Raman spectroscopy setup

Operation Instructions

MAKE SURE YOU SIGNED UP FOR TIME ON THE ON-LINE SCHEDULE FOR THIS INSTRUMENT BEFORE USING IT.

LOG YOUR USAGE IN THE LOGBOOK.

Specifications

Spectral Range: 400-1100nm (1.12-3.10eV) (9090-25000 wavenumbers)

Temperature Range: 1.5K-300K

Monochromator: SPEX Trilemate 1877 Triple grating monochromator.
Gratings available:
  600 and 1800 gr/mm for the double grating filter stage.
  150, 600, and 1800 gr/mm for the spectrograph stage.
Initial Setup

1) Cool the CCD detector.

   a. Verify that the CCD controller is on, and fill the CCD dewar with liquid N$_2$. **IMPORTANT:** The controller of the CCD (see Figure 1) must be **ALWAYS ON** when the CCD is cold, otherwise, permanent damage to the CCD chip will occur. The CCD takes about 20 to 30 minutes to reach a working temperature (-90 to -110 °C), therefore, plan to fill the CCD dewar at least 20 minutes before you intend to start the data acquisition. Once filled, the dewar should keep the CCD cold for the next 12 hours.

   b. Start the program Winspec on the Raman setup computer. Select the **Setup** menu tab, and choose **Detector temperature** (Figure 2) to monitor the CCD temperature. The target temperature should be -110 °C.

      ![Figure 1. CCD Controller.](image1)

      ![Figure 2. Detector temperature.](image2)

2) Mount and align your sample. (See Figure 3)

   a. Mount your sample on the appropriate sample holder or in the cryostat, if you intend to measure at low temperature. If you are using the cryostat, refer to the **Raman setup cryostat usage instructions**.

   b. Open the front slit (S1) of the monochromator to about 4 mm.

   c. Slide the pin-hole mask in, selecting a larger aperture in front of S1.

   d. Turn the TV monitor on, and lower the periscope behind S1, to allow for focusing the region of interest (ROI) on your sample.

   e. Position the sample holder close to the focus of the objective OB. Fine
tune the focus using the micrometers on the sample stage or cryostat support. You should obtain a crisp focused image of your sample’s ROI on the TV monitor. If your sample is very reflective and smooth, it may be difficult to see it’s surface on the monitor. In that case, move to an edge of the sample for a better focusing, then return to the ROI.

f. Adjust the height and lateral position of your sample to center the ROI vertically in relation to the pin-hole, and horizontally in relation to the slit S1.

3) Choose the excitation source (laser) and wavelength, which are appropriate to your measurements, and align it.

   a. Turn the laser on and select its wavelength emission (when applicable) following the laser operation instructions. Leave the laser at the minimum possible power to do the alignment.

   NOTE: Never operate a laser that you have not been trained to operate! Always use proper protective eyewear.

   b. Direct the laser beam to mirror M1, through the prism monochromator. This will clean your beam from the plasma radiation of the laser tube.
c. Insert the cross-hair target in the holder P1, and adjust M1 so the beam hits the center of the target.

d. Move the target to P2, open the neutral density filter ND1 and the iris A1 completely, and adjust M2 so the beam hits the center of the target.

e. Remove the target and place it on the table near P2.

f. Adjust the position of A1, so the beam goes through its center.

g. Use the filter ND1 to lower the intensity of the beam to a minimum.

h. Take the lens L1 out, placing it carefully on the table, caring that the surface of the lens doesn’t touch anything. Adjust M3 to get the beam centered on the sample’s ROI. Use the TV monitor to assure the laser is centered. Replace L1, adjusting it to focus the laser beam and center it on the sample’s ROI.

i. Raise the monochromator periscope.

j. Block the beam.
**Experiment setup**

1) Check the detector temperature, and make sure it is between -90 and -110°C.

2) Select the spectral range for your experiment.
   
   a. Calculate the wavelength range using your laser wavelength ($\lambda_L$), and desired initial ($R_i$) and final ($R_f$) Raman shifts from the equation:

   $\lambda_{i,f} = \frac{10^7 \lambda_L}{10^7 - \lambda_L R_{i,f}}$

   b. From the wavelength range, choose the appropriate grating to be used looking at table 1.

   c. Select the grating by lifting and turning the grating selector knob on top of the spectrometer clockwise.

   d. Move the grating to the center wavelength in your range.

   **Table 1. Diffraction grating selection.**

<table>
<thead>
<tr>
<th>Diffraction grating (gr/mm)</th>
<th>Range on CCD chip (nm)</th>
<th>Range through filter stage (nm/mm) (maximum slit width is 8 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>600 gr/mm</td>
</tr>
<tr>
<td>150</td>
<td>298</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>148</td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>74</td>
<td></td>
</tr>
<tr>
<td>1200</td>
<td>37</td>
<td></td>
</tr>
<tr>
<td>1800</td>
<td>24.7</td>
<td></td>
</tr>
</tbody>
</table>

3) Make sure the laser beam is blocked.

4) Choose the detection area of the CCD chip to be used.

   a. In the Winspec program, select the *Acquisition* menu tab, and choose *Experiment Setup*.

   b. On the *General* tab, check the *Use full chip* box (Figure 4).

   c. Turn the flashlight on and place it in front of the spectrometer slit (you can use the periscope to check if the light is coming in the spectrometer homogeneously).
d. Select an exposure time of 0.1 second, and click the **Acquire** button. It should take about 10 seconds for the program to show the CCD image (Figure 5).

e. Turn off the flashlight and put it aside.

f. Once the CCD image is displayed, select the **Acquisition** menu tab, and choose **Experiment Setup**. Select the **ROI** tab.

g. Select with the mouse, in the graphic window, an area which spans across the whole chip horizontally, and has the same height as the just acquired data.

h. Click on the button **Mouse**, then on the **Store** button, and confirm to overwrite the stored configuration.

i. In the **General** tab, choose **Use Region of Interest**, and click **OK** (Figure 4).

5) Calibrate the grating position.
Experiment setup

a. Chose the appropriate calibration lamp to use for the selected range. Use Figure 7 as a guide.

b. Place the calibration lamp (Figure 6) in front of the spectrometer, and power it on. **ALWAYS check if the correct power supply is connected to the lamp. Using a power supply for a different lamp may result in damage to the calibration lamp.**

c. Start with an acquisition time of 50 ms, and acquire a spectrum. If you see the lamp peaks, turn off the lamp, and proceed with the calibration. The calibration lamps have a short lifetime, so keep them on only for the necessary time to get the peaks for the calibration.

d. If you don’t see any peaks, check to see if the filter stage is appropriately set, and nothing is blocking the light (e.g. the periscope is down). If everything is right, increase the acquisition time to 200 ms. If you still don’t see a signal, and you are on a region that the calibration lamp has peaks, see Appendix 1)b.

e. Once you see the peaks, you can use them to calibrate the position of the spectrometer grating in relation to the CCD chip. The best is to have peaks covering the full range of your experiment, which is not always possible. See Appendix 1)a. for more details on this.

f. With the calibration lamp spectrum window selected, in the Calibration menu tab choose Setup. You will see a table like the one in Figure 8.
g. For each peak, enter the peak position on the CCD in pixels, and the correspondent value for the calibration lamp peak in the units defined on the right upper corner of the calibration setup window. To find the values for the calibration lamps peaks, use the spectra in Appendix 2. Choose rel. cm\(^{-1}\) as display units, and enter the wavelength of the excitation laser, then click OK.

h. The system is now calibrated and ready for data acquisition.
Data Acquisition

1) Make sure that the alignment of your sample didn’t change, by lowering the periscope, turning the illuminator on, and observing your sample surface.

2) Unblock the laser beam and make sure it’s centered on the slit aperture.

3) Raise the periscope.

4) Make sure you are effectively blocking the laser on the CCD by acquiring a short time spectrum (6-7 ms). If you see a strong laser peak, move the filter stage to block the beam from reaching the CCD.

5) Choose the acquisition time by clicking the button, or changing it on the Experiment setup dialog box (Figure 4), under the Acquisition menu tab. You should start with a small acquisition time, and increase it as necessary. The CCD saturates at 64000 counts. If you are reaching the saturation of the CCD, lower your acquisition time.

6) If the feature of your spectrum that interests you is much weaker than other spectral features, and you need longer exposure times, which would saturate the CCD with the other spectral features, you can choose to accumulate several acquisitions, by increasing the number of accumulations on the Experiment setup dialog box (Figure 4). Alternatively you could set the filter stage to block the other spectral features.

7) It may prove useful to use the check list on appendix 3 every time you acquire data. It may save you some time.
Shut down

1) Save your data.

2) Convert to ascii format the data you want to analyze or plot with a different program.
   a. Press the "button.
   b. Select all the files to be converted.
   c. Choose the output directory.
   d. Press the “Convert to ASCII” button (Figure 9).
   e. Press “Done”

3) Exit the program Winspec.

4) Terminate the laser beam with a beam block and close the shutter on the laser (when applicable).

5) Shutdown the laser, following the proper shutdown procedure for the laser in question. For the Ar ion lasers, ALWAYS allow the cooling water to flow (and the chiller to run, for the black I-90) for at least 10 minutes after turning the laser off, so the tube cools down. Failing to do so will result in catastrophic damage to the laser tube (~30 – 50 K US$).

6) LEAVE THE CCD CONTROLLER ON!!!!!!!
Appendix 1 – Calibrating the spectrometer position

a. How to ensure the best calibration

i. Select peaks over the entire range you are measuring at.

ii. Use only three or four peaks for the calibration, and confirm the accuracy of your calibration, verifying the position of other peaks.

iii. If too few peaks are visible, you can use more than one calibration lamp.

iv. Moving the filter stage doesn’t affect the calibration, therefore, you can move it to acquire more peaks over the entire range of the CCD camera.

v. Perform a new calibration every time you move or change the grating.

b. Troubleshooting for weak signal from calibration lamps. If you don’t see any peaks:

i. Verify that the lamp has peaks in the region you are acquiring at. If not, choose a different lamp.

ii. Check to see if nothing is blocking the light from the calibration lamp (e.g., the periscope is down, or misaligned pin-hole), and if the lamp is properly aligned in the spectrometer entrance. You can use the periscope to check the alignment (you should see the lamp very bright on the monitor), but don’t forget to raise it to acquire the peaks.

iii. Check if the filter stage is properly set at a wavelength in the range covered by the CCD with the chosen grating and center wavelength. Use table 1 to verify that.

iv. Make sure the lamp is on.

v. If all the above was verified and is OK, increase the acquisition time. As a rule of thumb, if you need more than 2 minutes of acquisition time to get peaks, something is wrong. Typical times range from 20 ms (strong peaks) to 10 s (very weak peaks).
Appendix 2 – Laser lines (some of the available laser lines for Raman excitation)

Ar ion
- 514.5 nm
- 501.7 nm
- 496.5 nm
- 488.0 nm
- 476.5 nm
- 457.9 nm

Nd:YAG (doubled)
- 532.0 nm

Dye (DCM)
- 620 – 680 nm
Appendix 3 – Measurement check list

Short measurement check list

1) Verify that the neutral density filter is appropriately set to the desired power
2) Make sure that the filter stage is correctly set.
3) Check the slits widths.
4) Make sure the laser beam is unblocked.
5) Check that the laser hits the spot on the sample that is focused on the slit/pinhole of the spectrometer (use the periscope and TV monitor, never look directly into the laser beam).
6) Check that the fiber optic sample illuminator is off.
7) Certify that the periscope is up, out of the light path.