	Department of Physics National University of Singapore		Ref. No <i>SOP/001</i>
	Standard Operation Procedure Title: Confocal Raman spectroscopy measurement system		Rev. No <i>002</i> Pages: <i>8</i>
Lab: Nanomaterials & Devices Group			
Written by Chen Yuan	Approved by A/Prof Eda Goki	Issue date <i>25/12/2021</i>	Review date <i>25/12/2024</i>

1. Purpose

The objective of this SOP is to provide guidelines to all the laboratory personnel on operating confocal Raman spectroscopy measurement system.

2. Scope

The procedure is applicable to all research staff, research students and technical staff working in the laboratory.

3. Responsibility

It is the responsibility of the PI in conjunction with the laboratory I/C to ensure that all research and technical staff and students are advised, prepared and trained.


3.1. Principal Investigator

The principal investigator is responsible for the implementation of these guidelines and takes ownership of all research and technical staff, graduate and undergraduate students under his charge in ensuring that they will carry out their activities in a reasonably practicable manner. The PI has to ensure that all the personnel mentioned above are adequately advised, prepared and trained.

3.2. Staff / Students

All research and technical staff and graduate students are under the obligation to work and behave safely and are responsible for taking care of their own health and safety and not placing themselves or others at risk of injury

4. Personal protective equipment

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At a minimum, laser-proof glasses with side shields, chemically resistant gloves, and closed toed shoes should be worn. This is to be considered as minimum protection and must be upgraded if necessary.

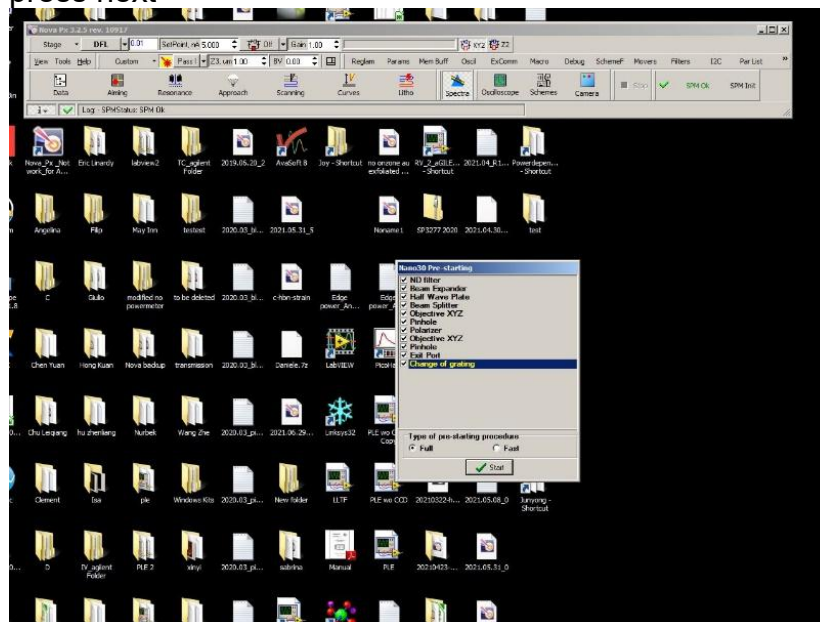
5. Safety precautions

Inspect equipment to be used and ensure all are in proper working condition. Report any equipment deficiencies prior to use.

6. Procedure

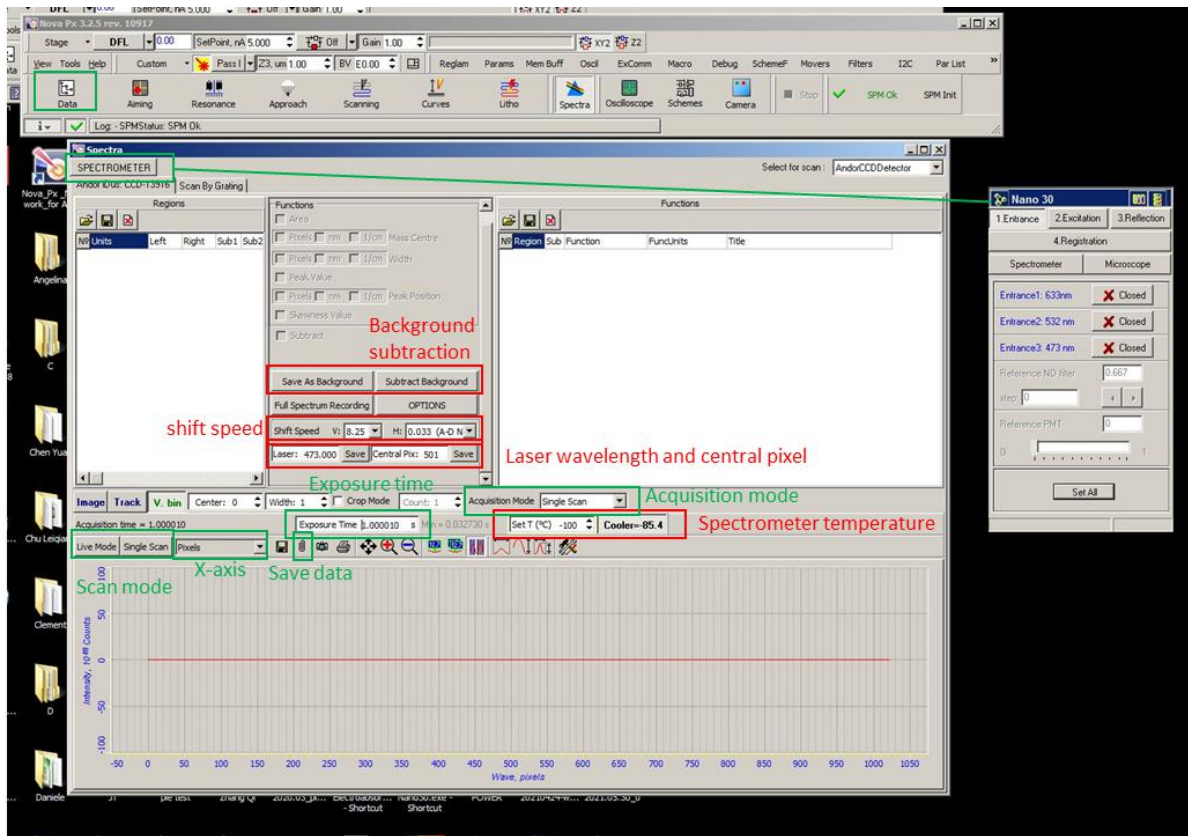
6.1 Photoluminescence spectroscopy measurement setup

- Personal protective equipment should be worn as required
- Turn on the laser that you intend to use (eg. 473nm)
- Turn on **Nova_Px** software on the third monitor (most right) (if is already turned on, skipped to step 5)
- Press **Spectra** and tick all the option and press start. After calibration, press next



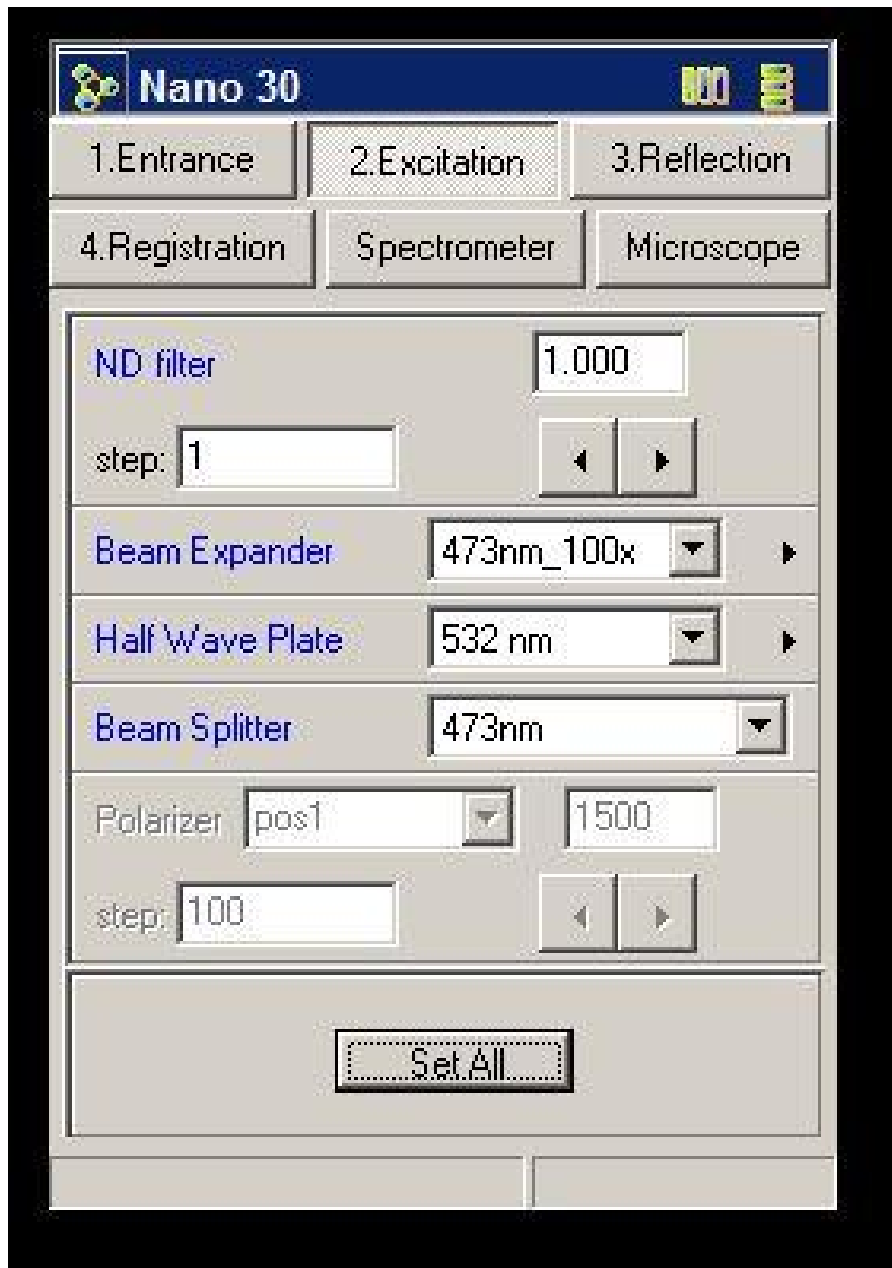
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Data storage



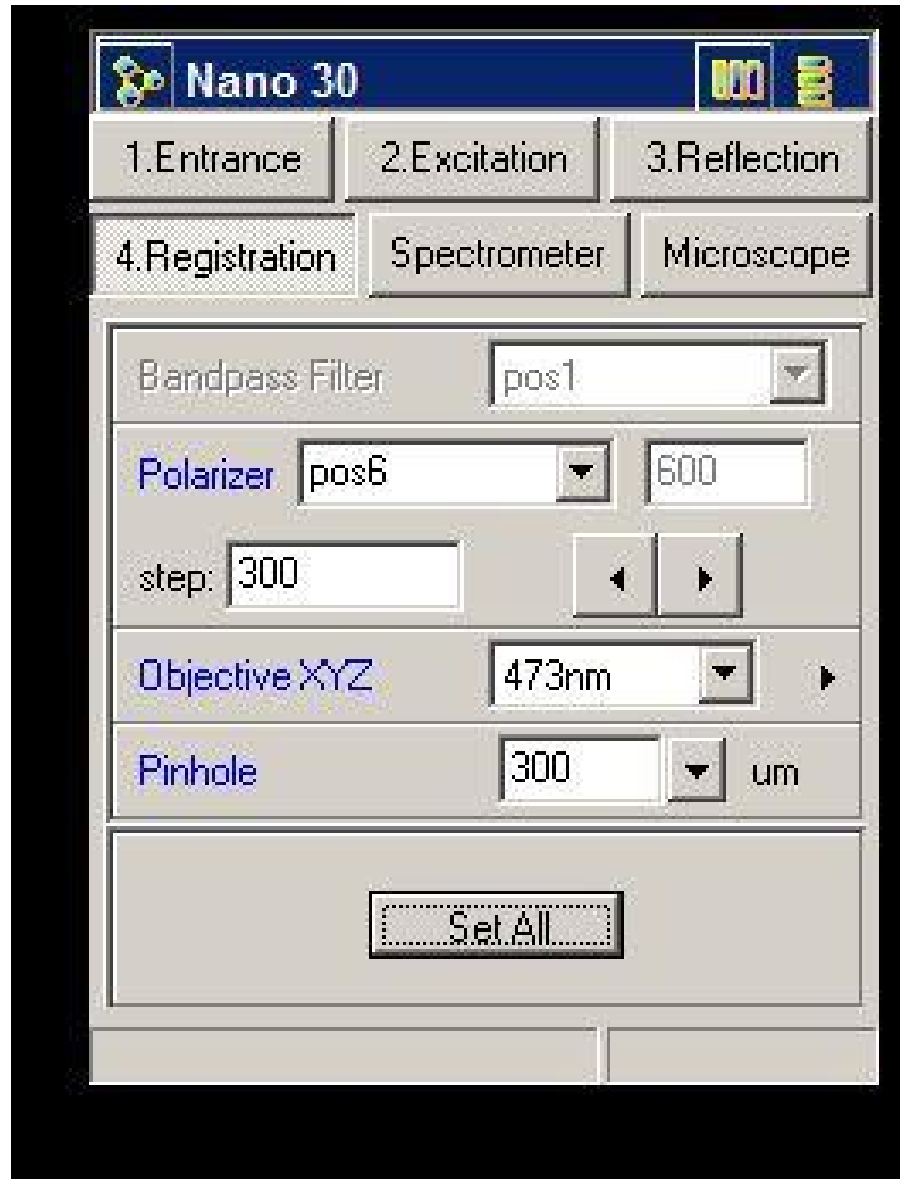
- This is the spectra interface. The ones in red will be set during calibration. The ones in green are changeable during measurement.
- **Set T (°C) to -100 °C**, and wait for the reading on cooler to drop to ~ -84 °C
- **Set shift speed V: 8.25 and H: 0.033**
- Click excitation, change the **beam expander** and **beam splitter** to the laser that you're using (473 in this case). Leave halfwave plate at 532nm (this is empty)

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- Click Registration, **set objective XYZ** accordingly (473)

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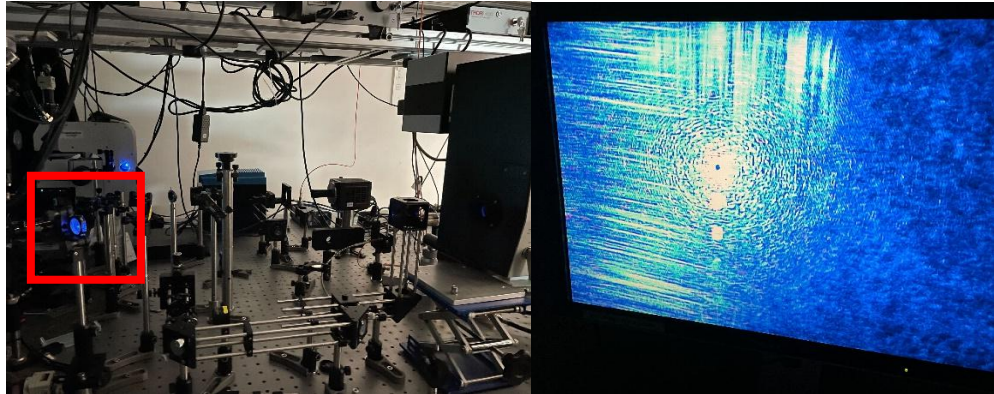
-
- Under spectrometer, change grating to **100/500** for PL and **2400/400** for Raman

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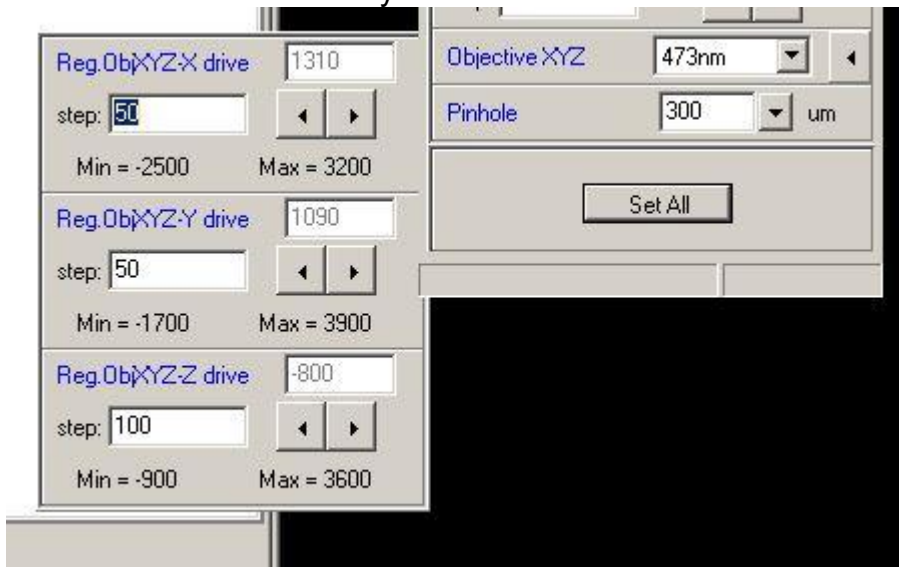


- Set **Wavelength to 0 nm** (this is **central wavelength**)
- Use normal **SiO2 substrate** for calibration
- Switch the **filter on the microscope (pink)**, open laser and align laser pathway. Try to use **this mirror only** to align the laser. then marked the center of laser on the screen.

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-
- Press **single scan (without laser)** to take background. press **save as background**, then **press substrate background**. (to retake background, uncheck these 2 boxes and repeat the step)
- Go to registration, change, press the left arrow before objective XYZ. Change Reg. ObjXYZ-drive and Reg. ObjXYZ-Y drive until you reached maximum intensity.



-
- You can change the size of pinhole to a smaller number (~ 100uM) for higher accuracy. Then **changed back to 300 um once you're done with calibration**.

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- Change **x axis to pixels**, and take the signal. Press save data, and **check what is the central pixel (highest signal)**. Change the central pixel accordingly
- Change **x axis to wavelength** and check if the signal is at **0 nm** (central wavelength that you set). If it is not, change the laser wavelength a bit, until the central wavelength is at 0nm. (eg. 473.25/ 473.5/ 472.5 etc.)
- Once the calibration is done, change the central wavelength accordingly. You can start measuring now.
- To **save data**: go to file > save all frames > your location
- To **export data**: go to file > export > ACSII (txt)
- Once you're done. Switch off laser and closed the shutter of the spectrometer.

6.2 Raman setup SOP


- The calibration is the same as PL
- Once you're done with calibration, change central wavelength to **540 nm**.
- Then, you change grating to **2400/400. Change grating will take a while**
- Tune the **laser wavelength** a little bit so that the Si raman signal is ~ 520- 521 cm^{-1} .
- Once you're done measuring, change the grating back to 100/500

7. Operation control

7.1. Administrative control

- All the keys of laser source is regulated by safety lead, only fully-trained personnel with NEA authorized laser license can use the laser system
- Laser precaution and safety label is pasted on the door of optical room

7.2. Engineering control

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- Laser optical path is enclosed by black foil

8. Revision History

Date Revised	Revision No.	Author	Revision Summary
20.12.2021	001	Justin Zhou Yong	
27.12.2021	002	Chen Yuan/ Chen Mingjun	Raman and PL spectroscopy measurement SOP