Acquiring Raman Spectra with Horiba XploRA Spectrometer



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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.





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

White light between sample and substrate





7. Focus standard by changing the height.





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



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.



Not focused

White light between sample and substrate



Focused





7. Focus standard by changing the height





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.

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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".

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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".

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

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21. Save spectrum in LabSpec 6 (.I6s), GRAMS (.spc), and text (.txt) file individually.

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

I LabSpec 6 - HORIBA Scientific





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