

# SOP

## Standard Operating Procedure: LabRAM HR Evolution-RAMAN

### Instruction for operators

#### Precautions

- Make sure room temperature 20-22 °C (check AC) and humidity is < 50% (check dehumidifiers; empty bucket if full)
- Only one detector (Syncerity (CCD1- Visible) or Symphony (CCD2-NIR)) can be used at a time. Typically only Syncerity is used
- Avoid direct exposure to Laser 532 radiation

#### Part 1: Switch on the 532 nm laser (Turn on the laser 10 minutes before measurement)

1. On the main extension board (wooden framed), switch ON the surge protector extension connected to the laser (marked as **3**)
2. Power ON the surge protector by pressing the power button, it should glow
3. Turn ON the power of RemoteBoxx (Black box) and rotate the safety key to “ON” position for laser emission and check status (Green LED). Wait for at least 10 mins for laser power to be stable



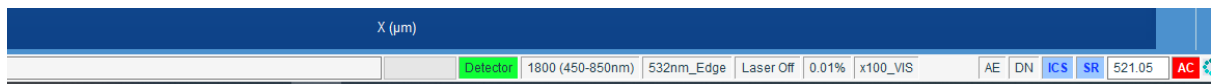
#### Part 2: Switch on LabRAM HR Evolution system

1. Since the laser has been switched on, the surge protector extension (**3**) should be already on by now. Next, switch ON the extension board (white, **marked 4**) connected to Syncerity, Stage controller, light source and General Power supply. Connect Symphony only if it is to be used
2. Turn ON General power supply (**white box, marked 5**)
3. Turn ON power of Stage controller (TANGO Desktop, marked 6)
4. Check the LED behind Syncerity. It should be orange now. Measurements can only be started when it turns green.

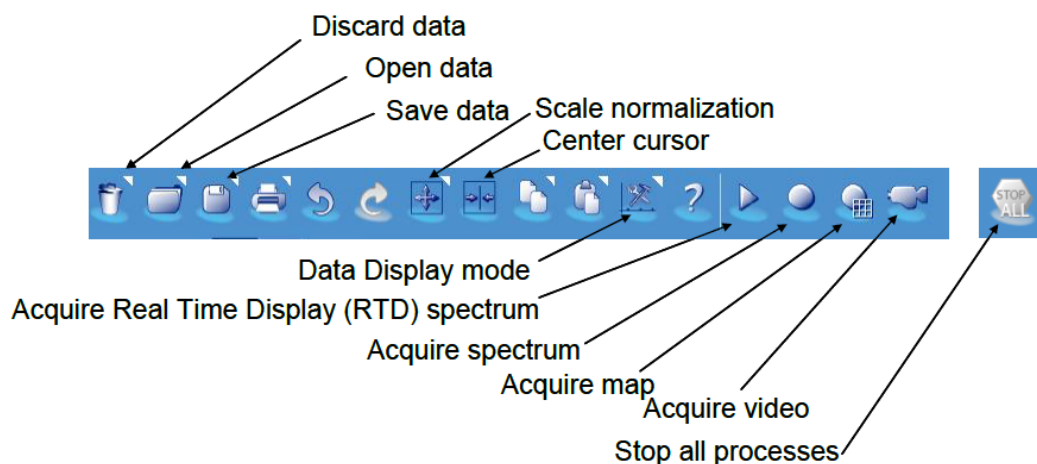
#### Part 3: Starting up LabSpec 6- HORIBA software (See manual for details)

1. Switch ON the extension board (black) connected to the CPU and monitor and turn ON the computer


2. Double click the LabSpec 6 icon on the desktop. Once the software opens, it will ask permission to reset the coordinates of the motorized stage. Select OK. Make sure there is no sample on the stage and the microscope objectives are set to the lowest magnification and in raised position
3. In instrument set-up (right panel) make sure the correct detector is selected. By default, it should be Syncerity (Si CCD)
4. The detector needs to be cooled to  $\sim -60^{\circ}\text{C}$  before starting measurements. By default, the detector starts getting cooled when the software is opened. If not, right-click on the 'detector' option and select 'Cool to Operating Temperature'
5. Wait till the detector is cooled. Initially it will be RED and then ORANGE. Then the color should turn GREEN and upon hovering the mouse pointer on the 'detector' tab, the pop-up should be 'Detector cooled'.



6. Check the LED denoting temperature on Syncerity (back side) and make sure it is GREEN.
7. Check if Autocalibration (AC) on lower panel is GREEN. If RED, the calibration needs to be performed using standard sample before beginning measurements
8. On the 'Acquisition parameters' section on the right panel, make sure the units are in  $\text{cm}^{-1}$ , then enter 521 in the Spectra tab and hit Enter on the keyboard. The spectro will move and finally set to 521.




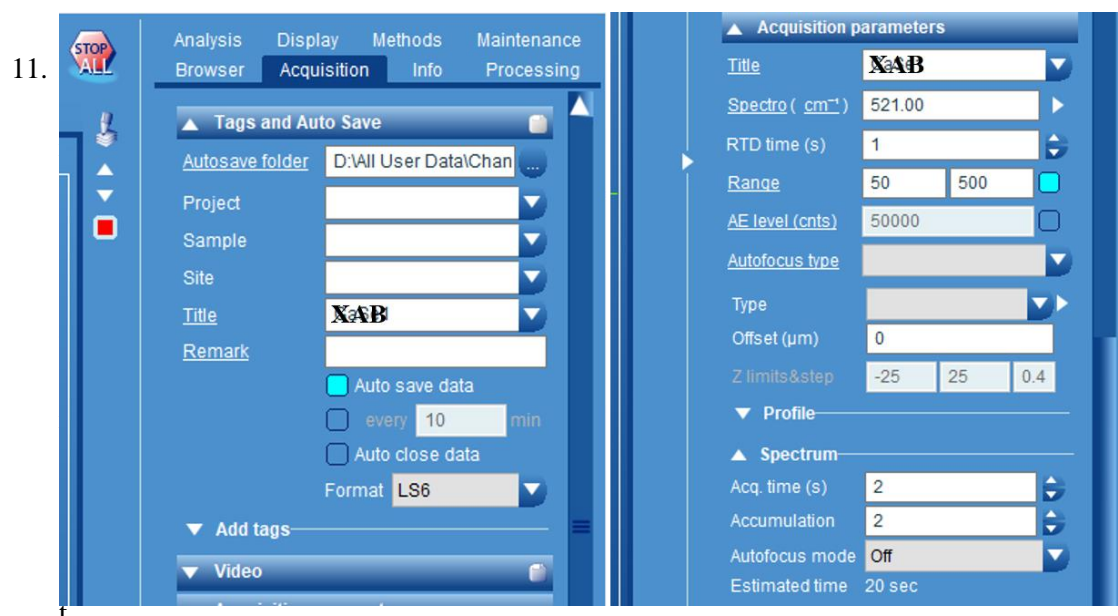
#### **Part 4: Acquiring a Raman spectra**

1. If Autocalibration (AC) is in RED, the system needs to be calibrated. The validity of auto-calibration is 24 hours.
2. Place sample on the microscope stage
3. Click on the Camera icon to 'Start video acquisition'. Click on the Video option in the second line of tabs (if software does not automatically switch to video mode)
4. Adjust intensity of white light source by using the knob
5. Observe the sample at lower magnification first (5X, 10X) and then switch to higher objectives (50X or 100X) as per requirement. The spot where Raman spectra will be acquired is the area denoted by the green dot on the screen. Align required area with that point
6. Click on 'Stop All' on the top right. 

7. Then delete the camera image by clicking the bin icon on the top right. (You can screenshot the desired region here. Deleting the image is to reduce load on software, else it might crash frequently)
8. Turn the knob to completely turn off white light source
9. In 'Tags and Auto Save' section, enter details of folder to save the file. Also enter details of sample name. Make sure autosave is turned on.

-----Only if RTD is required, else avoid step 10-----

10. For Real time display (RTD) acquisition, click on RTD in Spectra option  This will use the value set in Spectro as center point of the scan range. Now, the joystick can be used to improve sample positioning or other system parameters like Acq. time can be modified. Once the desired spectra features are obtained, click on 'Stop All'.




he spectra, check the following parameters:

Set laser power using **ND filter** tags

In 'Acquisition parameters' section on right panel: Select desired **Range**

Under Spectrum, set **Acq. time (s)** and **Accumulation values**

In Instant processing, switch on **FLAT correction** (if needed)

12. Click on '**Start spectrum Acquisiton**' button on top panel. 
13. Wait till the entire spectrum is collected. The status is displayed on the bottom panel, make further changes only after it displays 'Ready'
14. The data can be saved as .txt file manually

#### **Step 5: Performing auto-calibration (NOT COMPULSORY, NEEDED IF AC is RED)**

- a) Before performing Auto Calibration select Detector "Syncerity OE" in Instrument setup and Objective "x100\_VIS"
- b) Click on "AC" on the bottom panel and after that select "Current laser/grating". Upon completion of auto calibration, it will turn Green and provide reference line of the silicon peak position ( $\sim 520.8 \text{ cm}^{-1}$ )

- c) After that put that exact value of silicon peak position in Spectro ( $\text{cm}^{-1}$ ) and hit Enter

**After measurements are done, the system needs to be turned off.**

**Switch off the Laser 532**

- a) Rotate the safety key to “OFF” position and turn off Power of RemoteBoxx (Black box)

**Switch off LabRAM HR Evolution system**

**1. Warm the detector**

- a) Click “warm to ambient temperature” on detector from the software and color should turn RED
- b) Wait for 15-20 mins, the detector color should now turn ORANGE and should show the message “Detector is safe to turn off”
- c) The software can be closed now
2. Turn off the power of Stage controller (TANGO Desktop, marked 6).
3. Turn off the power of General power supply (white, marked 5) HORIBA Scientific
4. Switch off the extension boards (white, **marked 3, 4**)
5. Turn off computer and switch off the extension board (black) connected to PC