

Low temp PL (using the CCD) Standard operating procedure

1. Release the ventilator of the vacuum pump(open the vent valve, V).
2. Keep everything turned off except the main switch of the temperature controller of cold head.
3. Turn on the chiller.
4. Remove all shielding from the cryostat cold head and sample holder.
5. Load sample. Slide the cryostat on its stand to insert it into the Maple chamber as far as required and lock it.
6. Check alignment:
 - a) put on the pen torch at the detector end of the optical fiber attached to reflected PL collimator.
 - b) Adjust x-y positioners of the cryostat such that the light beam is incident on the desired part of the sample that will allow both reflective and transmittive PL to be done.
 - c) Fix the NEAR END stopper to mark the adjusted position.
7. Put on all shielding on the cryo-cold head and sample holder.
8. Turn on temperature controller.
9. Close the ventilator of the vacuum pump (close vent valve V). Keep the vacuum valve at the cryo end of the bellow closed.
10. Turn on vacuum gauge.
11. Turn on the main switch and the switch attached to vacuum pump.
12. After 30 seconds slowly open the vacuum valve at the cryo end of the bellow to align it along the axis of the bellow.
13. When the vacuum gauge reading is 2×10^{-2} Torr, turn on the water supply pump located near the chiller.
14. Slowly open the inlet and outlet valves for water supply in compressor.
15. Turn on the compressor. **At this time PC, Monocromator, LASER everything must be kept switched off.**
16. The ultimate reading of the vacuum gauge should be 10^{-3} Torr.
17. Turn on the LASER when temperature of the cold head becomes 100 K(reading B of the temperature controller) so that the LASER can stabilize before measurements start. Also turn on the cooling fan in sample chamber.

18. Within $1\frac{2}{7}$ hrs. of turning on of the cryostat the temperature of the cold head should go down to 10 K.
19. Turn on the switches in spike buster (Monochromator, PC, shutter controller, CCD power supply).
20. Move forward the cryostat cold head into the Maple chamber till it reaches the NEAR END stopper.
21. Turn on Monochromator.
22. Turn on PC.
23. Close input slit of the Monochromator.
24. Check whether the shutter of the sample chamber is closed ('NO' position of the shutter controller toggle switch).
25. Open Andor Solis software.
26. Click 'Video' icon to get real time signal → Click 'Runtime' icon and set exposure to 1 second (higher exposure gives better noise performance but it can also cause CCD signal to saturate). Check that the CCD temperature goes to -50°C (indicated at the bottom left hand corner of the Andor Solis software).
27. Open the shutter controller by putting toggle switch in 'NC' position
28. Adjust the Monochromator slit to 10-50 μm , depending on signal strength.

Make sure that signal strength is sufficient at 10 K since at higher temperature the PL intensity will decrease.

29. On the Andor Solis menu bar click 'Calibrate' and then 'x-calibrate by monochromator' → click 'Setup Monochromator' and fill in the following details:
 - a) Grating = 3
 - b) Grating descriptions 1200 line density and 750 nm blaze wavelength; 600 line density and 1.6 μm blaze wavelength and click OKClick 'Load Monochromator Defaults' → Fill in 'Center Frequency' → Click 'Calibrate'
30. Click 'Take Signal' icon and you should get the spectrum with the set Central Frequency and bandwidth 64 nm.
31. Click 'Abort' icon.

32. Click 'Acquisition Setup' on main menu bar → Go to 'Step and Glue' tab and fill in details like scan wavelength range, number of data points, etc.

For unknown sample it's better to go for 'step and glue' option since you get to scan over any wavelength range and locate the unknown peak positions. However, if the peak positions are known beforehand, the 'single scan' option can also be chosen.

33. Switch from 'NC' to 'NO' on the shutter controller to allow software control of the shutter.

34. Click 'Take Signal' to capture the spectrum. 'Save' as *.sif or 'Export As' ascii or other formats.

35. Change the set point of the temperature controller to next higher temperature and turn on the heater. Wait till sample temperature settles at the set temperature and repeat step 33.

36. At the end of the measurement exit the Andor Solis software.

37. Turn off heater by pressing "control" button on front panel of the temperature controller.

Shut down process

1. Turn off computer.
2. Turn off monochromator.
3. Turn off Laser.
4. Turn off all switches in the spike buster *except the temperature controller of the cold head.*
5. Turn off compressor.
6. After 10 min. slowly close the inlet and outlet valves for water supply to the compressor.
7. Turn off chiller and water supply pump.
8. Keep the vacuum pumps running for another 3-4 hrs so that the cold head reaches room temperature. Otherwise ice formation on shroud will damage the cryostat.

9. Slowly close the vacuum valve at the cryo-end of the bellow.
10. Turn off vacuum pump.
11. Don't vent the system after use.

Precautions:

1. Keep the Laser, Monochromator and PC off when turning ON/OFF the compressor. Otherwise the current spikes will damage them.
2. Do not touch the cold head with bare hands to avoid contamination. Always use gloves for sample loading/unloading.
3. Do not use your USB flash drive to transfer the data from operating PC. Use the dedicated USB drive or CD/DVD.
4. Do not shake the optical table when the measurement is in progress. It will give wrong readings.
5. When in doubt consult the SOP and User Manuals.
6. When you need to keep LASER on for long time but is not taking readings, keep the slit closed. However, do not keep LASER ON if the system is not in use since the life of the laser will diminish. Once the laser lifetime is crossed the laser power will drop drastically.
7. LASER power is 25 mW. Although it won't cause immediate injury but it's better to avoid direct incidence on eyes or body.