INSTRUCTIONS LASER SET-UP (Lab B45)

ASK FOR TRAINING before using the machine (Juan Cabanillas, compare details Anex I). The training process is MANDATORY before using the lasers, only qualified laser operators can enter the lab.

REPORT ANY ISSUES to the responsible people (Juan Cabanillas and Reinhold)

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Before starting: Safety rules

More exhaustive safety rules are detailed in the MANDATORY laser training documentation "Laser Safety at IMDEA Nanociencia". Contact Juan Cabanillas for more details.

- Make sure to check that the red light on top of the entrance is on or off. "On" means that a laser in the laboratory is turned on. "Off" means that all lasers are turned off. If you turn on a laser and don't turn on the warning light you will be **responsible** for any personal damage that might occur.
- If the light is on, knock at the door and wait for confirmation from the inside before entering.
- Avoid wearing reflective clothes, remove jewelry (rings, watches or necklaces) in the lab.
- Be extremely cautious when aligning the optical set-up (keep the table tidy, reduce the power of the laser sources, inform coworkers, etc.).
- Use beam blockers when inserting new optical elements.
- Wear appropriate laser safety glasses or goggles, especially when aligning the set-up.

Reservation

The use of the laser set-up is regulated through a paper **calendar** in the lab. Register there with the proper time margin (two-three weeks in advance) writing your name on the days you want to use it. Half-day reservations are possible, of course. No more details of the measurements or samples are needed. Keep in mind that last-time cancellations may occur, but let people know if you cancel. Do not make reservations if you are not sure that you need them.

Equipment and material

It is preferable to take **our own material from our lab** (Laser Complements drawer in lab 161) if available (see inventory for details). If necessary, there are lenses, filters, sample holders, etc. available in the laser lab cabinets.

If you need to borrow something from the Laser lab, **register** yourself in the **spreadsheet** next to the door. Remember to put them back afterwards.

Laser sources

There are various lasers available in the lab.

- **High-frequency laser** (MHz): 375nm, 405nm, and 785nm, all about 50-70 ps pulse width, these are controlled with SEPIA II (1a) driver, normally used with Hydra Harp (HH) TCSPC electronics.

  HydraHarp electronics: Input count rate must be below 5% of the "Sync" rate (repetition rate of the laser)

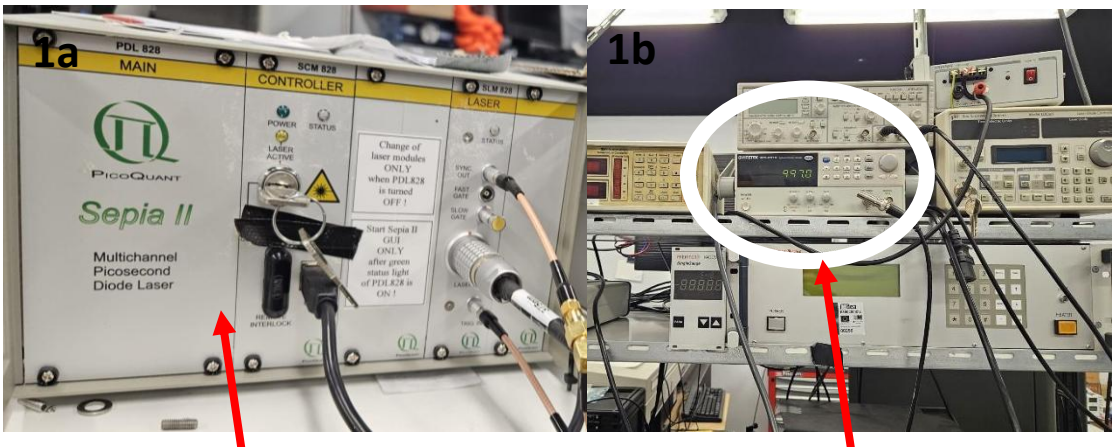
  HydraHarp and TimeHarp electronics: Input rate should not exceed 1MHz. Check that "Sync" shows the repetition rate of the laser.**Turn-on procedure of the SEPIA electronics:** Turn on the electronics, wait until the "Status" LED goes green without blinking, only then start the SEPIA software.

  Turn-on procedure of the HydraHarp electronics: Turn on the electronics, wait 5s, only then start the HydraHarp software software.

  Turn-off procedure of the SEPIA electronics: Quit the SEPIA software, only then turn off the electronics.

  Turn-off procedure of the HydraHarp electronics: Quit the HydraHarp software, only then turn off the electronics.

- **High-frequency LED** (MHz), 313.6nm, very low power, 1ns pulse width, control integrated in the device, normally used with Hydra Harp (HH) TCSPC electronics.
- **Low-frequency laser** (Single shot, **1kHz max**): 532nm and 355nm, triggered with the function generator (1b) and normally used with the TimeHarp module (TH, board plugged into the PC). Highest time resolution 1ns per bin. Time range up to seconds.
- 390nm or 284 nm femtosecond laser comes from the TAS set-up. Passes through the aluminium tube through the wall.
- Several continuous wave lasers (405nm, 532nm, 632.8nm)
- High-power OPO, tunable 220nm-1700nm, 6ns pulses, spectral width about 2GHz, 10Hz repetition rate, currently only to be operated by Reinhold



Detectors

The entrance and exit slits of the spectrometer are located 18cm above the table surface, consider that when aligning the laser beam path through the set-up. The sample and the centers of all lenses should be at that height.

For the time-resolved measurement detectors (2a and 2b) (single photon detection), there are two different hybrid photomultipliers are available:

-“red-sensitive detector” (PMA50, Fig. 2b,c, suitable to measure in red - visible range), SMA connector. Sensitivity up to 900nm.

-“blue-sensitive detector” (PMA06, Fig. 2a), Sensitivity up to 650nm max., but much higher sensitivity in the blue.

Ask Reinhold to change the detector in case you need to change it.

BOTH PMs:

- **Disconnect the shutter, before turning any lights on !**
- **Do not saturate the detectors ! Open the slits slowly. Strong saturation will lead to irreversible rise of background pulses not due to photons.**

One SMA connector

Two SMA connectors.

Connect the cable to the TCSPC electronics to the upper one.Do not remove the lower one !

Optical elements

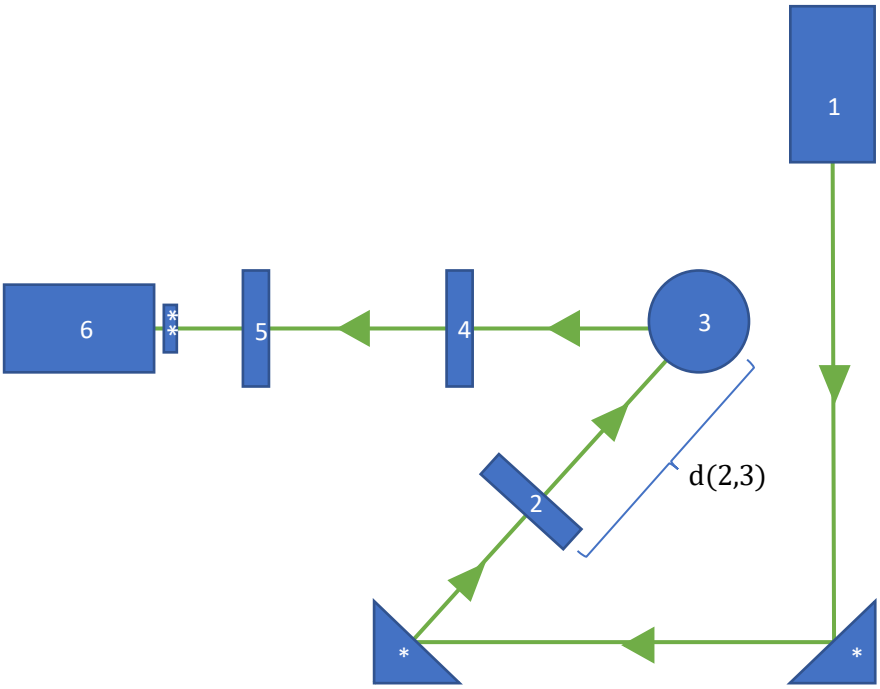
There are plenty of singlet and doublet lenses, mirrors, beam splitters, etc. available in the lab. When choosing the appropriate ones for your set-up, there are a few things to keep in mind. Some of them are:

- **Range of operation**: for achromatic doublets (3a) and mirrors (3b), is important to know in which spectral band they work properly. This is usually indicated on the side of the lens or behind the mirrors (F-01 = aluminium coating, P-01 = silver coating, E-01, E-02, E03 = dielectric coatings. All relflection curves as a function of wavelength available on the Thorlabs web page (www.thorlabs.com).
- **Focal distance f_f**: for lenses and non-plane mirrors, the focal distance is the distance from roughly the center of the lens to the point where the incident light will be focused (if the latter comes collimated).
- **Filters**: sometimes might be necessary to block some wavelengths from the optical path, for that we use filters. Depending on the spectral properties, these can be:
    - Short pass (SP): allows the pass of light up to some wavelength.
    - Long pass (LP): allows the pass of light from some wavelength.
    - Bandpass (BP): allows the pass of light in a specified range.

Polarizers: filter the light according to the polarization. All the important properties and specifications of each element can be found online by searching the reference name on the web pages mainly of Thorlabs and Newport (CGA LP filters). In case of doubt, please **refer to these online manuals**.

TCSPC set-up for measuring PL lifetimes

The setup for performing lifetime measurements is relatively simple, composed of:



1. **Laser source**
2. **Sample focus lens** (don't focus the 355 nm or 532 nm sharply at the sample, especially at 0 OD) (f_f> d(2,3)) where d(2,3) is the distance from 2 to 3. **Never focus those lasers at OD0 sharply on anything, even not on windows of cryostats or chambers!**
3. **Sample holder in a cryostat or vacuum or gas chamber or simply in the air.**
4. **Collimator** (□)(f_f=d(3,4))
5. **Lens** focusing the light on the entrance slit (□) (f_f=d(5,6))
6. **Spectrograph**

There are **mirrors** (*) and **filters** (**) depending on the geometry and requirements of the system. The ** filter should be an LP filter to filter out the laser wavelength.

The exact geometry of the set-up depends on the placement of the laser source and the sample holder.

Set-up preparation

1. Turn the PC on

1A. Launch the Windows 7 OS. The user is the one that comes up by default. You can find the password below the keyboard.

1B. Launch WinSpec program (on the desktop).

1C. To check the system temperature, go to Set Up > Detector temperature. It should be **~ -120C**.

2. Fill the camera dewar with liquid nitrogen (LN_2)

You only need to do this once and it lasts until the end of the day. This helps to reduce thermal fluctuations in the CCD camera, which reduces the noise. To fill up the dewar, follow these instructions:

2A. **Remove the keyboard and mouse** from the metallic **surface** where they normally are.

Use proper cold gloves and protection googles for the next steps

2B. **Introduce the LN_2 tank rubber hose into the dewar**, not too deep but past the first limit (you'll feel it). If you introduce it too deep, it will be difficult to take it out when frozen. If you don't introduce it correctly, it will spill.

2C. Open the valve of the LN_2 storage dewar slowly and **fill up the dewar** of the CCD camera until it starts overflowing.

2D. **Close the tank** and wait for about 15 minutes.

- If the system is warm, the LN_2 will evaporate. If this happens, **repeat step 2C**.
- If done correctly, after 15 minutes the temperature should be around the target of -120C. **Take out the hose** when is no longer frozen to avoid cracking it.

2E. **If the CCD is cold, dry off the condensation** of the cap with the heat gun

3. Switch the laser on

When starting the laser, it is important to switch on **first the Hardware and then launch the Software** so it doesn't lose the connection. The inverse order is required when shutting down the system.

The following steps should be done in the dark, switch the lights off

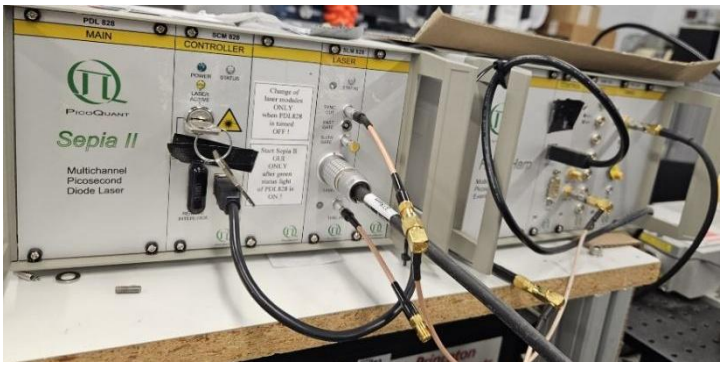
3A. **Connect the laser** (e.g. the one operating at 380nm) to the Sepia II electronics with Sepia II turned off.

3B. Only when the green light is steady (not blinking) **turn the SEPIA software on** (at the back of the electronics).

3C Turn of the Hydraharp electronics if you want to measure short lifetimes (nanoseconds) The TH electronics is inside the PC and does not need to turned on.

3D. **Turn on the TimeHarp or HydraHarp *Software*** (Sepia II) and the measurement control software (HH/TH).

3E. **Switch the laser key on** (at the front of the system). A red light should shine. The laser is now on, unless the software switch is off..



3F. **Block the laser beam** with a beam blocker or enable the soft-block in the Sepia II software.

Configure the laser repetition frequency (e.g. 10MHz) and intensity in Sepia II. The Max intensity for the 405 nm laser is 79%. The Max intensity for the 375nm laser is 67%. If more intensity is applied, you will have non-desired artifacts in your measurements. Once you set your parameters, press "Apply".

When using a low-frequency laser, you should connect the laser to the function generator, normally already connected, and connect the trigger diode to the Time harp Sync input (normally already connected). You use a mechanical shutter to block the laser light. Check that the Sync rate reflects the laser repetition frequency in both cases (HH/TH). If this is not the case the trigger input is missing or the Sync threshold is set to high.

4. Set up alignment and calibration

When preparing the set-up, there are a few tips and considerations:

- Keep the laser beam **height at 18cm** from the table at all times. This is important as this is the height of the entrance slit of the spectrograph.
- As a safety rule, always **use gloves** when aligning and use **paper cards** to track the laser path. If you do not do that you will not be covered by insurance.
- Use **blockers** to **block the reflections** that may be generated in the system
- **Soft-lock the laser** using the controller software (Sepia II) or the physical key in the apparatus when i**nserting new elements** in the system.
- **Alignment:** Set the lenses exactly on axis. Move the sample and the excitation until you have signal.
- **Do not move your eyes to the level of the laser beams.**

For the alignment, it is recommended to use a fluorescent reference (a silicate subtrate with a fluorescent polymer deposited on it) that emits really brightly.

4A. Place the reference sample in the sample holder.

4B. Unblock the laser. A bright dot should appear on the reference sample.

4C. Open WinSpec. You need to configure the data acquisition process: go to Adquisition > experimental Set-up.

- Exposition time: 200 ms
- Accumulations: 1

Now go to Spectrograph > Move. There you can set your acquisition wavelength.

- Move to: (emission wavelength of your reference, e.g. 570nm)
- Grating: 600g/mm, 500nm (for visible light)
- Mirror: Front (option is for steady-state measurements with the CCD camera).

4D. Go to Experimental set-up > Acquire > Focus.

4E. Open the spectrometer slit until you have sufficient signal using the nonius (up to 500 um). When measuring, keep track of the slit width, this is an important parameter.

A PL spectrum at the specified wavelength should appear. This is the signal received from the reference sample by the detector.

4F. Move the lenses 4 and 5 back and forth until the signal is maximized. Try to keep the distance 3-4 close to the focal distance of lens 4 and the distance 5-6 close to the focal distance of lens 5.

4G. Keeping the distance and the height (remember, 18cm above the table). Lenses should be perpendicular to the axis (not tilted) and properly centered to avoid aberrations.

4H. Block the laser and take the reference sample out of the sample holder. Place your sample in the same position. Now the set-up is ready to measure.

Data collection

5. Steady State

The Steady State spectra are measured using WinSpec Software.

5A. **Place your sample in the sample holder**. Don't fix it too tightly, just enough to hold it in place.

5B. Unblock the laser beam. A fluorescent dot should appear in your sample. if the dot is above or below the sample, regulate the height of the sample holder.

5C. Go to WinSpec. In the Experimental set-up, define the Exposition time (200ms) and number of accumulations (6). Accumulations are the number of spectra to be added, high acc are recommended for low values of fluorescence intensity.

5D. Go to **Spectrograph > Move** and set the wavelength of interest. **The mirror position should be front** (this is a common mistake).

5E. Go to Acquisition > Step and Glue to perform a measurement. Define the wavelength range (400-800nm) and the overlap (10-15 nm).

5F. (Normally) Create a new folder, give it an appropriate name and in "Step and Glue" choose this folder (Recommended to call it by the date of the measurement) and a file name for the measurement. This name should include:

- Sample name
- Excitation wavelength (the laser source)
- Excitation frequency
- Slit width
- Exposition time
- Grating (e.g. 600 gmm 500 nm)
- filter used for detection
- Number of accumulations

- if necessary, other relevant parameters (pureged/unpurged, sample temperature, sample in air or vacuum, etc.)

Example: Purge_A96_exc375nm_10MHz_50mu_LP375_200msec_600gmm_500nm_10acc

- It is recommended to record the background signal after measuring to correct the spectra. This is done by blocking the laser and repeating 5E and 5F (rewrite the same name adding "_BGR" at the end).
- Then you can subtract the second spectrum from the first one in Process > Math > Operation > Subtraction
    - You choose as element A the original spectra, and B the background spectra.
    - File C is the result of the operation, name it as file A but add "_corr".
- This spectrum needs a correction (ask Reinhold or Juan for the correction file). After correction it should resemble the ones obtained in the Fluorolog at the same excitation wavelength.

5G. To export the spectra to ASCII for further data manipulation, export it in Tools > Convert to ASCII.

- Choose the files to convert.
- Choose the output folder.

6. Time-resolved

Always keep the shutter of the photomultiplier disconnected except when measuring PL decays. Disconnect after each measurement.

6A. Open the TH/HH software. If the oscilloscope/sepia is connected correctly, you should see the frequency of the laser in the lower part of the screen.

6B. In WinSpec software, go to Spectrograph>Move

6C. Make sure to have connected the PM detector to the HH/TH, have connected PW to the power supply and the shutter disconnected until the moment you measure (**Never leave the shutter open if the ambient light is on**).

6D Close the exit slit of the spectrograph

6E. In the Winspec software ("Spectrograph/Move") select the wavelength of detection you are interested in (usually at the peak of the PL spectrum). In Mirrors, make sure to change the mirror to "side".

With the lights off

6F. With the laser on, open the shutter by connecting the additional connector at the back of the photomultiplier.

6F. Open the exit slit until you have signal without saturating the PM. You should see the number of photons collected per second by the detector within the window of the TH/HH software. For the HH, this number **should not surpass 5% of the frequency of the laser**.

6G. You can change the parameters of the measurement in the control panel:

- Max number of counts
- Max time. If you want to measure up to a certain number of counts set the "Time" value to a very large value, because the measurement stops whenever either the maximum time or number of counts is reached.
- Time axis / vertical axis of the graph
- Time resolution

6H. Once ready, click "GO" to start the measurement. Wait until the measurement finishes automatically. The results should look like a sharp rise followed by a more less slower decay, and the signal/background ratio should be ideally larger than 3 orders of magnitude. If the values at the end of the exponential decay tail correspond to those of the background signal before the pulse, the excited state has decayed completely in the time range of the measurement. If, on the contrary, the values in the tail are over those of the background, you should take longer decay times; this is, use lower frequency.

6I. To measure IRF, move the spectrograph to the laser wavelenght (using winspec), take out the filter in front of the spectrometer slit, and measure the decay there. This should be done with the same parameters as the measurement (this is, laser intensity, aperture of the spectrograph slit, resolution of the measurement, etc.) if you change any parameter when measuring a sample, you should retake the IRF.

Do not do anything that you are unsure about. Ask Reinhold or Juan first in that case. The instrumentation of the room is expensive, so please be very careful.

Annex I: Responsible contact information

If you have any questions or find any issues, please contact the responsible. Details below.

Contact information of the responsibles

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