Acquiring Simple Transmission Scan using the UV-3101PC Spectrophotometer

DISCLAIMER

Safety –the first !!! This presentation is not manual. It is just brief set of rule to remind procedure for simple measurements. You should read manual first.

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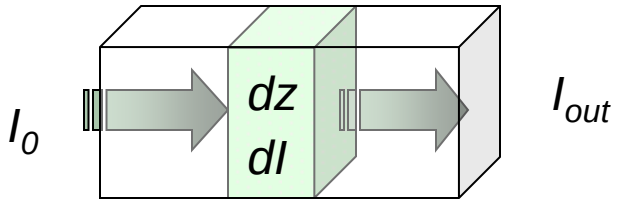
ABSORPTION SPECTROSCOPY

Lambert-Beer law (also known as Beer-Lambert-Bouguer law *)*

$$\frac{dI}{dz} = -\alpha I \qquad I_{out} = I_0 e^{-\alpha \cdot z}$$

In essence, the law states: when radiation propagates in the sample with concentration (***N***) of the absorbing impurities; the intensity of the radiation (***I***) decays exponentially with samples thickness (**z**),



Transmission

$$T = \frac{I_{out}}{I_0} = e^{-\alpha \cdot z}$$

absorption coefficient

$$\alpha = -\frac{Ln(T)}{z}$$

absorption cross-section

$$\sigma_{ab} = \frac{\alpha}{N}$$

Goal

One of the important goal of new samples characterization is measure absorption cross section of the impurities, since with this knowledge, one can calculate transmission for any impurity concentration and any crystal thickness.

Units typical in optical spectroscopy

Transmission (***T***)- dimensionless factor 0..1 or %

Absorption coefficient (α)- [cm^{-1}]

Intensity (***I***)- W/cm^2 or (Number of Photons)/cm^2

Concentration (***N***)- [cm^{-3}]

Absorption cross-section (σ) - [cm^2]

SHIMADZU SPECTROPHOTOMETER

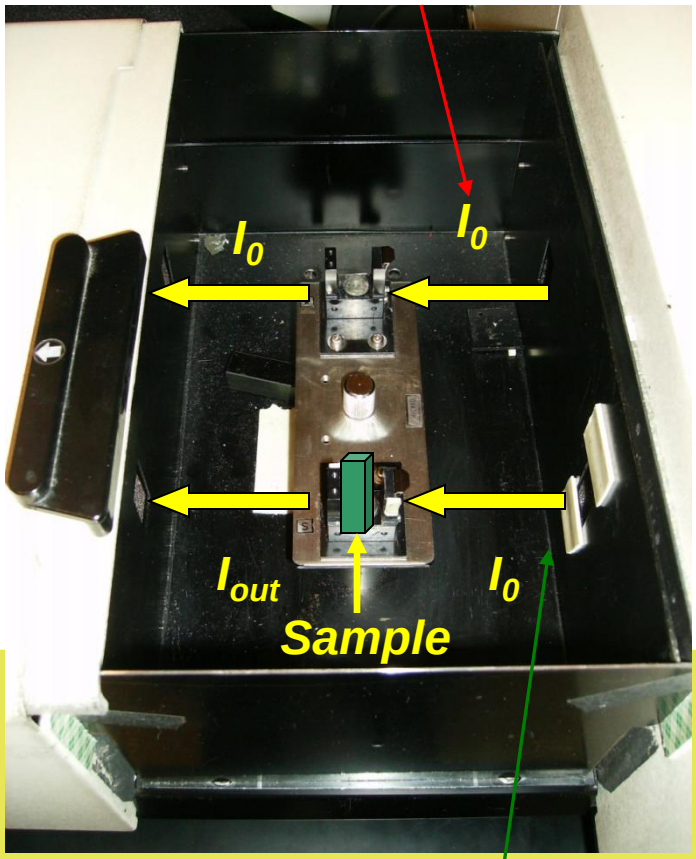
Spectrophotometer measures transmission T(%) of the sample at different wavelength (λ) of the incident light

There are two beam paths in the Spectrometer: Reference beam path (always empty) and Signal beam path (with studied sample)

First of all spectrophotometer select light with required wavelength (λ), then it measures intensity of the light in the reference beam path (I_0) and in the signal beam path after the sample (I_{out}). Then transmission is calculated as a ratio of these measurements

$$T(\lambda) = \frac{I_{out}(\lambda)}{I_0(\lambda)}$$

Reference beam path



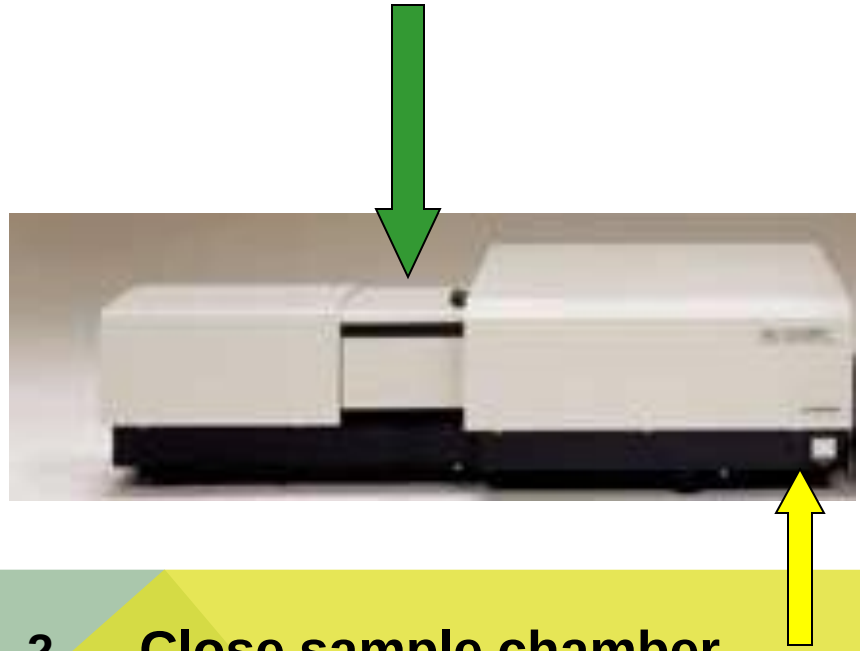
Signal beam path

Specifications

- Wavelength range 190-3200 nm
- Accuracy 0.3%(T)
- Number of Channels (scans) 10

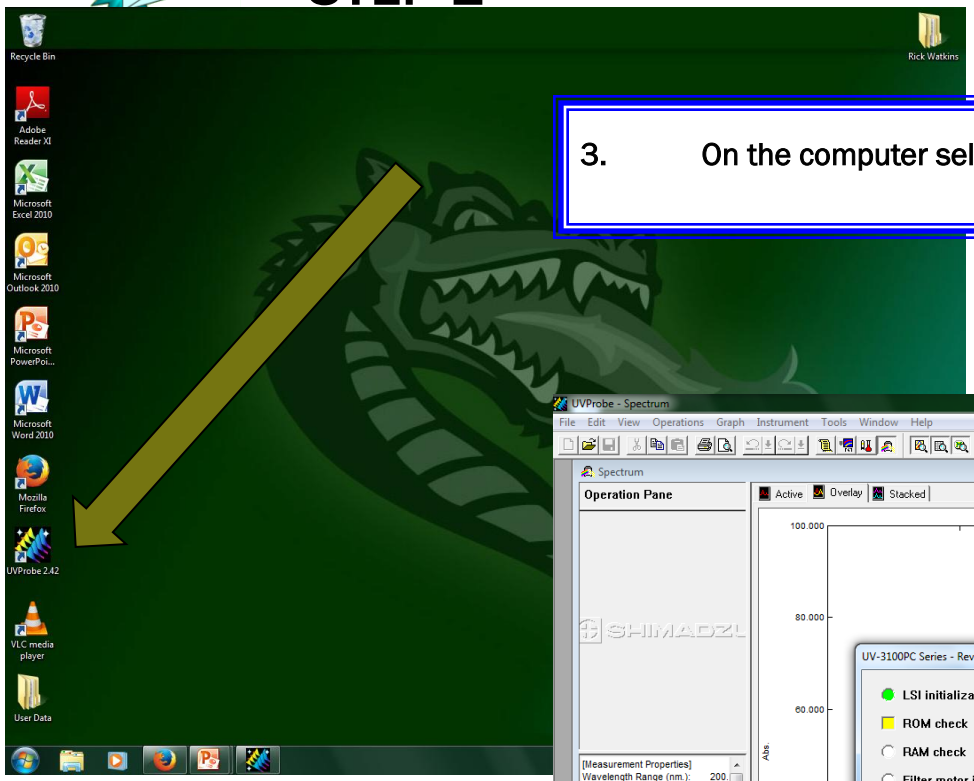
STEP 1

1. Before switching on power to the spectrophotometer, check sample chamber for unblocked sample and reference beam paths! (Some bad people left their samples in the chamber dot not follow this behavior)

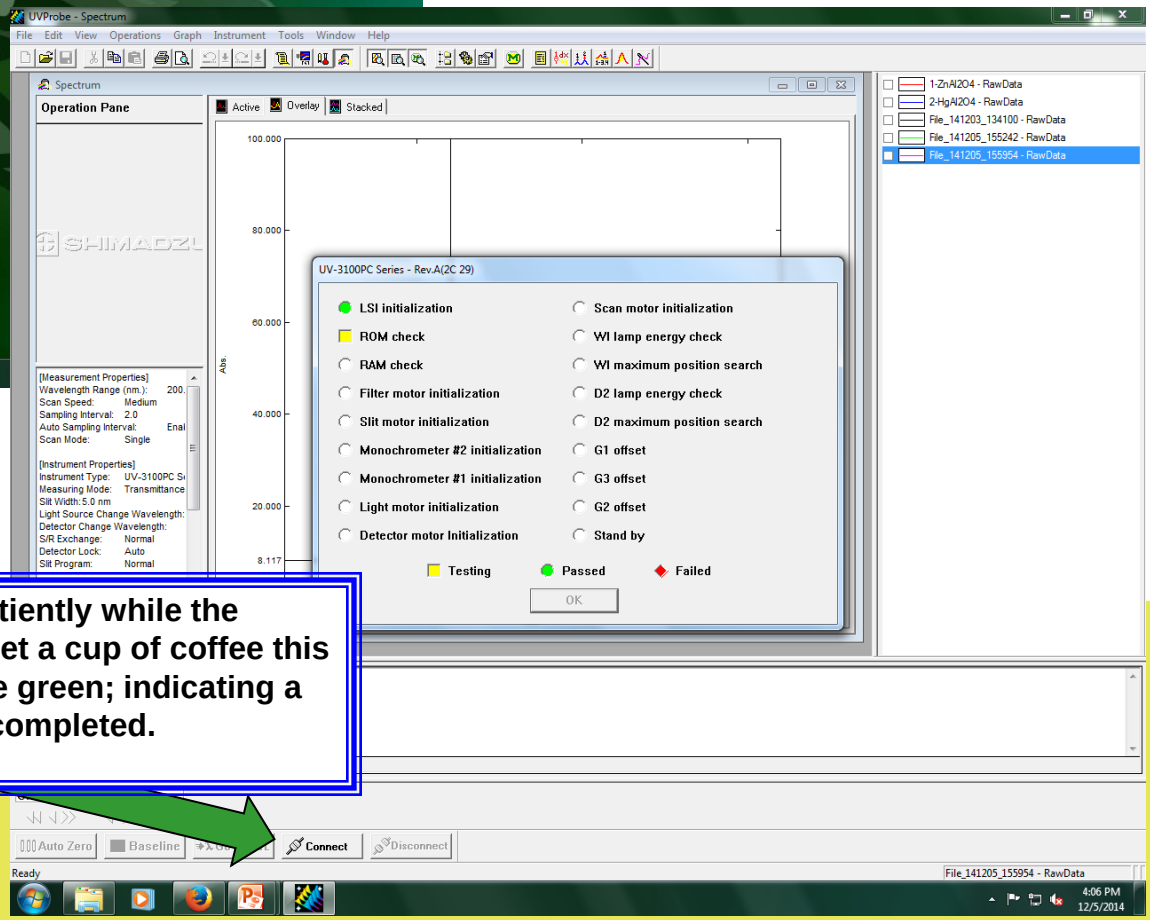


2. **Close sample chamber door** and turn power on with white rocker switch.

STEP 2

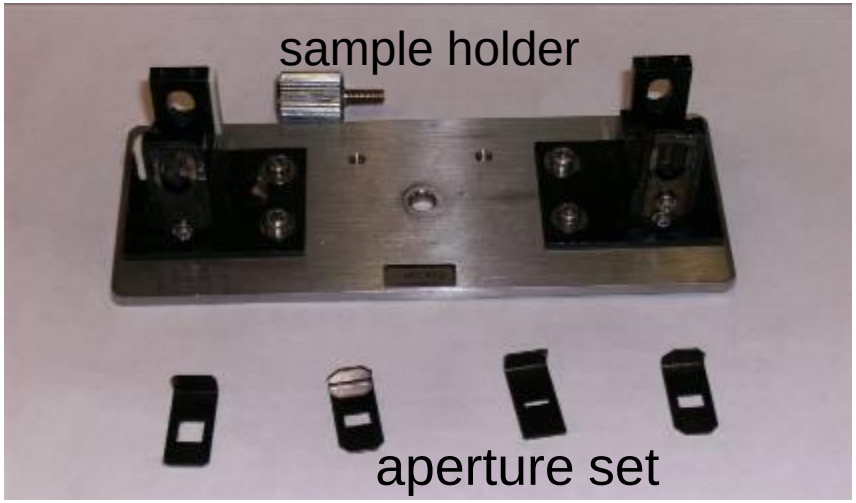


3. On the computer select the UVProbe software on the desktop.



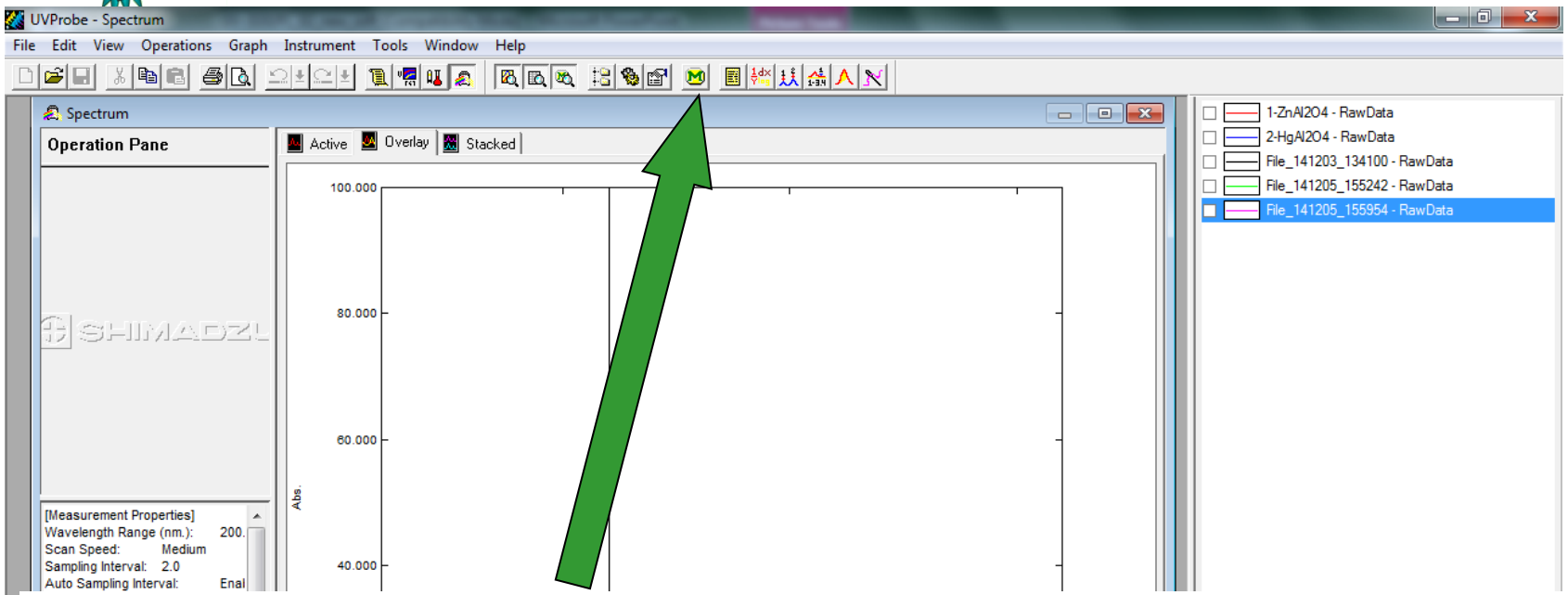
4. **Once opened, click connect . Wait patiently while the system performs its self checks; go get a cup of coffee this will take 5 minutes! All dots should be green; indicating a pass situation after the self check is completed.**

STEP 3



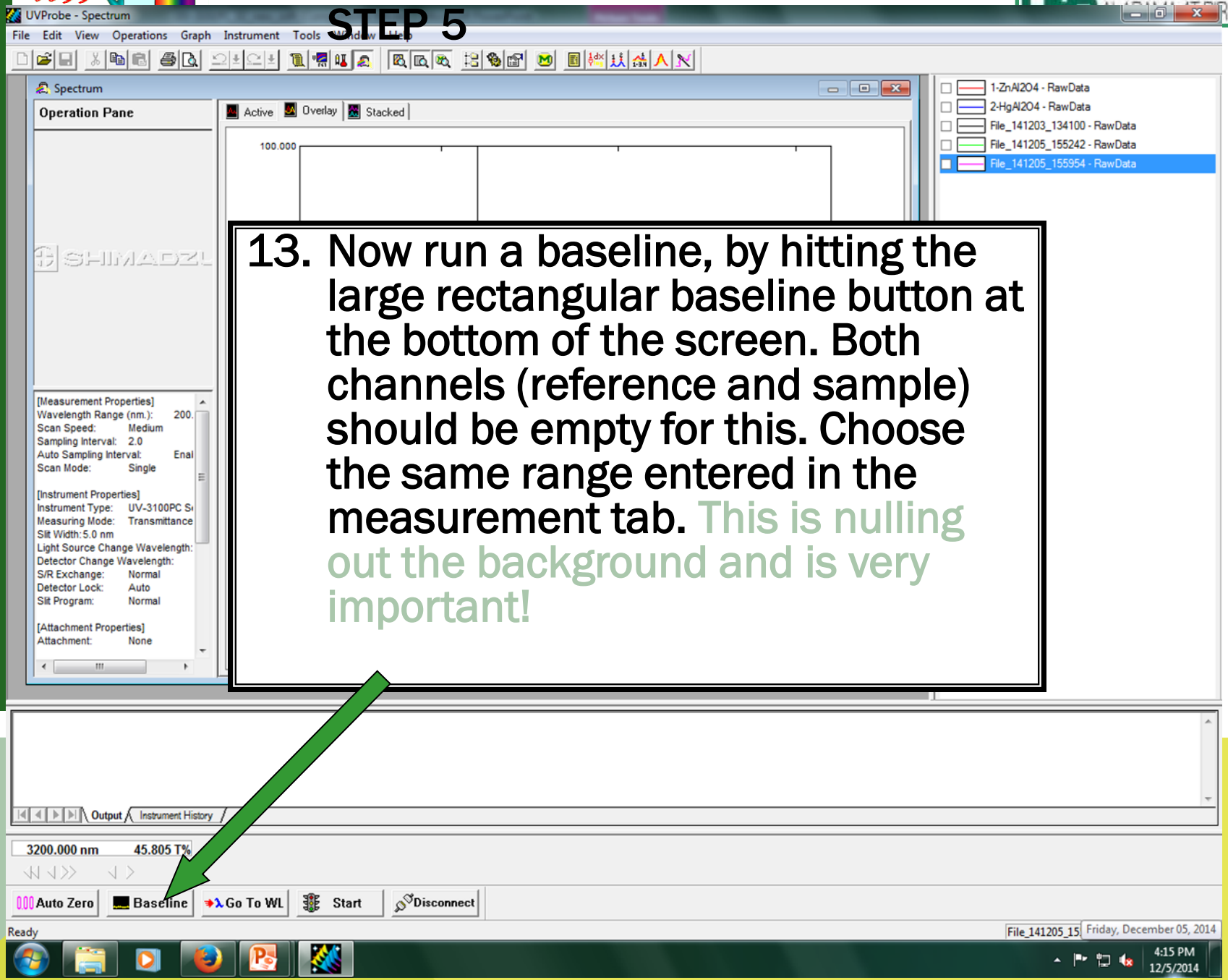
5. Check for an appropriate aperture set. The aperture should always be smaller than the sample size. However smaller aperture results in bigger signal noise. The same size aperture should be placed in the sample holder as in the reference holder. Fix sample holder in the chamber. Close sample chamber door

STEP 4



6. Select scanning range. Go to measure button, then select the measurement tab go to start and end wavelengths. Note the start wavelength should always be higher than the end wavelength.
7. Set the scan speed to medium. Slow can be done for more accurate results, but depending on the range, it may take too long to be practical.
8. Sampling interval-leave this on auto.
9. Now choose the instrument parameter tab.
10. The measuring mode should be in “transmission”.
11. The standard slit width is set to 8.0 but can be varied as dictated by the resolution needed for the sample lines. Narrower slits produce a higher resolution but also produce more noise. Wider slits have a lower resolution but also less noise. Proper slit selection can be estimated by knowing something about the material to be scanned line properties.
12. Press OK when these settings are entered.

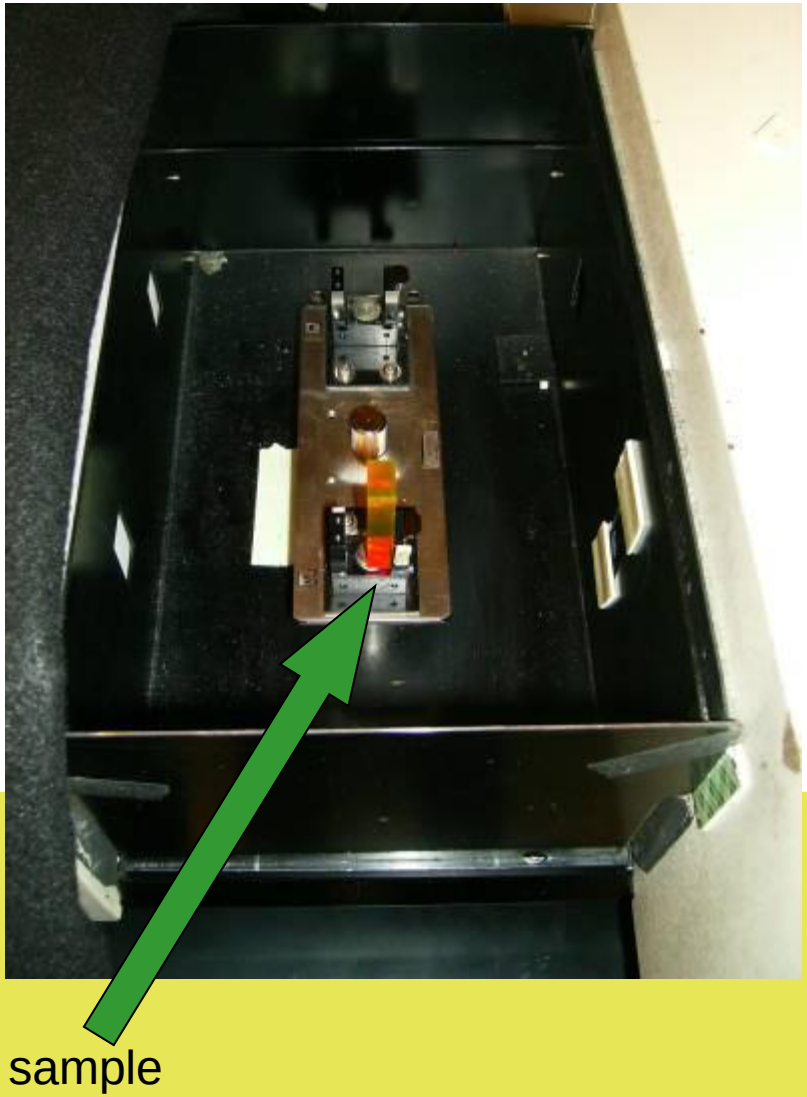
STEP 5



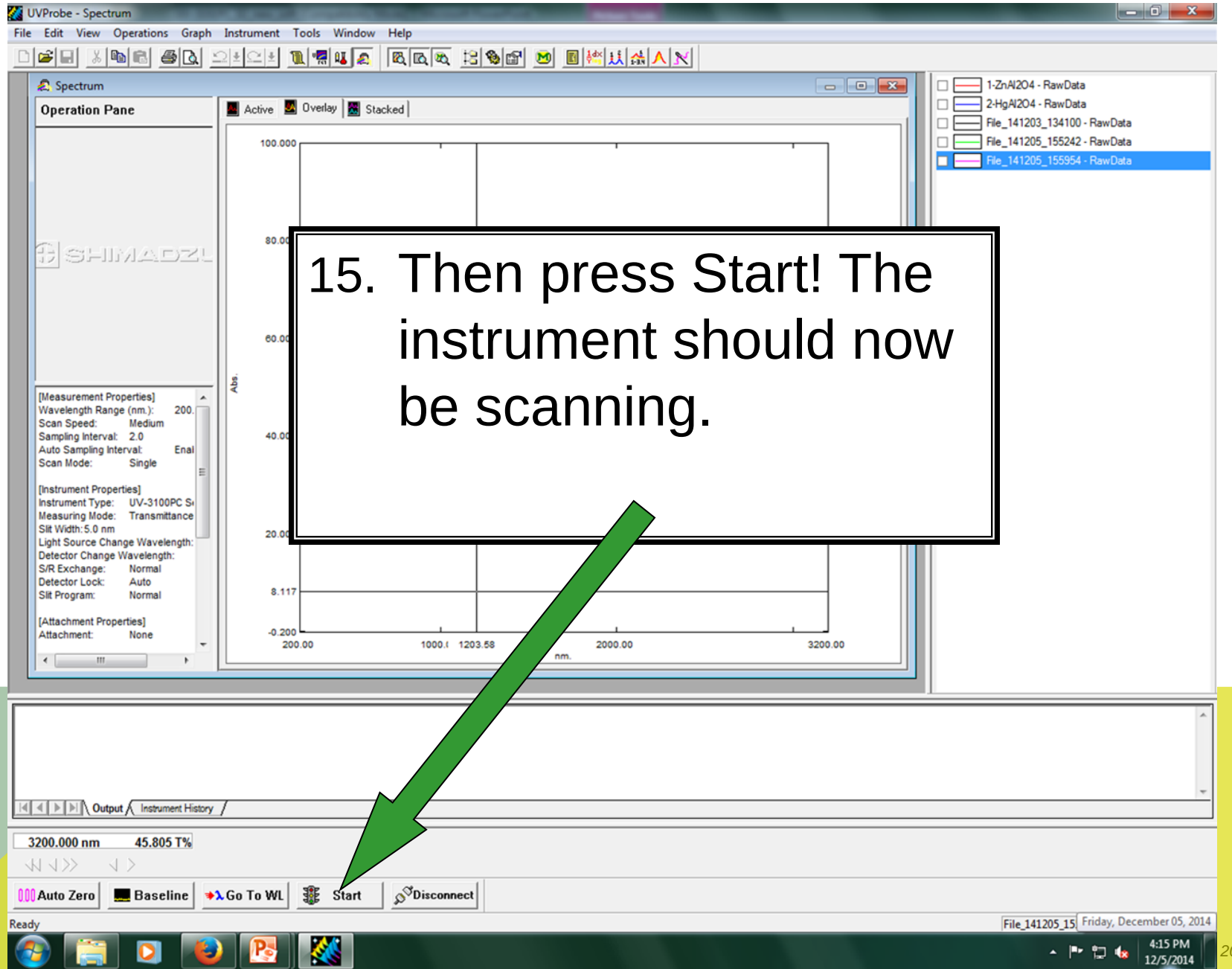
13. Now run a baseline, by hitting the large rectangular baseline button at the bottom of the screen. Both channels (reference and sample) should be empty for this. Choose the same range entered in the measurement tab. This is nulling out the background and is very important!

STEP 6

14. After the Baseline is completed. Place a sample in the sample holder. The sample holder is the one closest to you. Ensure that the sample completely covers the aperture and close the chamber, pulling the blanket a.k.a. “cloak of darkness” over the instrument.

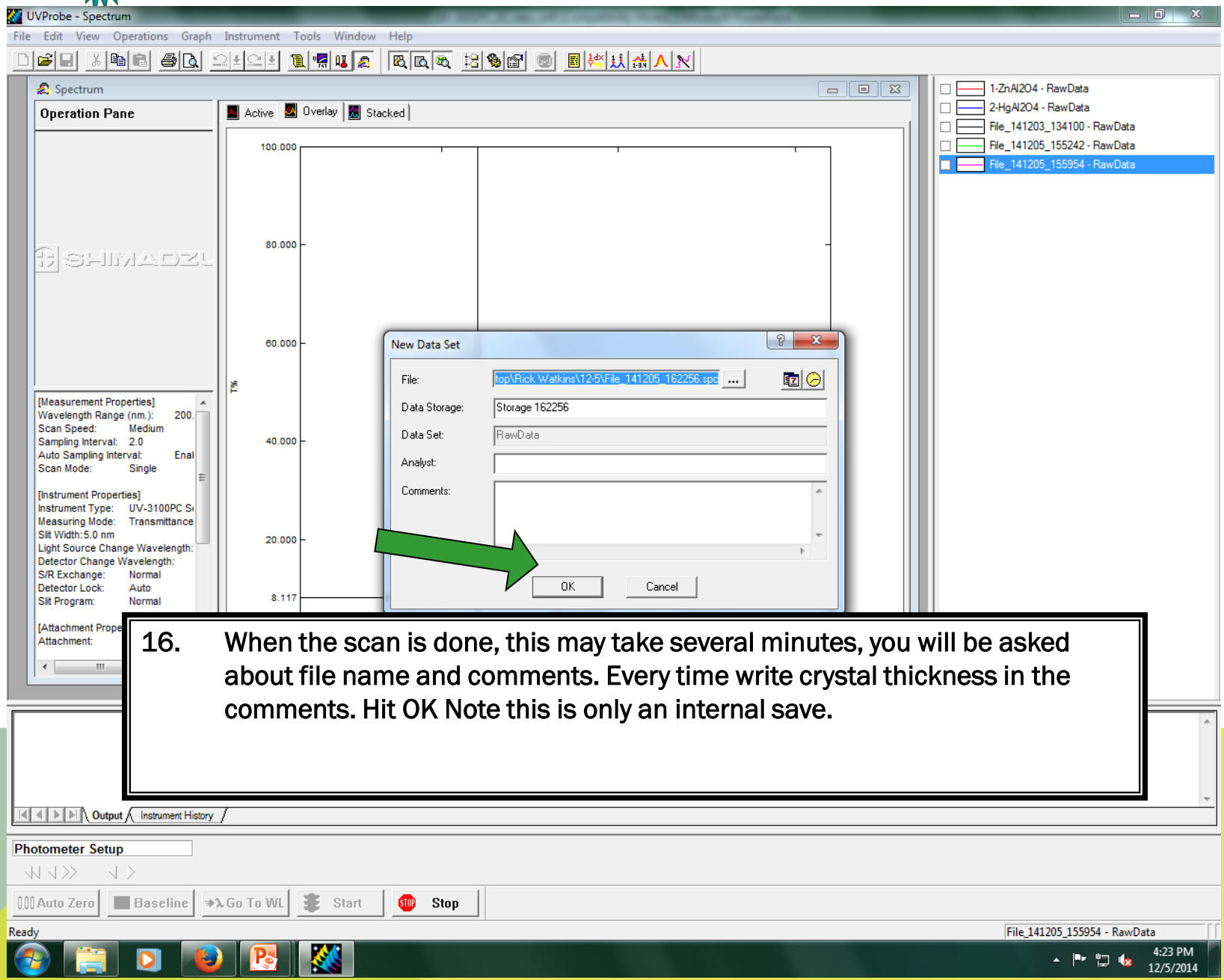


STEP 7



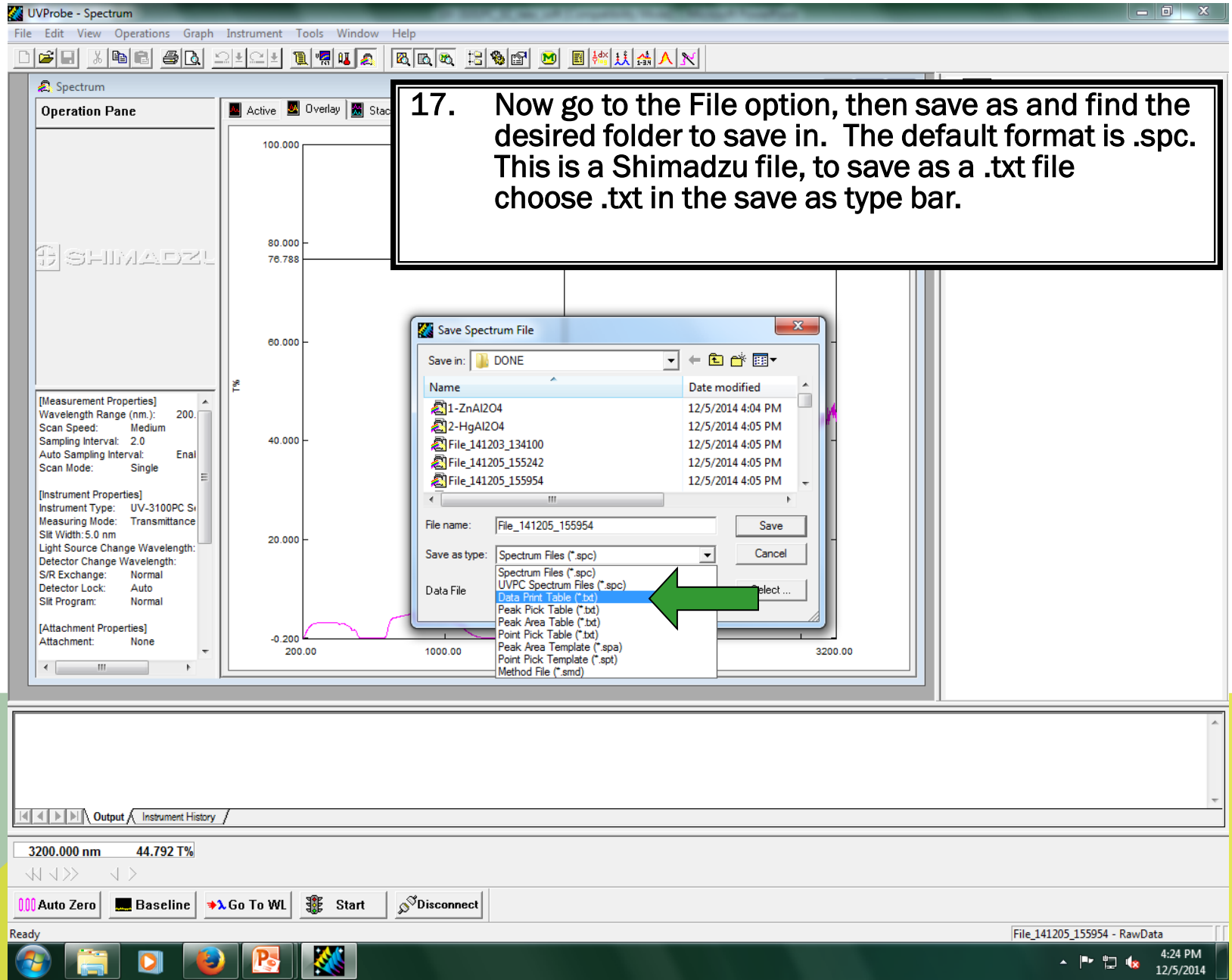
15. Then press Start! The instrument should now be scanning.

STEP 8



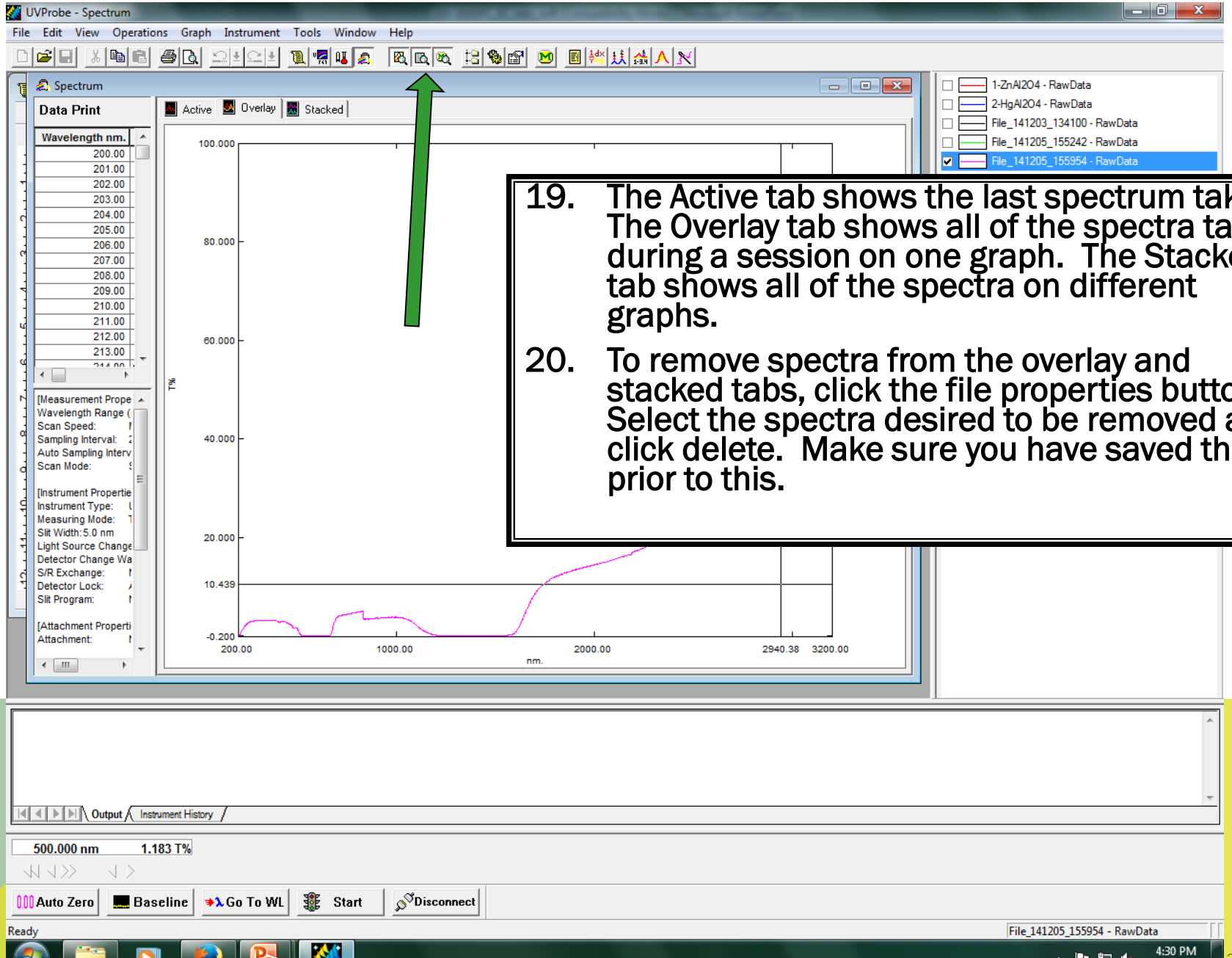
16. When the scan is done, this may take several minutes, you will be asked about file name and comments. Every time write crystal thickness in the comments. Hit OK Note this is only an internal save.

STEP 10



17. Now go to the File option, then save as and find the desired folder to save in. The default format is .spc. This is a Shimadzu file, to save as a .txt file choose .txt in the save as type bar.

STEP 10



19. The Active tab shows the last spectrum taken. The Overlay tab shows all of the spectra taken during a session on one graph. The Stacked tab shows all of the spectra on different graphs.

20. To remove spectra from the overlay and stacked tabs, click the file properties button. Select the spectra desired to be removed and click delete. Make sure you have saved them prior to this.

STEP 11

20. When done for the day, Close the program and turn off the Spectrophotometer by using the white rocker switch.

21. Make a copy of your files in your PC. Remember that files with “*.SPC” extension are written in internal SHIMADZU format; and files with “*.txt” extension are written in .txt format

TROUBLESHOOTING

- *Big Noise in Mid-IR Spectral Region*

To reduce noise in the 2-3 spectral range, one could take these precautionary steps:

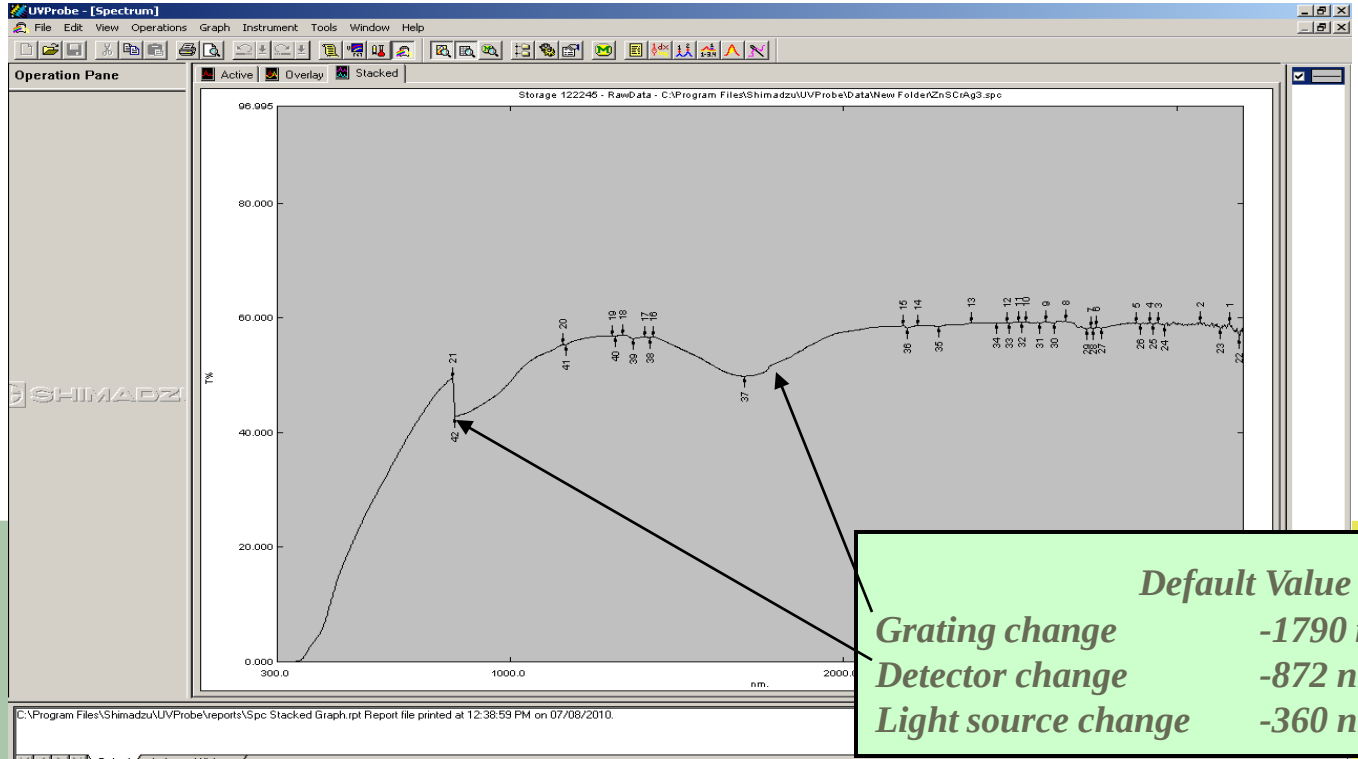
1. *Reduce scan speed (see slide 8)*
2. *Increase slits width (see slide 8)*
3. *Change absorber of the water vapor (silica gel)*

Ask assistants from Lab Personal !!!

TROUBLESHOOTING (CONT.)

- *Artifacts caused by Switching of the detector and Gratings*

There are several wavelengths where mechanical switching of the SHIMADZU *components are sensitive to the quality of the samples polishing and samples alignment. Spectral bump at these wavelength are artifacts and not related to the samples properties.*



	Default Value	*(Possible range)*
Grating change	*-1790 nm*	
Detector change	*-872 nm*	*(895-750 nm)*
Light source change	*-360 nm*	*(393-282nm)*

SHUT-DOWN
SPECTROPHOTOMETER

When done for the day, Close the program and turn off the Spectrophotometer by using the white rocker switch.
Remove your samples from the sample chamber