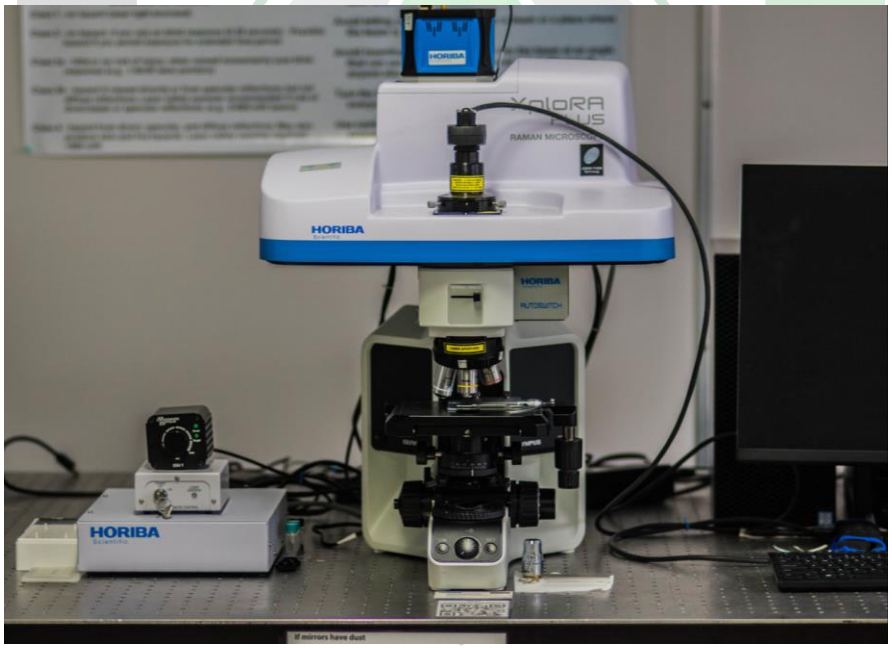
Acquiring Raman Spectra with Horiba XploRA Spectrometer



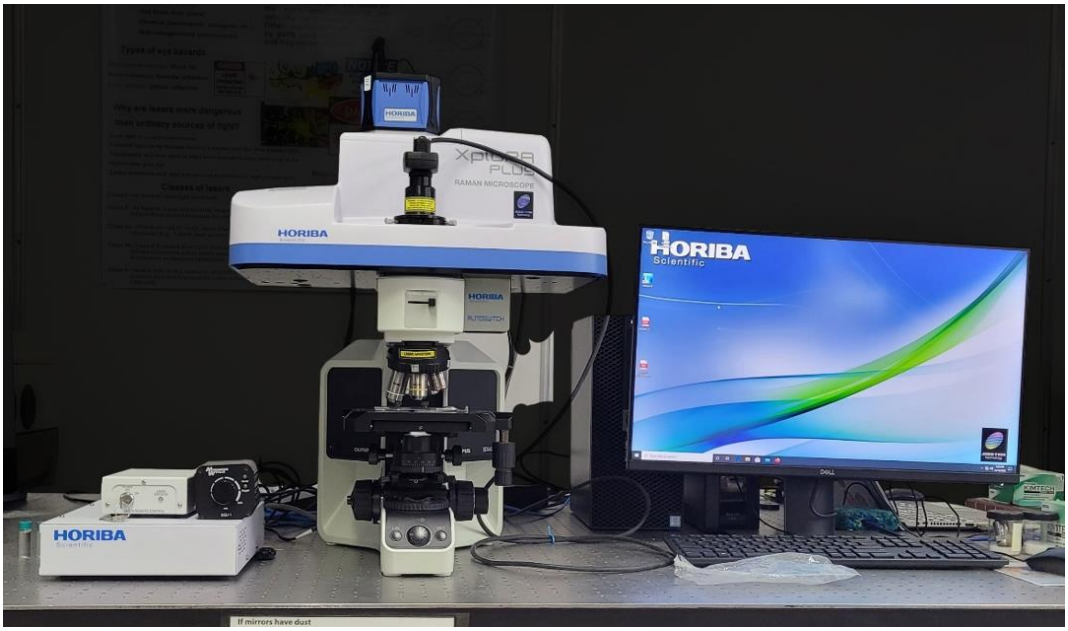
Created by: Annette Colón, Edwin Caballero

Manager: Samuel Hernández-Rivera

SOP-01	Annette Colón Edwin Caballero		University of Puerto Rico at Mayagüez
Effectivity: October/25/2022	Acquiring Raman spectra with Horiba XploRA Spectrometer		Revised by:
Revised:			Approved by:

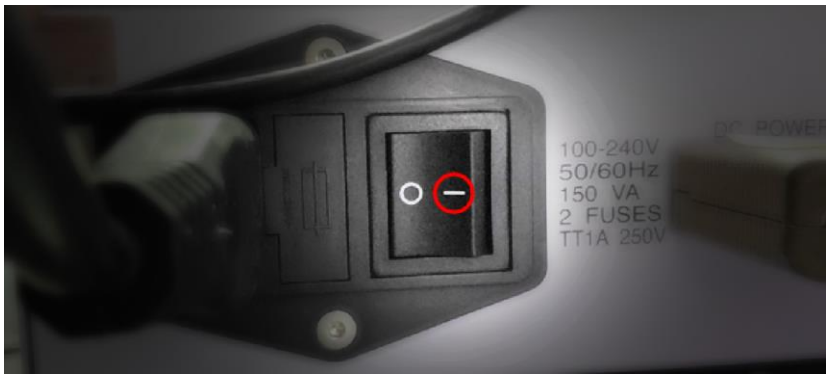
This SOP uses the following:

- Instrument: Horiba XploRA Raman spectrometer
- Laser: 638 nm
- Filter:
- Program: LabSpec6

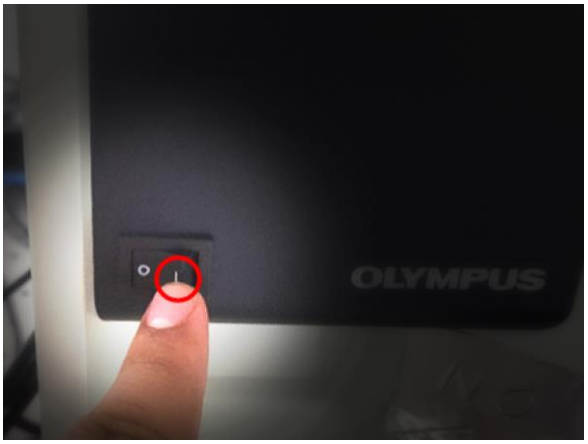


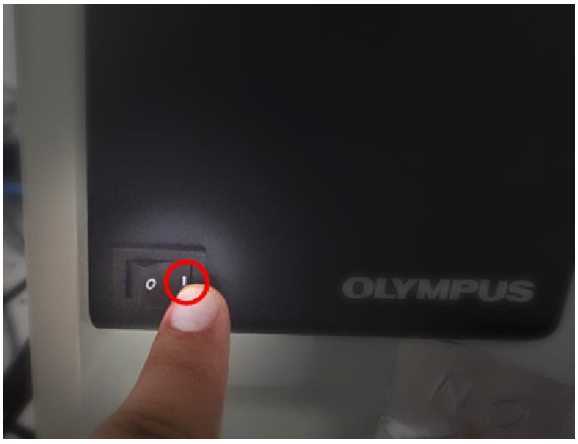
RAMAN SPECTROMETER SETUP

1. **Turn on** XploRA spectrometer by pressing the power button.

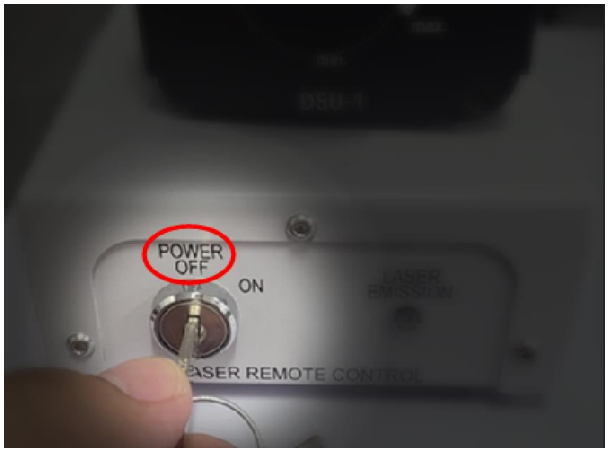


2. **Turn on** XploRA spectrometer by pressing its power (I/O) button.

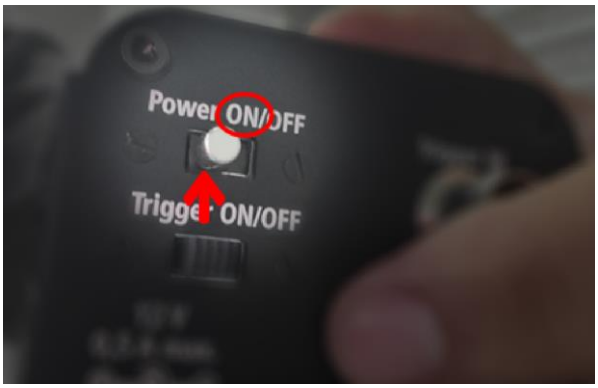




3. **Turn on** Laser remote control.



4. **Turn on** the white light power.



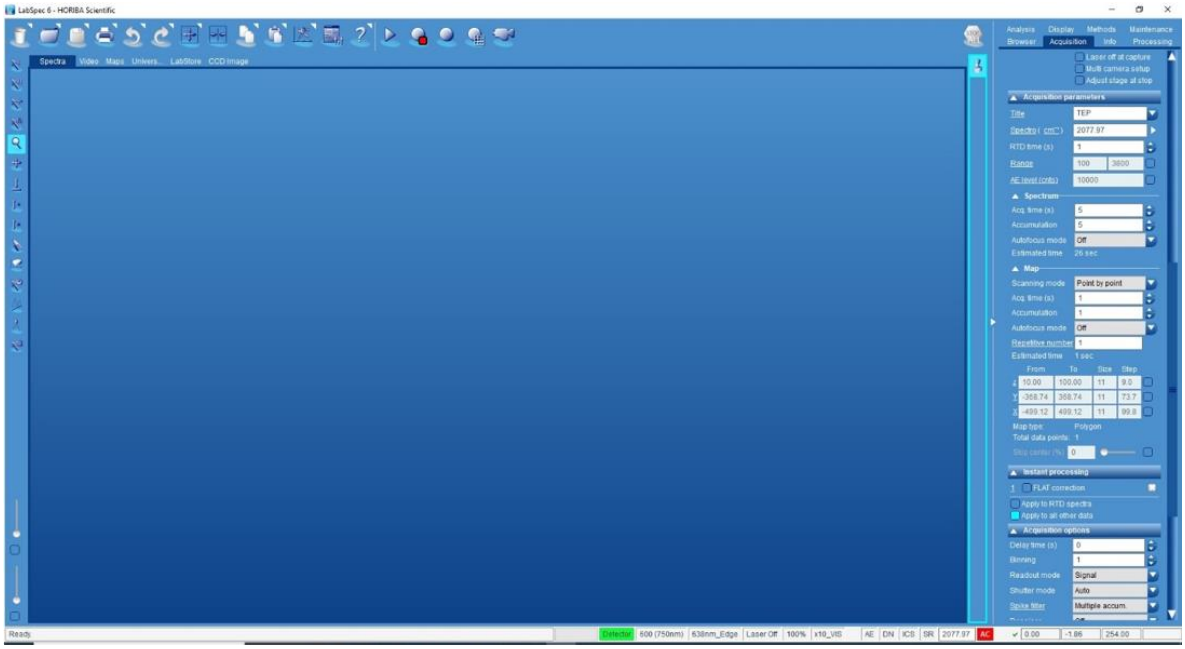
This is how the parts should look like.

5. **Open** the LabSpec6 program.


Recycle Bin
Raman Calibrations
HORIBA
Scientific
Stainless Steel Hori...
LabSpec6
Silicone Calibrations

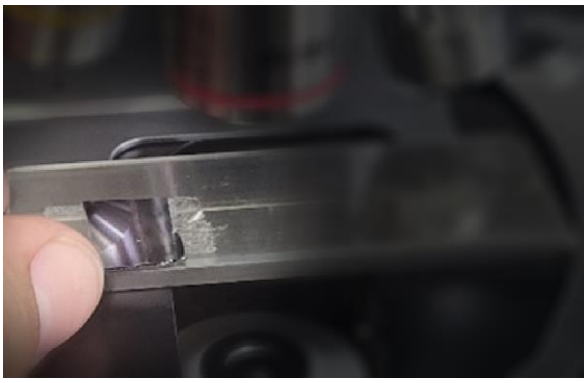

HORIBA
Scientific
LabSpec6 version: 6.5.2.11
License identification number: 2101806431_ls6_1
License reference person: Juan Lopez-Gamiga
Organization: University of Puerto Rico
LabSpec 6
Spectroscopy Suite
www.horiba.com/scientific

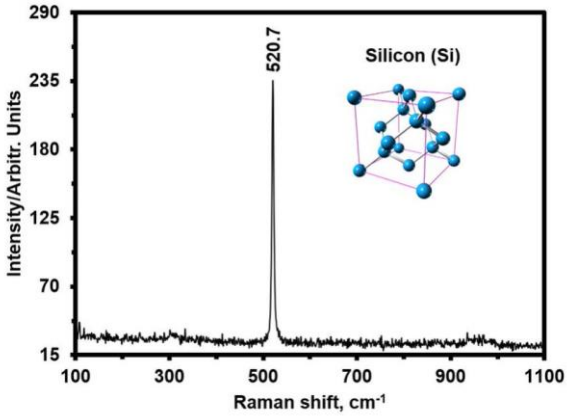
Main interface



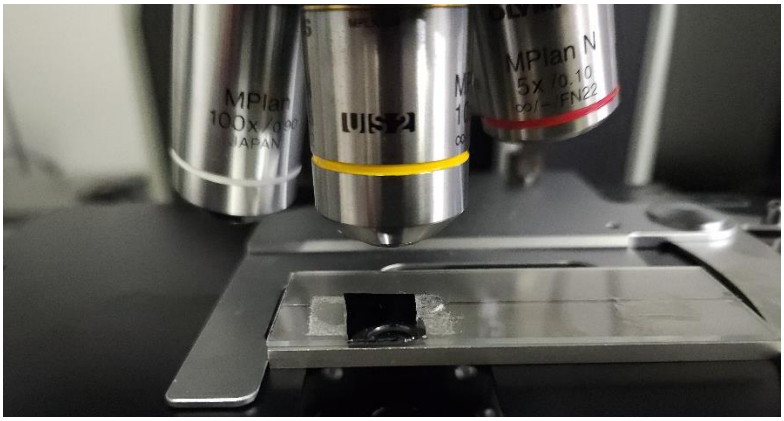
CALIBRATING SPECTROMETER

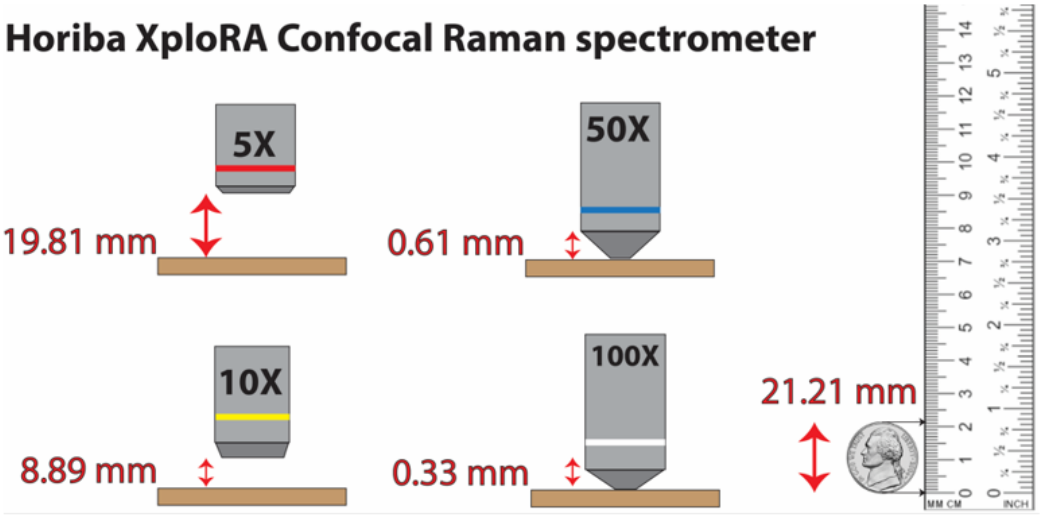
1. **Use** a silicon standard sample to calibrate the spectrometer.



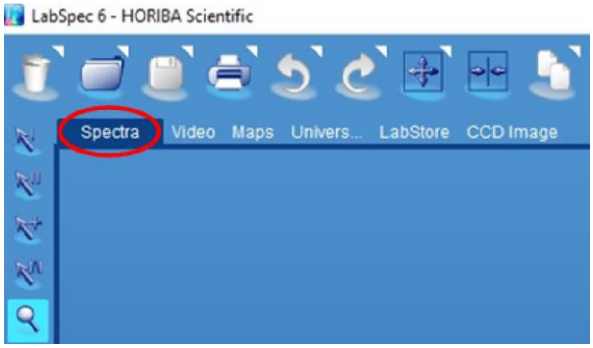


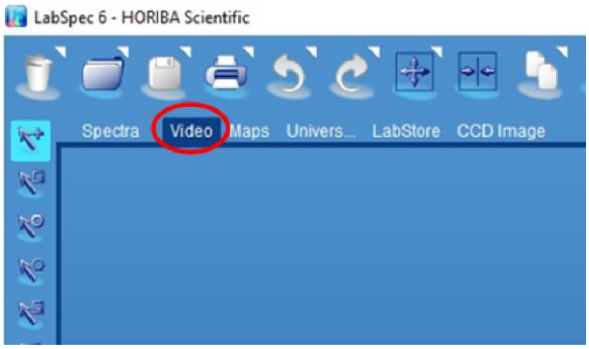
2. **Place** desired standard below the 10X optical objective.





3. **Change** mode from Raman to Viewing.

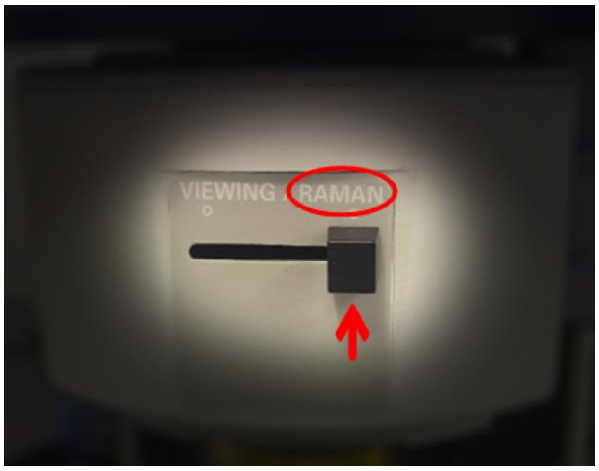


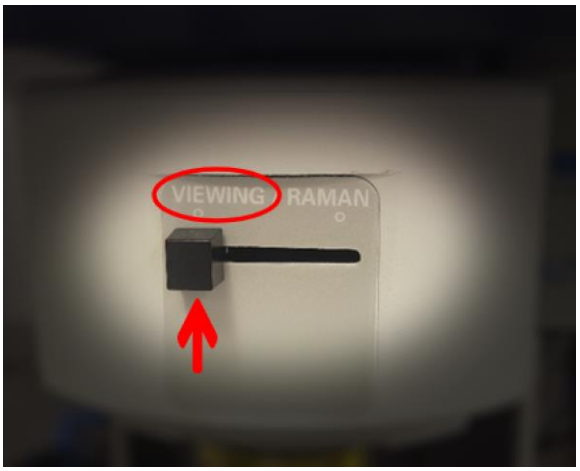


Raman spectrometer will move from the “RAMAN” mode to “VIEWING” mode.

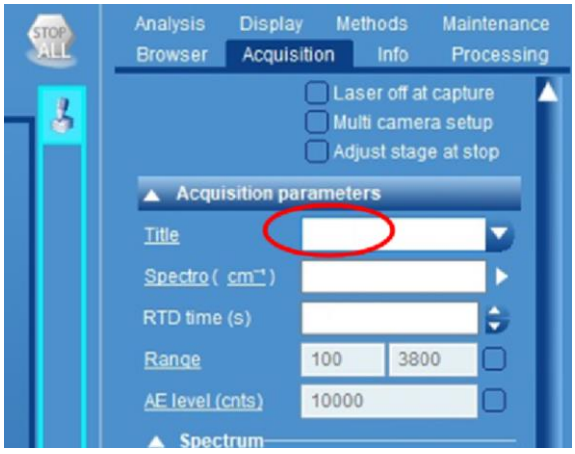
USER DOES NOT MOVE THE SLIDE. THE SLIDE MOVES BY ITSELF.

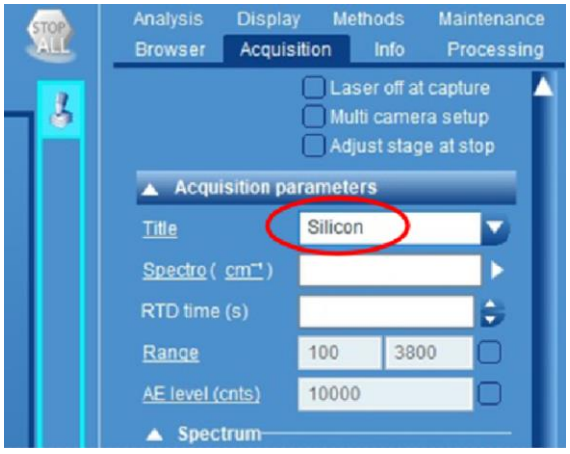
DO NOT TOUCH OR MOVE THE SLIDE



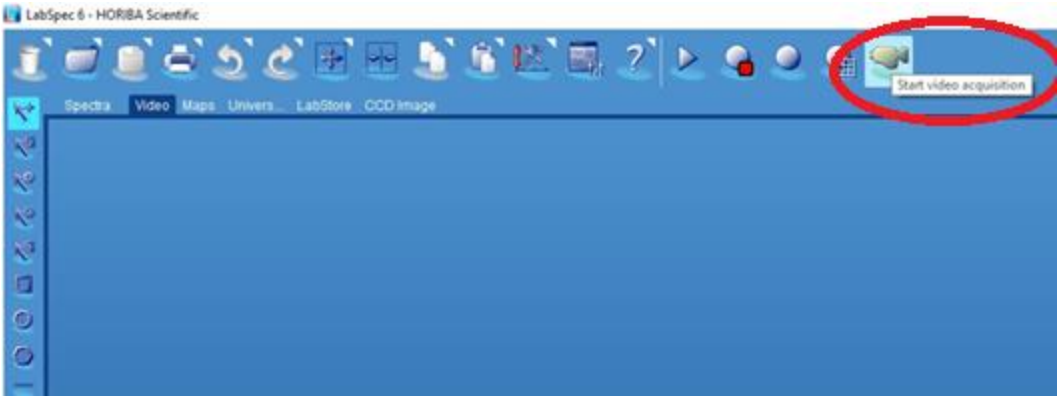


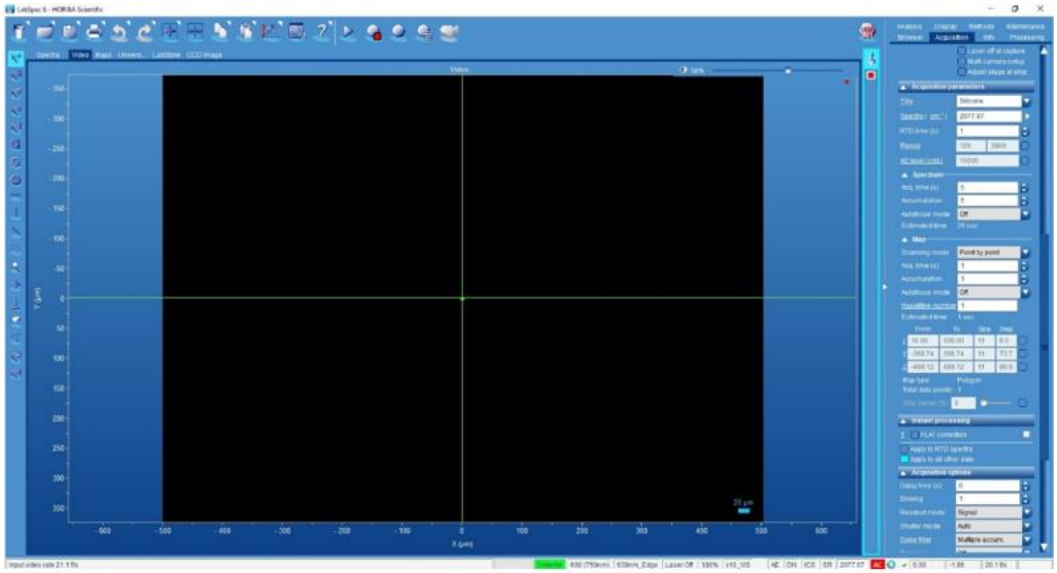
4. **Name** the sample in the Acquisition tab > Acquisition parameters > Title box.





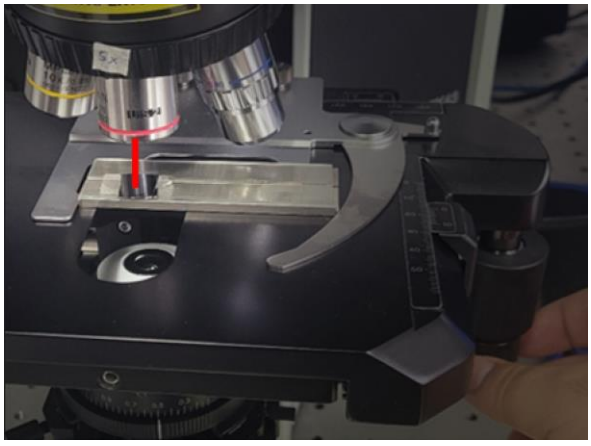
5. **Start** video acquisition to see the standard with the objective lens.



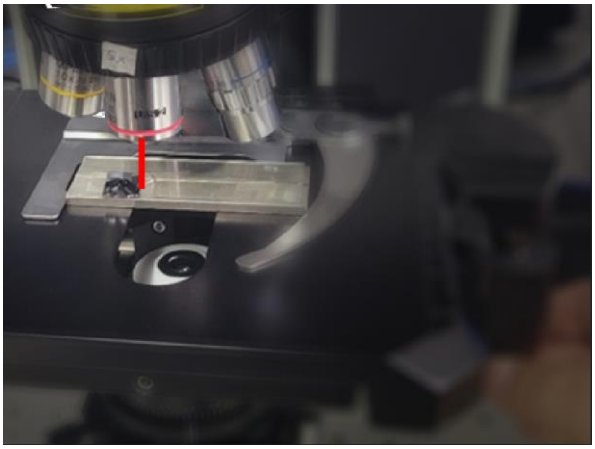


6. **Place** the border between the standard sample and the sample slide on the white light source.

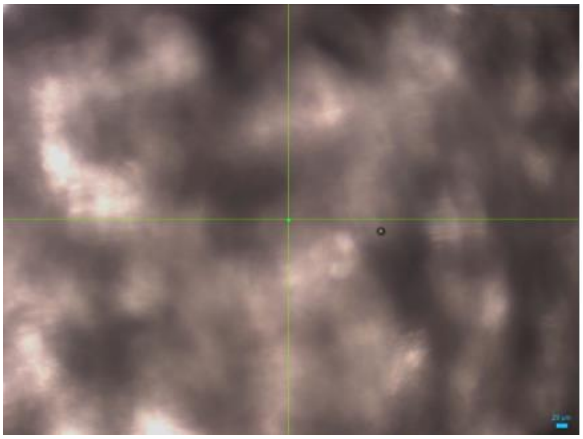
White light on the sample



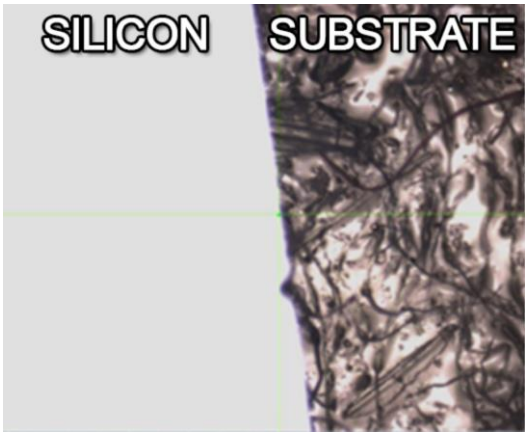
White light between sample and substrate



Not focused

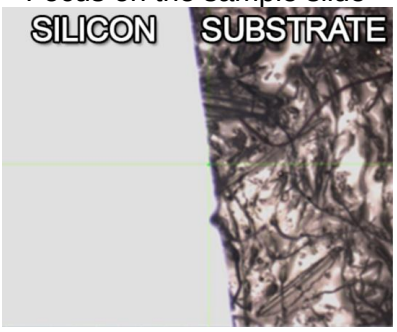


Focused

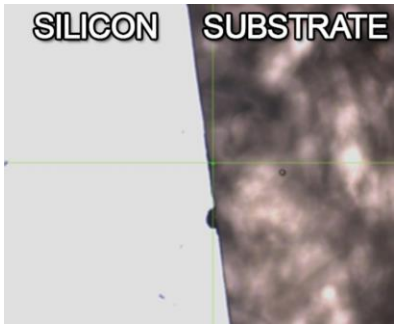


7. **Focus** standard by changing the height.

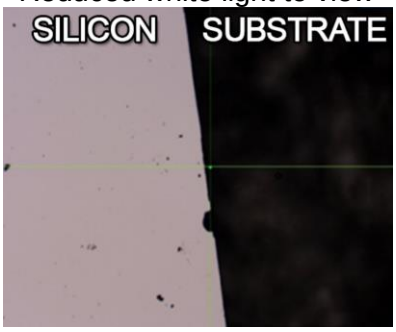
Focus on the sample slide



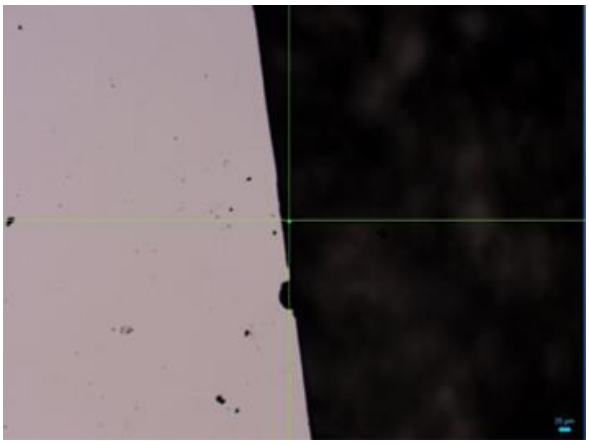
Focus on the standard

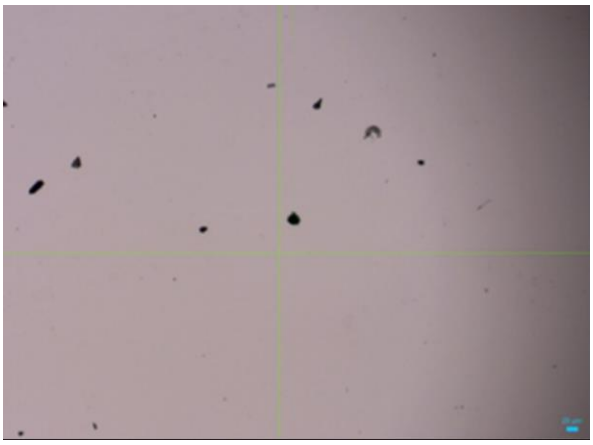


Reduced white light to view



8. **Move** substrate and center pointer on a dark spot.

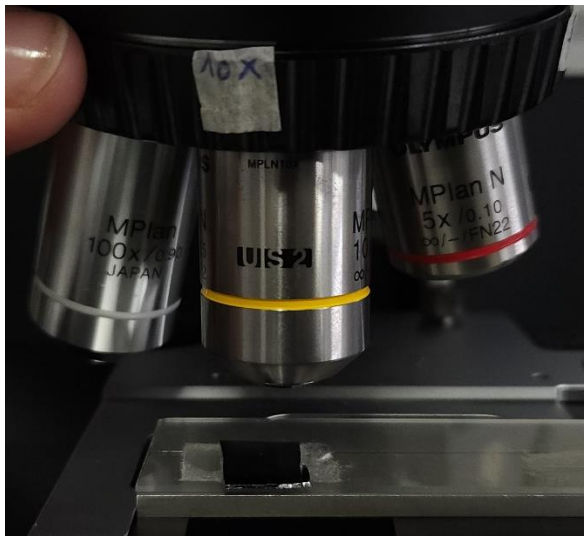


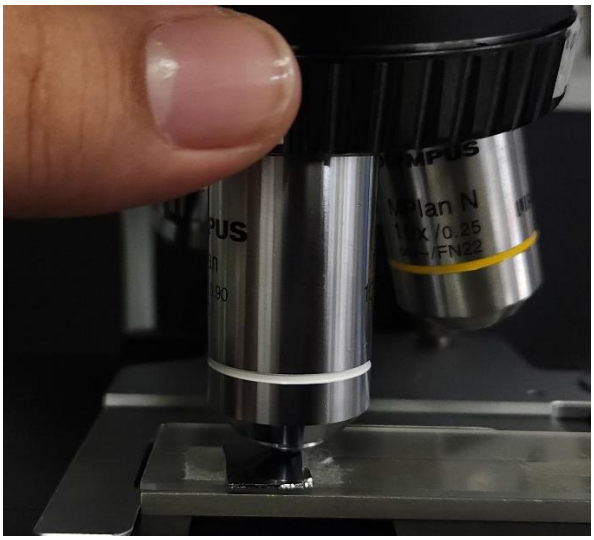


THE DARK SPOTS ARE INPURITIES!

We use the dark spots to focus when changing from 10X to 100X objective.

9. **Change** from 10X to an objective of 100X.





10. **Focus** 100X objective by changing the height.

NOTE: objective is focused when the focused image changes when moved in the x and y axis.

11. **Press** the AutoCal button.

12. **Select** the All lasers and gratings.

13. **Select** that the 100X objective and reference are placed.

The instrument will automatically calibrate the spectrometer taking samples of silicon with different gratings.

AutoCal with change from **RED** to **GREEN** when it is done.

Not calibrated

Calibrated

AutoCal usually lasts for 48 hours. If the button appears red, then you need to repeat the calibration process.

ACQUIRING SAMPLE SPECTRUM

1. **Place** 10X objective on the revolver of the Raman spectrometer.

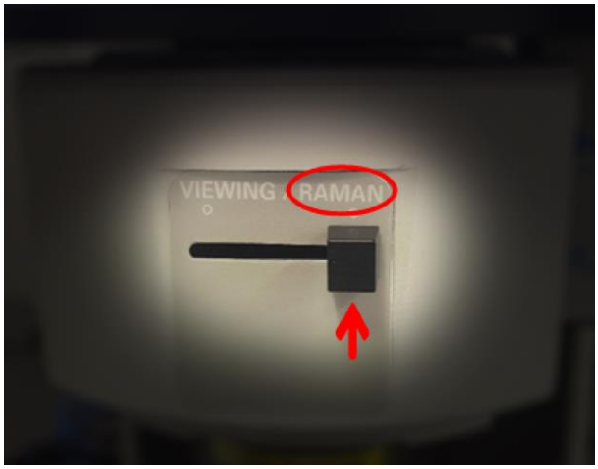
2. **Place** sample below the optical lens.

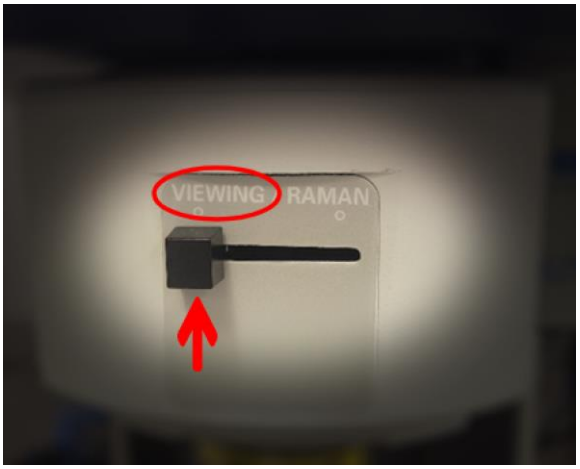
3. **Change** mode from Raman to Viewing.

Raman spectrometer will move from the “RAMAN” mode to “VIEWING” mode.

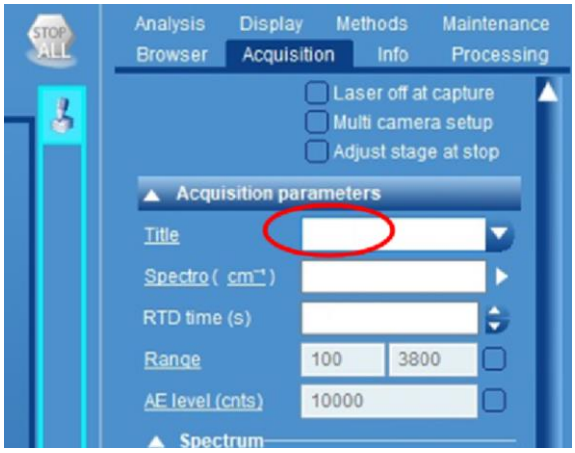
USER DOES NOT MOVE THE SLIDE. THE SLIDE MOVES BY ITSELF.

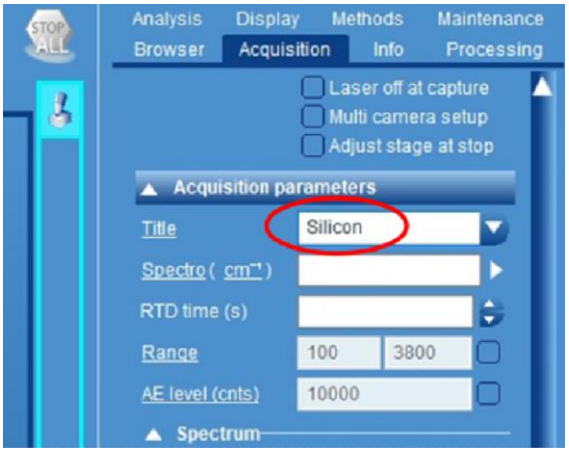
DO NOT TOUCH OR MOVE THE SLIDE



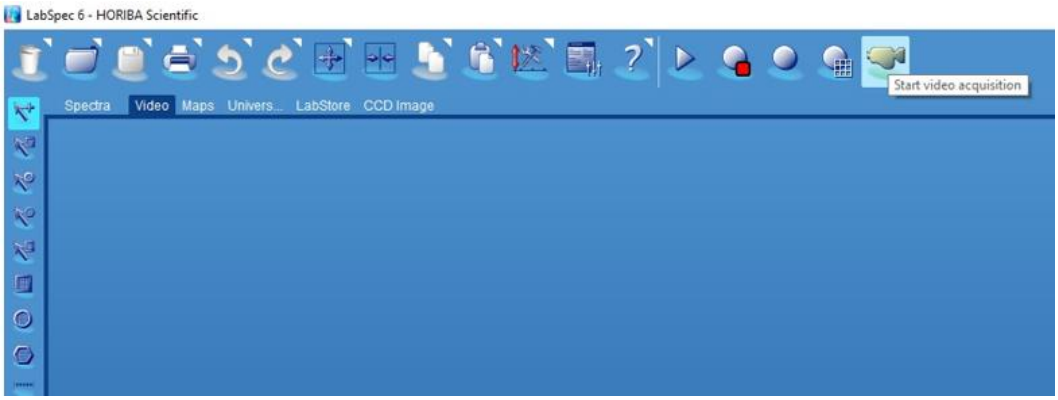


4. **Change** name of the sample in the Acquisition tab > Acquisition parameters > Title box.



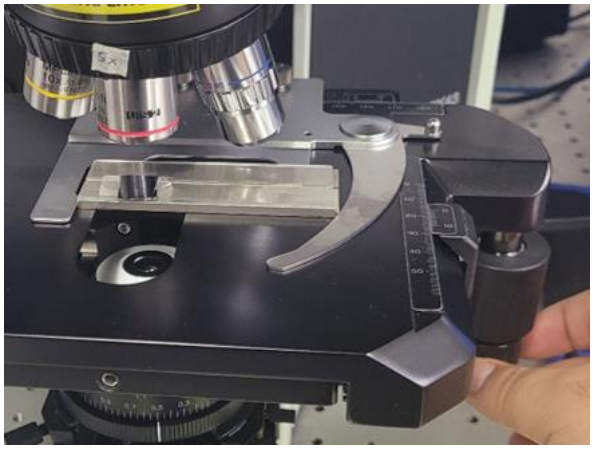


5. **Start** video acquisition to see the standard with the objective lens.

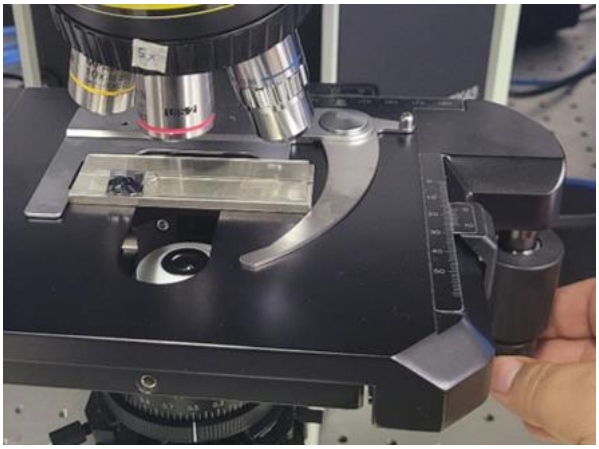


6. **Place** the border between the sample and the sample slide on the white light source.

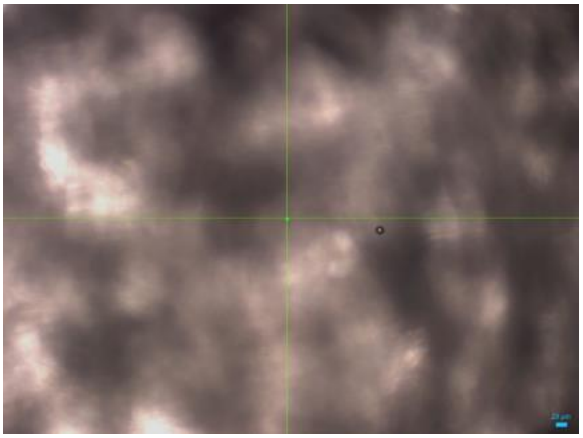
White light on the sample



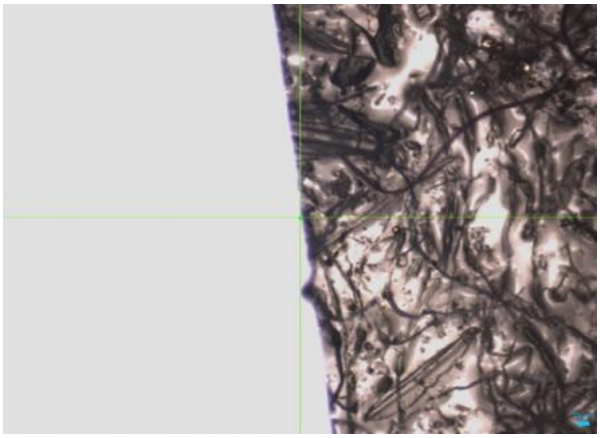
White light between sample and substrate



Not focused



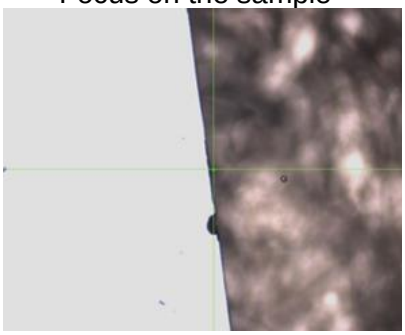
Focused



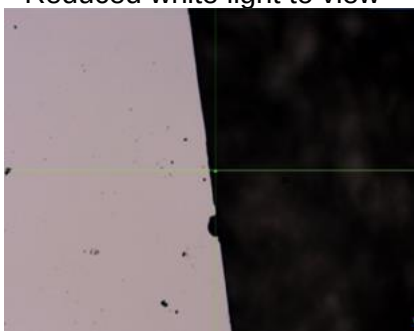
7. **Focus** standard by changing the height

Focus on the sample slide

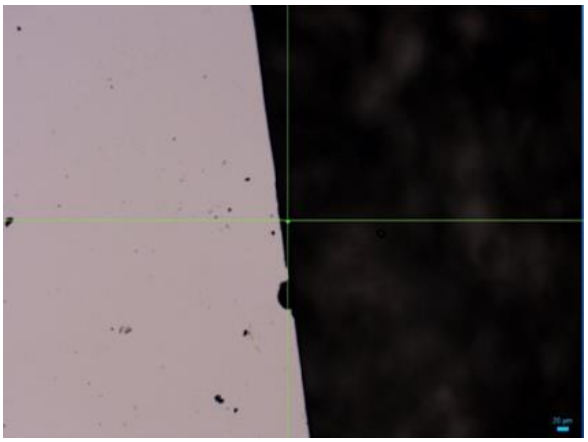
Focus on the sample

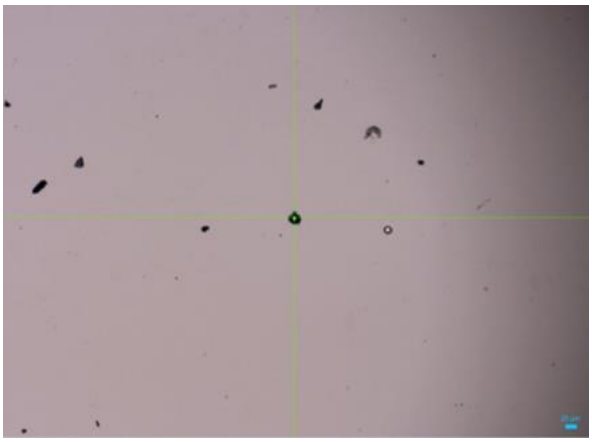


Reduced white light to view

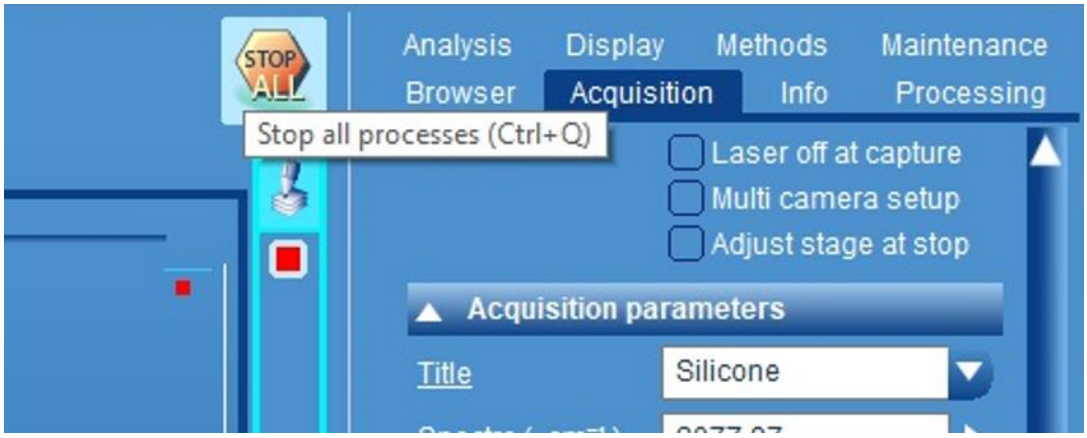


8. **Move** substrate and center pointer on the sample.

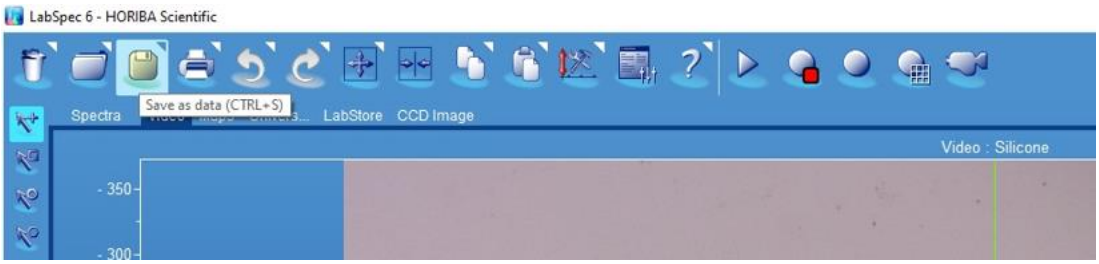




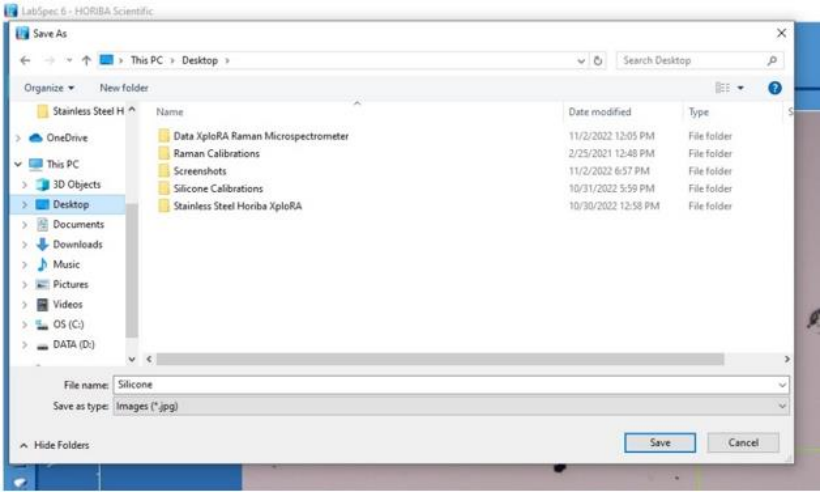
9. **Stop** video acquisition by clicking on the “STOP ALL” icon.



10. **Save** the acquired image by left clicking the floppy disk icon.



11. Find the desired place to save the image.



12. Save image in .jpg format.

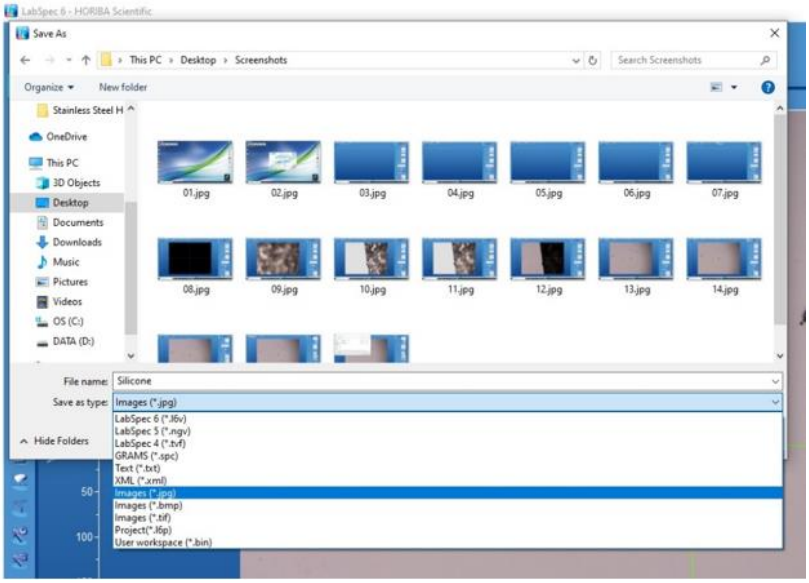
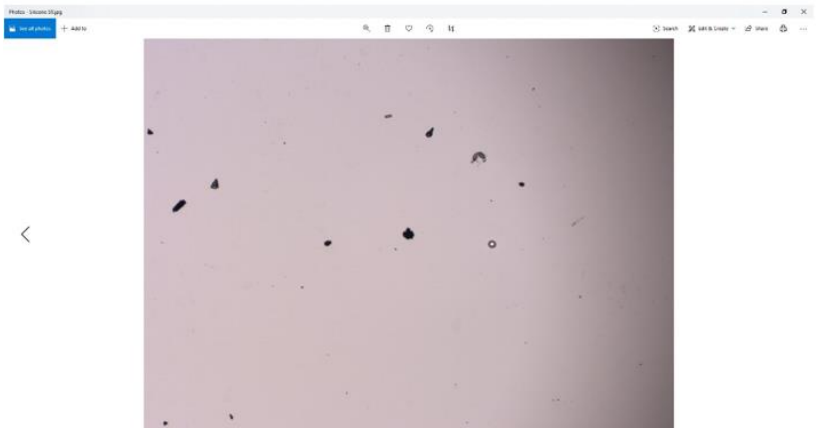
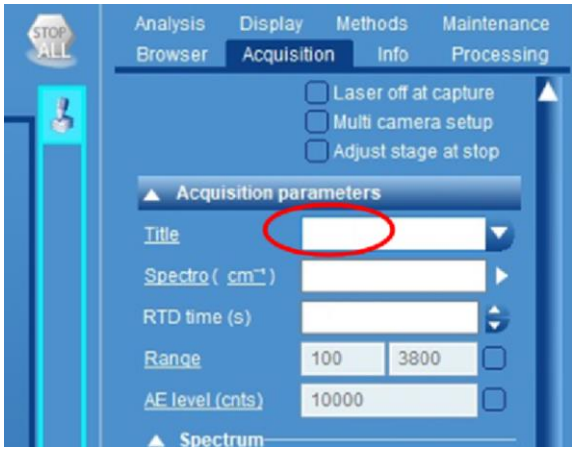


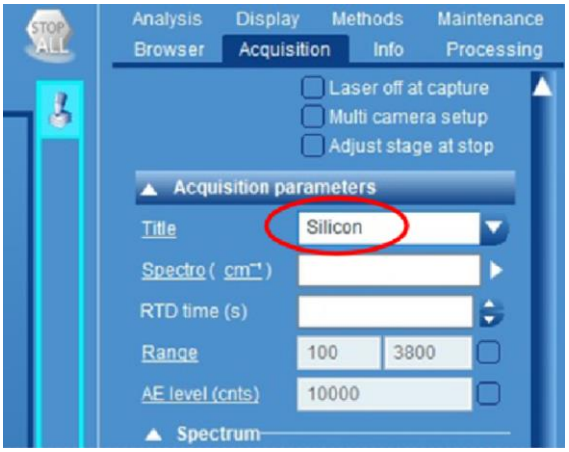
Image does not appear with scale.



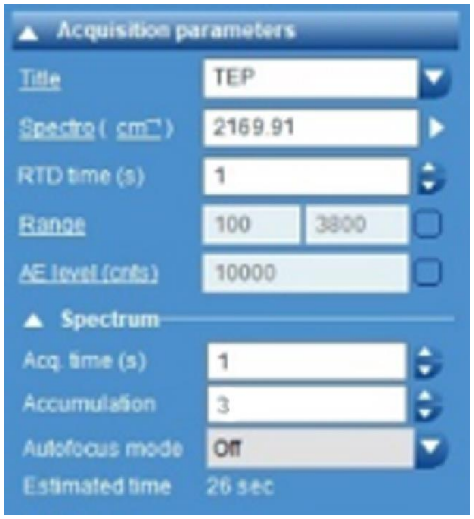
13. Click on “Spectra” tab.

14. Change name of the sample in the Acquisition tab > Acquisition parameters > Title box.

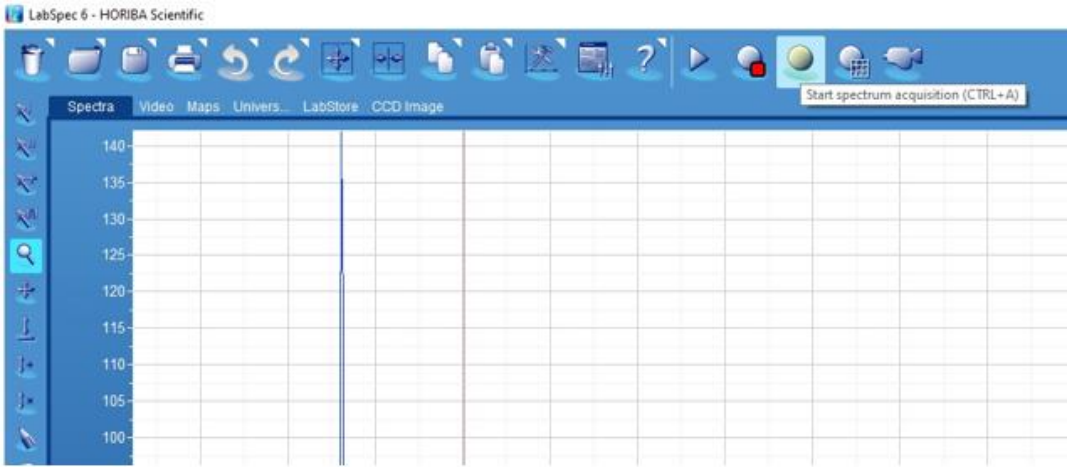




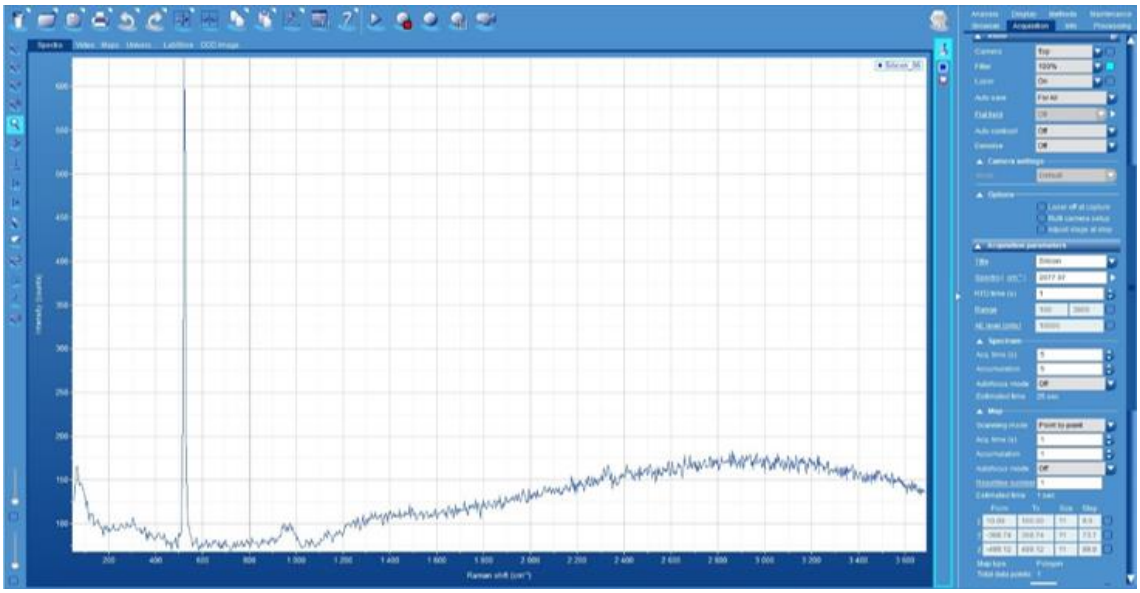
15. **Change** the acquisition parameters for the spectrum to 1 second of excitation time (ET) and 3 accumulations in the Acquisition tab > Acquisition parameters > Title box.



16. **Acquire** Raman spectrum by clicking on the “Start spectrum acquisition”.



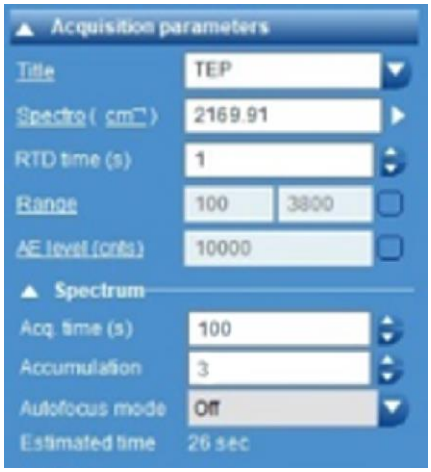
Resulting spectrum with acq time of 1 seconds and accumulation of 3.



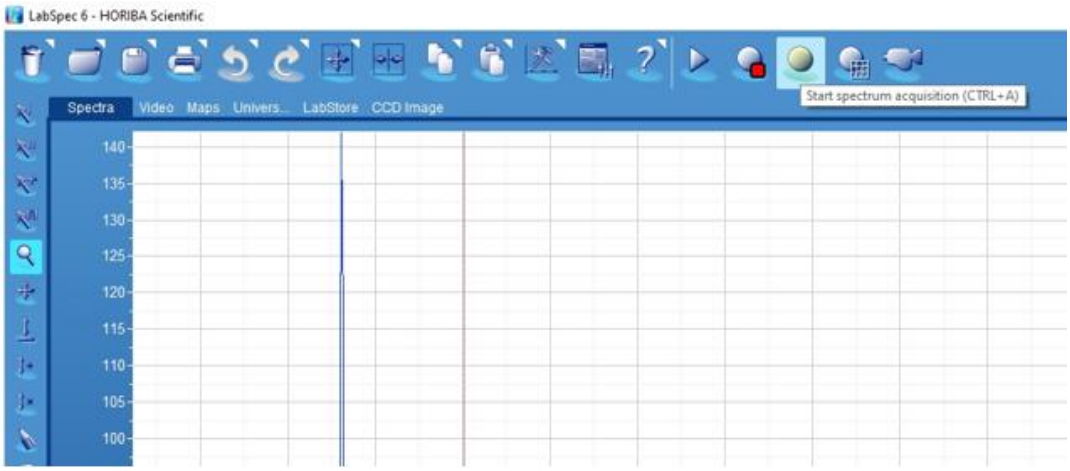
17. **Calculate** the amount of exposure time needed for the highest peak of the spectrum (e.g., 600 counts/s) to reach the maximum intensity counts without saturation (60,000 $(count)$).

$$60{,}000\ counts \cdot \frac{1\ s}{600\ counts} = 100\ s$$

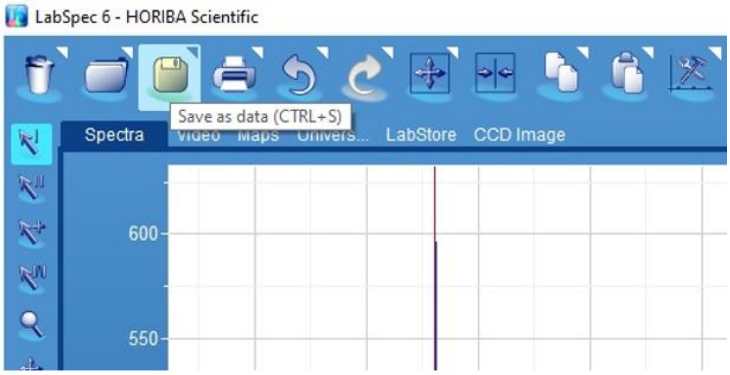
18. **Change** the acquisition parameters for the spectrum to 100 second of excitation time (ET) and 3 accumulations in the Acquisition tab > Acquisition parameters > Title box.



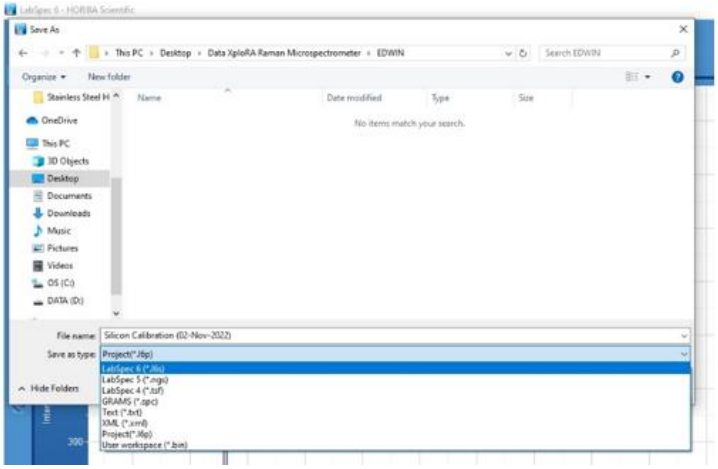
19. **Acquire** Raman spectrum by clicking on the “Start spectrum acquisition”.



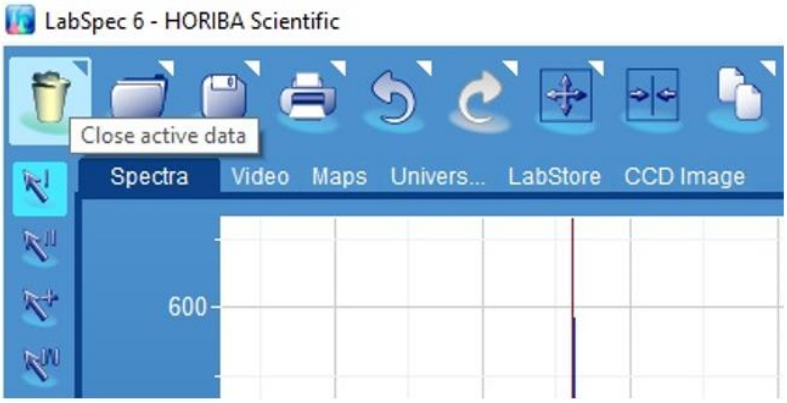
20. Save sample by clicking on the floppy disk icon.

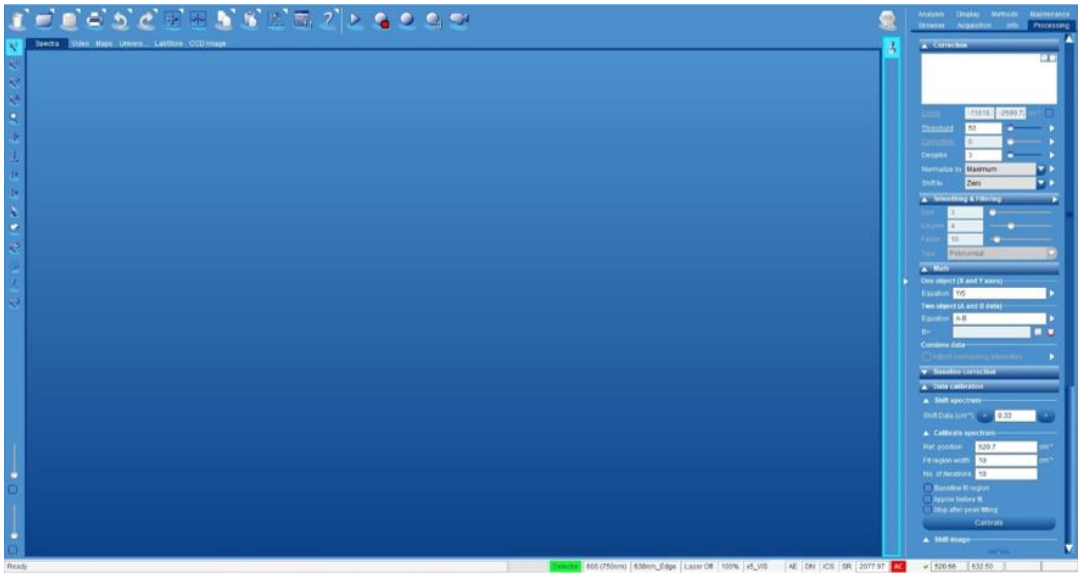


21. Save spectrum in LabSpec 6 (.l6s), GRAMS (.spc), and text (.txt) file individually.



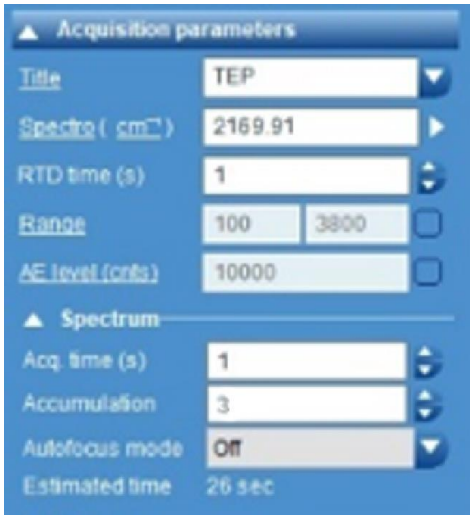
22. Delete spectrum by clicking on the trash can icon.


LabSpec 6 - HORIBA Scientific
Close active data
Spectra
Video
Maps
Univers...
LabStore
CCD Image
600

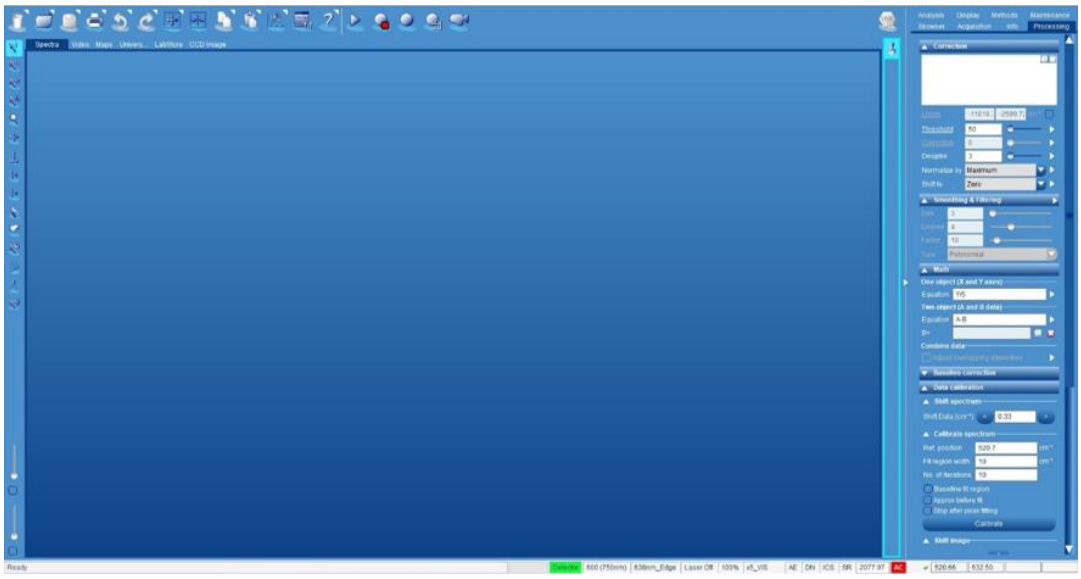


TURNING OFF SPECTROMETER

1. **Change** the acquisition parameters for the spectrum to 1 second of excitation time (ET) and 3 accumulations in the Acquisition tab > Acquisition parameters > Title box.



1. **Close** the LabSpec 6 program.



2. **Turn off** the white light power.

3. **Turn off** Laser remote control.

4. **Turn off** XploRA spectrometer by pressing its power (I/O) button.

5. **Leave** spectrometer on. The CCD detector can get damaged from humidity, the system prevents this when on.

TROUBLESHOOTING

1. **Stage does not go up to the desired height.**

Move objective out of the way from the stage and elevate the stage manually, setting a new base height. This change will allow the user to go up higher than before to accomplish a better focus.

Advisor Signature _____

Co-Advisor Signature _____