Ocean Optics System User Guide to measuring absorbance of a thin film sample

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The ocean optics system is capable of accurately measuring the band gap of thin films and single crystals and an estimate of the band gap of powders. The absorbance (α) of smooth thin films can also be approximated using this system. An InGaAs detector (>6.84 nm resolution) and Si detector (0.6-2 nm resolution) combine to create an overall measurement range of 248 – 2060 nm (0.6 – 5 eV). The basic principle used in analysis states that light shined onto a flat sample at a normal angle of incidence will be either transmitted through the sample, reflected off the sample, or absorbed. Therefore, by determining the initial amount of light shined onto a sample, and the amount of light that is transmitted and reflected, the total absorption of the sample can be determined.

Data Analysis

Therefore, Total Light Entering System = Transmission + Reflection + Absorptance can be simplified to 1 = T + R +A where T and R are defined as the percent of light transmitted/reflected through the sample. The program automatically subtracts out background noise in the "calibrated data" columns. In this formula, absorptance (A) includes scattering, refraction, diffraction, etc. Because the ocean optics systems lacks an integrating sphere where all of these terms can be accounted for, absorption (α) can only be estimated. Estimation of absorption (α) begins by using the calibrated transmission and reflection data which contains background subtracted T and R information. Plot the transmission and reflection data as a function of energy in eV. Look for good agreement between the NIR and UV/Vis measurements (see Troubleshooting 1). Beer's law, $\frac{T}{1-R} = e^{-\alpha d}$, can be used to approximate absorption (α) at each wavelength, if the film thickness (d) is known. Then plot the absorption (α) as a function of energy to determine the band gap and learn about the approximate absorption at each wavelength. Exact absorption cannot be found using this system because any surface roughness on the film will cause light scattering that is not measurable by this system.

Preparation

Thin films should be on substrates that have at or near 100% transparency over the wavelengths of the measurement, such as fused silica (impurity free SiO_2 glass). Also, the smoother the sample, the more accurate the results.

Bring a blank substrate that has undergone the same processing.

Trouble shooting

1. IR detector temperature will not stabilize – Close the Lab View program. Unplug the IR detector USB cord from the computer. Wait 15 seconds. Plug the IR detector back into the exact same USB port as before. Reinitialized program.

2. Almost no reflectance signal from sample – Your sample is either not flat against the sample holder or is really rough.

3. No transmittance signal from sample – Film is too thick or substrate is blocking the signal in that range. For example, films on TOX will have no transmittance below 1.1 eV (the band gap of silicon).

4. Sample holder will not maintain it's set height. – Try propping it up with something or holding it in place if you have a steady hand.

Thin Film Set-up

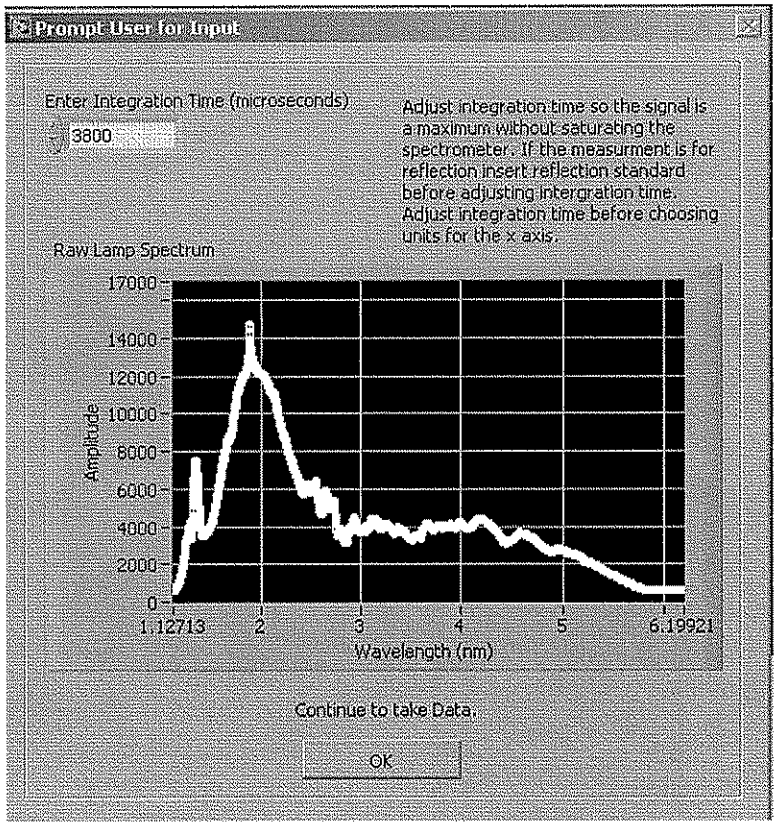
1. Flip on the small power strip and reset it. Turn on "system power" on the large grey servo box.

2. NIR measurements are made using the halogen lamp contained in a blue tube. UV/Vis measurements are made using a halogen and deuterium lamps that are turned on by a red and blue buttons on the a white boxed light source, respectively. Turn on the appropriate light sources and mark your start time in the log book.

3. Log onto the computer and open "Shortcut to OpticFiberSpectrometerProgram.vi". Run the program (click arrow in upper left).

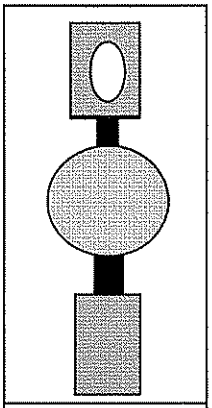
4. Fill in your name and choose the "spectrum range" that you want to measure. For UV/Vis, go to instruction 5. For NIR, go to instruction 16. Click "Energy (eV)" to continue.

5. Maximize the signal by making sure the grey bifurcated cables are aligned at the source and the receiving ends. This is done by slightly unscrewing the input source optics cable. Gently twist the cable. The signal should be maximized within a 180 rotation of the cable. Retighten screw. This adjustment only needs to be made if the previous user measured diffuse reflectance.



The signal should match the intensity in the screen shot. If it is too low, the system is out of alignment. If the signal is higher, the reflectance measurement will be too low and the system must be calibrated for the reflectance measurement before the transmittance calibration. Get someone to help you calibrate the system.

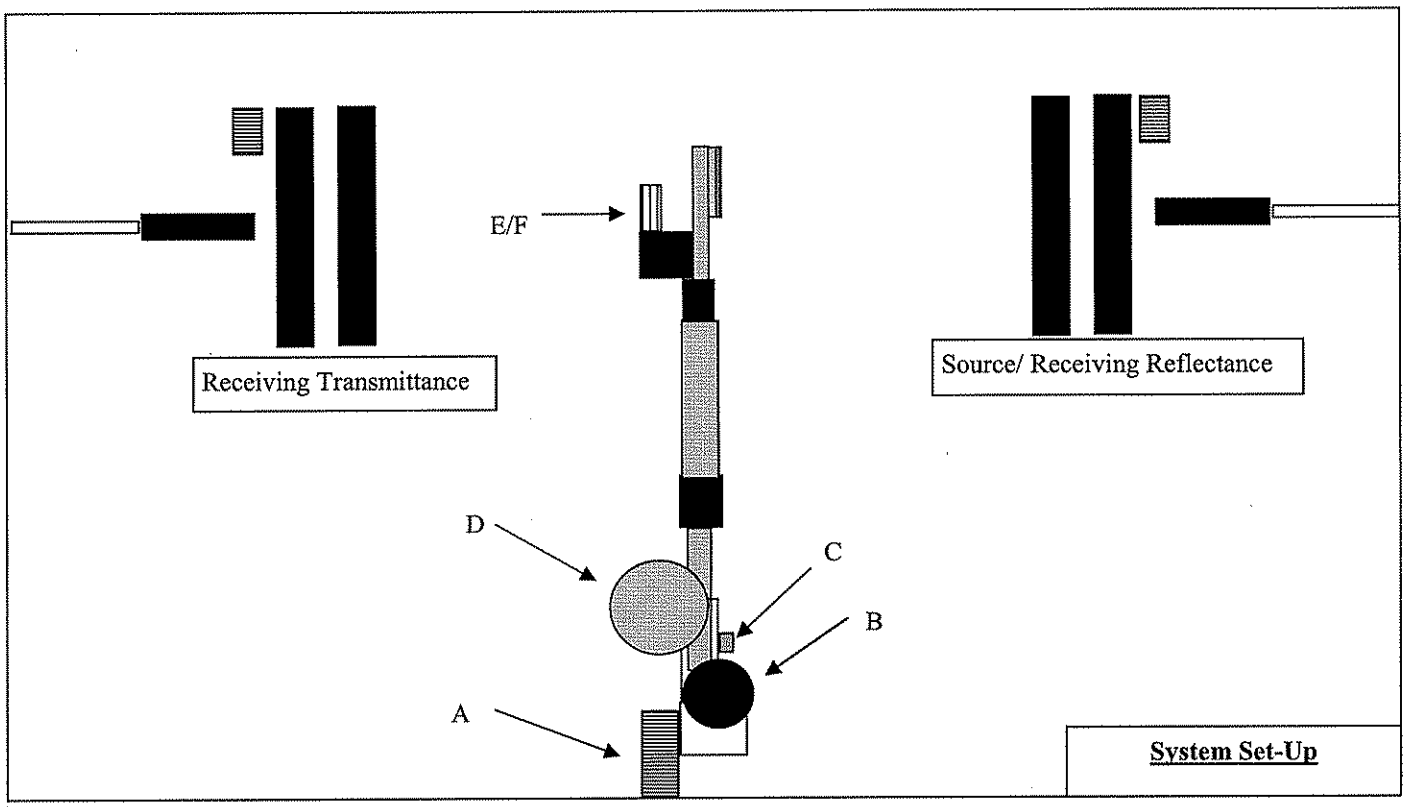
Click "ok" once the signal is maximized.


Sample Holder

6. When the green light comes on under the "Take Transmittance Dark Spectra" lights up, click the lit button next to it. Wait for the green light under "Take Transmittance Reference Spectra" to light up before continuing.

7. Apply double sided tape on the edges of the hole in the sample holder. No tape should cover the hole. Place a blank substrate on the sample holder as flush with the sample holder as possible. Unscrew the mirror cap.

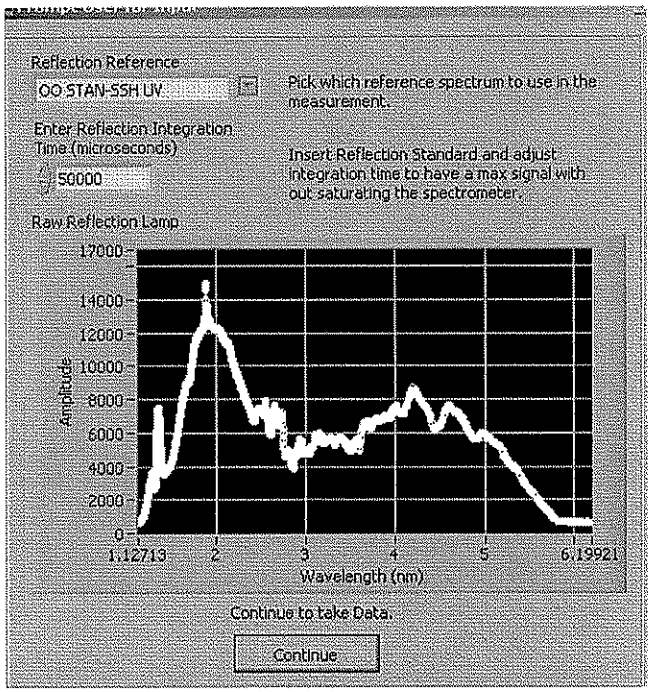
8. Insert the sample holder into the system. Use knob A to center the sample holder in the appropriate beam (red optics cable for NIR, grey optics cable for UV/Vis). Use knob B to move the sample holder as far away from the source optics cable as possible. Knob C for making sure the sample holder sits upright in the holder. Use knob D to adjust the height of the sample holder and make sure the beam passes through the substrate.



9. Click the lit button under "Take Transmittance Reference Spectra".

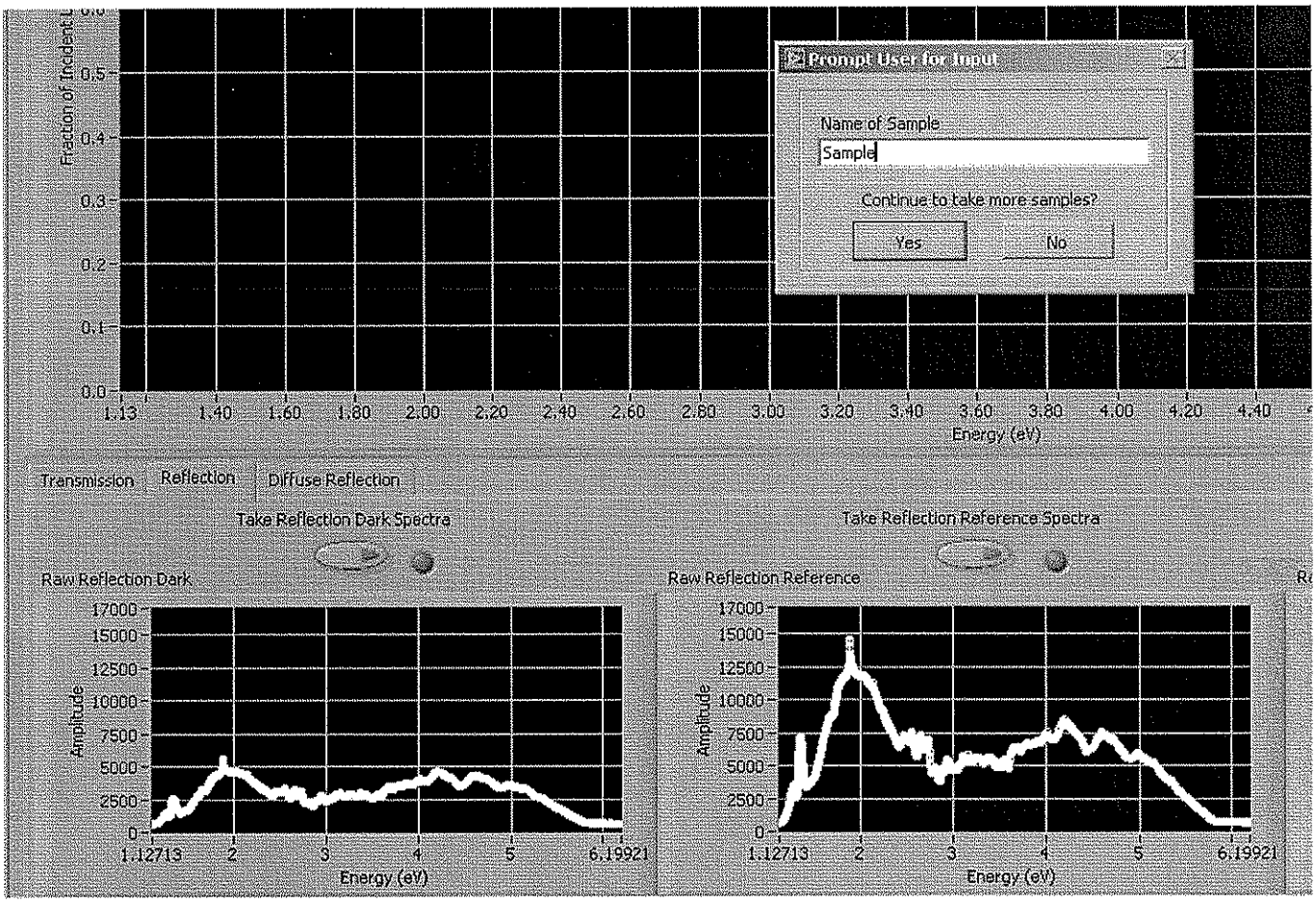
10. The next prompt will ask for the reflectance signal to be maximize. Raise the sample holder height so that the mirror is in the beams path. Use knobs E and F to angle the mirror so that the signal is maximized. Increase the integration time to 50,000. If the signal does not look like the screen shot, the system is out of alignment. Please get help aligning the system.

Click "Continue".



11. Adjust the sample holder height so that the beam is passing through the blank substrate. Click the button to "Take Reflection Dark Spectra". When the "Take Reflection Reference Spectra" green light to lights up, return the mirror to the beam path by adjusting the sample holder height.

12. Click the "Take Reflection Reference Spectra" button. When the "Name of Sample" prompt appears, type the samples name and click "yes".



13. Loosen knob 3, and remove the sample holder. Recap the mirror. Place your sample on sample holder with the film facing the beam source (not on the tape side) and is FLAT against the holder. Return the sample holder to the system and retighten knob D. Adjust the sample holder height so that the beam is passing through the sample, Adjust knobs E and F to maximize the signal intensity.

Click the "Record Reflection Sample Data" button. Click the "Record Transmission Sample Data" button.

14. If done with the UV/Vis measurements, click "no" at the prompt and select "T, R and Raw Data". Save the file. You can access this file in Excel.

If you have more samples to measure in the UV/Vis spectrum, click "yes" and return to step13.

15. If you have sample to measure in the NIR range, jump to step 16. Otherwise, remove sample, close up the aquarium, turn off the lamps, log off the computer, switch off the power strip and grey servo box, mark the log book with time.

Good luck analyzing your data and may the Flying Spaghetti Monster aide you in your research!

16. Fill in your name and choose NIR under "spectrum range". Click "Energy (eV)" to continue.

17. Allow the detector to cool to a consistent – 9.9 to -10° C.

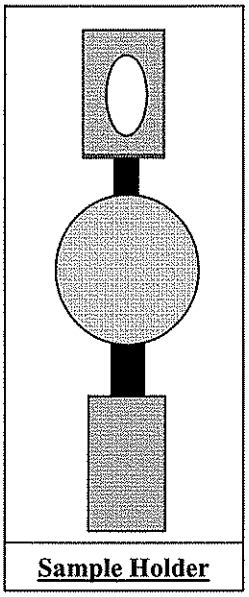
18. Maximize the signal by making sure the red bifurcated cables are aligned at the source and the receiving ends. This is done by slightly unscrewing the source optics cable. Gently twist the cable. The signal should be maximized within a 180 rotation of the cable. Retighten screw. This adjustment only needs to be made if the previous user measured diffuse reflectance.

The signal should match the intensity in the screen shot. If it is too low, the system is out of alignment. If the signal is higher, the reflectance measurement will be too low and the system must be calibrated for the reflectance measurement before the transmittance calibration. Get someone to help you calibrate the system.

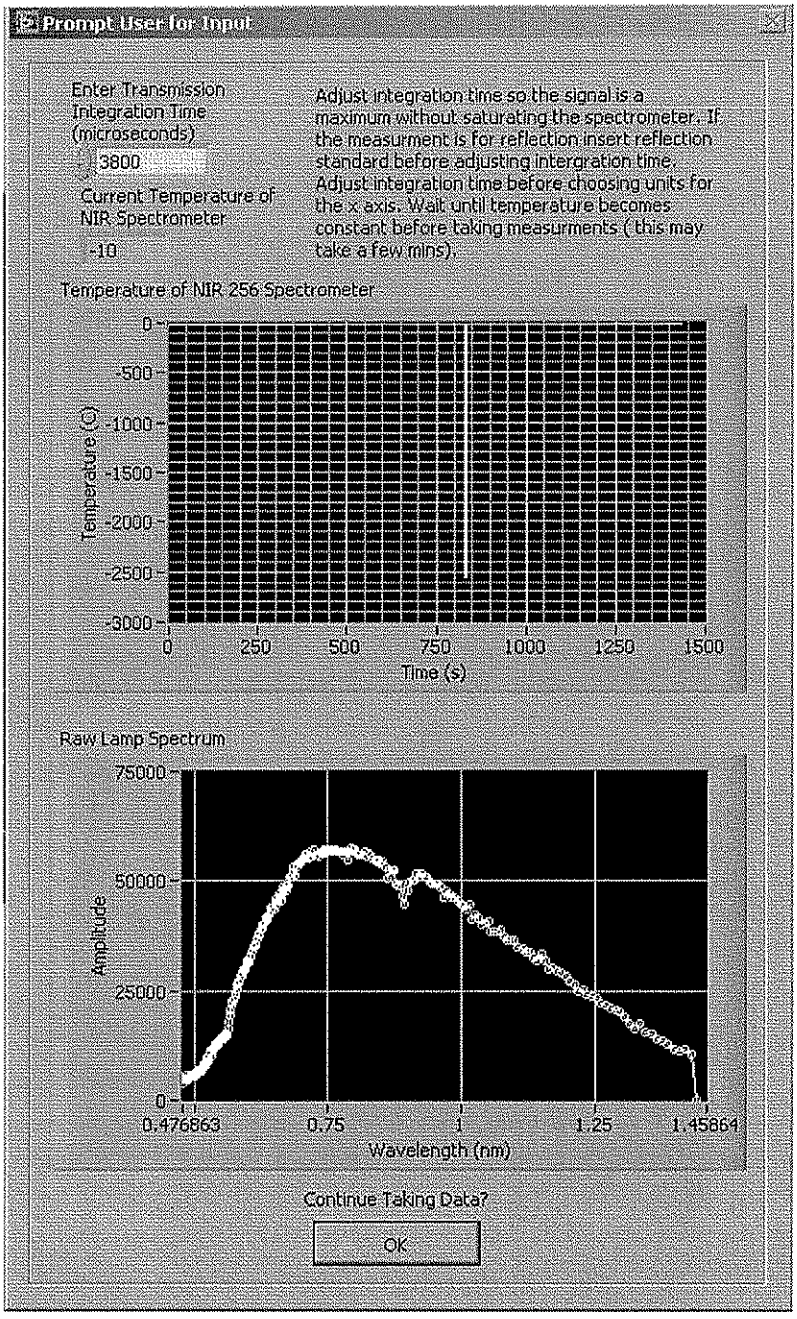
Click "ok" once the signal is maximized.

19. When the green light comes on under the "Take Transmittance Dark Spectra" lights up, click the lit button next to it. Wait for the green light under "Take Transmittance Reference Spectra" to light up before continuing.

20. Apply double sided tape on the edges of the hole in the sample holder. No tape should cover the hole. Place a blank substrate on the sample holder as flush with the sample holder as possible. Unscrew the mirror cap.

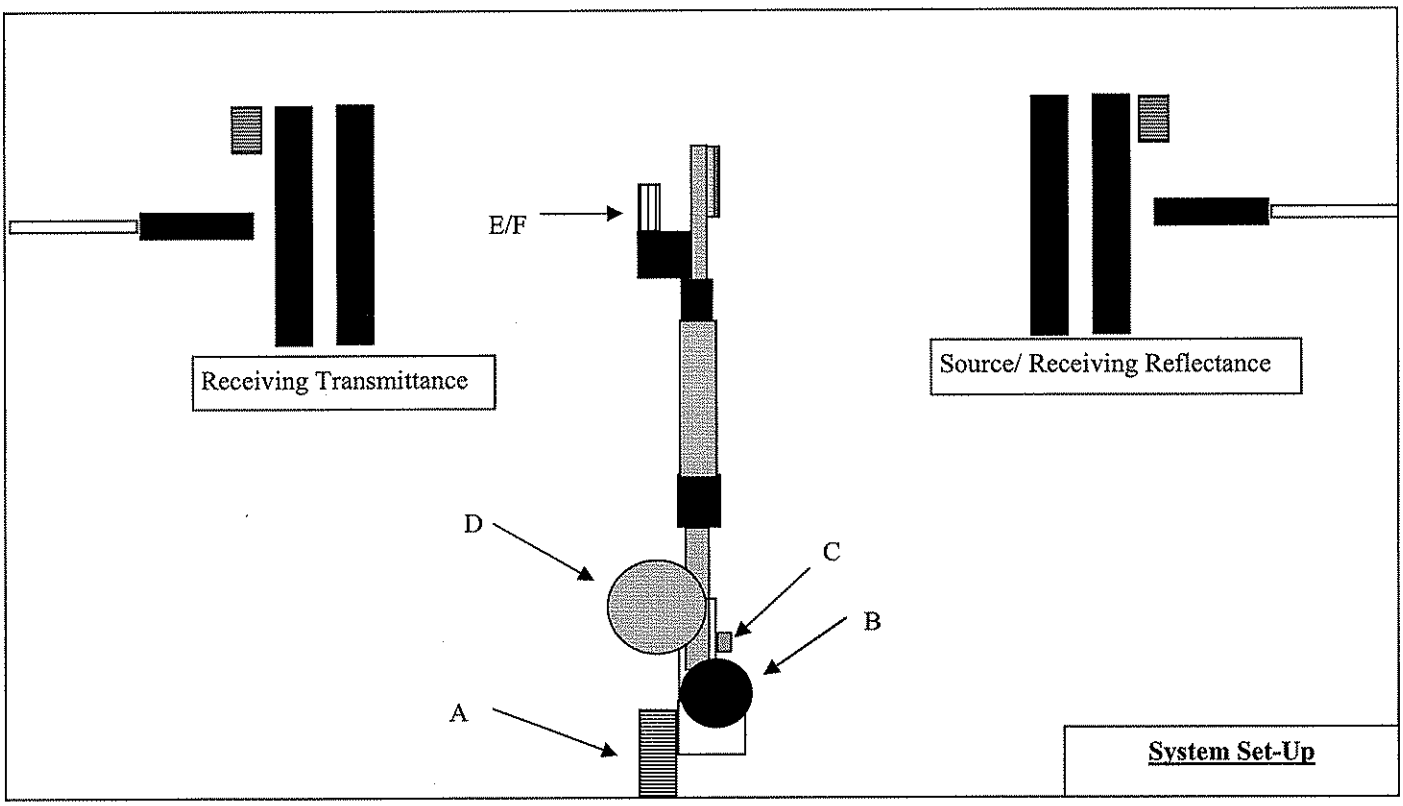


Sample Holder



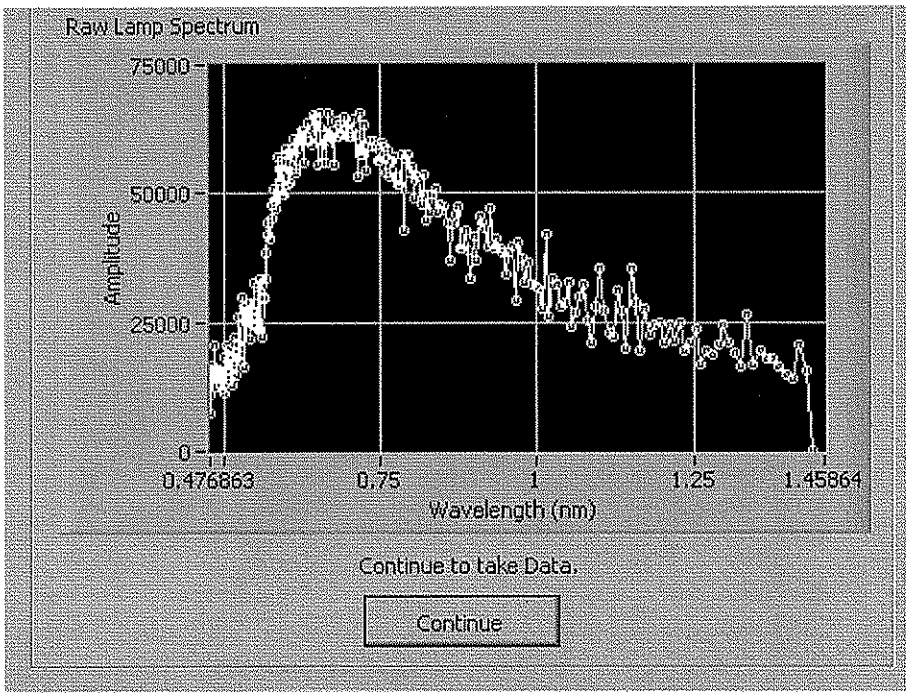
21. Insert the sample holder into the system. Use knob A to center the sample holder in the appropriate beam (red optics cable for NIR, grey optics cable for UV/Vis). Use knob B to move the sample holder as far away from the source optics cable as possible. Knob C for making sure the sample holder sits upright in the holder. Use knob D to adjust the height of the sample holder and make sure the beam passes through the substrate.

22. Click the lit button under "Take Transmittance Reference Spectra".



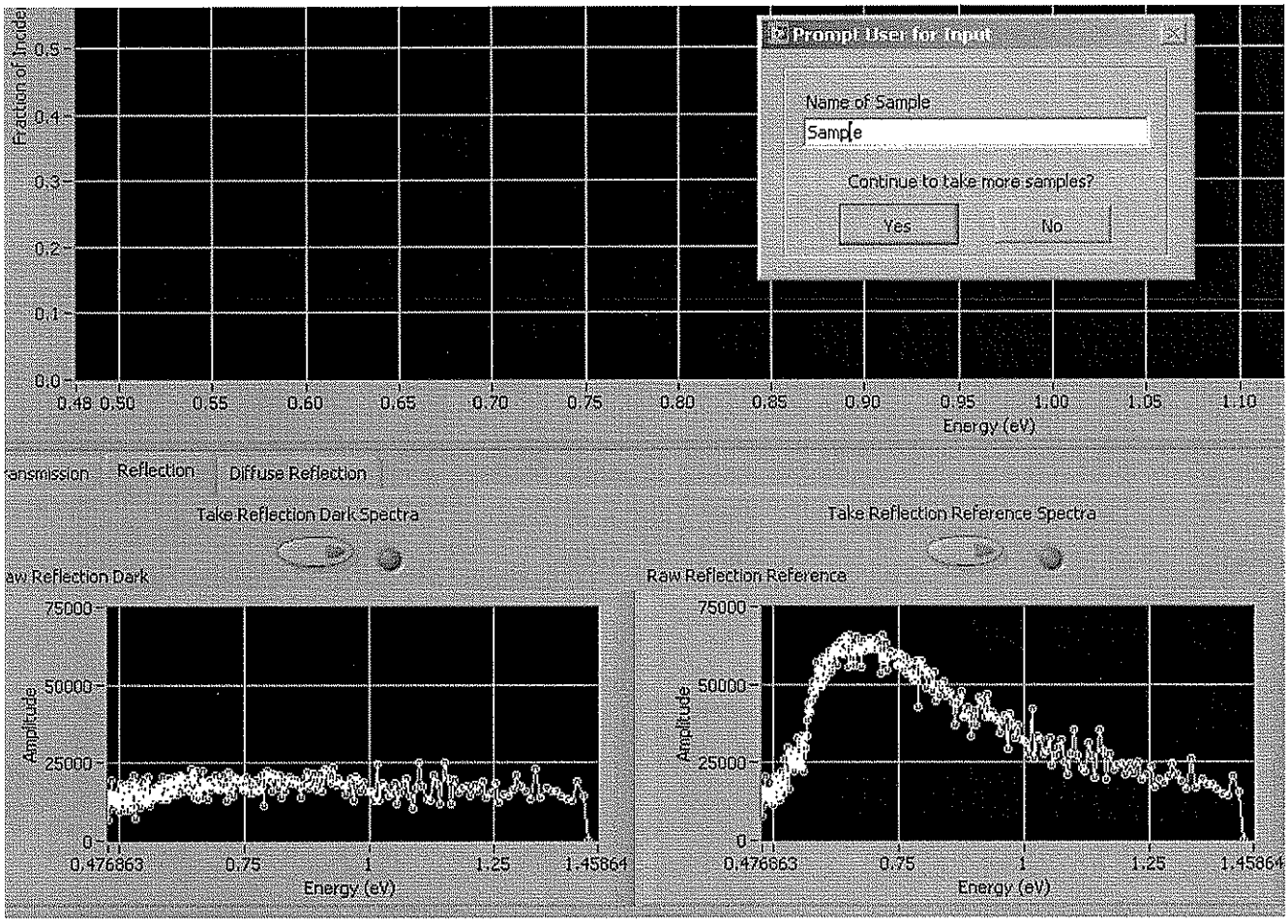
23. The next prompt will ask for the reflectance signal to be maximize. Raise the sample holder height so that the mirror is in the beams path. Use knobs E and F to angle the mirror so that the signal is maximized. Increase the integration time to 30,000. If the signal does not look like the screen shot, the system is out of alignment. Please get help aligning the system.

Click "Continue".



24. Adjust the sample holder height so that the beam is passing through the blank substrate. Click the button to "Take Reflection Dark Spectra". When the "Take Reflection Reference Spectra" green light to lights up, return the mirror to the beam path by adjusting the sample holder height.

25. Click the "Take Reflection Reference Spectra" button. When the "Name of Sample" prompt appears, type the samples name and click "yes".



26. Loosen knob 3, and remove the sample holder. Recap the mirror. Place your sample on sample holder with the film facing the beam source (not on the tape side) and is FLAT against the holder. Return the sample holder to the system and retighten knob 3. Adjust the sample holder height so that the beam is passing through the sample, Adjust knobs 6 and 7 to maximize the signal intensity.

Click the "Record Reflection Sample Data" button. Click the "Record Transmission Sample Data" button.

27. If done with the NIR measurements, click "no" at the prompt and select "T, R and Raw Data". Save the file. You can access this file in Excel.

If you have more samples to measure in the NIR spectrum, click "yes" and return to step 26.

28. If you have a sample to measure in the UV/Vis range, rerun the program and jump to step 4. Otherwise, remove sample, close up the aquarium, turn off the lamps, log off the computer, switch off the power strip and grey servo box, mark the log book with time.

Good luck analyzing your data and may the Flying Spaghetti Monster aide you in your research!