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### 1. Application to:

All personnel working on the Low Temperature Photoluminescence set-up (LT-PL)

- 2. Pre-requisite for Operation of LT-PL:
  - 2.1. Must know how to run PL software, lock-in amplifier, monochromator controller and Laser controllers.
  - 2.2. Must have attended Safety Orientation.
  - 2.3. Must have N3 Laser user license.
  - 2.4. Able to operate vacuum pumps.
- 3. Hazards that may be present:

3.1. Eyes damage.

4. Personal Protection Required:

4.1. Special Goggles, Gloves.

5. Procedures: 5.1. Operating the LT-PL

Put on the PPE as per section 4 before start work with Laser.

- 1. Open the Main Water Valve supplying cooling water to the respective taps at the side of the dark room. (Note: *Valve is open when it is parallel to the direction of the pipe.*)
- 2. Turn on the taps for the individual water supply to Laser (*only for Argon Laser*), Photomultiplier (PMT) detector and Compressor.





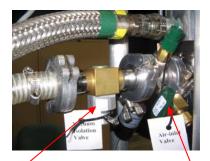




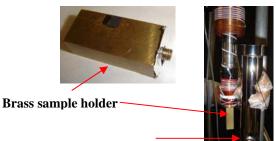
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# A) Mounting/Loading Sample

- 1. Close Vacuum Isolation Valve.
- 2. Open Air-inlet Valve to equalize pressure in chamber.
- 3. Pull down Vacuum Shroud (heavy easier with hand supporting base)
- 4. Unscrew the Radiant Heat Shield.







**Radiant Heat Shield** 

vacuum isolation valve air -inlet valve

Va

Vacuum shroud

- 5. Unscrew the Brass Sample Holder.
  - i. Apply a little vacuum grease to hold sample onto holder.
  - ii. For ease of focusing, place sample on the left side of holder.
- 6. Screw back the Brass Sample Holder and Radiant Heat Shield. Ensure the openings are perpendicularly aligned to each other.
- 7. Return the Vacuum Shroud while supporting the base, do the following:
  - i. Close the Air-inlet Valve.
  - ii. Start the Vacuum Pump.
  - iii. Open the Vacuum Isolation Valve.
- 8. Wait for chamber pressure to drop to about 20mTorr (<u>~40min</u>) based on the reading on the Pressure gauge before proceeding to switch on the compressor.
- 9. Leave vacuum pump "ON" until temperature in the chamber drops to 150K. Upon reaching 150K, close Vacuum Isolation Valve before switch the pump "Off".

<u>CAUTION:</u> – Negative pressure in chamber may suck oil from the Vacuum Pump into the chamber will <u>contaminate chamber</u>.

10. While waiting, check the water supply.

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## B) System setup

- a. Turn on the following:
  - i. Switch on main switch at the wall and turn on Cryostat Compressor (when pressure gauge is 20mTorr or less)
  - ii. Switch on Temperature Controller (set to the desired temperature)- (Minimum) 4 K and (Maximum) 300K

**Temperature Control Setting** 

Press the "set-point" button in the temperature controller, key in the desired temperature, then press "Enter".

When heater is needed, press the "Heater on/off", select "Low" or "Med" range then press "Enter". Do not use "High" heater range.

Remember to turn "off" the heater after using.

iii. Switch on PMT cooler (at least 30min before turning PMT power supply on)





temperature controller



PMT cooler



Ar-ion power supply

- iv. Laser power supply
  - A. He-Cd Laser
    - a. Turn on laser power supply.
    - b.Wait for 20 minutes to warm up the laser.
  - B) Argon ion Laser
    - a. Turn on the laser controller.
    - b.Set laser power at 10mW on the remote controller
    - c. Adjust & focus the laser

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- 11. Turn on the following
  - i. Chopper: check frequency 276Hz
  - ii. PMT power supply: slowly increase to -1100V and lock the knob (*1 hour before doing the measurement*)
  - iii. Monochromator controller and lock-in amplifier (software must be closed before turning on the lock-in amplifier and monochromator controller)



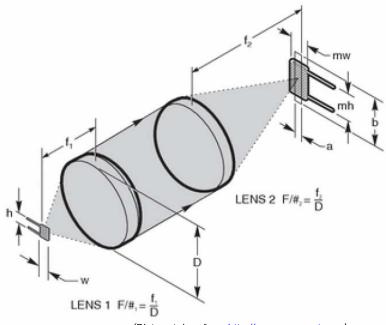
PMT power supply

- 12. Set the Monochromator slit width to your need (usually set to 200 µm (1 complete rotation))
- 13. Start PL software. Enter calibration wavelength before starting measurement.
- 14. Wait for temperature to reach the desired set-point. (Typically ~90min to reach 4K from room temperature).

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# C) PL Measurement

- a. Focus laser beam onto sample by adjusting mirror
- b. Two lens are used (condenser and secondary focusing lens) to collect light from sample into the Monochromator.



(Picture taken from <u>http://www.newport.com</u>)

- c. f1=124mm, d1=55mm, f2=300mm, d2=65mm (coated)
- d. Check lens alignment by adjusting L1 and L2 and ensure light is focused into the slit of the Monochromator.
- e. If needed, use appropriate filter to cut off light from laser. (e.g. 370nm filter for 325nm laser). Do not scan past the laser line.

### A. Rough Scan

- i. Set scan parameter through software named PL measurement on the computer, including start  $\lambda$ , stop  $\lambda$  and motor speed (20A/s). *Always check for calibration wavelength before scanning.*
- ii. Attempt to start with the alignment when a temperature of 4 K is attained and do a scan.

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- B. Final Scan
- i. Check wavelength (monochromator position).
- ii. Change motor speed (1-5 A/s or less).
- iii. Do a scan after getting the optimum intensity.
- iii. If signal is saturated, change the slit width or control signal sensitivity using lock-in amplifier.
- iv. If signal is not saturated, go to the highest peak and maximize the signal intensity by optimizing the optical alignment. Check signals using lock-in amplifier and do the final scan upon getting the optimum signal intensity.

## D) Power off after measurement

- a. Power off in the following sequence:
  - i. Turn off the laser.
  - He-Cd: Switch off laser power supply.
  - Ar-ion: Switch off the power on the remote controller, turn off the laser power supply & turn off the power laser on the wall.
  - ii. Close the monochromator slit.
  - iii. Turn off PMT power supply (slowly reduce to zero and switch off).
  - iv. Switch off Monochromator and lock-in amplifier.
  - v. Switch off chopper controller.
  - vi. Switch off cryostat temperature controller and compressor.
- b. Close Vacuum Isolation Valve and switch off Vacuum Pump.
- c. Wait for 20 mins for equipment to cool down before switching off the PMT cooler.
- d. Wait for 30 mins before closing water valves.
- e. Exit from the software for PL measurement.

### Unloading Sample

- 1. Wait for the temperature to reach room temperature (usually 8 hours).
- 2. Follow the loading sample procedure to remove sample.

### User record

1.Identify process steps and record in respective record.

2. Inform the super user /supervisor if encounter the problem.

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- 5.2 Maintenance of the LT-PL
- 5.2.1 Check Laser sources to keep at idle
  5.2.2 Check the vacuum pump efficiency.
  5.2.3 Check the monochromator position at zero.
  5.2.4 Check water valves & power switches are switched "Off" before leaving (<u>FLOODING</u> of the lab can take place if the water supply is not switched off)

#### 6. Communication Frequency:

The regular communication is every one year. However, the Lab Supervisors shall arrange for this Safe Work Procedure to be communicated as & when required.

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