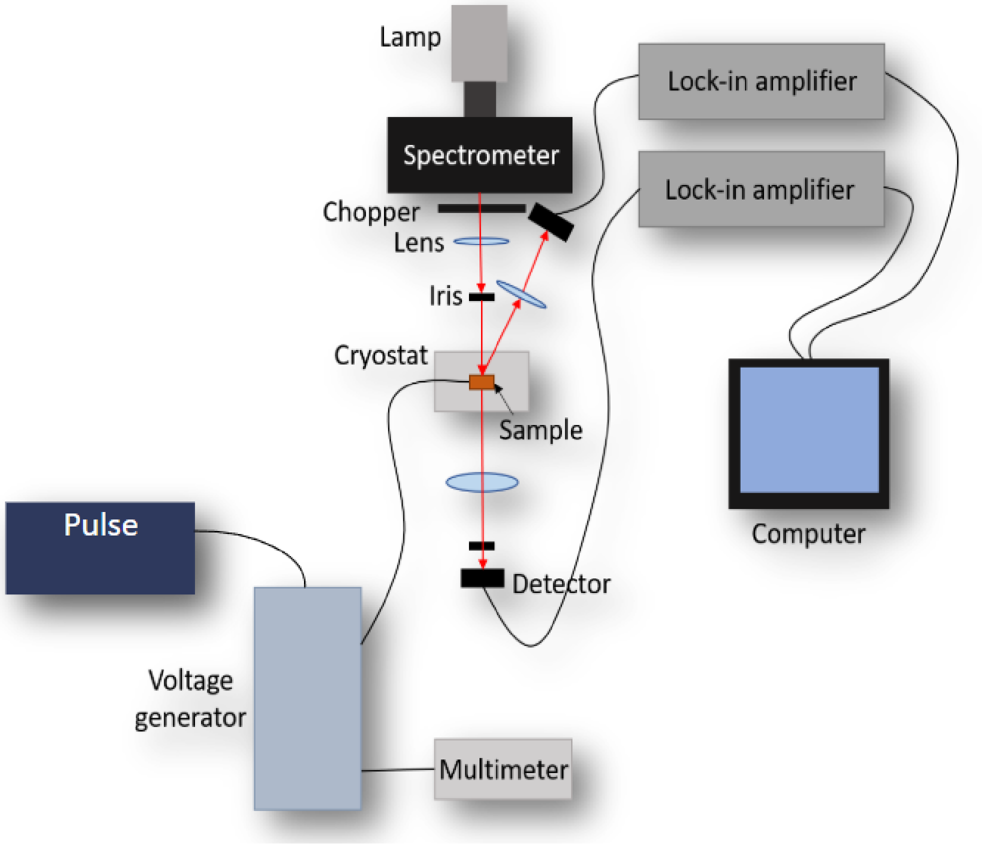


Figure 2.1 Setup for Electroabsorption. The preovskite sample is put in the cryostat and absorption data is collected with an without an applied electric field. Oscillations in the absorption data are seen when an electric field is applied, allowing the band gap energy to be calculated. The detector collecting the reflected beam beam off the sample and the attached lock in amplifier aren't used in EA, but are used in electroreflectance scans to compare against EA scans. Electroreflectance scans should give similar information as EA about exciton binding energy, but can be used to collect data from thicker samples.

Chapter 3

Results

3.1 Organic Semiconductors

Based on Wan's research [7] that used electroreflectance to observe an intermolecular charge transfer, we hoped to see direct excitations from LUMO of donor to HOMO of the donor (also known as intermolecular charge transfer) from electroabsorption. We observed, however, that there was not any evidence of the charge transfer state using our EA technique.

3.2 Hybrid Organic-Inorganic Perovskite Data and Uncertainty

Using phenylethylammonium-x tin iodide (x = 2-F, 3-F, 4-F, 2-CF_3, and 2-methyl), the band gap energy of hybrid organic-inorganic perovskites was found to be impacted the most by octahedral tilt (see Figure 3.1 for a depiction of octahedral tilt). Octahedral tilt is increased most by an increased dielectric constant, which depends on the material added to the octahedral ring, or by the position of the modifying material in the ring (for example 2-F, 3-F, 4-F PESI). The position of the modifier on the ring seems to effect exciton binding energy much more than the modifier itself. The exciton binding energies of the materials added to different positions the phenylethylammonium ring can be seen in Figures 3.2, 3.3, 3.4, 3.5, 3.6, and 3.7.

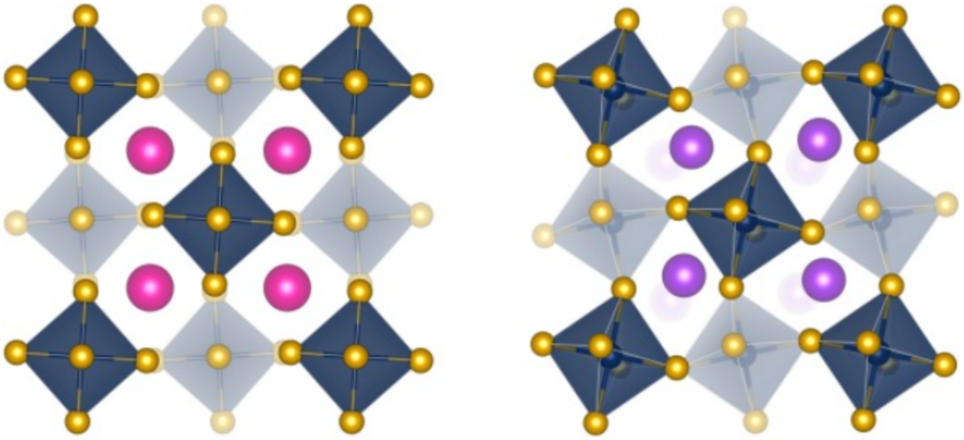


Figure 3.1 Octahedral tilt. Defining the plane of the page to be the x y plane, and the z axis to be into / out of the page, the atoms can rotate clockwise / counterclockwise in the x y plane, as well as sideways in the z axis.

Some experimental techniques that support that octahedral tilt in one of the biggest factors in determining exciton binding energies include ionization energy (IE) and time-resolved terahertz spectroscopy (TRTS). IE is used to measure the amount of energy needed to remove an electron from the perovskite structure. The data shows that more distorted crystal structures, meaning structures that have more tilt in their octahedra, have a blue shift in the IE data. This blue shift, which indicates a decrease in the wavelength of emitted light, corresponds to ionization occurring at a higher energy. [10] The increased ionization energy in more distorted crystal structures supports the claim that when the position of the modifier on the phenylethylammonium ring induces more octahedral tilt, and thereby more disorder in the crystal structure, the exciton binding energy increases. Ionization energy and exciton binding energy are related in that they both refer to the energy needed to separate an electron or electron-hole pair from a molecule or solid. TRTS is used to investigate how octahedral tilt and crystal structure distortion impact charge-carrier mobility in different time regimes. TRTS uses a pump-probe technique and shows that there are shorter charge carrier lifetimes with more energetic pumps. This indicates that crystal structures with more disorder, and therefore more octahedral tilt, will have smaller charge carrier lifetimes which corresponds to more tightly bound charge carriers and a higher exciton binding energy. [10]

Octahedral tilt appears to affect binding energy, with a transition observed around 100-150 K—potentially due to phase transitions or other effects. [10] Tilt can be observed in 2 directions: θ (in-plane distortion) and ϕ (out-of-plane distortion). Table 3.8 shows a summary of the EA results for each material. The EA scans of phenylethylammonium-x tin iodide where x = 2-F, 3-F, and 4-F (measured at 16K or 17K) give insight into how exciton binding energy changes based on the position of fluorine on different parts of the phenylethylammonium ring. In tin-based compounds there is more disorder so the electroabsorption signal is not as clear in comparison to other metals. [8]

The EA spectra for phenylethylammonium-2-F tin iodide and phenylethylammonium-3-F tin iodide show similar exciton binding energies (see Figure 1.5 for a visual representation of where the modifier will be added to the PESI structure, and see Figure 1.4 for the numbered positions on the phenylethylammonium ring). Their similar positions in the perovskite structure could give similar spacings between the crystallographic planes, and thus give similar exciton binding energies. In contrast, the exciton binding energy for phenylethylammonium-4-F tin iodide decreases. The 4th position on the phenylethylammonium ring is the position that can cause the largest spacing between the crystallographic planes because it is the position furthest from the tin iodide plane layers, and will increase octahedral tilt and disorder in the perovskite structure. The increased octahedral tilt and spacing between crystallographic planes will decrease exciton binding energy because the energy required to separate the electron and hole and effectively make them free charge carriers. Free charge carriers are electrically charged particles that are not bound to an atom and are free to move within a material. The exciton binding energy would be less with there already being more space between crystallographic planes.

We can also see that different molecules (F, CF_3, and methyl) added to position 2 on the phenylethylammonium ring change the exciton binding energy. For these materials, we can see that the exciton binding energies are noticeably different at the same position on the phenylethylammonium ring, most likely because of the different dielectric constants for each material.

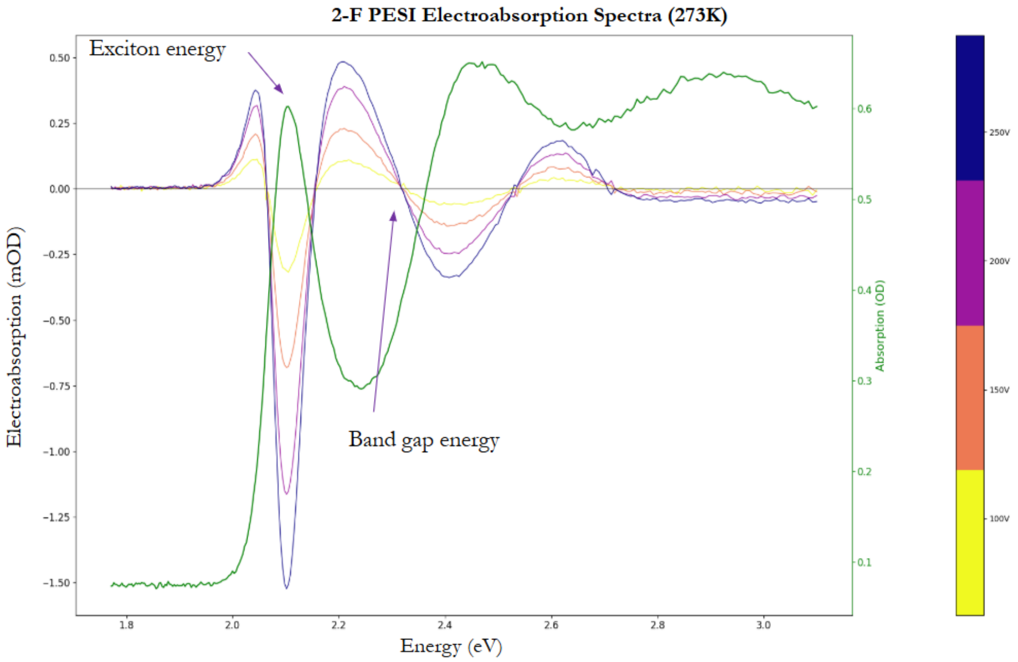


Figure 3.2 Electroabsorption and absorption spectra for 2-F PESI (273K). The green line is the absorption spectra, and the colored lines correspond to the electroabsorption spectra taken at different voltages as indicated by the bar to the right of the graph. The exciton energy is found at the first (1s) peak of the green absorption line. The band gap energy is found at the first zero crossing in the electroabsorption spectra after the exciton energy. Exciton binding energy is the exciton energy subtracted from the band gap energy.

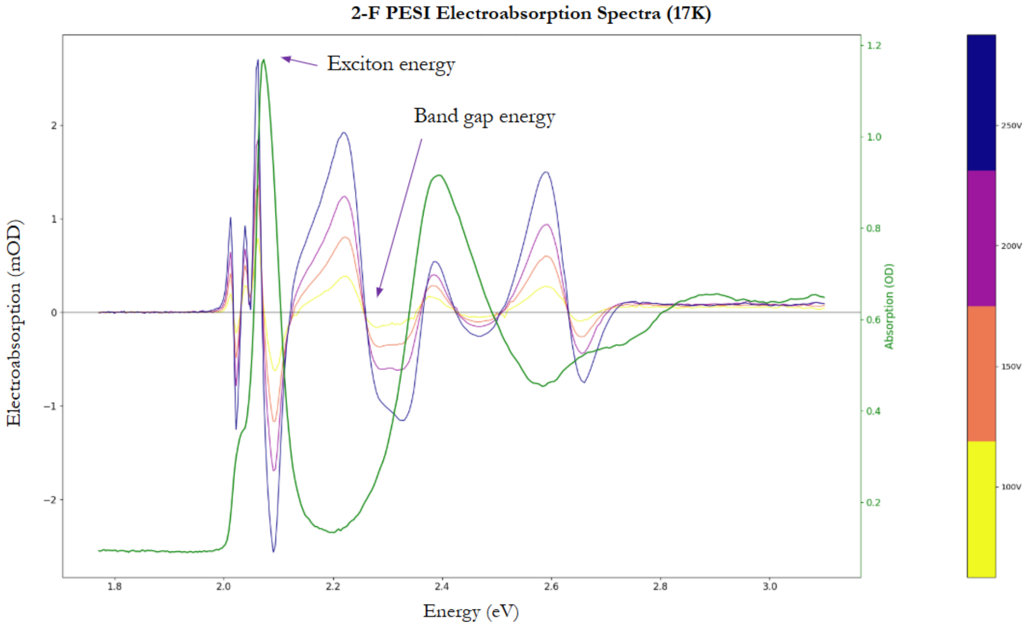


Figure 3.3 Electroabsorption and absorption spectra for 2-F PESI (17K). See Figure 3.2 for an explanation of the figure components.

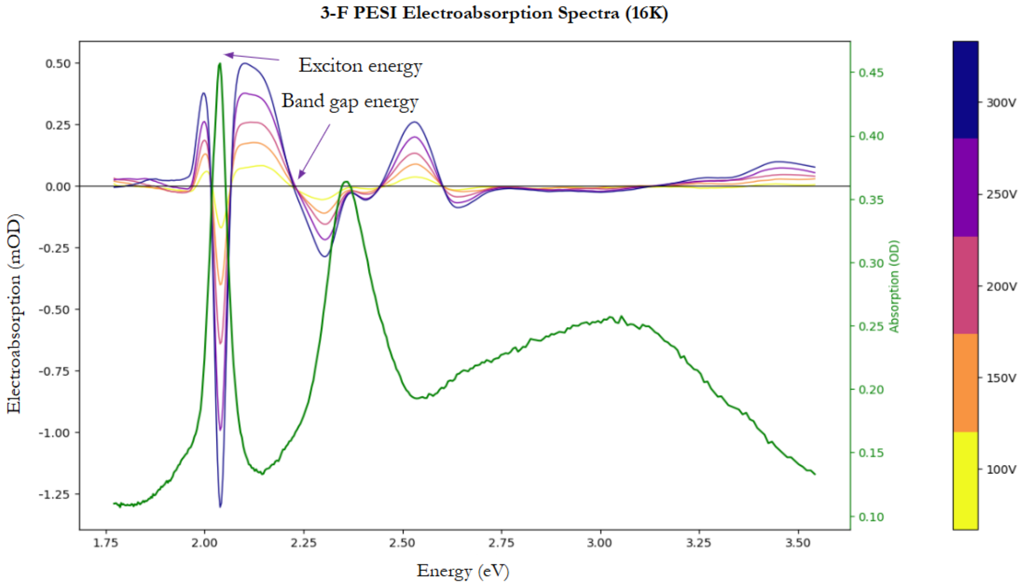


Figure 3.4 Electroabsorption and absorption spectra for 3-F PESI (16K). See Figure 3.2 for an explanation of the figure components.

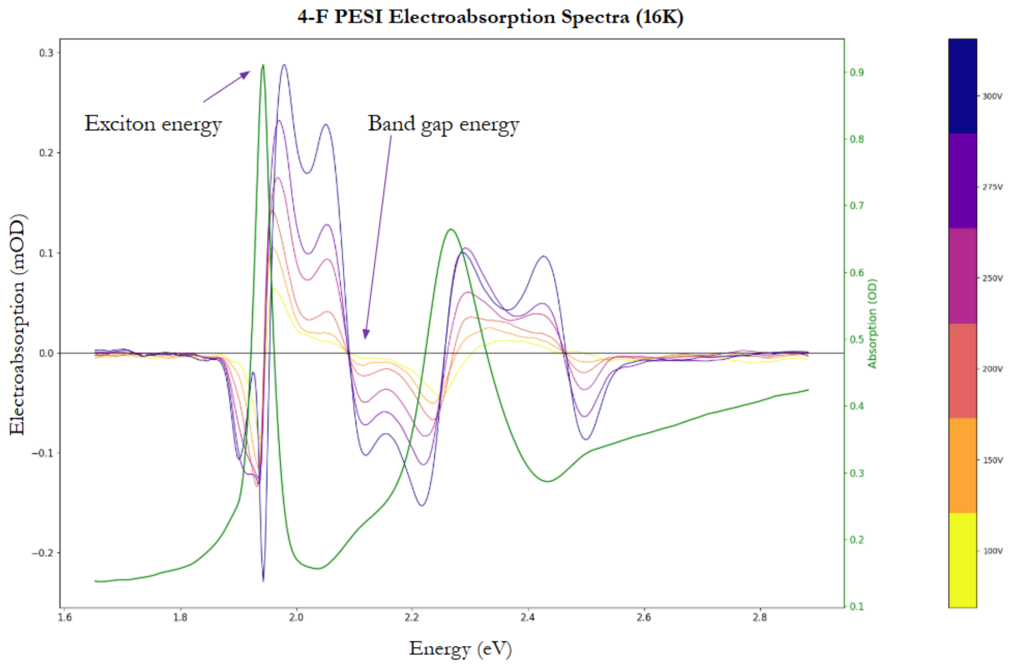


Figure 3.5 Electroabsorption and absorption spectra for 4-F PESI (16K). See Figure 3.2 for an explanation of the figure components.

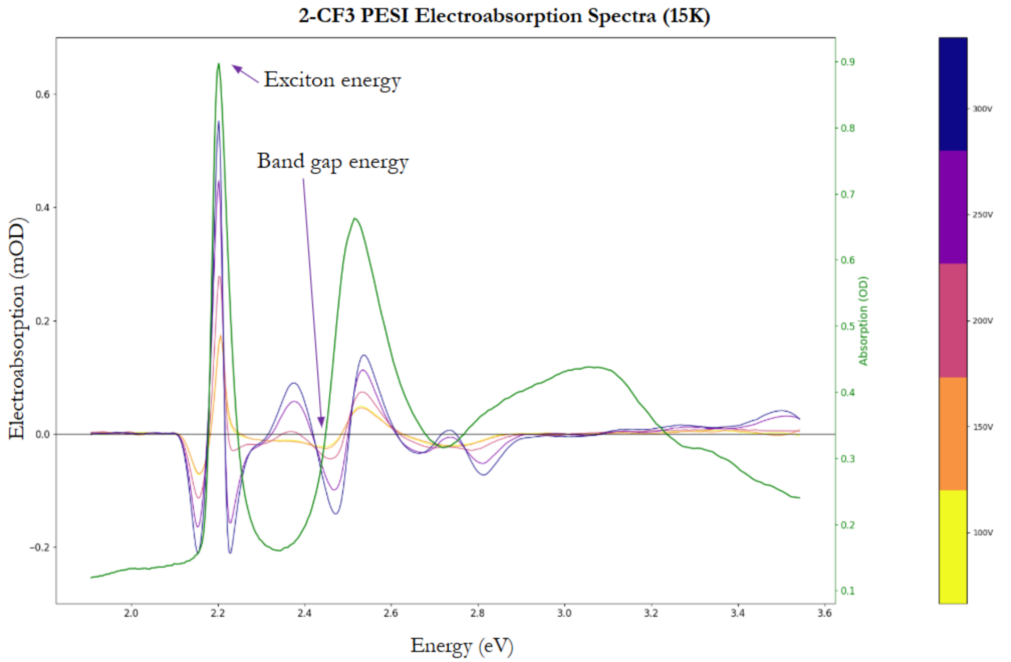


Figure 3.6 Electroabsorption and absorption spectra for 2CF3 PESI (15K). See Figure 3.2 for an explanation of the figure components.

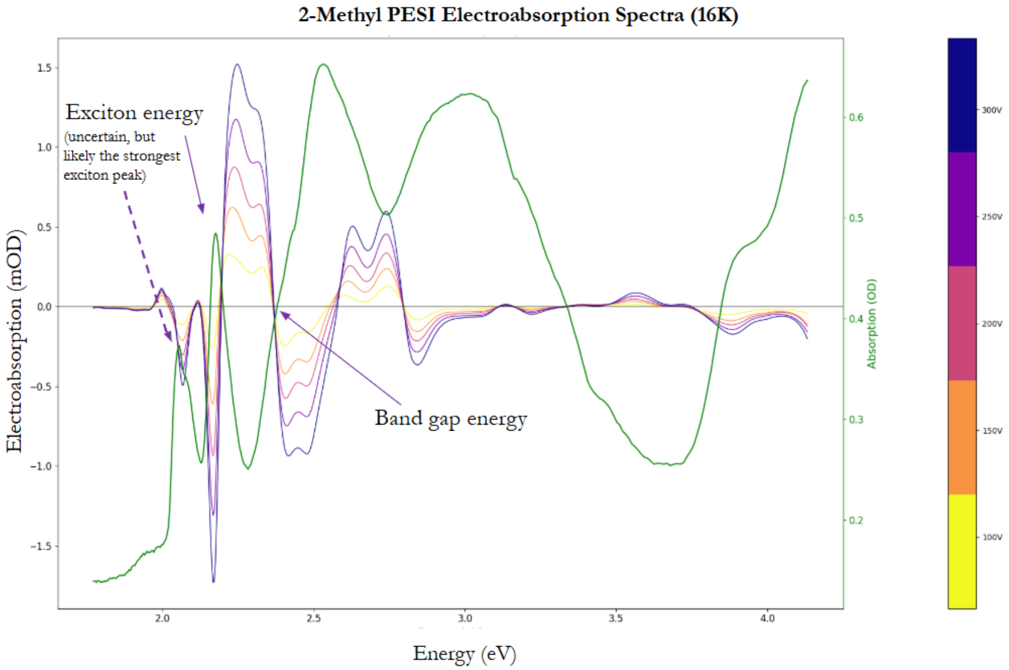
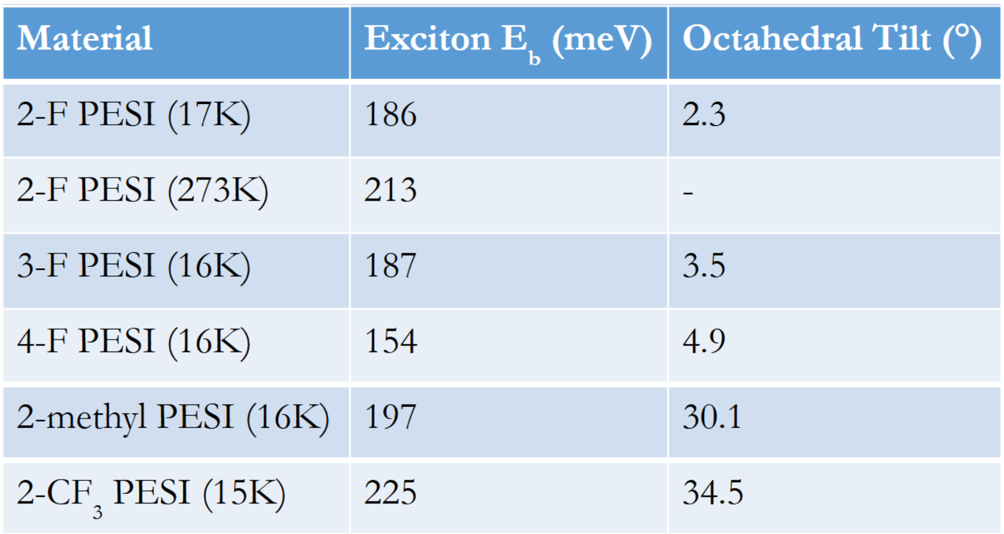


Figure 3.7 Electroabsorption and absorption spectra for 2-Methyl PESI (16K). See Figure 3.2 for an explanation of the figure components.



Material	Exciton E_b (meV)	Octahedral Tilt (°)
2-F PESI (17K)	186	2.3
2-F PESI (273K)	213	-
3-F PESI (16K)	187	3.5
4-F PESI (16K)	154	4.9
2-methyl PESI (16K)	197	30.1
2-CF_3 PESI (15K)	225	34.5

Figure 3.8 Exciton binding energies summary. The exciton binding energies (Eb) are calculated by subtracting the band gap energy from the exciton energy. Absorption and electroabsorption data from exciton energy and band gap energy is found in Figures 3.2, 3.3, 3.4, 3.5, 3.6, and 3.7.

Chapter 4

Discussion and Conclusion

4.1 Organic Semiconductors

The electroabsorption (EA) data does not show an indication of a charge transfer state. Further research directions include verifying the electroreflectance data from Wan's research that indicate a charge transfer state from electroabsorption. [7]

4.2 Hybrid Organic-Inorganic Perovskite Data and Uncertainty

The position (2, 3, or 4) of the modifier (F, CF_3, methyl) on the phenylethylammonium ring seems to effect exciton binding energy much more than the modifier itself because of octahedral tilt. Octahedral tilt can increase based on its position on the phenylethylammonium ring because position can increase the spacing between the layers. Octahedral tilt can also increase depending on the material because the material can change the dielectric constant which can increase octahedral tilt. Materials with a greater octahedral tilt seemed to have a greater exciton binding energy.

Defining the degree of disorder (disorder meaning difference from the chemical in an octahedral alignment instead of having octahedral tilt) has also been a challenge. One example of this is in

2-CF_3 which looks like it has more disorder because of octahedral tilt, but 2-methyl looks more disordered if it is quantified through the Sn-I-Sn bond angle variance—disorder in these materials being defined as deviation from either an ideal octahedral network (for example phenylethylammonium with no added modifier).

The electroabsorption findings alone are not enough to conclude that the position of the modifier affects exciton binding energy more than the modifier itself. To conclude this with more certainty, we can use electroabsorption on more of a variety of materials corresponding with the same modifiers at different positions on the ring. Other experiments like ionization energy and time-resolved terahertz spectroscopy also help support this conclusion with more certainty.

Appendix A

Experimental Concepts and Definitions

Band Gap Energy (Eg) is the amount of energy it takes to excite an electron from the valence band to the conduction band.

Cryostat maintains low temperatures using a vacuum insulated device (uses a roughing pump, turbo pump, and cryo pump) to make the samples at very low temperatures.

Electroabsorption (EA) is the difference of the absorptions with the electric field on and with it off. Introducing an electric field to an metal-halide-perovskite sample shifts its absorption spectrum allowing an accurate determination of a sample's exciton binding energy (Eb). First, a red-shift of the 1s exciton peak is observed because of reduced electron and hole quantum well energy levels, known as the Stark effect. Secondly, the Franz-Keldysh effect results in absorption below the bandgap and oscillations across the x-axis afterwards. The bandgap energy is given by the first x-axis crossing of these oscillations.

Exciton is a bound state between a photo-excited electron and the positive hole it leaves behind.

Exciton Binding Energy is the energy required to dissociate an excited electron–hole pair into free charge carriers. The exciton binding energy, can be measured as the difference in energy between band gap energy and the 1s exciton peak, where a low exciton binding energy (Eb) indicates easier separation of exciton components into a free electron and hole, and a high Eb indicates easier recombination of the electron and hole because of proximity

Franz-Keldysh Effect is a change in optical absorption by a semiconductor when an electric field is applied.

GE Varnish is used to glue the perovskites to the substrate. We use it because it works well at low temperatures and pressures, and takes a small amount of time to outgas from the vacuum system. GE varnish can be removed with isopropyl alcohol or acetone.

Interdigitated Electrodes are made of conductive material deposited onto a substrate in an interlocking comb-like pattern.

LabVIEW has the code for the equipment controls.

Lattice is a synonym for the framework of a crystalline structure.

Lock-In Amplifier is used to locate a signal from noise.

Metal Halide Perovskites (MHPs) are any material with a crystal structure following the formula ABX_3. Perovskites have low formation energy, rapid anion exchange, and ease of ion migration, which enables the rearrangement of their overall appearance by self-assembly or regrowth. There are 2 types of Perovskites: Dion-Jacobson, Ruddlesden-Popper.

Neutral Density (ND) filter controls the intensity of the laser light passing trough at that point without changing the power of the light coming from the laser. We use ND filters in the lab to make sure that when we are aligning the lasers that the power is below 1mW. It doesn't change the direction of the laser light.

Nitrogen Box is used because the perovskite samples may rust or corrode if oxidized or if they get water on them, so we put them in a container full of Nitrogen to prevent that from happening.

Oscilloscope displays the voltage signal and shows how it changes over time.

Photon is an electric field traveling through space. Photons have no charge, no resting mass, and travel at the speed of light.

Proportional-Integral-Derivative (PID) Controller is used in our lab to control the temperature of the smallest cryostat. A PID reads the sensor (like the temperature sensor used in our lab), and by summing the proportional, integral, and derivative responses at that level, it adjusts the power of the system to get the desired output (like how we keep our system at a specific temperature).

Power Meter is used to measure how much power is in the laser beam so we can keep it at a level that isn't as dangerous for our eyes while we are aligning the optics. If the laser power is too high we can decrease the voltage in the laser beam, or add ND filters to the laser beam.

Semiconductors are materials with bandgap energies that fall between those of conductors and insulators, with average values of 1.0 – 3.0 eV. If the bandgap energy is small enough that thermal excitation of a semiconductor allows a small fraction of the electrons to "jump" from the valence band to the conductance band, giving rise to a small conductivity which can then be manipulated by an applied electric field. When an electron leaves the previously full valence band, it leaves behind a "positive" hole which can be occupied by a different electron, leaving another hole. This can create an electric current, or conduction. An N-type semiconductor has one too many electrons, and a P-type needs one more.

Spectroscopy is the study of the absorption and emission of light and other radiation by matter.

Stark Effect is the shifting and splitting of spectral lines of atoms and molecules due to the presence of an external electric field. It is the electric-field analogue of the Zeeman effect, where a spectral line is split into several components due to the presence of the magnetic field.

Substrate is an underlying surface used in photolithography where light transfers a pattern onto it. It is typically a silicon wafer.

Vacuum Grease fills in the cracks in a vacuum system to get rid of leaks.

Zeeman Effect is similar to the Stark effect but is the response to a magnetic field instead of en electric field.

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