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Last updated: 03.01.2016

1. Raman facility

CW Raman Table

The University of Rochester Chemistry Department Continuous Wave (CW) Raman System, was designed and built to be a multi-user facility for the collection of Raman spectra for users with differing experimental needs and setups. The system is comprised of eleven independent diode lasers spanning from the Ultraviolet (UV) to the near-Infrared (IR) regions of the electromagnetic (EM) spectrum. The sample position is easily accessed so that a variety of sample setups (as well as a cryostat) can be placed in beam focus. Once Raman scattering is achieved, it enters the slit of a Princeton Instruments TriVista555 Triple Spectrograph equipped with three monochromators housing 9 gratings and a CCD camera. The TriVista triple spectrograph can be run in triple subtractive mode for stray light reduction, additive mode for enhanced spectral resolution, or as a single monochromator for low level scattering conditions.

2. Laser lab safety

ALL users must provide evidence of completing the annual Laser Safety Training course provided by EH&S before utilizing the instrument.

Additional on-site training will be provided to new users.

3. Laser Table Layout

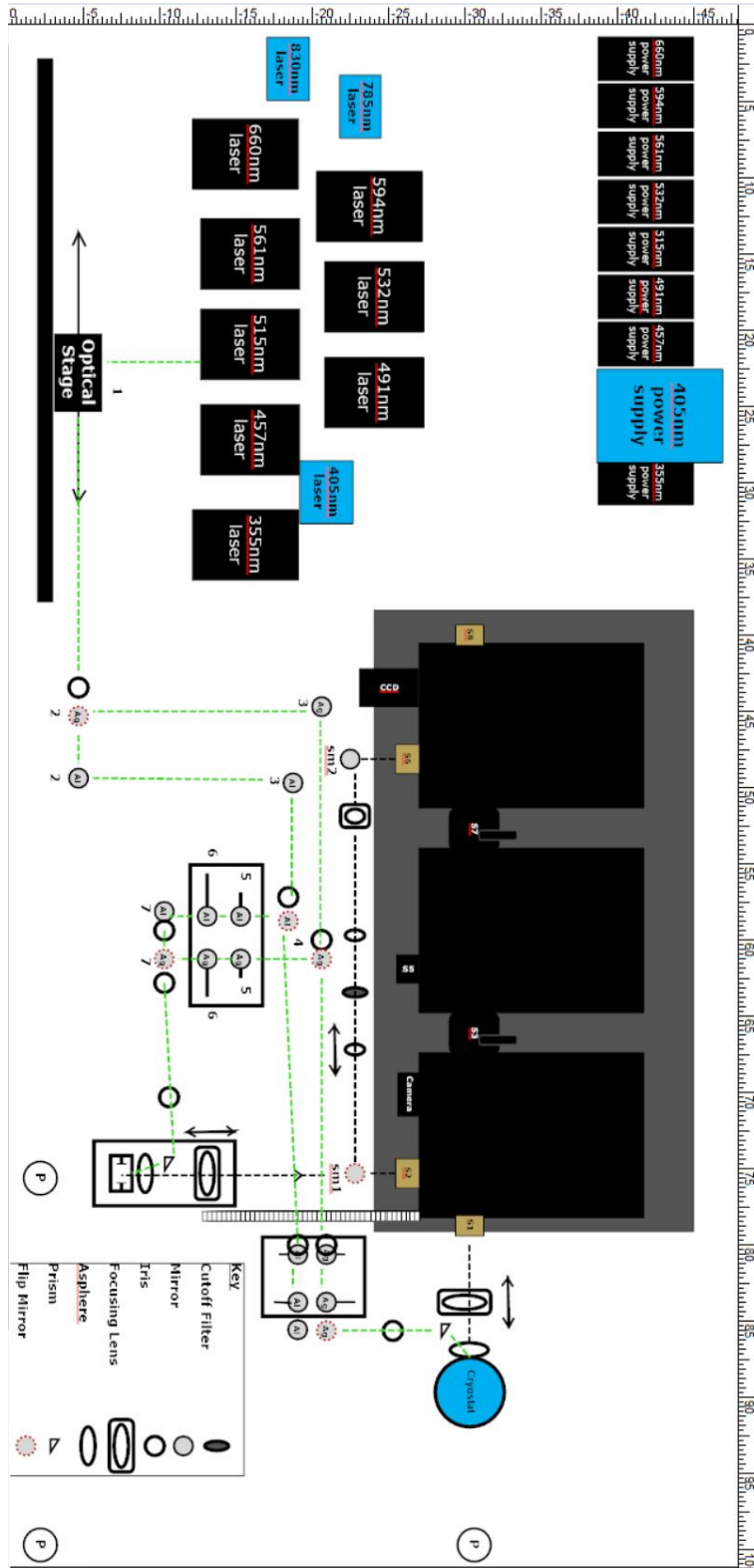


Figure 3.1. Overhead layout of the CW Raman table.

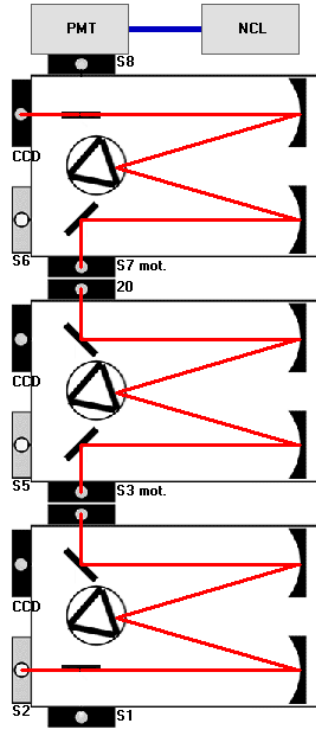


Figure 3.2. Schematic of TriVista triple spectrograph with illustrated beam path.

4. Setting up the table for your experiment.

4.1 Laser operation

The CW Raman system is houses 11 different diode lasers ranging from the UV to the Near-IR region of the EM spectrum. The lasers come from two different manufactures, Cobolt and Innovative Photonic Solutions (IPS).

*****Note: Always make sure the “Laser In Use” sign is on before turning on ANY of the lasers!*****

- The Cobolt^c lasers include 355 nm, 457 nm, 491 nm, 515 nm, 532 nm, 561 nm, 594 nm, and 660 nm wavelengths. Basic operation: of these lasers begins with turning the key on the back of the power source from “off” to the “on” position. Upon turning the key, the orange LED will be replaced by a blinking green LED on the back of the power supply. *Note: once the key is turned the laser will commence lasing, though not yet completely stable and ready for experimental use. Once the blinking stops, the laser is considered stable and is ready for use. The shutter must be flipped to the open position to allow the beam to exit the laser head.



Figure 4.1. Picture of Cobolt laser head and power source.

- The Innovative Photonics Solutions (IPS) lasers include 405 nm, 785 nm, and 830 nm wavelengths. The IPS lasers include 405 nm, 785 nm, and 830 nm wavelengths. Basic operation: To turn on the laser, turn the key on the back of the blue power supply. Push black on button on front of laser power supply. **** Note: There is no shutter on the laser head of the IPS lasers! Be sure the beam is following a controlled path before tuning on power button.**



Figure 4.2. Picture of IPS laser head and power source.

4.2.1 Selecting correct laser path: Triple monochromator

The CW Raman system is designed to measure the Raman spectrum over a broad region of the EM spectrum. To do this, laser wavelengths must be reflected off several mirrors before making it to the where they can interact with the sample. Due to the broad spectral region, mirrors made of different materials are used to maximize reflection efficiency such that maximum power will reach the sample. In general, silver mirrors should be use for wavelengths spanning the visible to near-IR region of the EM spectrum (457 to 830 nm) and aluminum mirrors for the shortest (UV) wavelengths (≤ 405 nm).

- If using the Ag track, mirrors Ag-2 and Ag-7 must be flipped **up**, see diagram on pg. 3 for mirror locations.
- If using the Al track, mirrors Ag-2 and Ag-7 must be flipped **down**, see diagram on pg. 3 for mirror locations.

4.2.2 Selecting correct laser path: Single monochromator

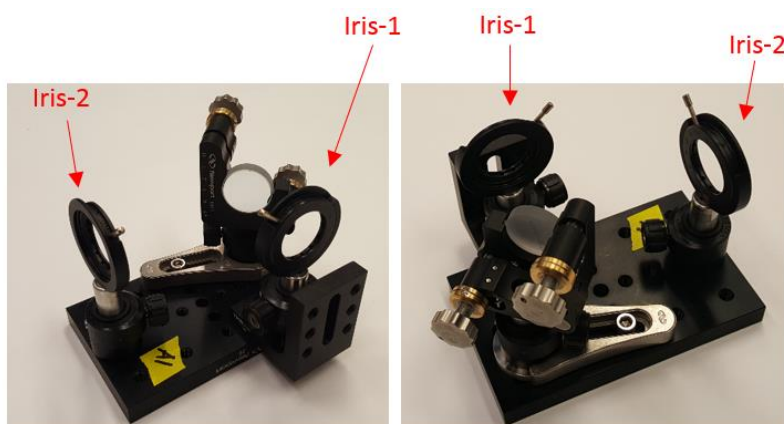
In special cases, such as low scattering samples or for grating efficiency reasons, the triple spectrograph setup may not be the optimal choice for Raman collection. In this case, the CW Raman system has been setup with the option to collect in a single monochomator setup as seen in figure X. Here, the Raman scattering path bypasses the first two stages of the TriVista triple spectrograph and light enters in directly through stage 3, see figure 3.1.

- To select the single monochromator setup flip mirror SM-1 up, this redirects the scattered Raman to slit S6. Be sure to remove entrance slit cover.
- Put the notch/cut-off filter that corresponds to the laser being used in the holder between the two focusing lenses before collecting data.

4.3.1 Beam alignment

Once the proper beam path is selected, it is time to align the beam along the proper path such that it makes it to the sample.

- First, select the translations stage (see below) containing the first mirror and iris which matches the mirror path of choice (Silver or Aluminum).



Front View

Back View

Figure 4.3. Picture of front and back view of translation stage used for selecting laser wavelength.

- Once the translations stage is in place open the shutter to allow the laser to reflect off the first mirror and align it such that it passes through the center of the second iris.
*****Note: Iris-1 is only used for aligning lasers which are invisible to the human eye. This will be discussed in the Near-IR alignment section.** This does not need to be precise. Once aligned, close the shutter on the laser/turn laser off (IPS) and carefully screw the metal foot for the translation stage to the table ensuring that it will not move during the experiment.
- Open the shutter/turn on the laser. If the beam is still in the same place, open iris-2 and align the beam through iris-3 using the adjustment knobs on mirror-1 which is located on the translation stage.
- Open iris-3 fully and follow the beam to the next iris. Align the beam through the iris using the mirror immediately following iris-2. ****Do not adjust any other mirrors on the table.**
- Once aligned the beam should be hitting the prism and being roughly focused through the aspheric lens located before the sample position.

4.3.2 Prism Adjustment

The CW Raman system is designed to have a backscattering geometry where the laser of use is focused through an aspheric lens and the backscattered Raman signal is at collected 180° , see figure. Because there are 11 different laser sources, the parallel beam alignment through asphere must be adjusted with the prism angle in order to focus properly in the sample.

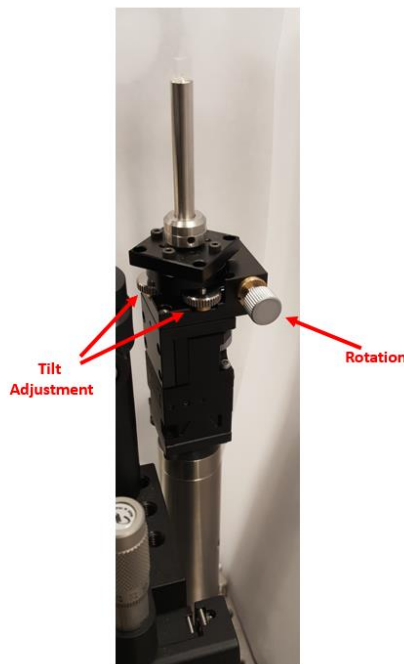


Figure 4.4. Prism adjustment stage located between aspheric and focusing lenses. Main adjustment knobs are shown.

- There are 5 knobs on the prism stand that adjust the location and angle of the prism.
- In general, the position of the prism will not have to be changed making adjustment the x, y, and z controls unnecessary.
- Adjustment of the two knobs for tilt and one for rotation must be adjusted iteratively such that the beam passes through the aspheric lens in a parallel fashion, see below.

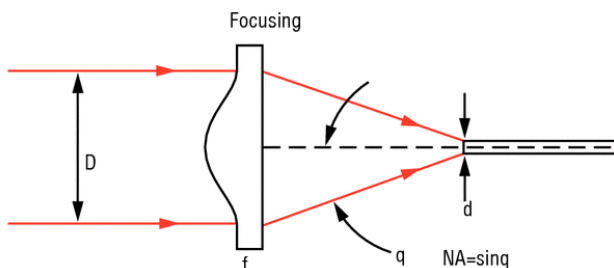


Figure 4.5. Diagram of beam focusing through aspheric lens. The beam must pass through the lens parallel to the focal plane in order for the beam to have the correct focus spot size and be at the correct distance at the sample.

*****Note: For prism adjustment, a solvent standard will be used to maximize scattering while the system is running in “Focus Mode”, see section 4.4 for sample setup and section 6 for computer controlled data acquisition.**

4.4 Sample setup

The system was designed to be able to collect Raman of a diverse number of sample types. For optimal Raman collection:

- The sample must be positioned 3.6 cm behind the asphere. This is the focal length of the lens which will ensure maximum Raman scattering.
- Nothing should be blocking the backscattered Raman signal.

4.5 Focusing lens and slit width adjustment

After collection of Raman scattering, the aspheric lens will collimate the scattered light and send it to the focusing lens. The focusing lens is designed to take the scattered light and focus it to a point (at the slit) then it will disperse in the monochromator. In the current setup, the lens is a fused silica plano-convex lens that is f-matched ($f/5.9$) to the TriVista555. Because we are capable of using several different wavelengths of light, we must accommodate for index of refraction in the fused silica medium. This requires adjustment of the focal length of the lens to that of the slit. Below is a table which gives the specific focal length adjustments for the focusing lens based on wavelength. Distance is measured from the entrance slit to the center of focusing lens 1. See diagram 3.1.

Table 4.1. Wavelength dependence based on index of refraction of focusing lens.

Laser Wavelength	Index of Refraction (Fused Silica)	Focal Length
355	1.47612	289.2
457	1.46498	296.1
491	1.4628	297.5
515	1.46156	298.3
532	1.46071	298.8
561	1.4595	299.6
594	1.45824	300.4
660	1.45637	301.7
785	1.4536	303.5
830	1.45282	304.1

***Put in diagram and put ruler on table.

- The focusing lens is situated on a movable stage. To maximize signal, adjust the stage such that the center of the lens holder is the correct distance from the top of entrance slit 2.

5. Computer control for table setup.

The TriVista triple monochromator system, with the exception of the entrance slits, is almost completely computer controlled via the S&I software package. The program has control over internal slit widths, grating selection and combination, grating calibration and CCD acquisition. The software also has Winspec integrated into it which allows for more precise CCD control as well.

Below is an image of the “Measuring” panel in S&I. From the “Settings” window (top), the user can select the mode of choice, x-axis display units and the laser wavelength being used. In this case the system is set to collect data using the 3-3-21 Sub 438.833nm mode which will display Raman on a wavenumber axis and set to use a 405 nm laser.

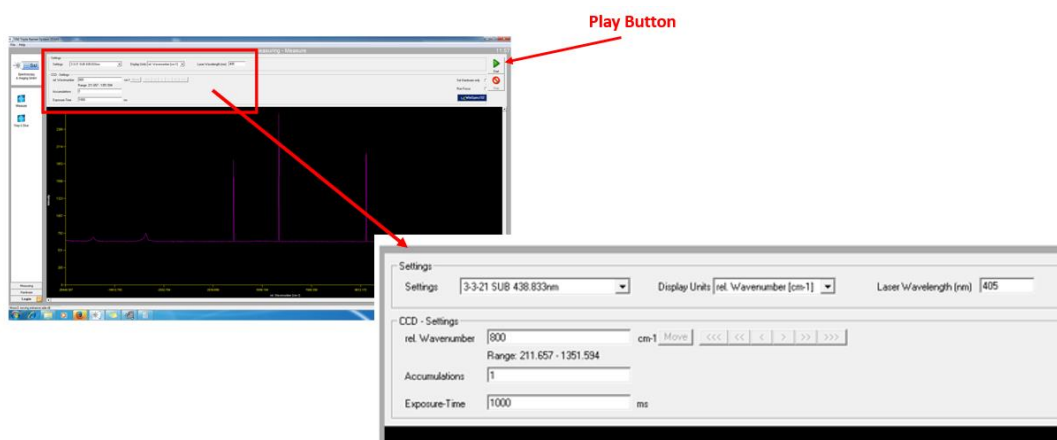


Figure 5.1. Basic S&I parameter setup for data collection.

*****Note: Each time a new method is selected in the S&I software, the default laser setting is 532 nm. Be sure to select the correct laser as it is possible to damage the CCD camera if laser radiation enters the spectrograph.**

Pushing the play button (green triangle, see above diagram) will tell the TriVista to change whatever settings are listed on the screen and it will automatically collect data once it is in position.

5.1. Choosing the correct mode

The TriVista triple monochromator requires that light passes correctly through each of the three monochromators which are each separated by a optical slit. This important for all collection, however, becomes especially important when running the system in triple-subtractive mode as slit 3 selects the spectral window of choice and slits 1 and 7 the resolution.

The user should choose a mode which is calibrated for near the laser line of choice, but not encompassing the Raleigh line. For example: say you want to collect stokes scattering of a compound between 500 cm⁻¹ and 1500 cm⁻¹ using a 532 nm laser. On an energy axis the 532 nm laser is at 18797 cm⁻¹ making the Raman shift window of interest 18297-17297 cm⁻¹. This corresponds to a range of 546-576 nm on a wavelength axis, centered at 561 nm. Below is an example of cyclohexane collected under similar settings. Figure a, shows cyclohexane plotted against the energy axis in wavenumbers (cm⁻¹)

where figure b shows the same data plotted against a wavelength axis. Since calibration is done using atomic emission lines, all modes are calibrated against a wavelength axis. A method calibrated to this region ensures that the maximum amount of light can be transmitted through the spectrograph in this region. Most methods calibrated to this general region will work adequately.

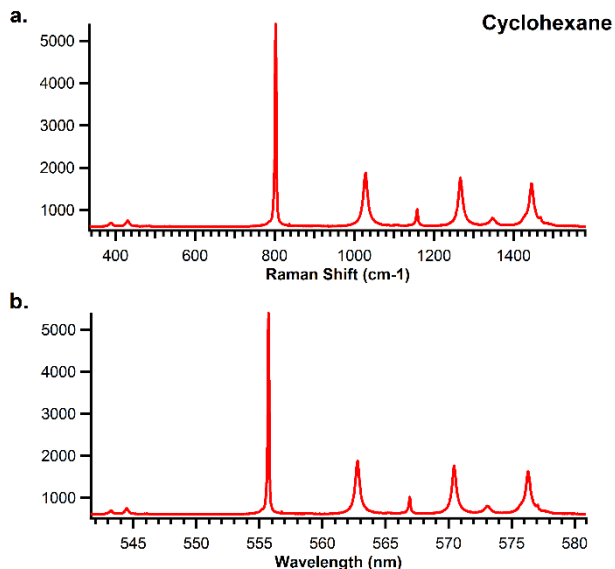


Figure 5.2. Raman shift vs wavelength axis for cyclohexane standard.

5.2 Creating a new mode

Due to the diverse nature of Raman spectroscopy and the limitations of single grating combinations, in many cases it will be necessary to create new “modes” which will allow the user to focus in on specific regions of the spectrum or increase/decrease spectral resolution based on their needs. New “modes” are created in the “Hardware” window found in the S&I software. See figure 5.3.

Basic instruction to make a new mode. In the Hardware menu of S&I:

1. Click on an existing mode with similar parameters.
2. Click the “New” option under the scroll down menu of modes.
3. Rename the method with an appropriate name.
4. Click the “Accept Changes” option. This will create a new mode with the same parameters as the original mode.
5. After the new mode is made, **make sure it is highlighted in the scroll down menu**, then make any and all necessary changes. This includes selecting grating combination, the light path through the spectrograph and slit widths. Once you are satisfied with these changes, click “Accept Changes” to store the new parameters.
6. To use the new mode, **make sure it is highlighted in the scroll down menu** then click “Set Mode.” This will initiate the TriVista to begin changing to the new parameters.

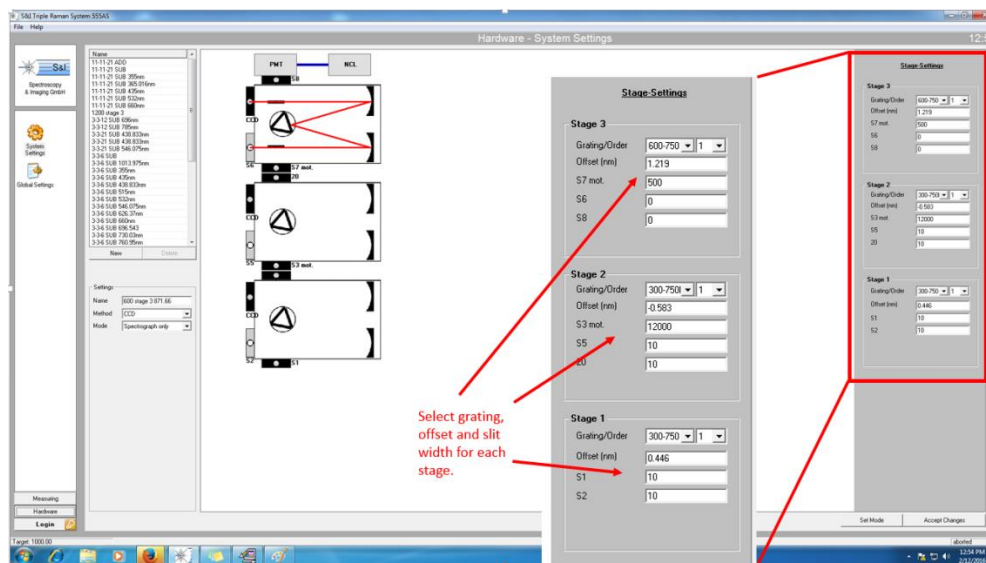


Figure 5.3. Image of “Hardware” control in the S&I software. Highlighted is the grating combination and slit width adjustment window.

5.3 Selecting the appropriate wavelength region and calibrating grating turrets

****Data collection in S&I is covered in section 6.**

Calibration of the TriVista grating combinations is very important to successfully collecting Raman spectra. In any case, calibration of the spectrograph is done using a HgAr lamp which gives known atomic emission lines, see below. Depending on the setup used (Triple subtractive, Triple additive, single monochromator) the system is calibrated differently.

****Note: In many cases there will already be a preset mode created for your needs. Be sure to check the premade methods before creating a new one.**

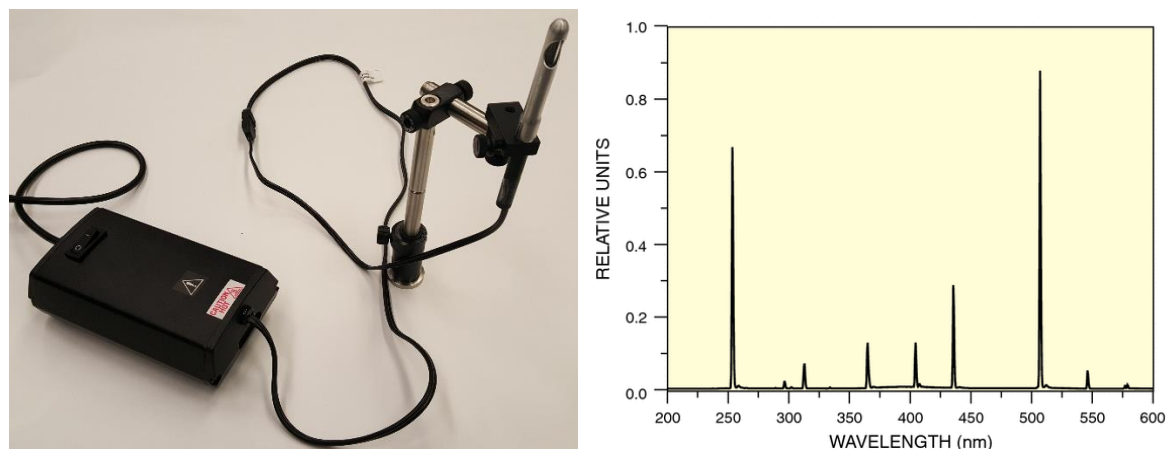


Figure 5.4. HgAr lamp setup (left) spectrum of atomic calibration lines given by lamp (right).

Calibration Instruction:

- Regardless of the setup, the HgAr lamp should be placed in front of the slit which Raman scattering is going to enter the spectrograph. When calibrating, the slit may need to be closed and/or the lamp may need to be moved further away from the slit as not to saturate the detector. An intensity of several hundred to several thousand counts is sufficient for calibration.
- After the lamp is situated, in S&I set the appropriate mode from the pull down menu. Next, change the display units to “Wavelength (nm)” the laser line to the value of the atomic emission line used for calibration and the relative wavelength to 0 nm. See blow figure A. This will ensure that the emission line selected for calibration appears at 0 wavelength when data is collected.
- Next, set the instrument to collect with a 100ms exposure an select to run in “Focus” mode.
- In the “File” option, while collecting, open the “Advanced Slit Commands” option, see figure B and C.
- For further calibration, see sub categories of calibration below.

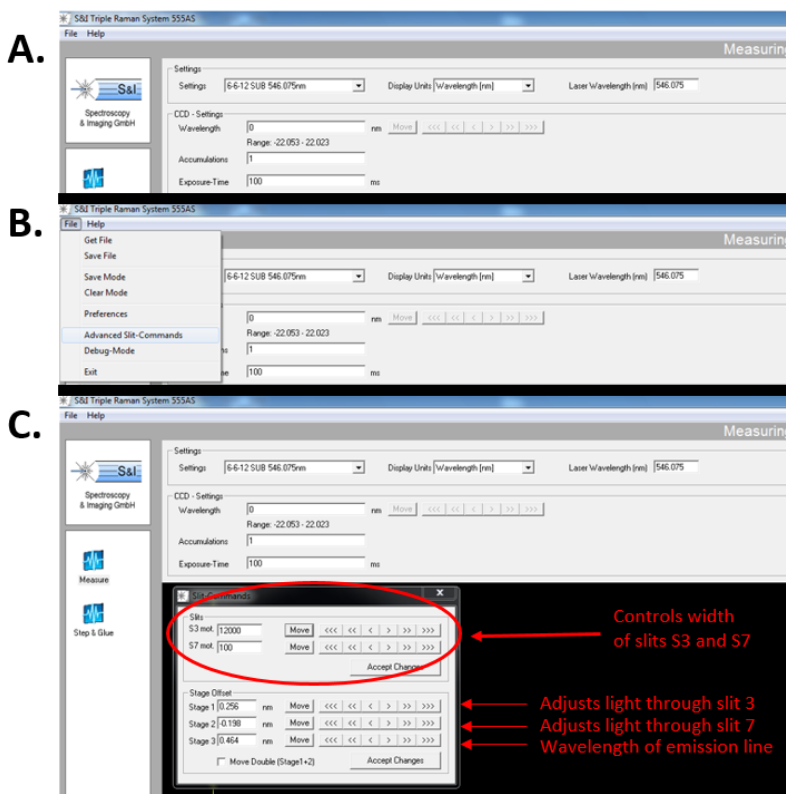


Figure 5.5. S&I software setup for calibration of grating turrets.

Subtractive Mode:

- While running in “focus” open both slits S3 and S7 to 12000 (fully open). Incrementally decrease slit S3 while simultaneously maximizing the signal with the “Stage 1” arrows, see figure C. When

S3 has been successfully decreased to 30 μm or less, and the signal of the atomic transition has been maximized the grating is calibrated.

- Next, reopen slit S3 fully to 12000 μm and begin the same process for slit S7 whilst changing the offset for "Stage 2." When this is complete, reopen slit S7 fully.
- Finally, adjust "Stage 3" such that your calibration wavelength is set to 0. Click "accept changes" to finalize the settings.
- Be sure to set S7 to match the entrance slit width to obtain proper resolution.

Additive Mode:

- Triple additive mode is calibrated in the same fashion as triple subtractive, however, the first slit is set to be half that of the second after completing the calibration. An example of this is if S3 is set to 6000 then slit S7 should be set to 12000.

Single Monochromator

- The single monochromator setup required only the "Stage 3" setting to be adjusted such that the atomic line of choice is located at a wavelength of 0 nm when the instrument is ready to be calibrated.

Section 6: Software control for data collection.

6.1 S&I Software

Collection of most data can be done using the Princeton Instruments S&I software. As seen in figure X, this software controls both the TriVista settings and the CCD detector settings. The TriVista settings in the case of figure X show that the mode is set to “600 Stage 3 871.66” the display units are in rel. wavenumbers and the laser being used is 785 nm. The detector settings show the data will be centered at 1000 relative wavenumbers, 1 accumulations will be taken with an exposure time of 1000 ms (or 1 second).

When the proper settings are inputted in the system, the green “play” button sets the parameters and collects the data as shown in Figure 6.1.

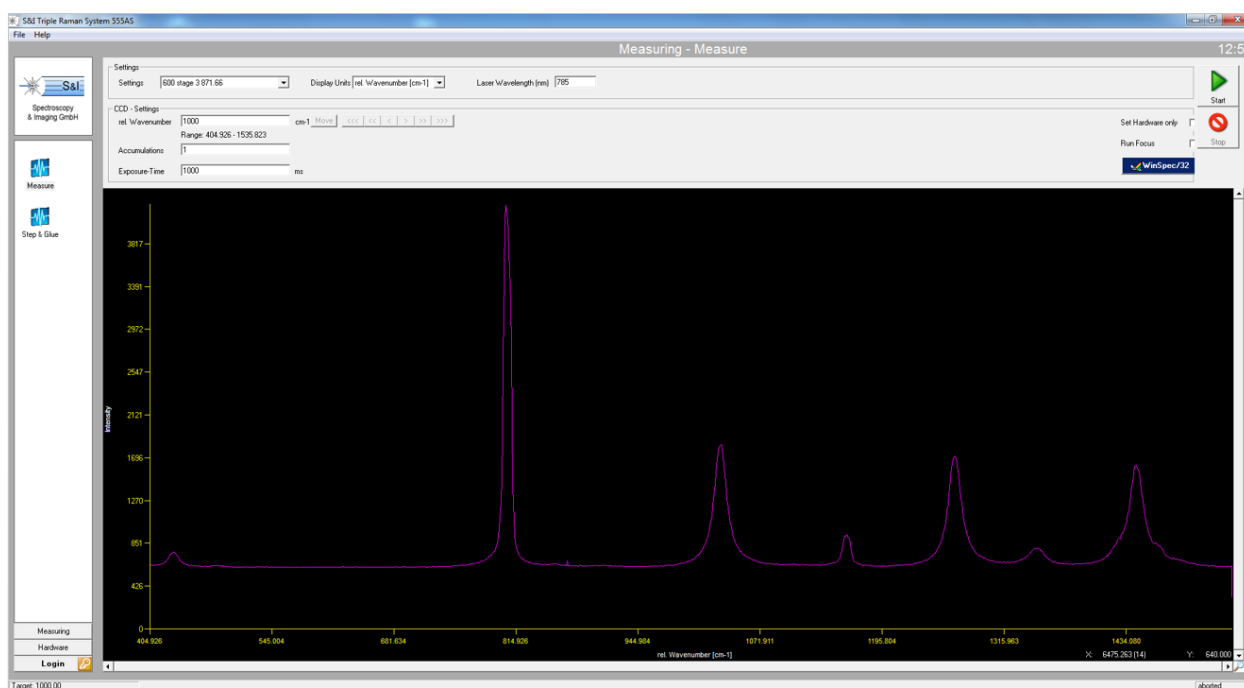


Figure 6.1. Screenshot of “Measuring” screen in S&I software with Raman collected.

6.2 Winspec

For more advanced settings, WinSpec can be used to control the CCD camera. WinSpec can be opened via the desktop icon or with the WinSpec button integrated into the S&I “Measuring” window. The main Winspec operation window is seen below in figure 6.2.1.

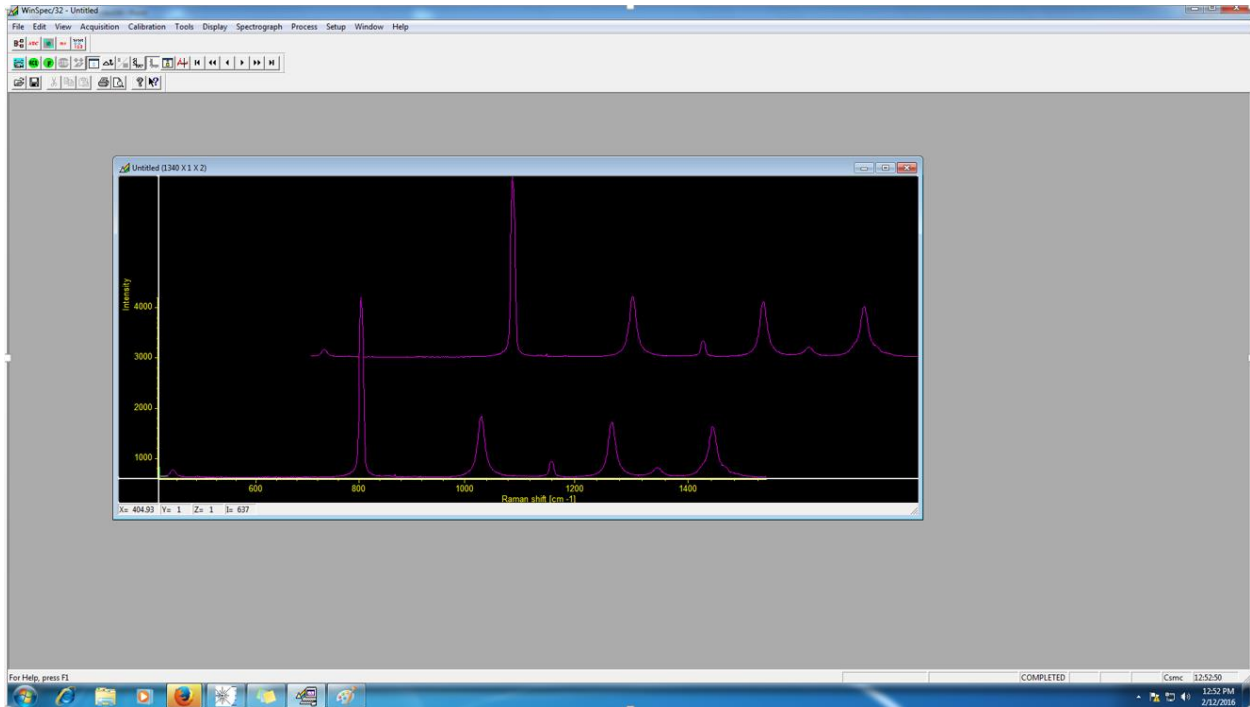


Figure 6.2.1. Screenshot of Winspec data collection window.

Basic camera control can be found in the “Acquisition” pulldown menu and by selecting the “Experimental Setup...” option, see figure 6.2.2.

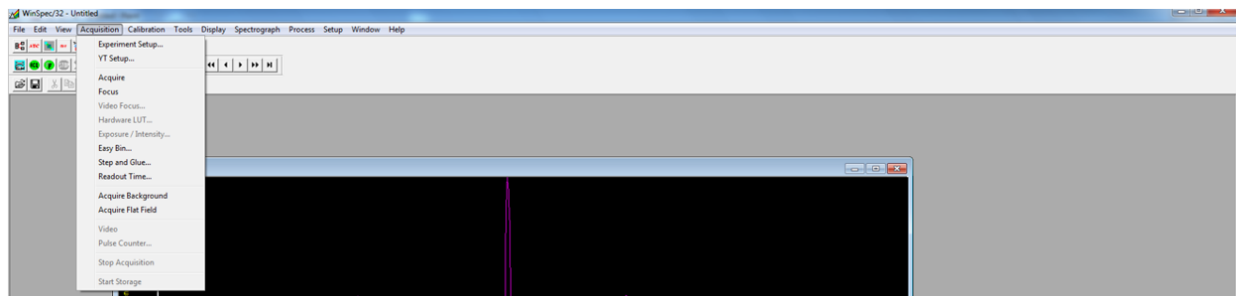


Figure 6.2.2. Screenshot of how to access “Experimental Setup” window in Winspec.

In this window, setting such as exposure time, accumulations, number of spectra obtained and CCD region of interest can be changed. Other advance options such as cosmic ray removal can also be toggled for the advanced user. See figure 6.2.3.

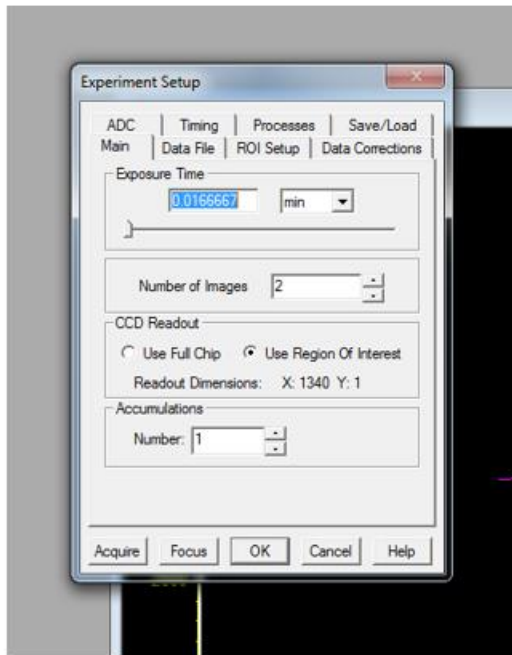


Figure 6.2.3. Screen image of “Experimental Setup” window in Winspec.

When you have the desired settings, quick commands can be accessed from the command bar as seen below. Here the Experimental Setup, Acquire and Focus can all be accessed with ease.

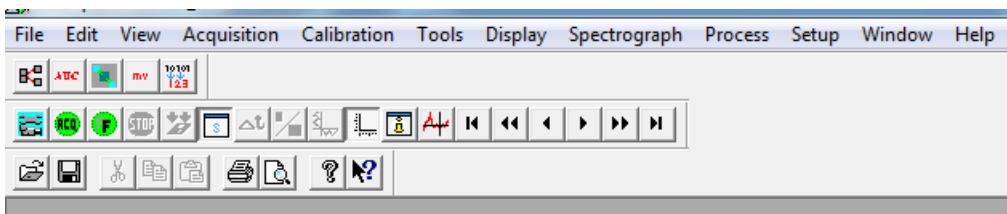


Figure 6.2.4. Zoomed image of command bar for quick access of data collection options in Winspec.

Quick Collection Guide

All material in this list can be found in the Users' Manual in more detail. This list should be used as a reference *only* after the user is comfortable collecting data on his/her own.

1. Before beginning any experiment be sure the laser sign is turned on and you have the appropriate safety equipment (goggles).
2. Decide what laser to use.
 - a. Have proper neutral density filters ready (if in UV-region you need to use reflective ones)
 - b. Decide on Ag vs Al mirrors
 - i. Ag (silver) is for visible and NIR lasers
 - ii. Al (aluminum) is for UV-region (405nm and 355nm lasers)
3. Select the desired mode in S&I Triple Raman System software. Example: "3-3-6 SUB 532nm"
 - a. Make sure to update laser wavelength value every time you change/update mode
 - b. Input "rel. Wavenumber" (to update range in which data will be collected – if numbers are red you will be collecting your Raleigh line - this is *not good* so you need to change the method or rel. wavenumber setting (click "off" box for it to calculate "range"))
 - c. IF LASER LIGHT HITS THE CCD DETECTOR IT WILL BE PERMANENTLY DAMAGED!
 - d. Under Hardware, check first mirror inside spectrograph to make sure it is aligned properly for RT(S2) vs. cryostat set-up(S1)
 - e. Click "Hardware Only" box next to green play button, then click play
4. Turn on laser (remember IPS lasers have no shutters - so make sure to have proper eye protection)
5. Place/align neutral density filters and beamstop in front of laser for all alignment work
6. Place translation stage in front of laser such that the beam passes through Iris-1 and reflects off mirror-1. (remember to select Ag vs. Al track)
7. Open iris-1 and adjust stage position (not mirror) so beam passes through the center of iris-2.
8. Shutter the laser. Then, bolt the translation stage to the table.
9. With laser still off/shuttered, select the appropriate beam track (Ag, Al, single spectrograph).
10. Check flip mirrors are allowing laser light to get to desired set-up (RT vs. cryostat).
11. Place solvent cuvette (for example: cyclohexane) 3.6 cm behind aspheric lens (for RT set-up). See section 4.4.
12. Open laser shutter and check the beam path to the prism. *****Use of a neutral density (ND) filter is recommended for eye safety. ONLY two mirrors are used for alignment to prism** Do not change position/move other mirrors/equipment (if you don't know which two, you shouldn't be working alone)
13. Check the general prism alignment with a card, you can adjust with mirror-2.
14. Remove slit covers (make sure method has been set properly beforehand or you will burn pixels off our CCD!)
15. When ready to optimize the prism alignment, turn off lights. Remove neutral density filters (remember eye protection) and Collect data in "Focus" mode -- using WinSpec with 100msec-1sec exposure time. While running in focus, adjust the prism tilt and rotation to maximize signal.
16. Once optimization is complete, replace solvent cuvette with sample cuvette and collect data.
 - a. Additional focus alignment could be necessary with sample depending on what it is

*****WHEN DONE: (1) TURN OFF LASER SIGN (2) TURN OFF LASERS, AND FANS IF APPROPRIATE
(3) PUT ON SLIT COVERS*****