Laser-Induced Breakdown Spectroscopy (LIBS) System (Operation Procedure)

Context

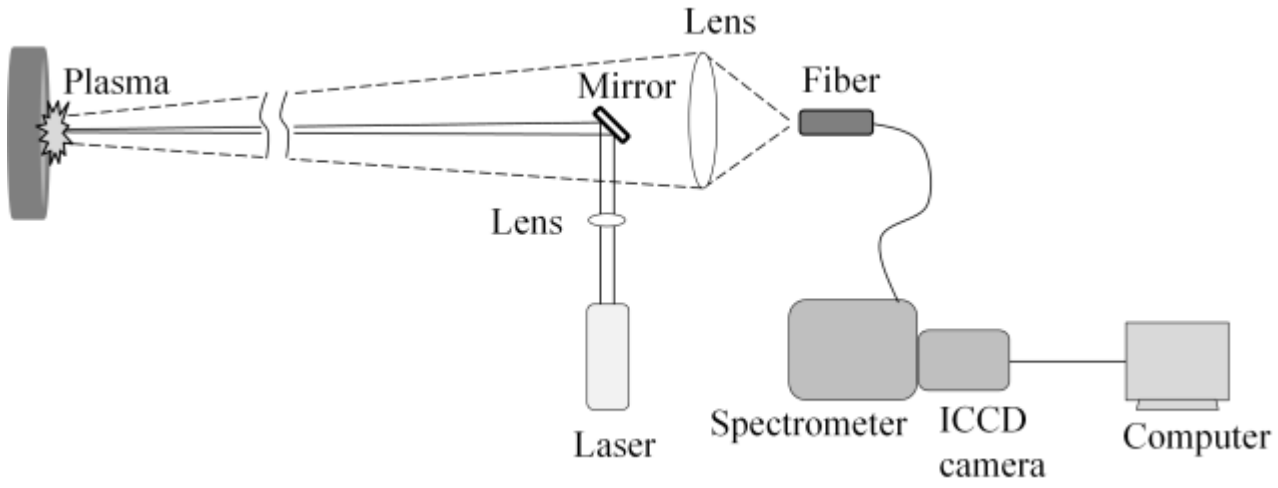
Disclaimer

Safety – first !!! This presentation is not manual. It is just a brief set of rules to remind procedure for simple measurements. You should read manual first and follow all requirement for operation with electrical and laser devices

Introduction

What is LIBS

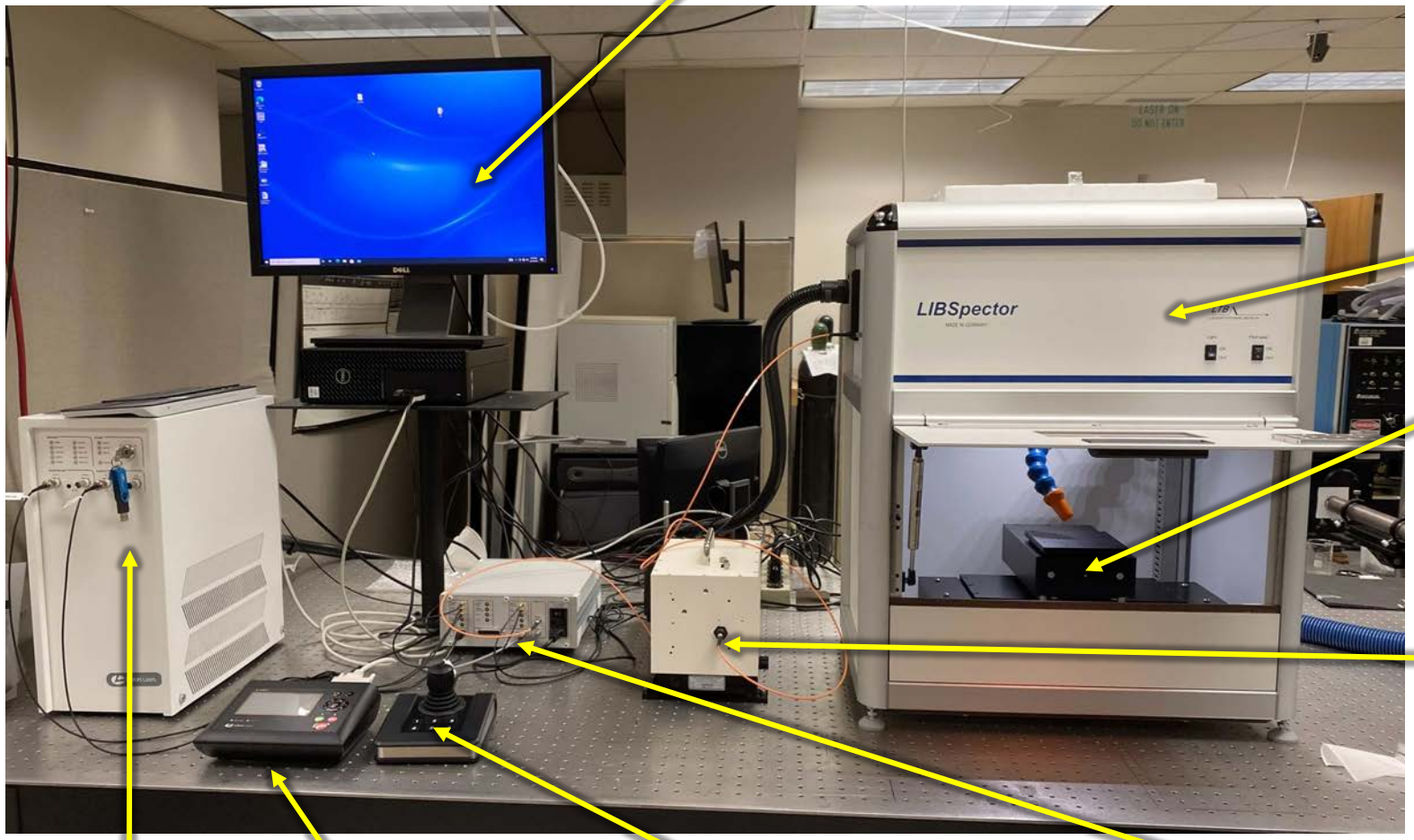
Laser-induced breakdown spectroscopy (LIBS) is a straightforward and versatile spectroscopic technique based on the analysis of the spectral emission from laser-induced plasmas.
LIBS has the far-reaching capability to provide rapid, in situ multielement detection of any material – solid, liquid, or gas. The field of LIBS has been rapidly maturing as a consequence of interest in LIBS for a broad spectrum of applications and the recent development of LIBS analytical systems by the commercial sector.



Introduction

Experimental Setup

PC + Sophi software control settings and measured data



LIBSpector Chamber (enclosure) with built-in laser head

XYZ Translational Stage

Echelle spectrometer and attached CCD camera measure emission spectra of the ablated plasma. Spectrometer is connected to the LIBS chamber and Calibration lamp by Optical Fibers

Laser Power Supply

Laser Remote (LUCi) Controller

Laser control units

Stage Joystick controls position of the sample in the LIBS chamber

LIBS Controller manages electrical triggering pulses between LIBS components

Introduction
Technical Specification

Detector (Andor iStar):

- ✓Model DH334T-18F-03-27A
- ✓ICCD 1,024 x 1,024 pixels,
- ✓13 x 13 μm^2 image area,
- ✓Minimum optical gate width, 5ns.
- ✓Dynamic range: 15 bit,
- ✓SN ICCD-60447; Y 2021

Echelle spectrograph :

- ✓ Aperture: f/10
- ✓ Focal length: 200 mm
- ✓ Slit width: 40 μm
- ✓ Wavelength range: 200 to 750 nm (max 600 nm simultaneous)
- ✓ Spectral resolving power: 7,000-15000
- ✓ Spectral resolution FWHM: 22 to 83 pm
- ✓ Absolute accuracy: spectral resolution / 4
- ✓ Order crosstalk: 2 x 10-3 (CCD) measured at 253.652 nm and full slit height

Litron Laser Nano 120-20 :

- ✓Wavelength 1064 nm
- ✓Output energy 120mJ
- ✓Repetition 20Hz
- ✓Pulse Duration 4-7 ns
- ✓SN LM3089; Y 2020

Switch On Procedure

Step 0:

- Check all connections according to the scheme shown in the Appendix.
- Switch ON LIBS Computer
- Run **.txt** file ”**LIBS Operation Sheets**” with operation records and fill in your name, date and time of experiment
- Also make the same note in the operation notebook near LIBS System

Switch On Procedure

Step 1 (Switch On Litron Laser):

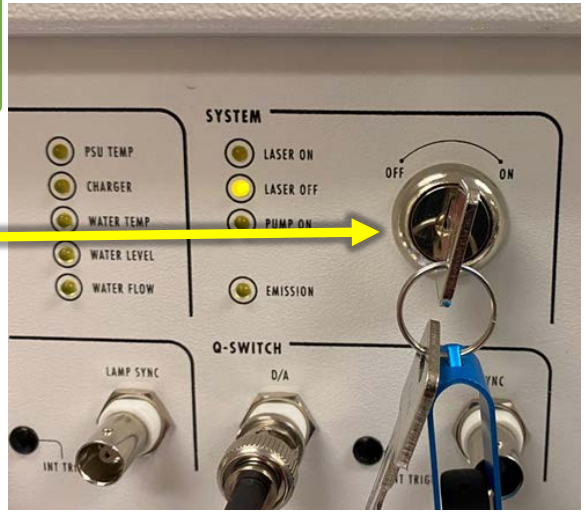
On the back side of the ***Laser Power Supply Unit,*** check the coolant level using glass window. Water level must be higher than minimum coolant level.

Step 2 (Switch On Litron Laser):

Right above the glass window, still on the back side of the ***Laser Power Supply Unit,*** turn ON Power Switch.

Step 3 (Switch On Litron Laser):

Holding the lock by its base, turn the key on the front panel of ***Laser Power Supply Unit*** to position ON. Look at the system indicator situated on the left of the key you turned.



Note: Check status of all activated systems on **LUCi controller**

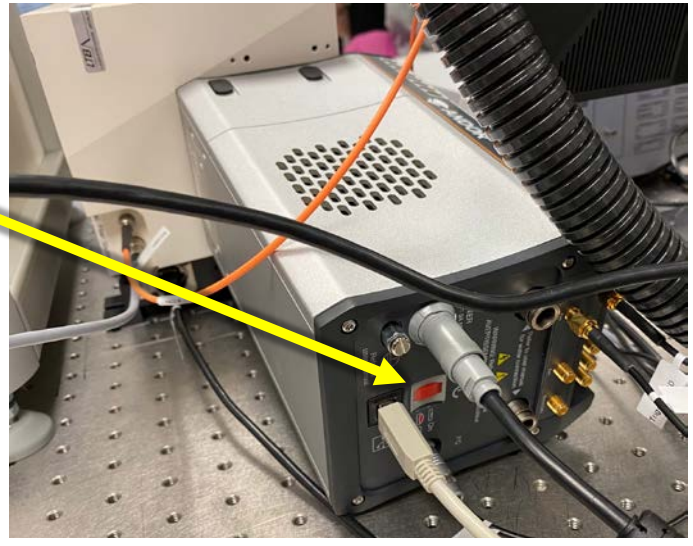
Switch On Procedure

Step 4 (Switch On LIBS CHAMBER UNIT):

Turn on Light and Pilot Laser switches on the front panel of **LIBS CHAMBER UNIT**.

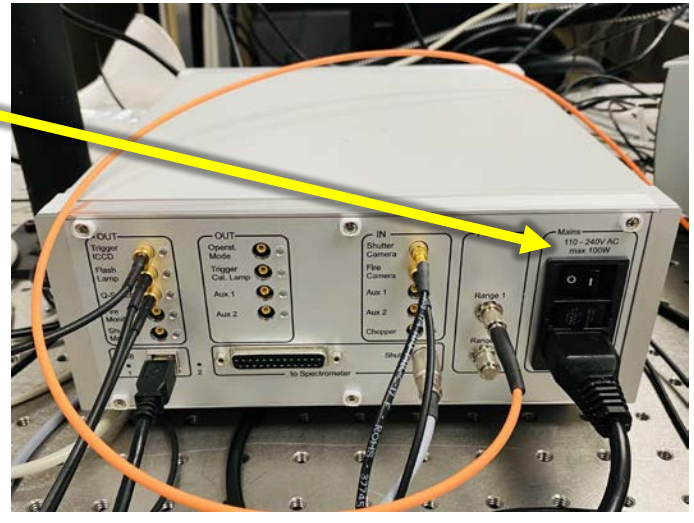
Step 5 (Switch On ICCD camera):

Push the red button <u>on the back panel</u> of **ICCD camera** to On position. You will hear the fan start to work.



Step 6 (Switch On LIBS Controller):

Turn On main switch on the front panel of **LIBS Controller**.



Switch On Procedure

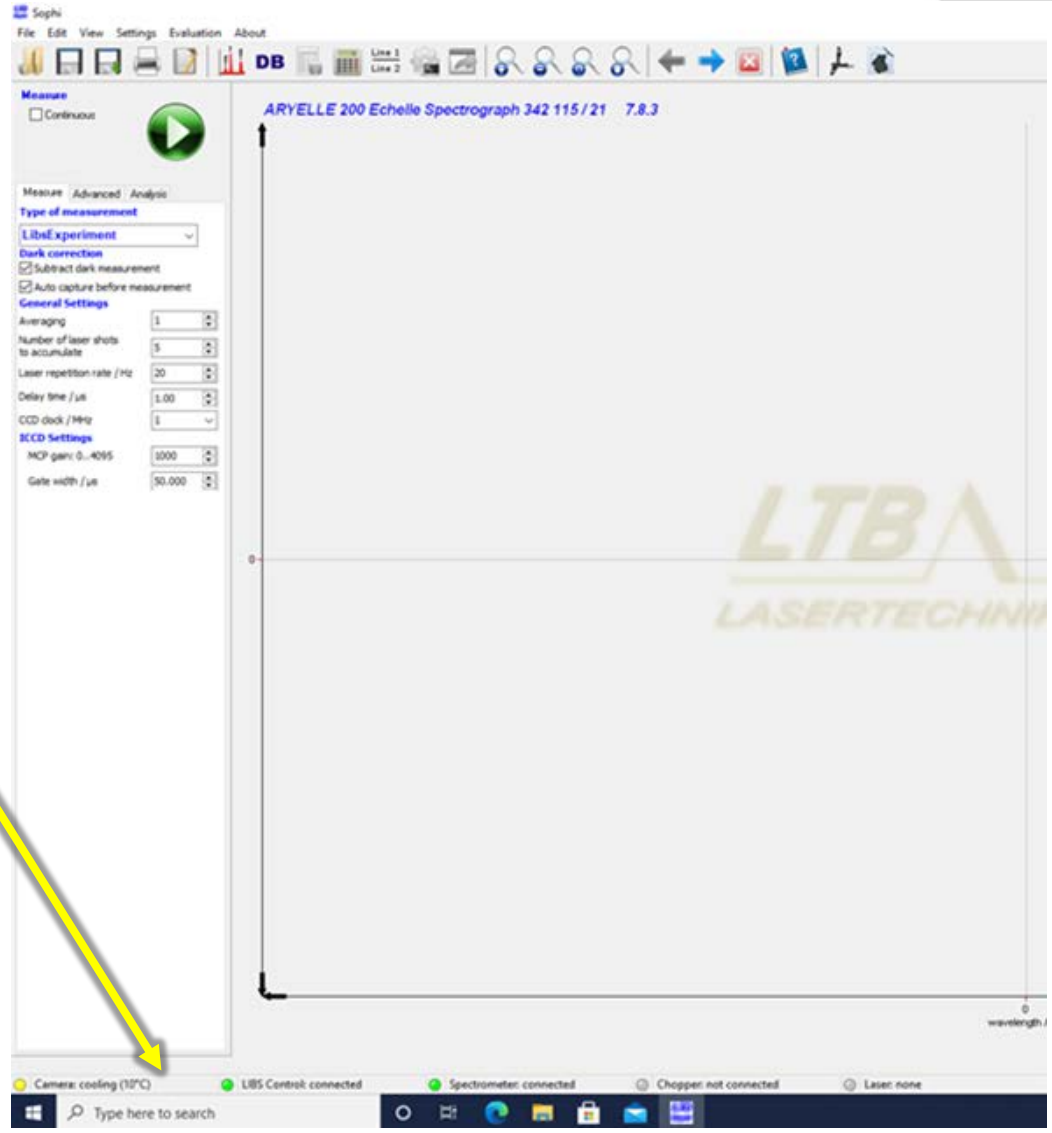
Step 6 (Run Sophi Software):

Start the ***Sophi*** **program** on the desktop of the PC. Check the indicators in the left lower part. If everything is fine, you will see three active indicators: **Camera**, **LIBS control** and **Spectrometer**.

- At first Camera status will be yellow.
- You must wait for 3-5 minutes until camera has cooled down enough. The indicator will automatically change its color to green.

Note: Choper, Laser and Energymeter indicators should not show anything. In our setup we don't have them integrated.

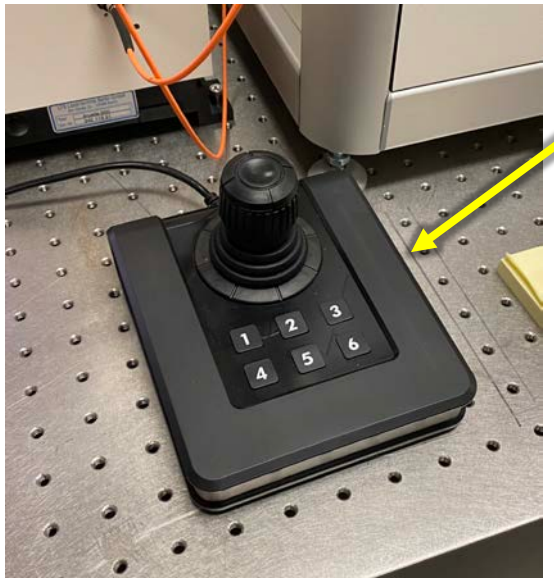
- When system is ready, all three indicators must be green.

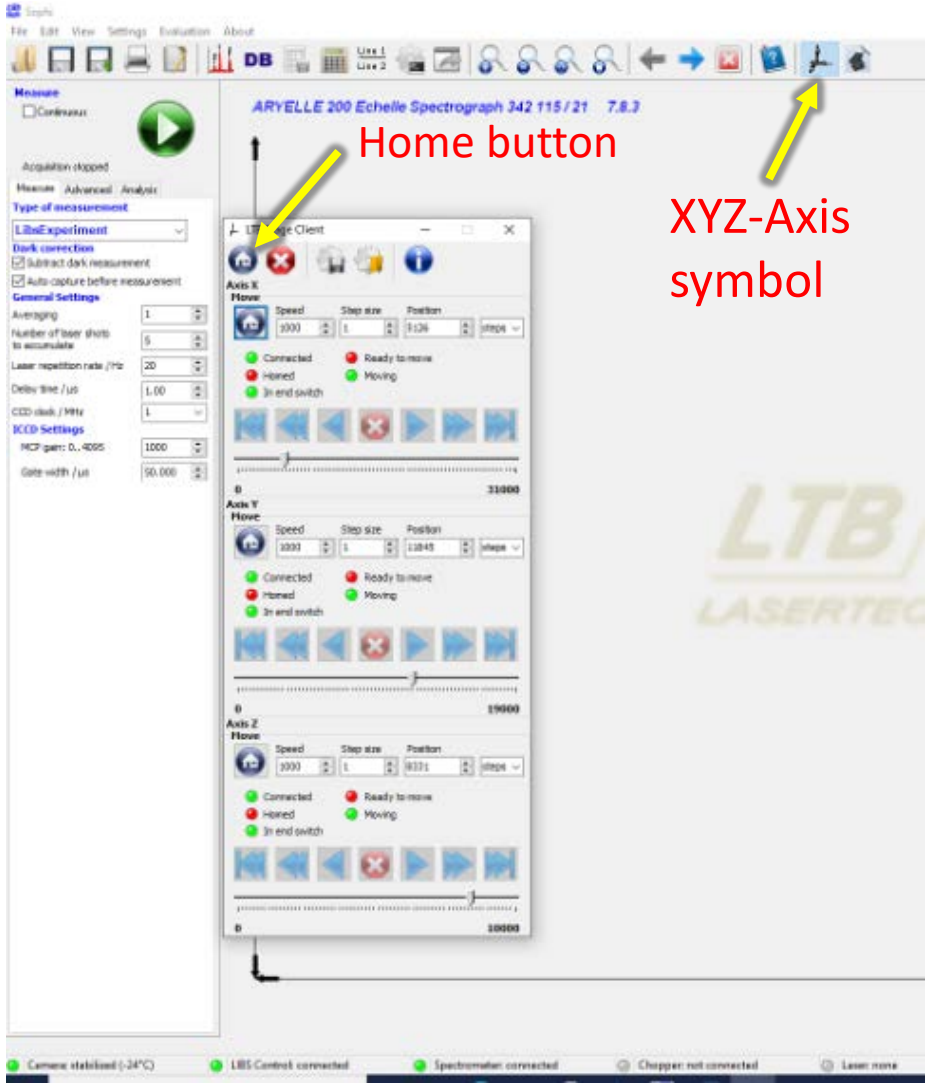


Switch On Procedure

Step 7 (initialize the motorized sample stage in the LIBS Chamber):

- Now we need to initialize the stage in the Sophi software. To do so, in the upper part of the screen in the second row below the settings find the *XYZ-Axis symbol.*
- It opens *LTB Stage client*. Here in the upper left corner, we have *home button* (blue house sign). Press it and the stage should now move to its “home” position on the lower left part of the chamber. Wait until it stops.
- Now stage is initialized, and we can control it with *joystick.*

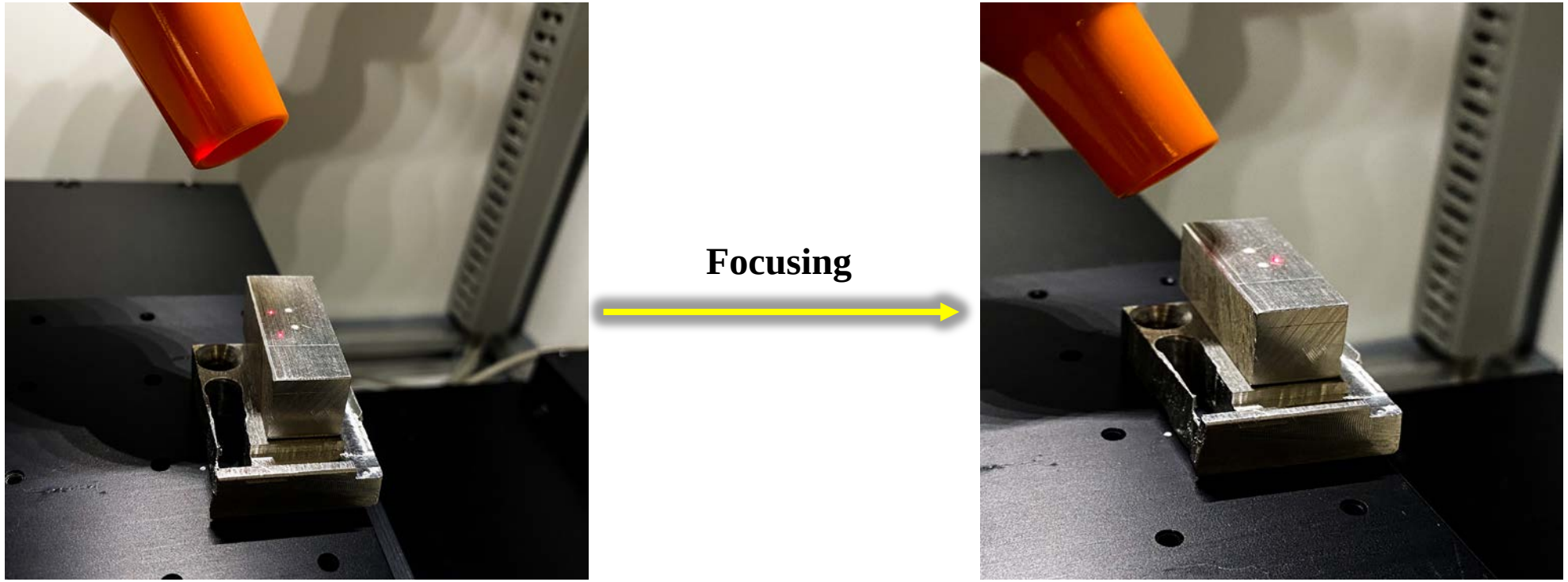




Sample Placing

Step 8(Focusing laser I):

- Put sample inside LIBSpector chamber on translational stage
- Look inside the LIBSpector chamber. *Two red dots* inside should be visible. Sample surface is in focus when these two dots covering each other.
- Move the joystick to control position of stage with sample, rotate joystick to control its height. You have to make them covering so that you could only see one bright dot on the sample.
- Close chamber

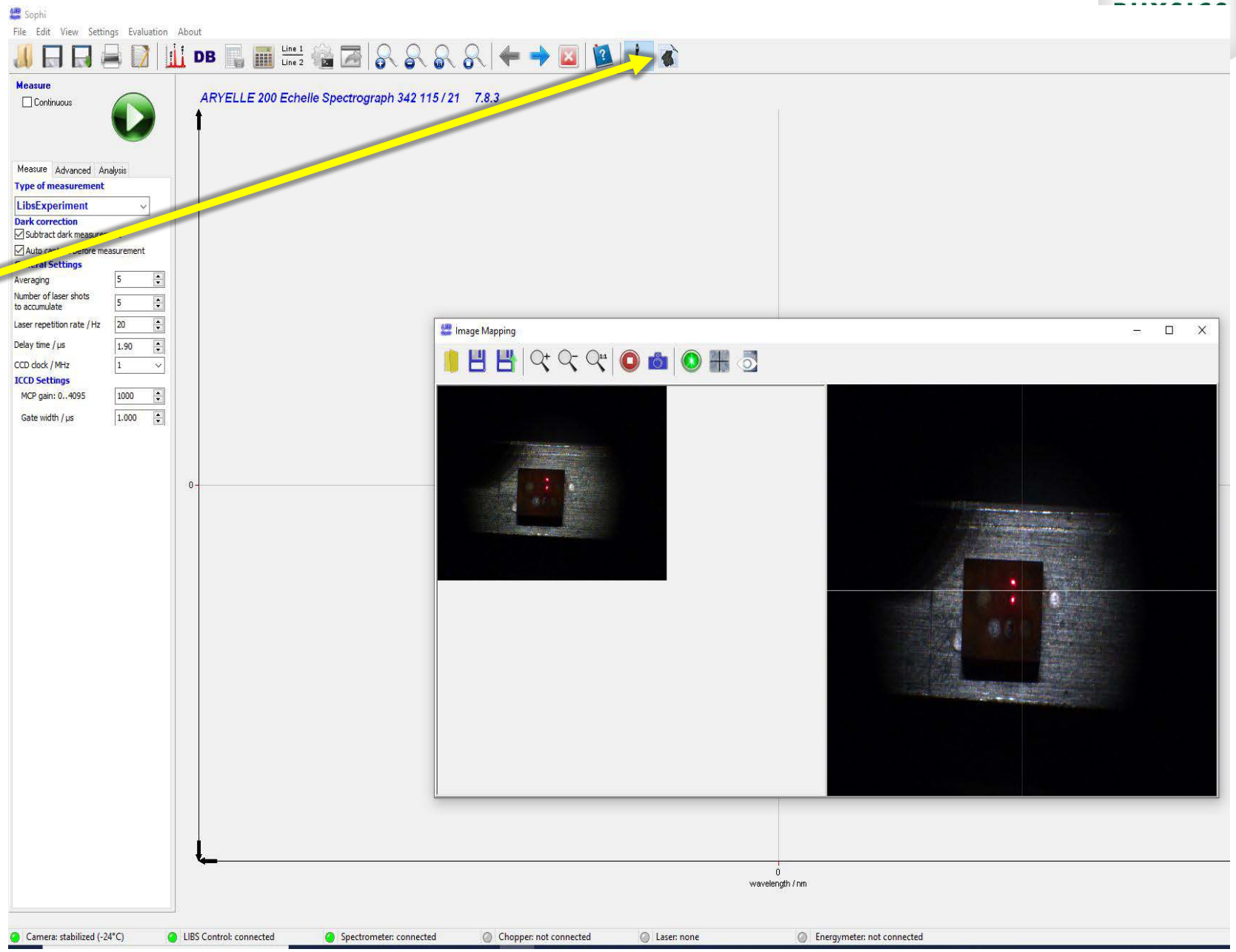


Step 8 (Focusing laser II, built in Camera)

1. Once you have your sample inside no need to open the hood of LIBS and look at the red dots for focusing. We have a built in camera to help us focus and move to a next spot.

2. ***Open Image*** mapping option can be easily used to focus the pilot laser onto the sample.

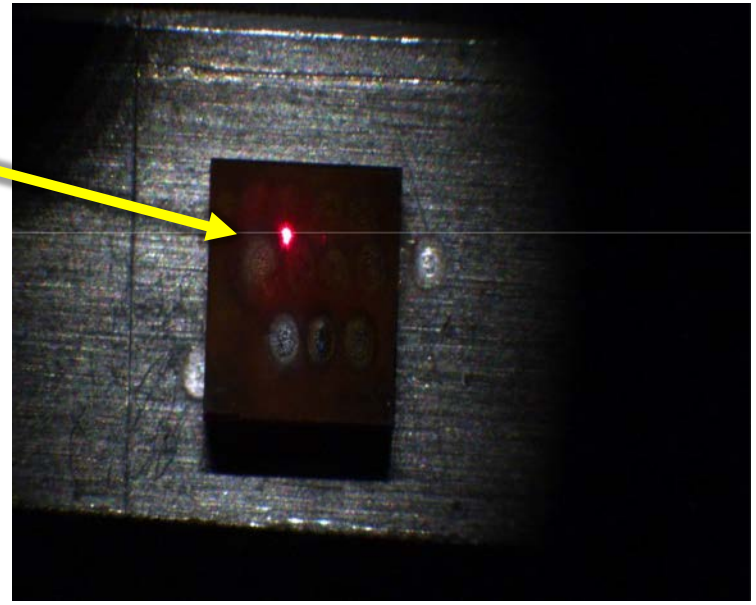
3. On the right a defocused laser consisting two dots is visible.



Step 8 (Focusing laser II, built In Camera and its usage)

4. Use the joystick to move the laser dots so that they keep coming closer and form one red dot.

5. When you have a ***focused laser light*** illuminating the sample it should look like this.



6. Saving the picture option does not work, instead use snipe to copy the image and paste it where you want to be.

7. Using joystick each time find a new and fresh spot for data collection.

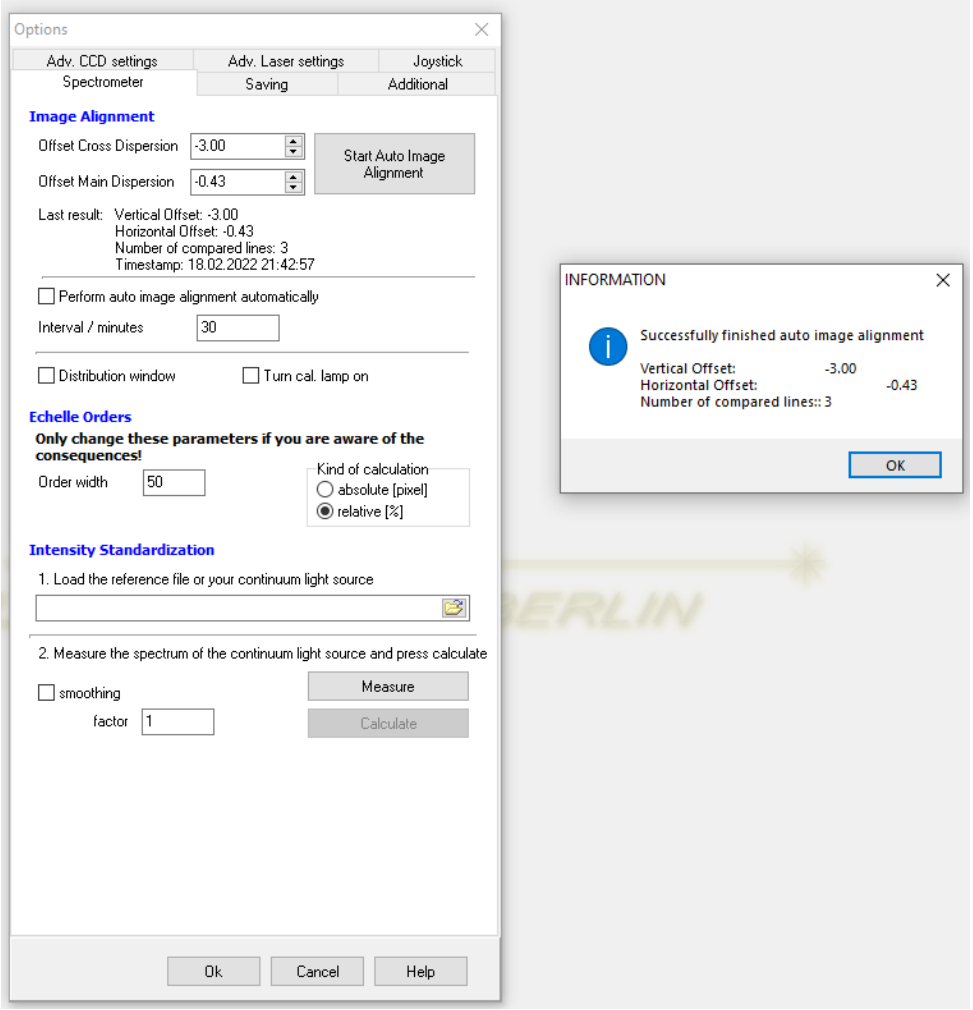
8. There is a time delay between the joystick and laser light moving. Move the joystick slowly for correct position.

CCD Camera Calibration (Outer image alignment)

This procedure could be done periodically during measurements:

Important: Outer image alignment needed to be done every day before starting measurements of samples.

- To find this option in Sophi software on the left upper corner go to Settings ⇒ Options ⇒ Spectrometer ⇒ Outer image alignment. CCD camera will be calibrated automatically.
- After calibration is done Sophie will show you offset values.
- Note for unstable lab conditions: If intensive fluctuations of air or temperature changes take place in lab, you might need to do outer image alignment every hour to make sure system works precisely.

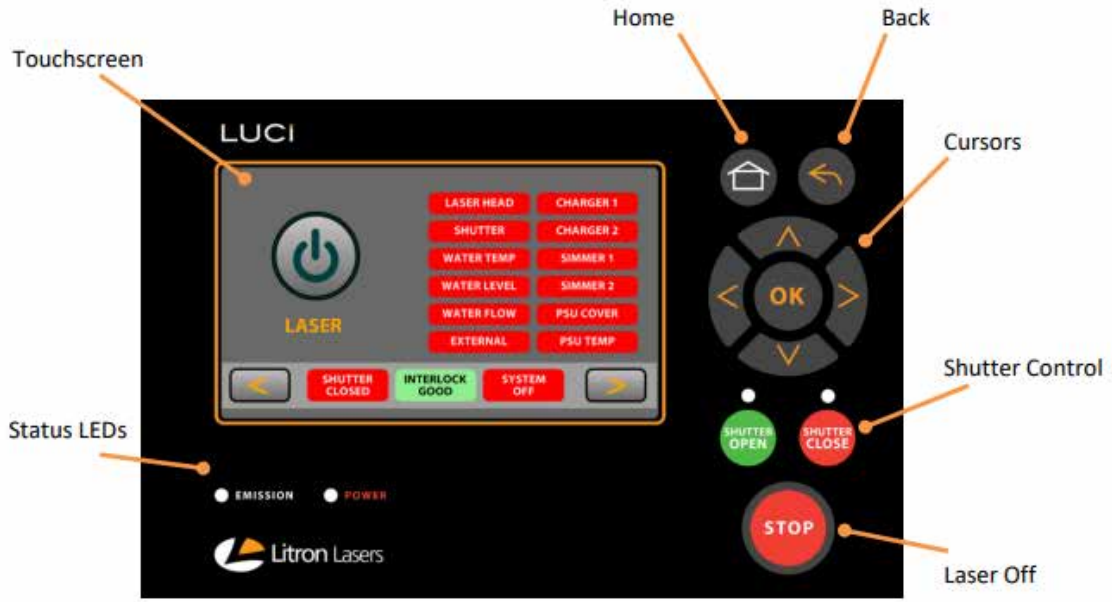


After Calibration Procedure, the LIBS system is ready to work

LUCi (Litron Universal Controller) quick overview

The LUCi control box can be used to operate a selection of Litron laser systems. It automatically detects the laser model and presents a number of control screens with which you can control and monitor the system.

- This screen displays the state of the individual laser interlocks.
- If the laser trips out, this screen will show the interlock which caused the laser to turn off
- RED indicates a fault condition. Green indicates OK.
- It is usual for some interlocks to be shown in RED until the laser is powered on. If the laser will not power on, the interlock indicators will help diagnose the probable cause



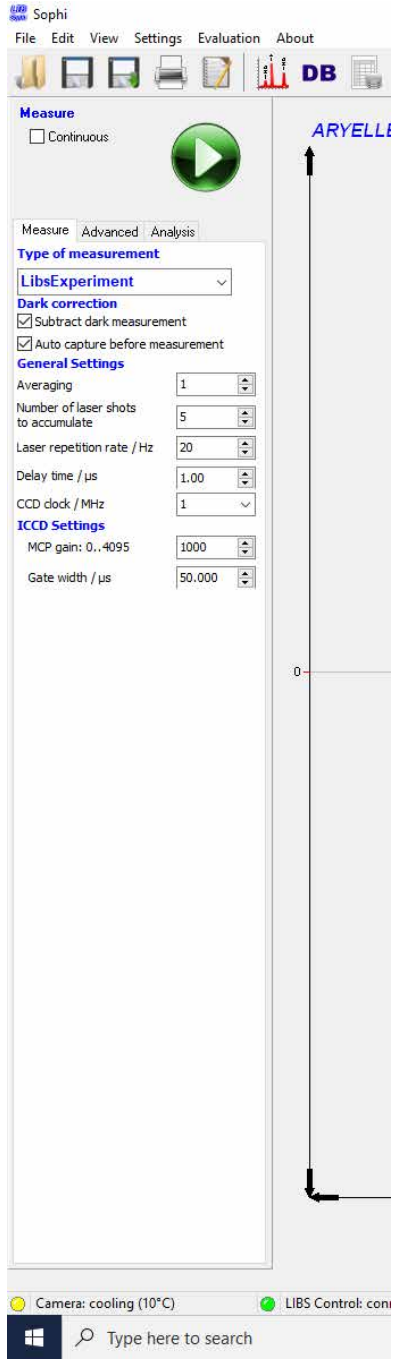
Note: Laser Repetition rate in SOPHI and LUCi must be same value otherwise in LUCi “Shutter Inhibited” will appear instead of stutter closed and immediately the system will stop working.
To change repetition rate- first change value in LUCi then change the value in SOPHI.

Starting measurements

- Under big green button on the left side of the screen choose **Parameters** ⇒ **Type of measurement** ⇒ **LibsExperiment**
- Here you have to set up parameters you need.
  - **Averaging** – number of measurements system will average in one spectrum.
  - **Number of shots** – number of laser shots system will made in one measurement.
  - **Laser repetition rate** – frequency of shots in one measurement.
  - **Delay time** – time between laser shot and CCD camera shot of spectrum.
  - **CCD Clock** – frequency of CCD camera. (CCD camera read time for one whole measurement, not just for one shot)
  - **ICCD settings** – in progress, no info yet.

Note: There is also **continuous measurement** checkbox. If this option is activated system will continue measuring until you stop it by yourself.

Important: Camera saturation not more than 180000-200000 intensity on spectrum.



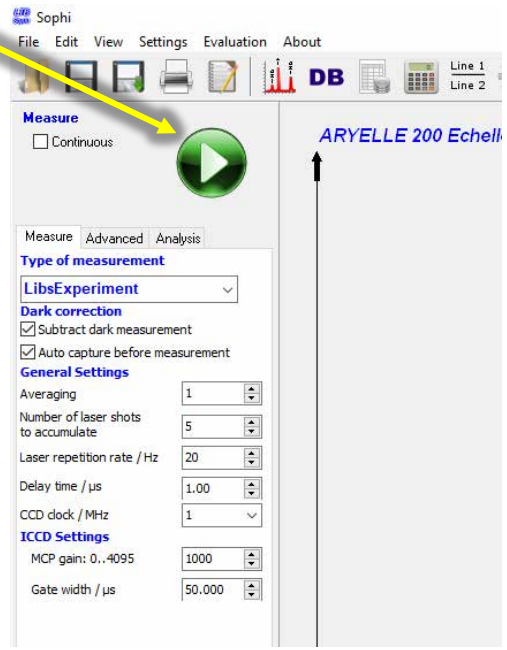
Starting measurements

After all the parameters are set up, we are ready to start the laser:

- On LUCi controller press big **LASER button**

Note: You will hear the water pump. In the left lower corner of LUCi screen you will see the **timer**. **Wait** until it will allow you activate the laser. You will hear the flashlamp noise.

- Open the shutter with green button **SHUTTER OPEN**
- In Sophie press green **start button**
- **Now LIBS starts measurement.**



Stop measurements

After measurement is done there are two options:

- Close the shutter with red **close shutter** button
- Then press **LASER button** on the LUCi screen. This will stop flashlamp. Water pump will continue working.

OR

- Just press **STOP** on LUCi. (same thing but in one step)

Note: If you are not completely done yet, do not turn off the water pump

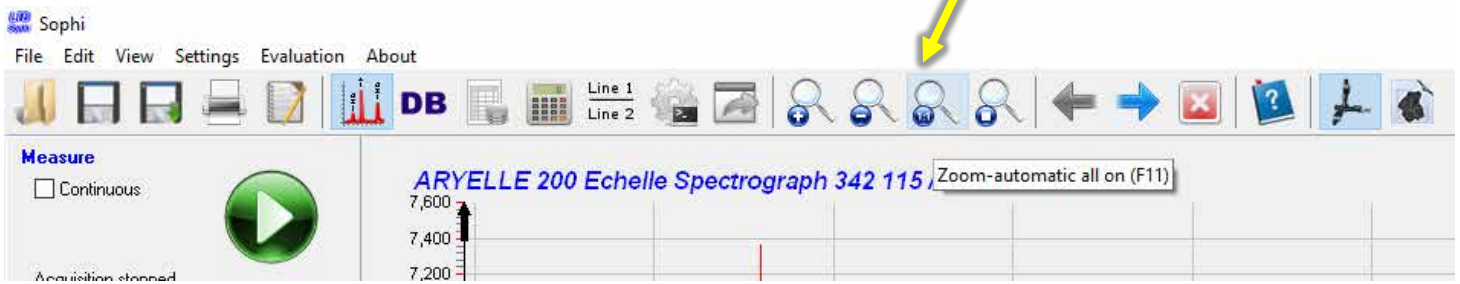
- To turn off the water pump, press **STOP** on LUCi again.

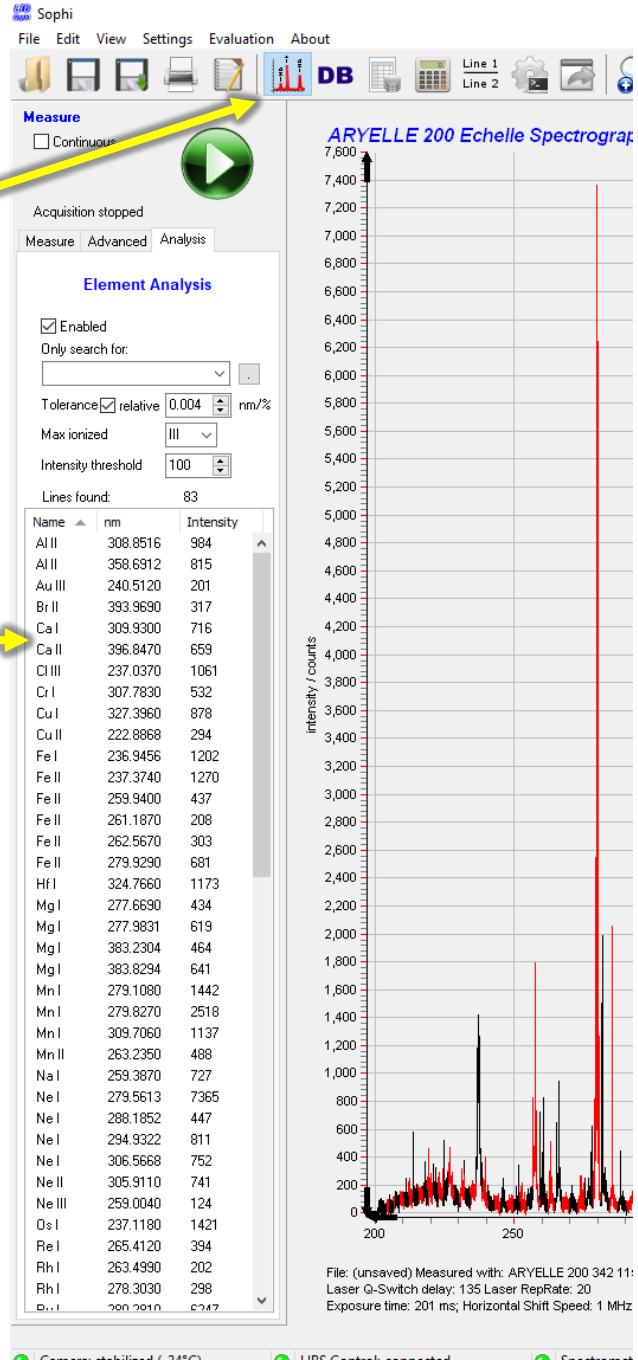
If you open the chamber, laser stops automatically.

Spectral lines database (elemental analysis within Sophi)

Sophi has integrated **NIST databases** for spectral lines. So you can make elemental analysis within Sophi software.

- On the left upper corner in Sophi activate **elemental analysis**
- On spectrum holding the left mouse button choose the area you want to analyse.
- Sophi will zoom to this area and show you what lines it has identified from **NIST** database. Also on the left side of the screen you will see the column with all identified elements in spectrum.
- To return to full picture of spectrum use **blue zoom buttons in Sophi**.





Saving data.

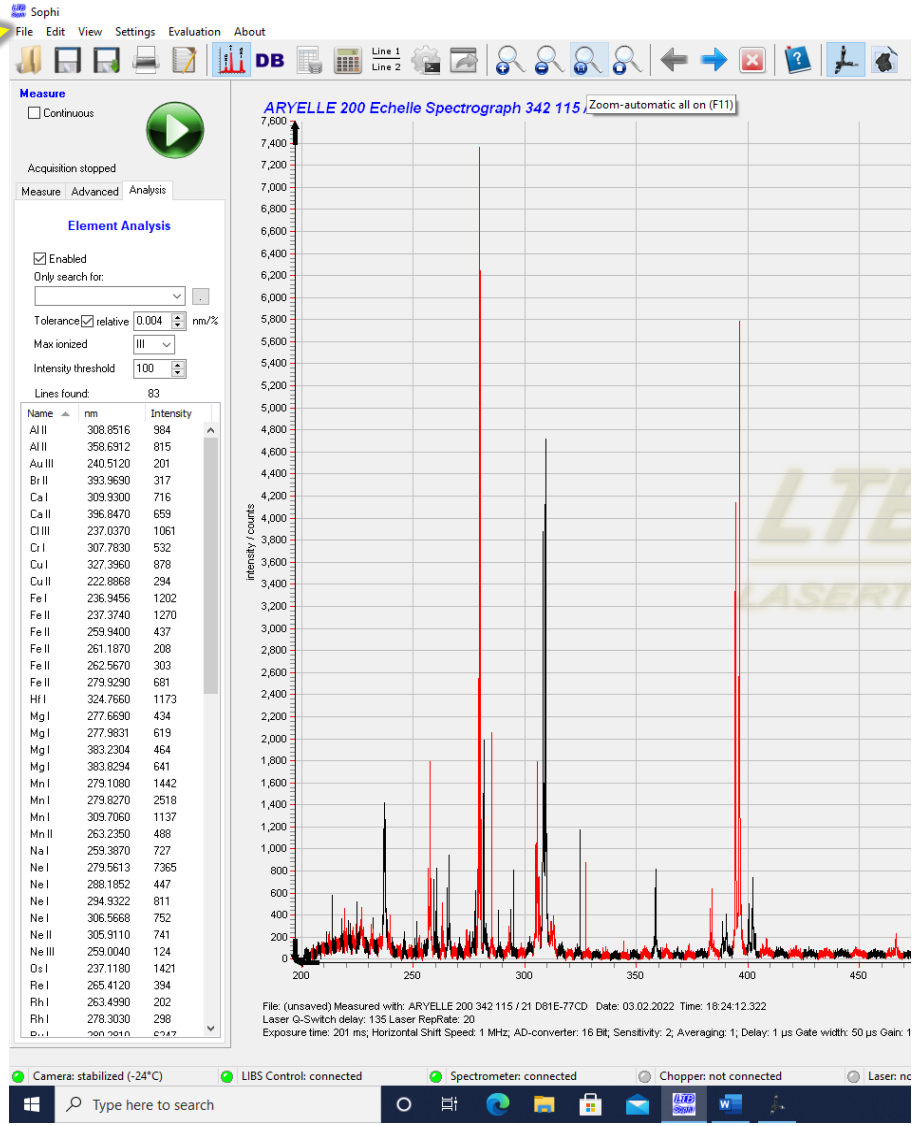
After measurement is done, you will see obtained spectrum in Sophi.

- To save the spectrum in **.ary** format in left upper corner in Sophi **File ⇒ Save ⇒ Choose folder to save file**.

Note: **Every student must** create their own folder named with last name of the student.

Note: **.ary** file can be opened again in Sophie for analysis.

- To save the spectrum in **.txt** (or other formats, but **.xls** currently not working) **File ⇒ Export ⇒ .txt** (or others) ⇒ **Choose folder**.

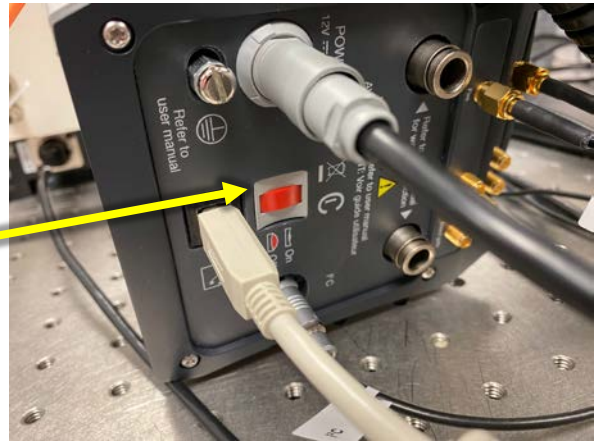


Shutting down the system.

Step 1

After you finished working with LIBS system you have to switch the whole system off.

- Make sure shutter is closed (look at the LUCi) and laser is stopped. No flashlamp noise. (**page 15**)
- Make sure the water pump is turned off. (**page 15**)
- Close **Sophi** software. (Just X in right upper corner)
- Turn the **CCD camera** off, red button on its back panel
- Turn off Light and Pilot Laser switches on the front panel of **LIBS CHAMBER UNIT**.
- Turn the key on **POWER SUPPLY** to OFF position.
- Switch off the **POWER SUPPLY** on its back panel



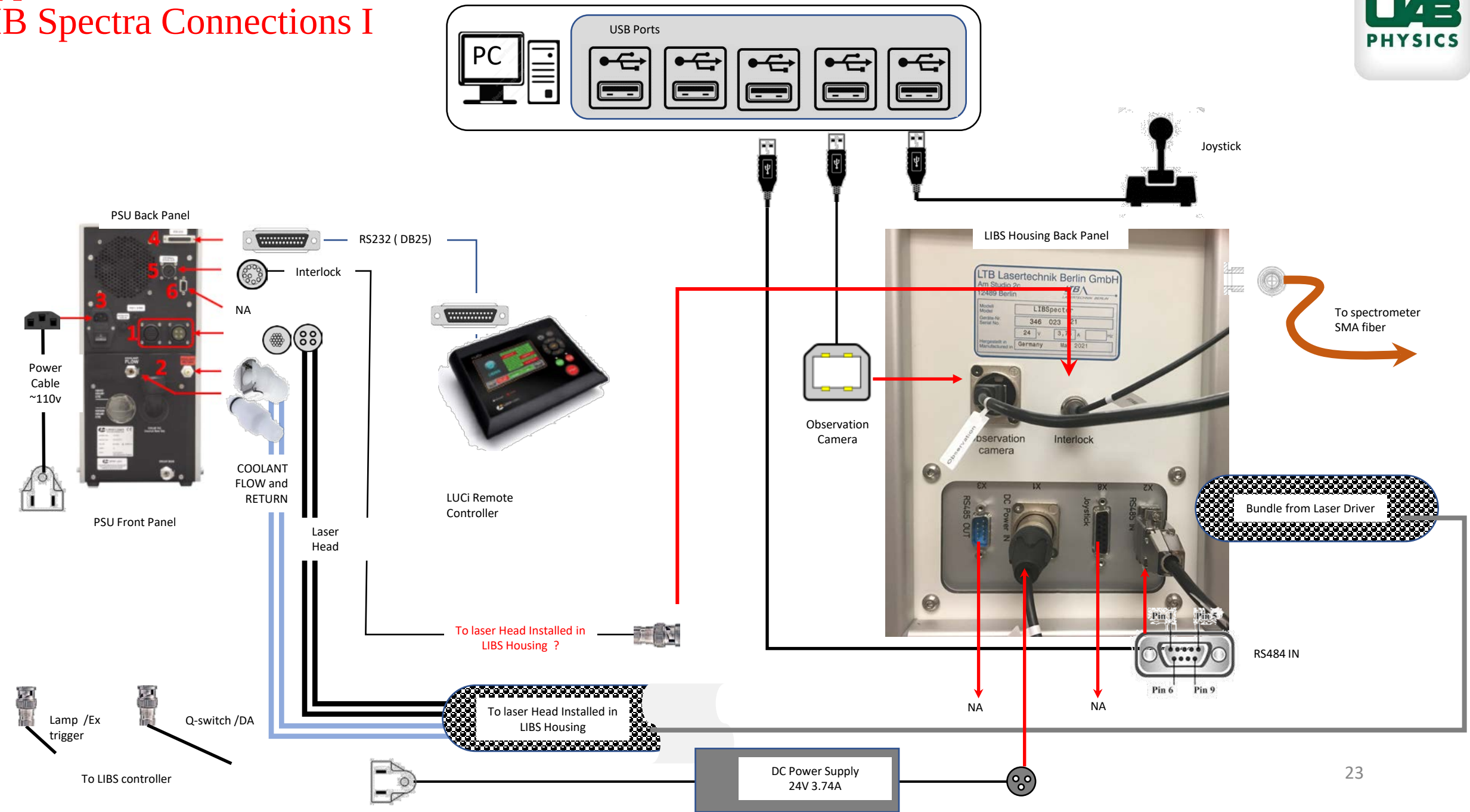
Shutting down the system.

Step 2

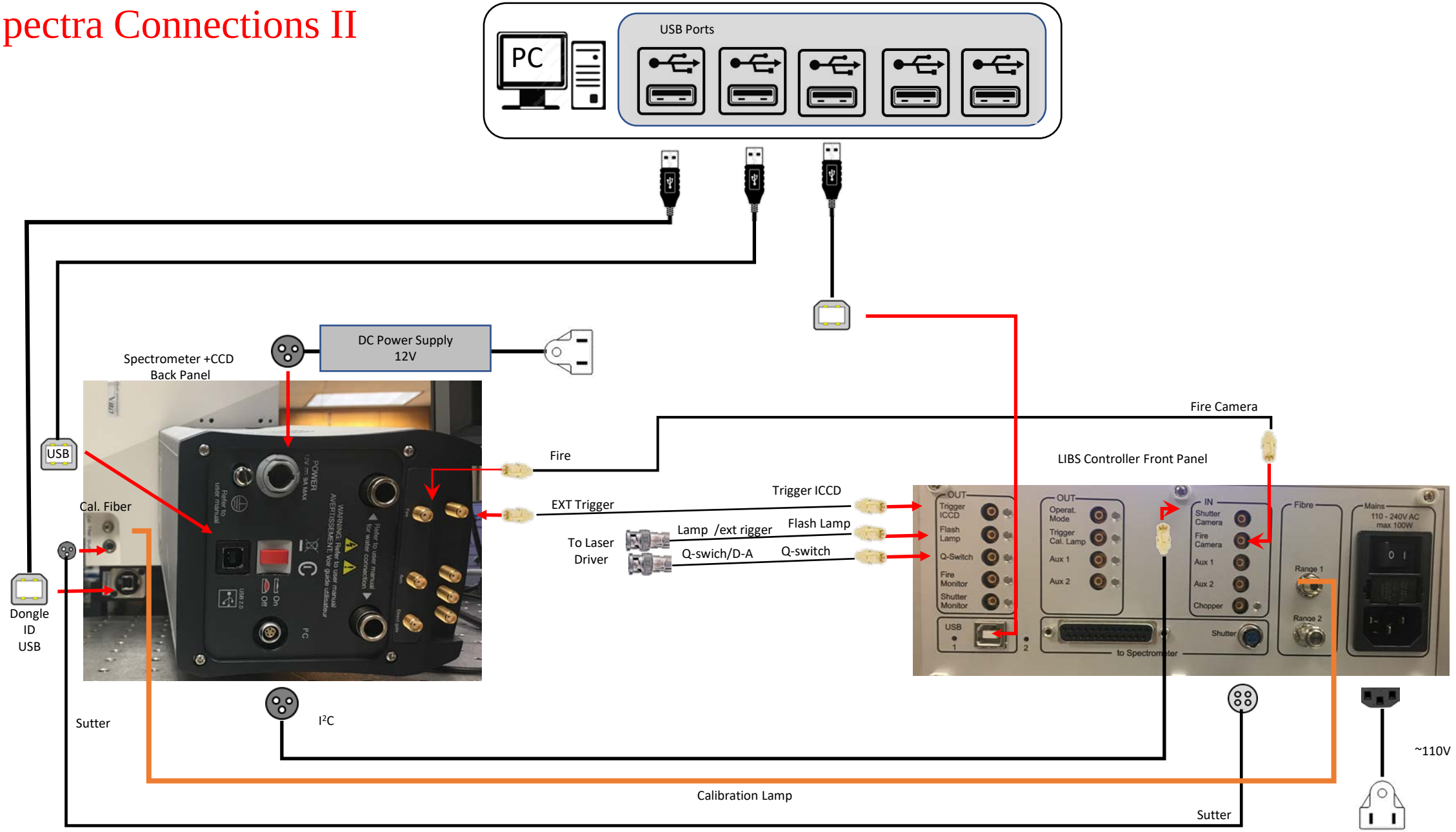
- Turn **the key** on **POWER SUPPLY** to **OFF position**.
- Switch off the **POWER SUPPLY** on its back panel
- Turn off the PC (additionally)

Now system switched off completely.

Appendix 1
LIB Spectra Connections I



Appendix 1
LIB Spectra Connections II



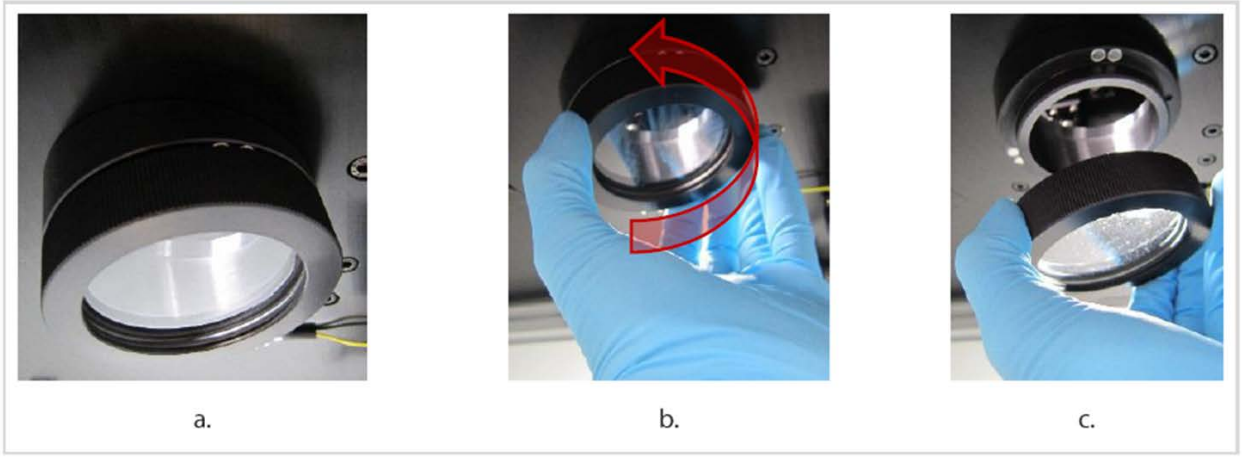
Appendix 2
Cleaning of LIBspector optics I

General Notice : Always keep the protective windows clean to avoid possible damage from the applied LIBS laser radiation

Step 0: Required aids for cleaning the protective window

a) Disposable gloves
b) Acetone
c) Lint-free paper wipes.

Step 1: Remove protective window



a. Open the from door of the LIBSSprctor
b. Turn the magnetic holder counterclockwise
c. Remove the magnetic holder from the housing

Appendix 2
Cleaning of LIBspector optics II

Step 2: Cleaning protective window

d. Remove protective window from the holder. Observe the two sealing rings
e. Clean the protective window with acetone and tissue wipers.

d. e.

Step 3: Mounting protective window.

f. g. h.

f. Place the clean protective windows between two sealing rings inside holder
g. Turn the magnetic holder clockwise to mount the holder
h. Close the front door