

3D Printed Diode Array Spectrophotometer
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May , 2015
Chemistry 522
Instrumental Analysis

Introduction:

A typical photo diode array (PDA) consists of a series of diodes arranged in order for each diode to measure one single wavelength. ¹ Thus, the typical PDA has a high wavelength specificity when used to obtain transmittance or absorbance spectra. ¹

A charged coupled device (CCD) is made on a silicon wafer and it can be used as a spectral analysis tool because light that hits the silicon of the CCD generates

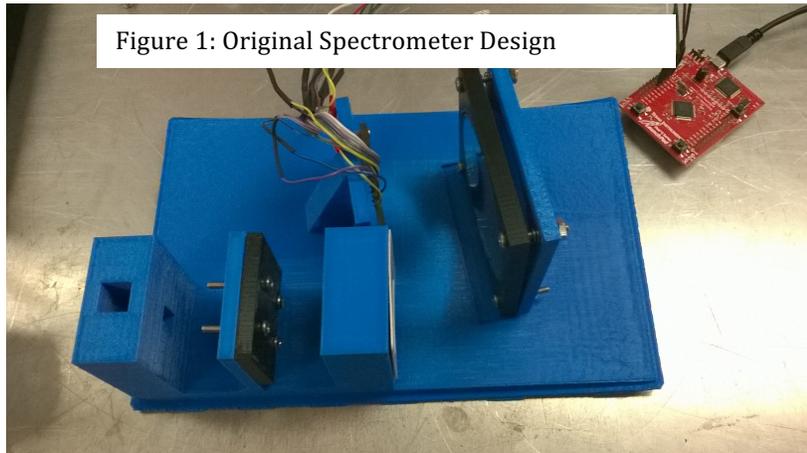


Figure 1: Original Spectrometer Design

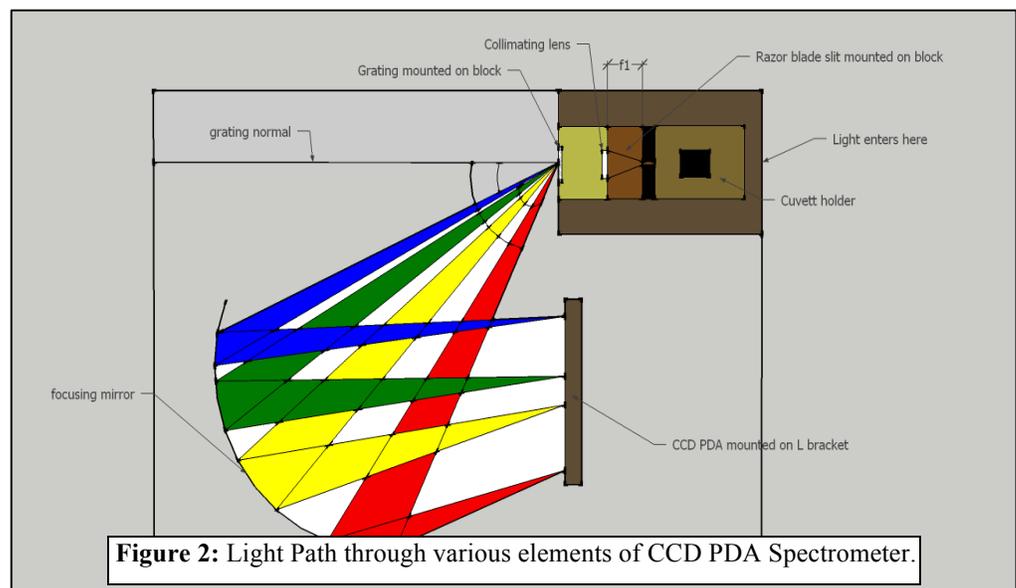
electrons, which are collected by the pixels of the CCD, and the amount of electrons generated by the light are read by the controller attached to the CCD. ¹

CCD-Spectrometers are relatively simple to calibrate, and such a process can be carried out using a polynomial and a calibration light source. ² Because of the ease of use and low cost of CCD-Photo Diode Array, this instrument is utilized in industry in several ways, such as inspection of food products or even the inspection of weld defects. ² In the case of a small scale CCD-PDA, this ease of set up and cost does not diminish, as there are various detailed methods by which to calibrate and even create such an instrument. ^{3,4}

Moreover, because of the availability of microcontrollers, methods by which to program CCD-PDA's and transfer their information to a suitable GUI (Graphical user Interface) abound. ⁴ All that is required for the construction of a spectrometer that utilizes a CCD-PDA would be a diffraction gradient, a light source, and a CCD-PDA at the correct angles. ⁴ However, with the addition of a focusing lens, a focusing mirror, and a slit, a high degree of resolution might be obtained using a small scale CCD-PDA. During this

experiment, the construction of a Diode Array Spectrometer using a CCD Photodiode array was carried out. This design was based on a previous design, displayed in **Figure 1**. The assembly of this instrument involved both hardware and software components, which were synced together to form a working spectrometer, which was then used to obtain spectral measurements. These spectra were displayed on a computer screen within the visible range of 400-700 nm. The design of this project was created with reproducibility as a primary goal and therefore a 3D printable base was utilized in order match precisely the necessary angles and distance measurements between the focusing mirror, grating, and CCD PDA.

The base provided a high degree of reproducibility in line with the original design, and provided a more expedient



and less labor-intensive construction method. The base design also provided for an unobstructed light path through which the proper reflections of the light were allowed to proceed, as is displayed in **Figure 2**. The construction also utilized the coding software Energia and Processing (both based on Java), which, allowed for the visualization of the spectrum in a GUI (graphical User Interface). This GUI allowed for the utilization of the Spectrometer to take Absorbance, Transmission, and a Series of spectra from the CCD PDA, and clearly displayed the spectra on screen, allowing for further analysis of the spectra.

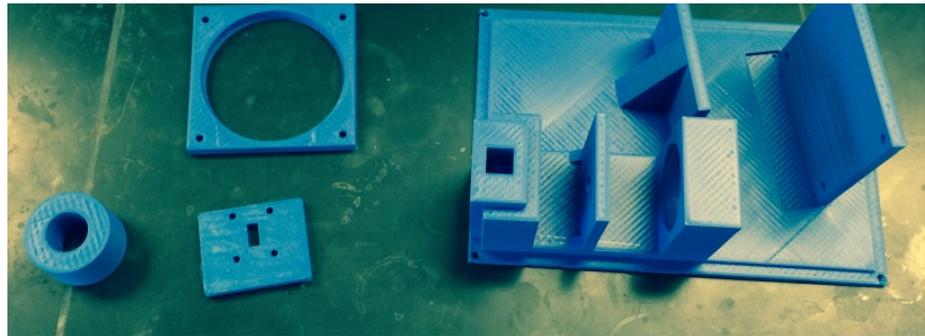
Methods and Materials:

Materials:⁴

The spectrometer was made with a 3D printed base, a holographic diffraction grating film (1000 lines/mm), an acetate collimation slit (0.09mm), a concave spherical mirror (75mm diameter x 7mm thick x 75mm focal length), a condenser lens (20.4 x 25mm focal length), a one element photodiode array (PDA) sensor (TSL1406R 768 X), a Tiva C series Launchpad Microcontroller (to send data from PDA to computer), a 3D printed base, insulated small electronic wire (various lengths), eight #8 screws, two #2 screws, 4 screw

springs, a cardboard box cover, a halogen lamp with mercury, a halogen lamp without

Figure 3: 3D printed base, lens adapter, mirror holder, and slit holder.



mercury, a plastic cuvette, a Windows compatible computer, and the software programs Energia and Processing. For the construction of the instrument, the following tools were utilized: sandpaper, a small metal file, bubble wrap, a screwdriver, a 2 inch long metal needle with a wooden handle, a lighter, black electrical tape, metal wire (not for circuit), and a pair of scissors.

Pre - Lab:⁴

The pre-lab set up for this experiment involved creating the 3D base of the spectrometer with a 3D printer. Using a 3D printer and the material PLA (Polylactic Acid), the base was printed, along with 3 smaller separate parts: the lens adapter, mirror holder, and slit holder (see **Figure 3**). The original 3D design was redesigned in order to reduce the amount of 3D plastic required to make the print and in order to cut down on

the time required to make the print, but no actual alterations were made to the distances or the angles of the design. Ordering each part previously listed was also essential to the pre-lab, and the orders were placed weeks in advance of the first lab day. After arrival in the mail, each element was carefully inspected for defects and the serial number of each part was confirmed in order to assure that each part received matched what was ordered.

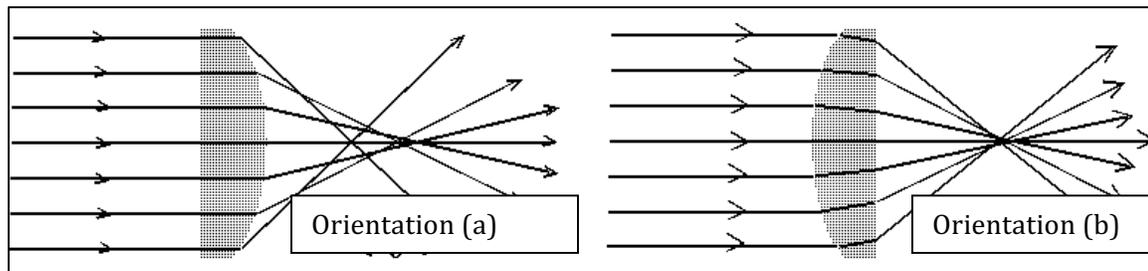
Procedure:

The first set up involved sanding down both the lens adapter and the inside slot surface on the base that the lens adapter fit into. Sand paper and a metal file were used for the sanding. **Figure 4** displays the lens slot, **Figure 5** displays the lens adapter, **Figure 6** displays the Sanding process, and **Figure 7** displays a failed attempt to fit the two parts together before the sanding was complete, which resulted in the parts becoming jammed together. When attempting to remove the adapter from the slot, the slit stand on the base broke (cleanly) off of the base, as displayed in **Figure 8**, which also displays the (see end of report for **Figures 4-8**). This was fixed easily due to the nature of the 3D printed base, which was constructed in a grid of lines that allowed for wooden toothpicks to be placed inside the slit holder and the base. The re-joining of the two broken sections back together is displayed in **Figure 9**, (see end of report). The slit holder was screwed onto the slit base using 4 screws and 4 nuts, and the acetate slit was placed in between the slit holder and the base stand.

Next the collimating lens was affixed to the lens adapter, and only handled with gloves to avoid getting oils on the lens (which can cause long term damage to the lens unless wiped off immediately). There was a size difference between the lens and the lens holder because of the expansion of the plastic that occurred after as the plastic cooled after the being printed. In order to account for the size difference between the lens and the adapter as is displayed in **Figure 10**, the edges of the lens were tapped with thin

sections of black electrical tape (which made it fit snugly into the lens adapter), and then the lens was tapped into the adapter with black electrical tape, as displayed in **Figure 11** (see end of report for **Figures 10-11**). This black electrical tape also served the purpose of absorbing (as opposed to reflecting) any spare light that doesn't hit the lens after passing through the slit. The collimating lens is a plano-convex lens, and so needed to be affixed to the base in a certain way as to properly focus light toward the slit. **Figure 12** displays the proper formation of the lens in reference to the light source, which was orientation (b).

Figure 12: The spread in the focal length for the plano-convex lens with two possible orientations. Orientation (a) displays a wider spread than orientation (b). Orientation (b) was used for this experiment.



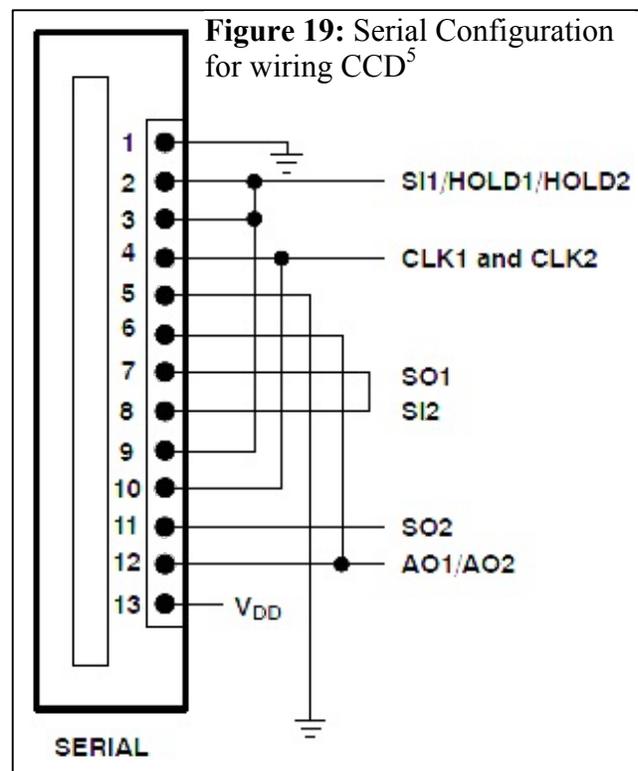
The Holographic diffraction grating film was attached to the end of the lens holder stand with black electrical tape.

The focusing mirror was then affixed to the base, using 4 screws and 4 screw springs, taken from Pilot G-2 pens. The screw springs were placed to allow for the fine adjustment of the focusing mirror in its holder after placement, with the concave portion affixed inward, in order to properly reflect the light. A layer of padding (bubble wrap) was placed behind the mirror in the holder in order to protect the back of the mirror (avoid rubbing the mirror against the base) and to push the mirror out and in place against the mirror holder. **Figure 13** displays the focusing mirror, **Figure 14** displays the mirror holder with the four screws and four screw springs attached, **Figure 15** displays the

mirror, the mirror holder, and the padding behind the mirror, and **Figure 16** displays the mirror attached to the base (**Figures 13-16** are attached at the end of the report).

The CCD Photo-Diode Array was then attached to the base, though a problem presented itself when the screw holes already present in the 3D printed base were too far apart to match up to the screw holes on the CCD. Because the 3D base was made of plastic (PLA), a new hole was punched on the proper level on the base using a 2 inch metal needle with a wooden handle and a lighter flame to heat the metal. The heated metal melted through the PLA base and, because the hole was so thin, wire was threaded through the CCD PDA and the new base holes in order to affix it to the base, as seen in **Figure 17** and **Figure 18** (attached at the end of the report).

The CCD PDA was then wired according to the serial configuration diagram on the CCD's data sheet (located online), as seen in **Figure 19**.⁵ This was done with various lengths of wire, and black electrical tape. The wiring in progress is seen in **Figure 20**, and is



displayed fully wired and attached to the base in **Figure 21** (**Figures 20-21** are attached at the end of the report). For this experiment, the SO2 connection is not necessary. The wiring of the CCD to the Tiva TM4C123 Launchpad microcontroller was carried out and the power (pin 13) was connected to the 5V pin on the Tiva, the ground pin (pin 1 & 5) was connected to ground on the Tiva, the SI1/HOLD1/HOLD2 pin (pin 2, 3 & 9) was connected to PD_1 on the Tiva, the CLK1/CLK2 connection (pins 4 & 10) was

connected to the PD_0 on the Tiva, and the AO1/AO2 connection (pins 6 & 12) were connected to PD_2 on the Tiva. **Figure 22** displays the wiring of the CCD to the Tiva microcontroller (attached at the end of the report). A cardboard box of an appropriate size was cut so that there was a slot through which the light source could pass through only the cuvette holder. At this point the hardware was completely set up, and the software programming began. **Figure 23** displays the completely set up CCD-PDA instrument without the cardboard box cover, **Figure 24** displays the cardboard box cover over the CCD-PDA with the slot cut out for the light (**Figures 23-24** are attached at the end of the report).

The software installation and set up was originally attempted on a macintosh computer with OS X operating system, and this proved to be problematic in various ways, so the software installation was set up with a Dell computer with a Windows 7 operating system instead. The following set up outlines the Windows 7 installation process. First, the development system Energia was downloaded, and the drivers were downloaded onto the computer.⁶ The drivers were In-Circuit Debug Interface drivers (ICDI) and allowed the program to communicate with the Tiva microcontroller, just like a driver on a computer might communicate with a mouse or a keyboard. The driver was installed according to the Driver Installation Instructions, located online.⁸ The coding necessary to program the Tiva microcontroller to take data from the CCD was loaded into Energia and the program was run. Once Energia signified that the program had successfully loaded onto the microcontroller (no error signs were displayed), the development system Processing was downloaded. Processing did not require any drivers, but there were several libraries (folders of code files to be referenced by the main program) that had to be loaded into the Processing “sketchbook” (which is essentially a file folder that contains all of the code necessary to run a program) before the code could

operate the necessary functions. The code was loaded and the program was run. Processing worked in conjunction with the program already loaded onto the microcontroller by Energia to operate a GUI, or a Graphical User Interface, which allowed for the interaction with the Tiva microcontroller to take data through the buttons and commands displayed in the Graph. **Figure 25** displays this GUI without any of the possible programs running (attached at the end of the report).

Then an attempt was made to calibrate the GUI by taking a blank from the Microcontroller (by hitting the blank command on the GUI), with various blanks taken at each different setting - absorbance blanks, transmittance blanks and series blanks - using two different halogen lamps, one with mercury and one without. The blanks were taken with nothing in the cuvette holder, with the empty plastic cuvette in the cuvette holder, and with DI water in the plastic cuvette in the cuvette holder.

Results and Discussion:

Hardware:

The hardware was successfully set up successfully. The wiring of the CCD to the Tiva C series Launchpad microcontroller was proven to be successful when there was a measurable response from the CCD to different light intensities.

Software:

Because of the difficulties involving the set up with the Macintosh OS X software, using Windows software is currently the only feasible method by which to obtain and display the spectra through a GUI using Processing and Energia, despite the extra step of having to install the drivers on the Windows system (which is a process that is not necessary on Macintosh). The problem with OS X is not that it cannot run energia or processing, but that the code for operating the CCD-PDA instrument utilized in this experiment is not compatible with Macintosh for various reasons related to the coding, which are unclear even to the original coding team.^{4,7}

Spectrum on GUI:

Figure 26 displays the experimental set up from which the following spectra were taken using either the halogen bulb with mercury or without mercury, which were simply exchanged in the silver lamp shown (attached at the end of the report). The attempt to take a blank in order to calibrate the instrument was meant to begin with a spectra taken from an transmittance of halogen light, which was then meant to be saved as a file of each wavelength vs the pixel on the CCD that absorbed the wavelength.⁴ Using excel, these data points were then meant to be run through a calibration polynomial, in this case a 3rd-degree polynomial, whose coefficients would have been obtained through the use of a calibration lamp, which in this case would have been a spectrum of a white halogen light.² The wavelength peaks that correlated were meant to be matched up to the blank

file, and that blank file was then to be manually saved as the blank in the internal data files of the GUI program.⁴ Instead of this process, all of the spectra that were taken were blanks of absorbance, transmittance, and series, and were taken in an attempt to display that the instrument is capable of taking data and displaying this data in a graphical format.

Much of the initial absorbance blanks were problematic at first, with no data at certain sections of the spectra. **Figure 27** displays an absorbance blank taken with no cuvette, and displays spectra absorbance problems. This lack of useable data on the high end was caused either by the angle of the mirror in the spectrometer, so the mirror was adjusted and **Figure 28** was taken, which displays the same conditions as Figure 27, but with the mirror adjusted to curve more to the right than the previous orientation. These runs were all run at an integration time of 100 ms, but the integration time was increased to 1000 ms and more concise spectra were taken (less outliers). Taking this sort of data two times in a row proved problematic as well at first, but after several runs and several adjustments to the focusing mirror, **Figure 29** was the first blank spectra to display a full spectrum of absorbance after the blank run before it did the same thing (with a 1000 ms integration time). **Figure 30** displays the absorbance peak with the halogen with mercury, which was problematic because of the intensity of the light, but after increasing the lights distance from the cuvette holder hole, the spectra in **Figure 31** was taken. **Figure 32** displays the operation of the transmittance blank with 1000ms integration, the halogen light with mercury and with the plastic cuvette (no water). **Figure 33** displays the same parameters as **Figure 32**, but with the addition of DI water. **Figure 32** provides very shaky data, while **Figure 33** provides more linear, smooth peaks. The difference in stability in the two might be brought on by the lower intensity of the light when it must pass through a water filled cuvette in **Figure 33**. **Figure 34** displays the functionality

of the series command in the GUI, and has parameters of 100 integration (msec) with DI water. This is a set of three blanks spectra – two at the higher intensity with the halogen light closer to the spectrometer – the middle intensity spectra with the halogen lamp further away from the spectrometer – and the lowest spectra even further from the spectrometer. The decrease in peak intensity and resolution as the light source gets further away from the spectrometer indicates the sensitivity of the PDA to the light.

References:

1. "CCD Spectrometers 101" <https://www.cigem.ca/pdf/CCDSpectrometers101.pdf> (date accessed: May 1, 2015).
2. Mirapeix, J.; Cobo A.; Cubillas, A.M.; Conde, O.M.; Lopez-Higuera, J.M. "In-process automatic wavelength calibration for CCD-spectrometers." Proc. of SPIE Vol. 7003.
3. Perret, E.; Balmer, T.E. "A High-Precision Calibration Method for Spectrometers." <http://www.americanlaboratory.com/914-Application-Notes/1596-A-High-Precision-Calibration-Method-for-Spectrometers/> (accessed May 3, 2015).
4. Hickman, B.; Summers, J. "3D Printed CCD Spectrometer (WheTrometer 3.0)." <http://publiclab.org/notes/bhickman/08-27-2014/3d-printed-ccd-spectrometer-wheetrometer-3-0> (accessed March 5, 2015).
5. "TSL1406R, TSL1406RS 768 × 1 Linear Sensor Array with Hold." <http://www.digikey.com/product-detail/en/TSL1406RS/TSL1406-RS-ND/3095065> (accessed May 5, 2015).
6. "Guide to the TM4C123 LaunchPad (EK-TM4C123GXL)" http://energia.nu/pin-maps/guide_tm4c123launchpad/ (accessed April 20, 2015).
7. Summers, J. "Getting Your Wheestat Set Up" <http://publiclab.org/notes/JSummers/07-04-2014/getting-your-wheestat-set-up> (accessed April 20, 2015).
8. "Stellaris® In-Circuit Debug Interface (ICDI) and Virtual COM Port" <http://www.ti.com/lit/ml/spmu287b/spmu287b.pdf> (accessed may 5, 2015).

Figure 4: Lens slot (during sanding process) **Figure 5: Lens adapter (during sanding)**

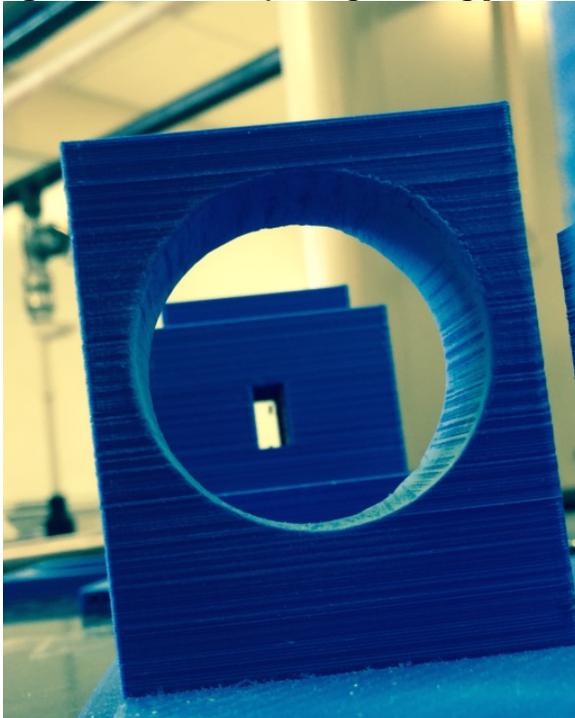


Figure 6: Sanding in progress.



Figure 7: Lens adapter and slot stuck together.

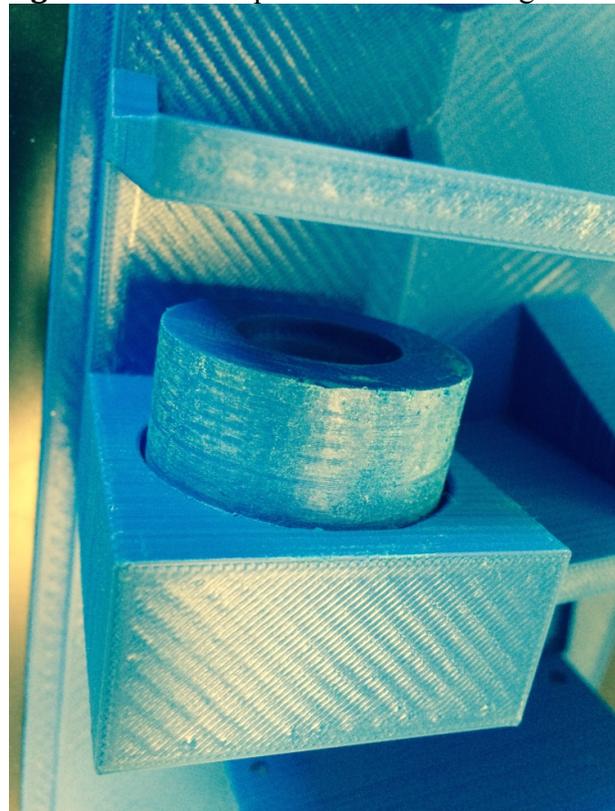


Figure 8: Broken slit holder

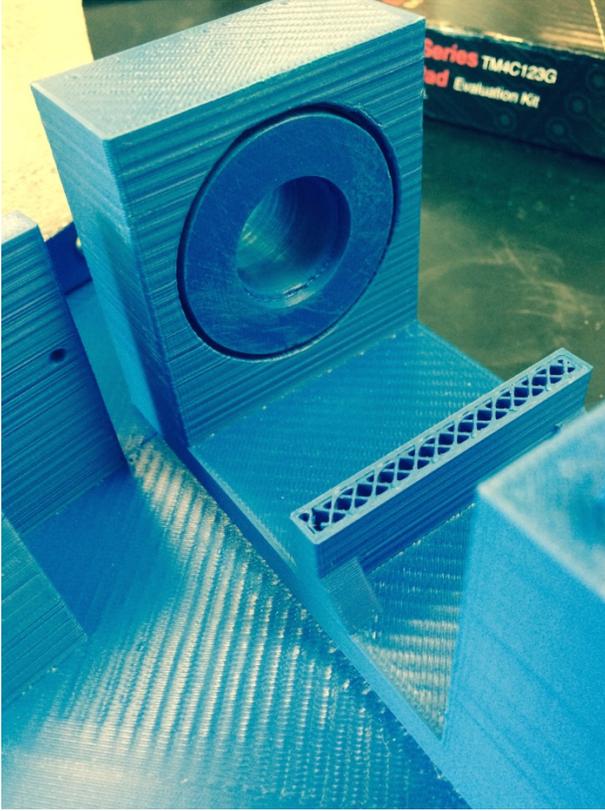


Figure 9: Joining slit holder to base with toothpicks

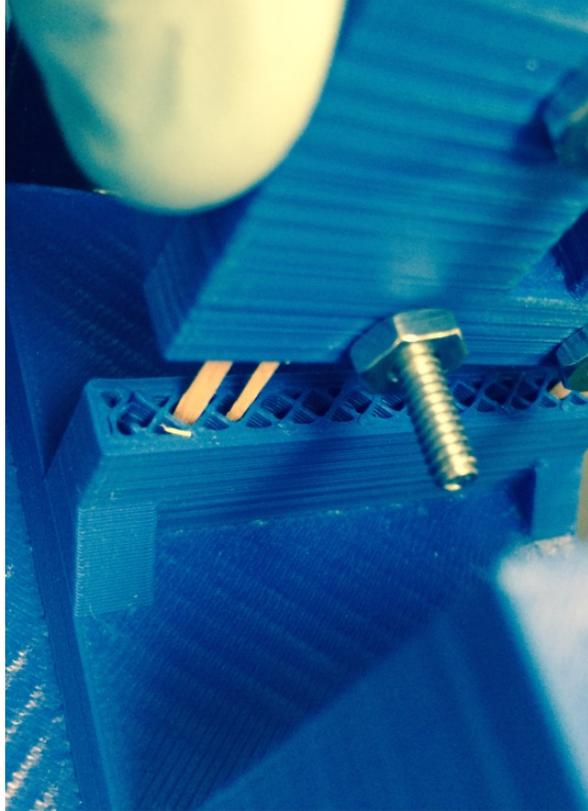


Figure 10: Lens is too small for Adapter.

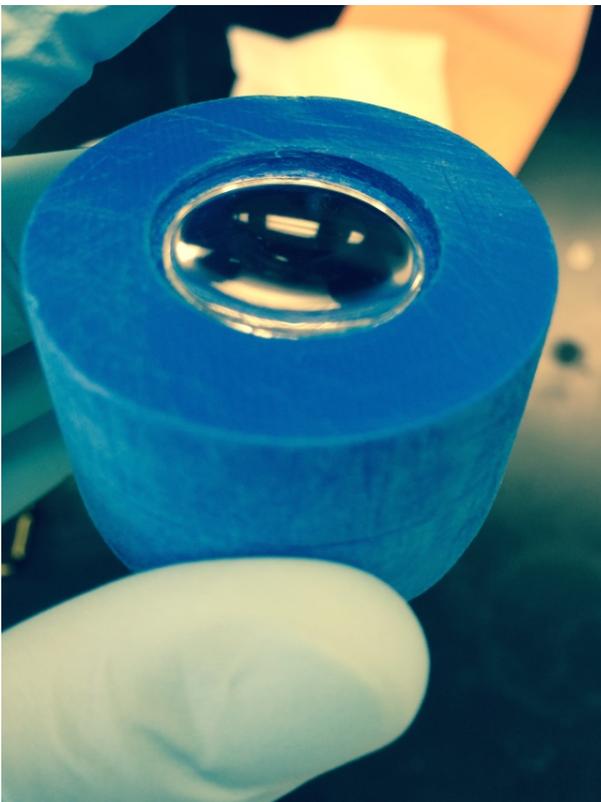


Figure 11: Fitting the lens with electrical tape.

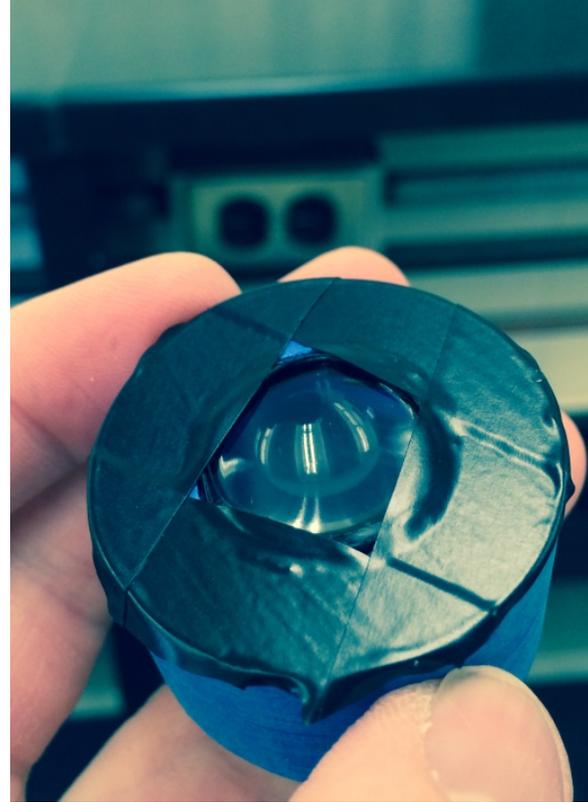


Figure 13: The Focusing Mirror (75mm diameter x 7mm thick x 75mm focal length)

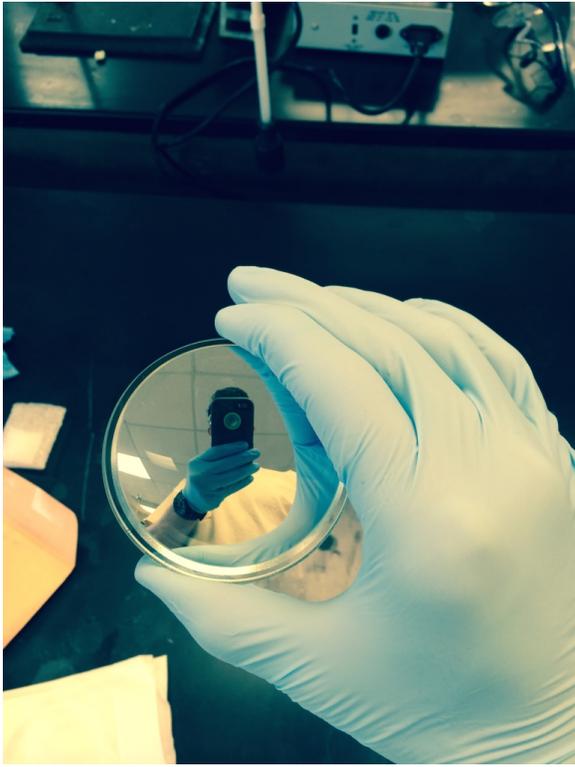


Figure 14 Mirror holder, Screws, Screw Springs.

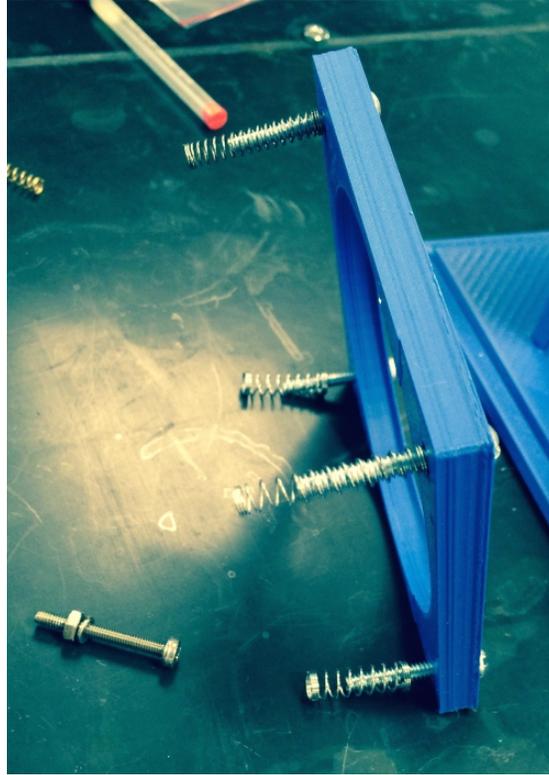


Figure 15 mirror, mirror holder, and padding



Figure 16: Mirror attached to base.



Figure 17: Affixing the CCD to the Base



Figure 18: CCD attached to the Base

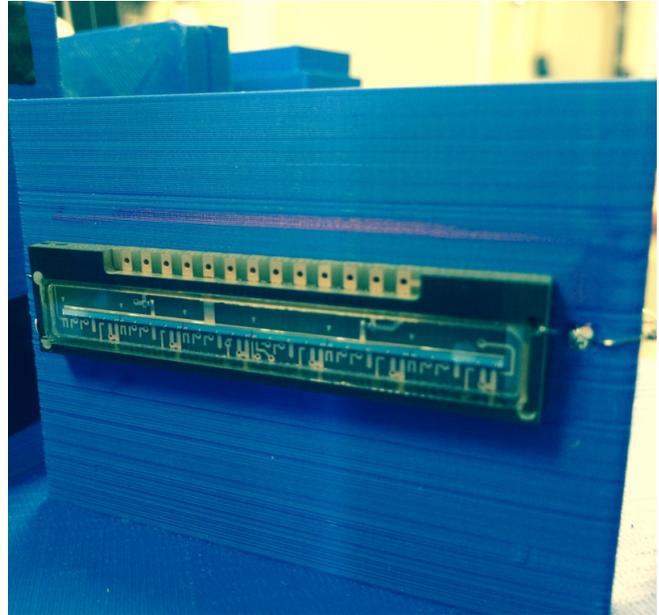


Figure 20: Wiring of CCD in progress.

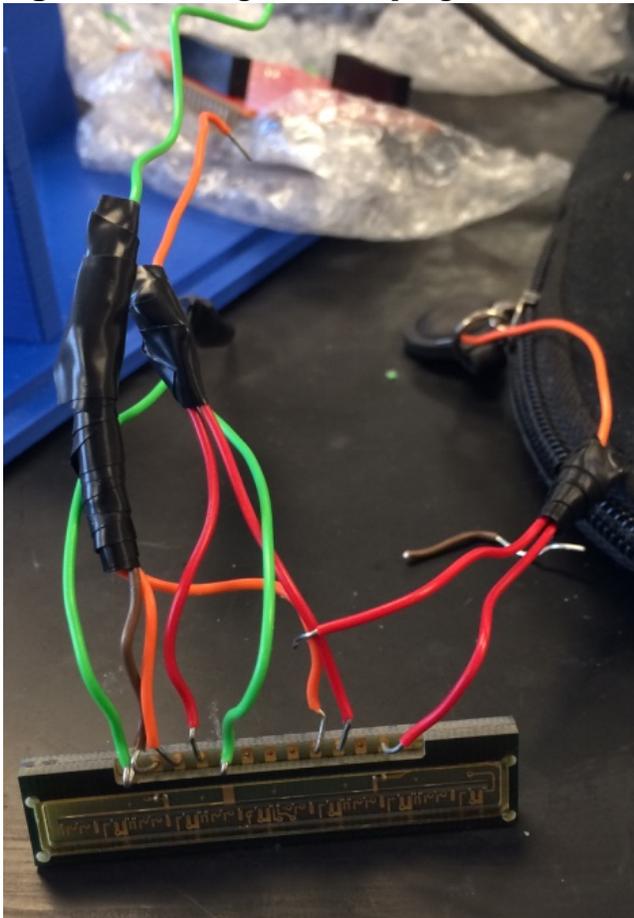


Figure 21: Fully wired CCD attached to the base.

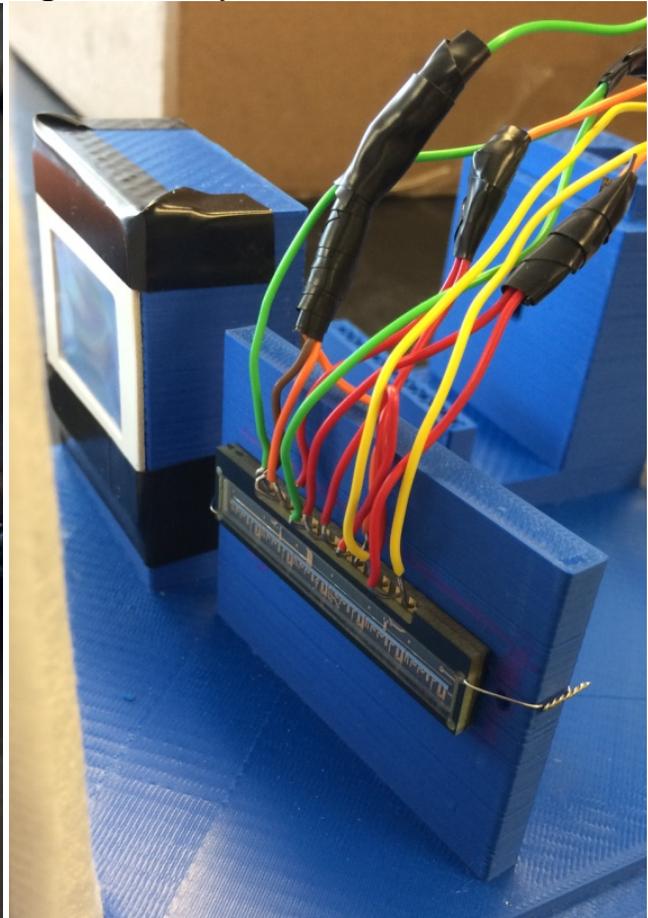


Figure 22: Tiva wired to the CCD

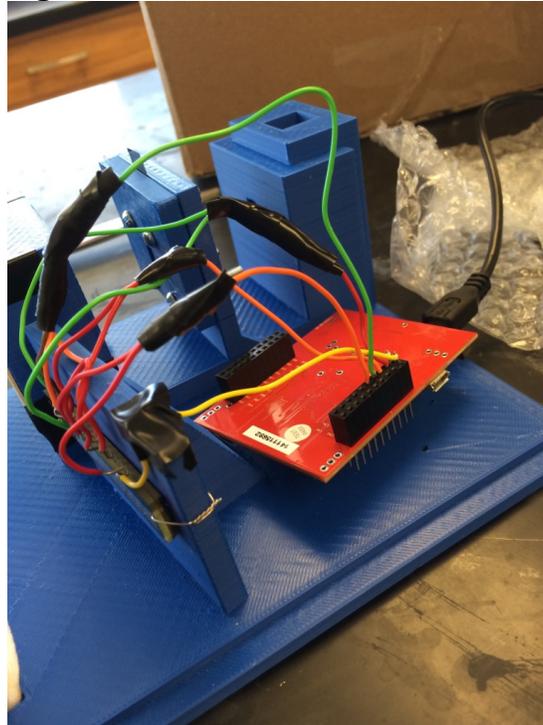


Figure 23: Completely Assembled CCD-PDA instrument. Connected to computer.

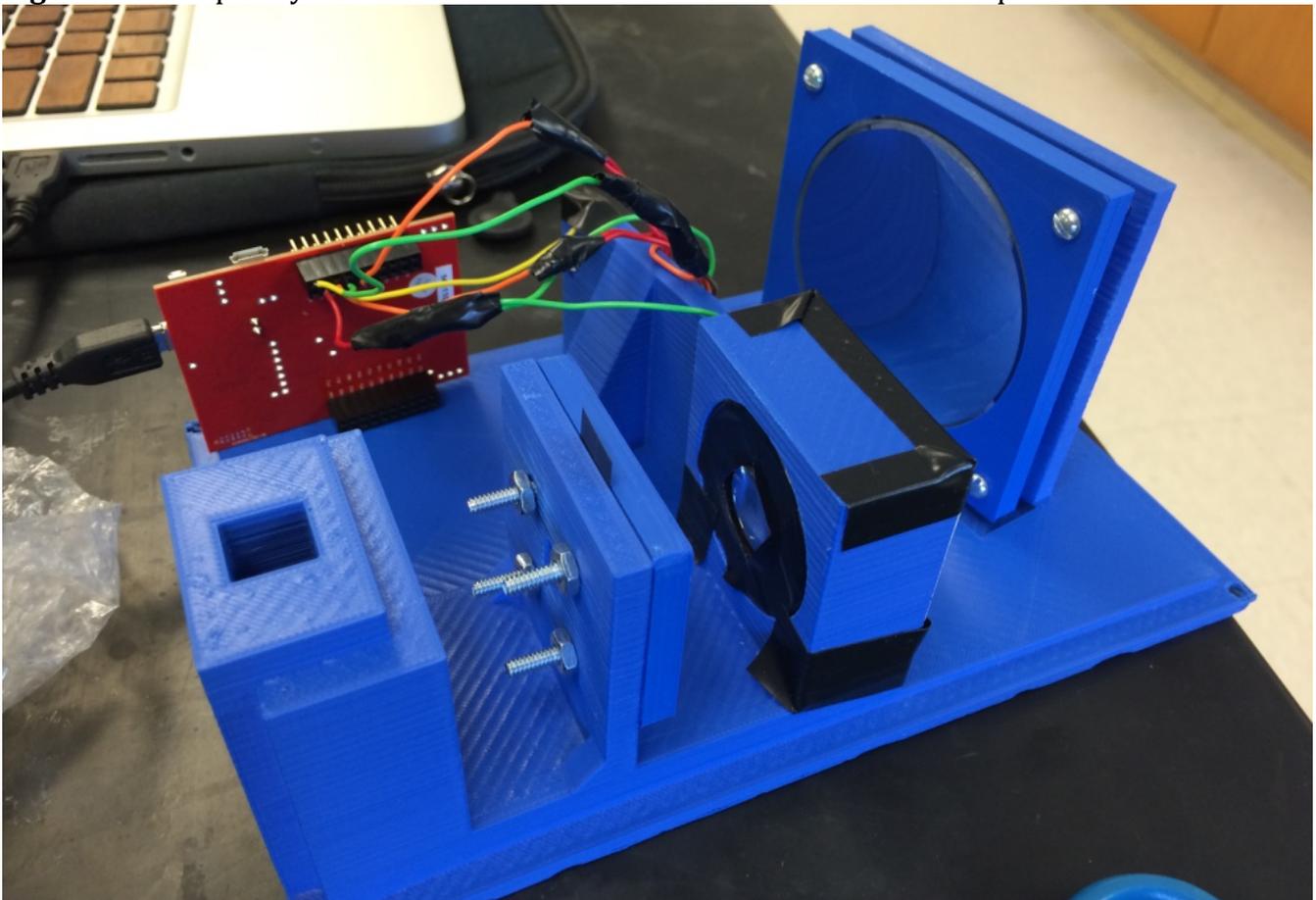


Figure 24: Card-board box cover with slot.



Figure 25: GUI from Processing. No mode is selected.

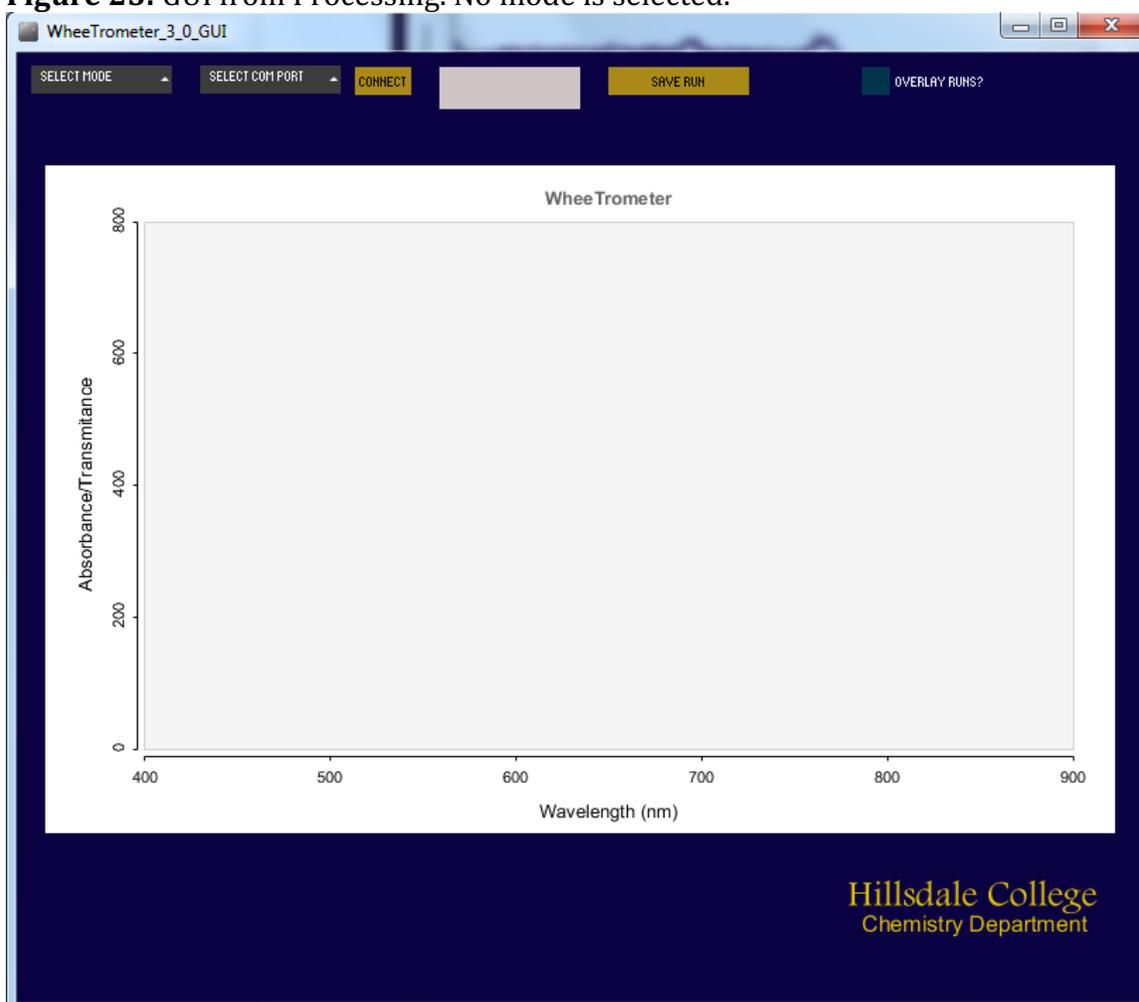


Figure 26: Spectrometer set up with halogen light.

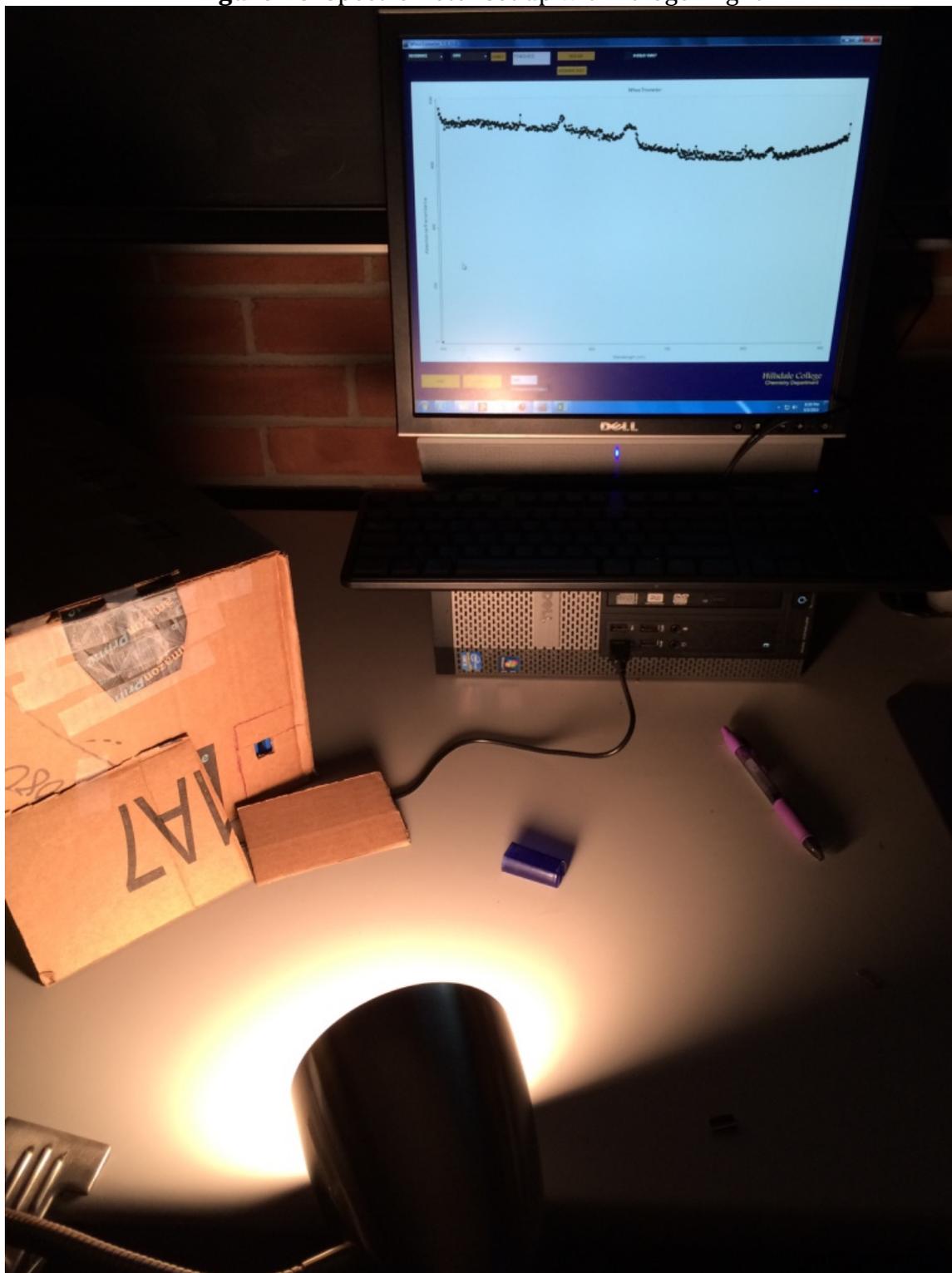


Figure 27: Absorbance blank Spectra with Halogen Light (no Hg).

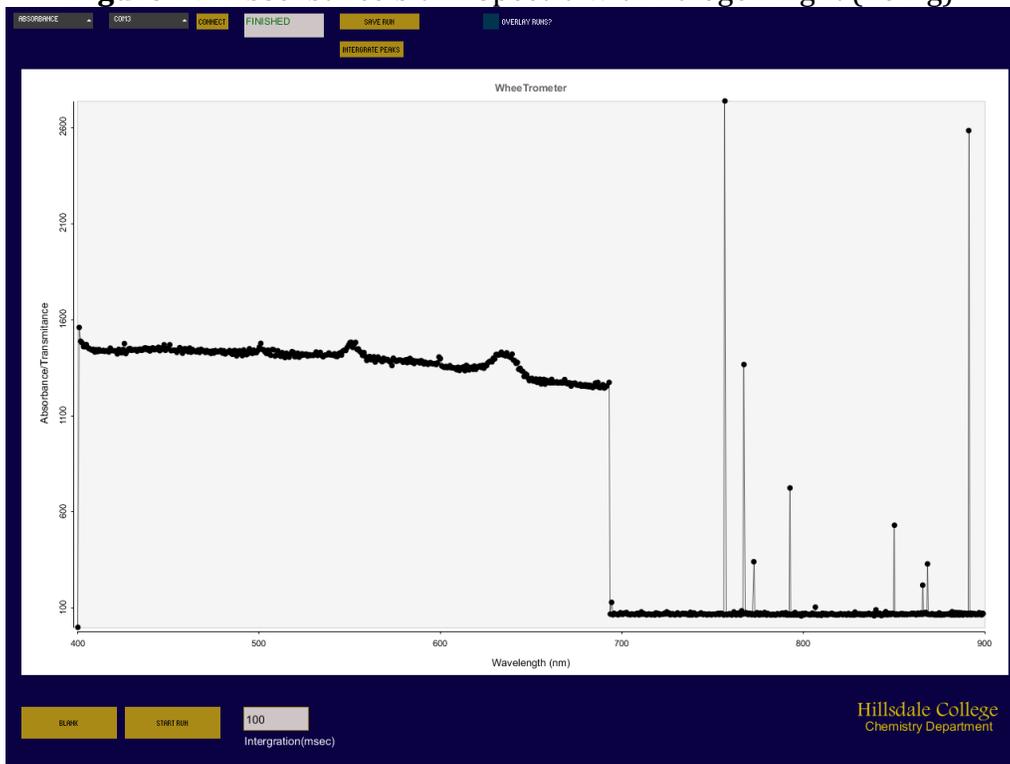


Figure 28: Absorbance blank Spectra with halogen light (no Hg)

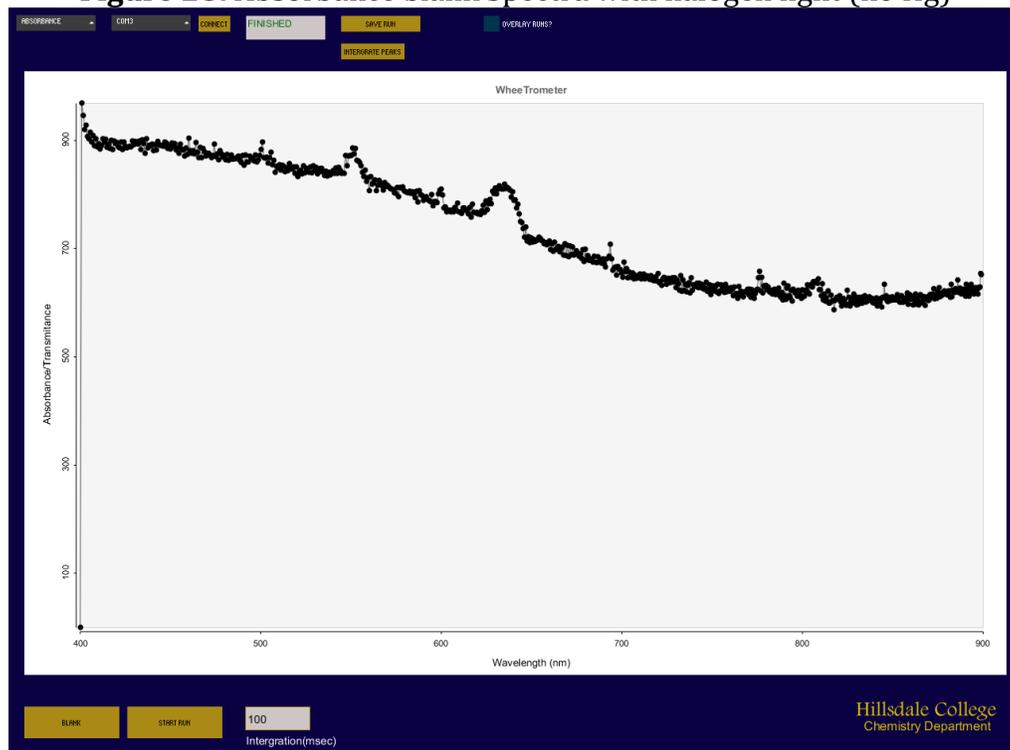


Figure 29: Absorbance blank spectra, halogen light (no Hg), 1000 ms integration.

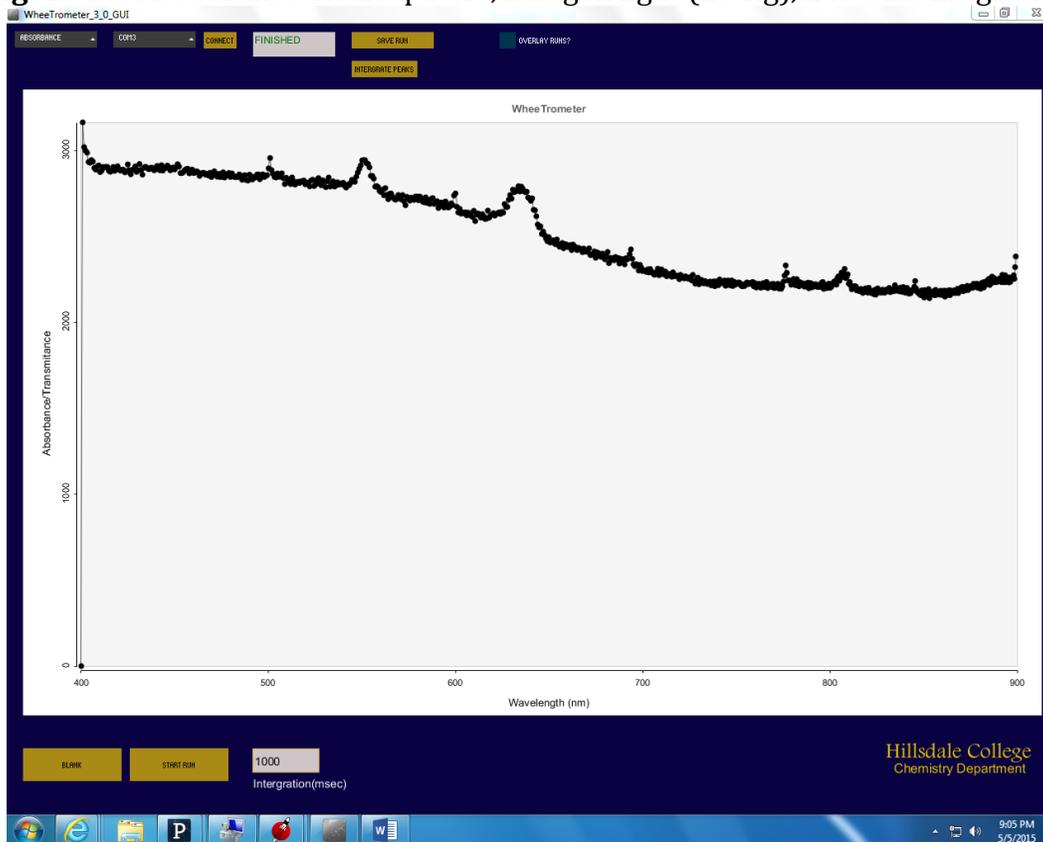


Figure 30: Absorbance spectra blank, halogen light with Hg

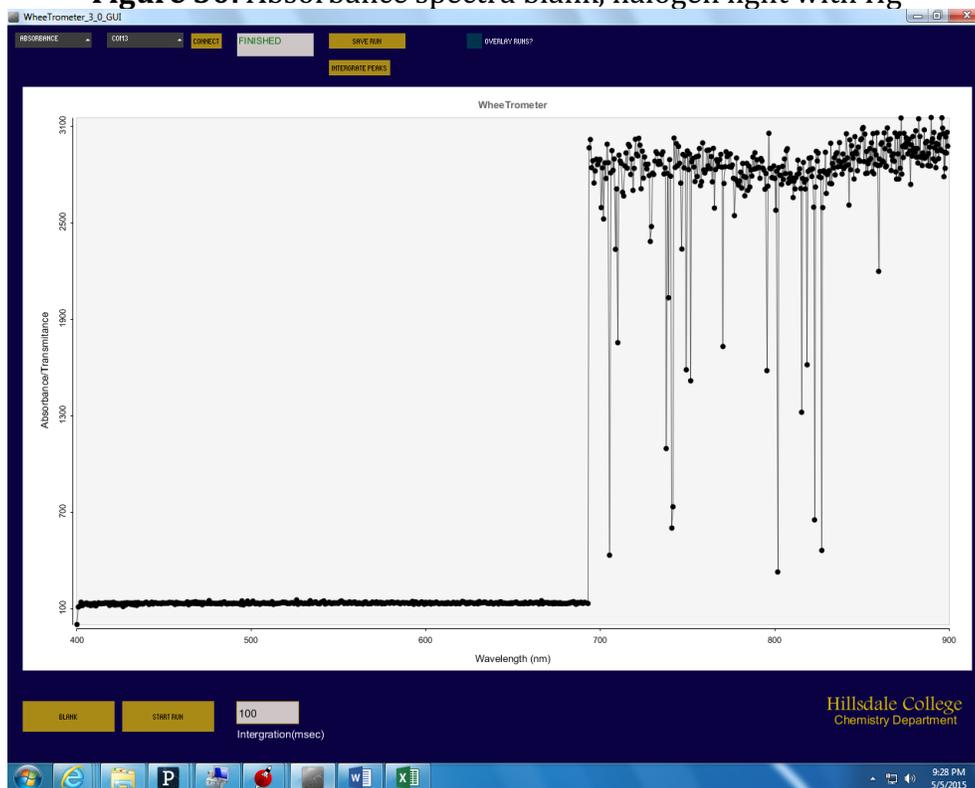


Figure 31: Absorbance spectra blank, halogen light with Hg, light distance increased.

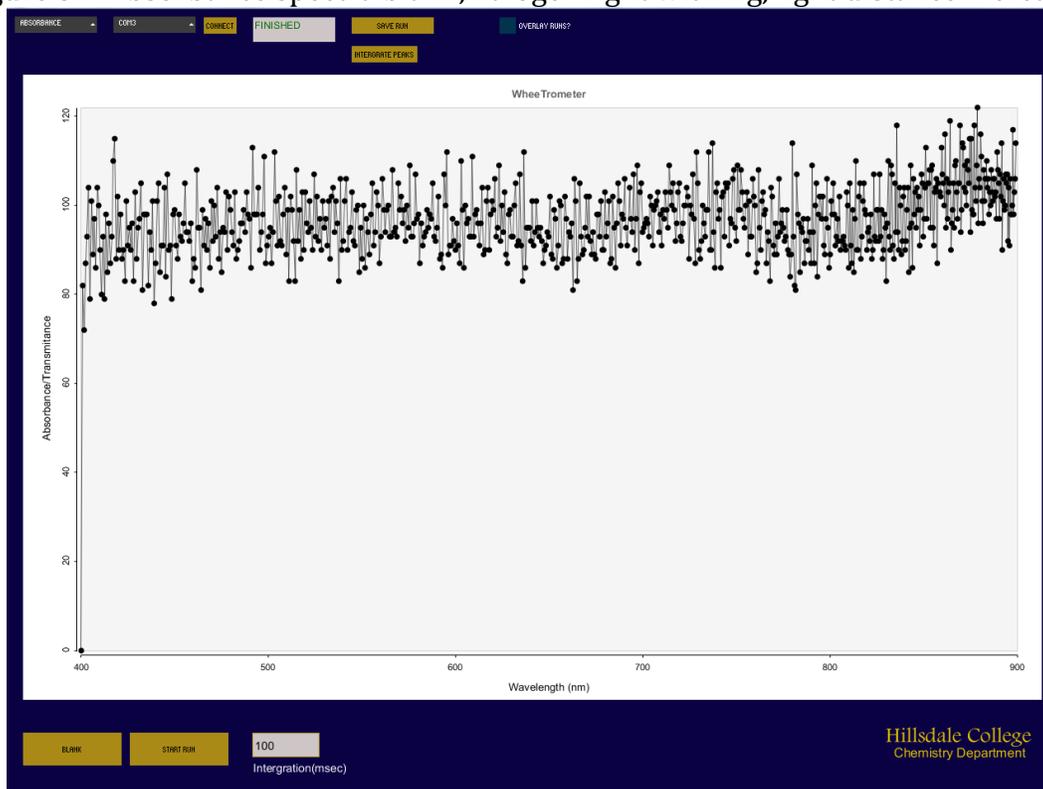


Figure 32: Transmittance blank – 1000ms integration– halogen w/mercury, with plastic cuvette

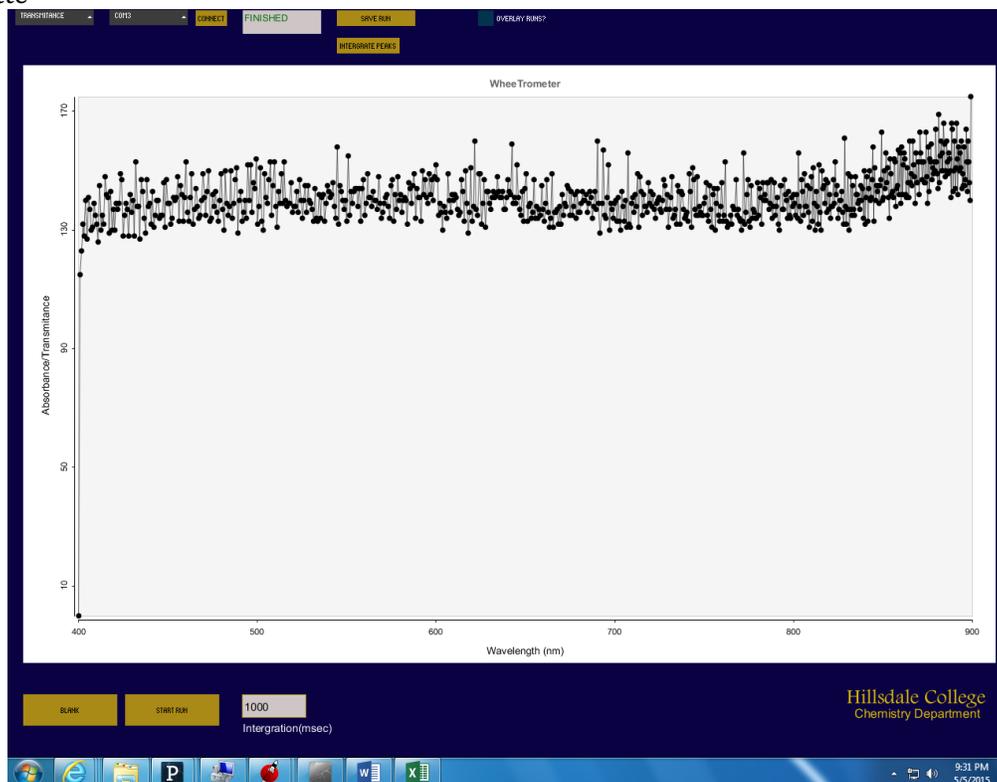


Figure 33: Transmittance – 1000 ms integration time – halogen (no Hg) plastic cuvette with DI water – holding paper up to block off other light from computer screen.

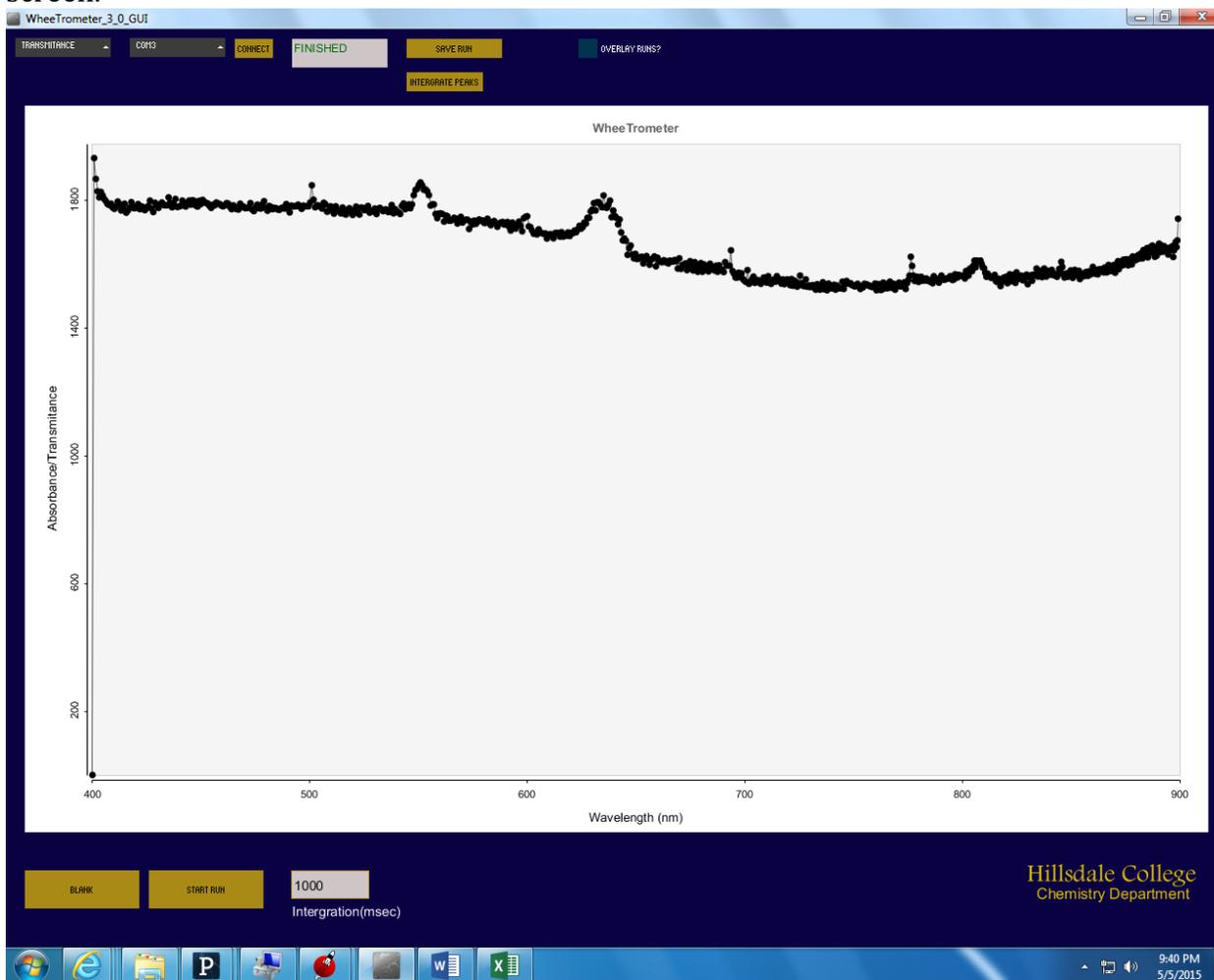


Figure 34: The functionality of the Series command. 100 integration (msec) with peak spectra – water – plastic cuvette

