Raman One by Wilson Analytical

Raman Spectroscopy Instrument

User's Manual

May 2022

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Contents

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Identifying Parts

Raman One

Figure 1. Raman One parts identification. Sampling accessories are sold separately.

Accessories

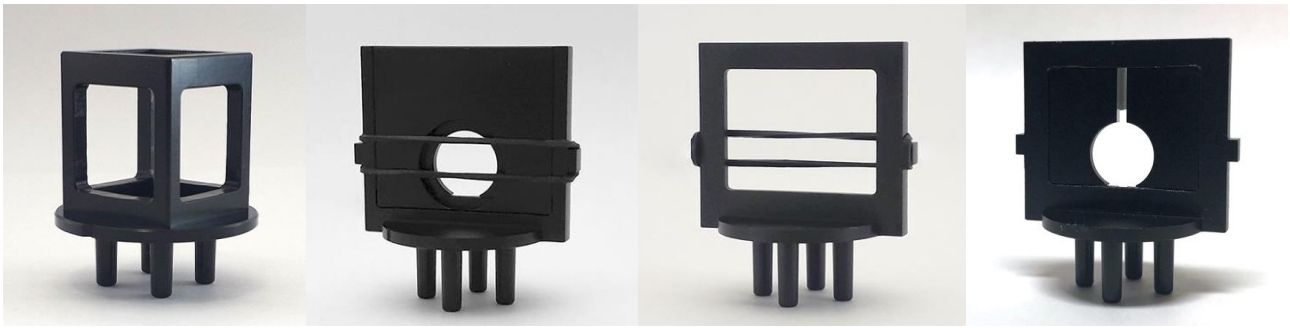

Figure 2. The four sample holders: cuvette holder (left), pellet holder (left centre), microscope slide holder (right centre) and capillary tube holder (right). The pellet holder and capillary tube holder can convert to the microscope slide holder. The four pegs on the bottom of the holders fit into the sample holder rotation stage in the sample chamber.

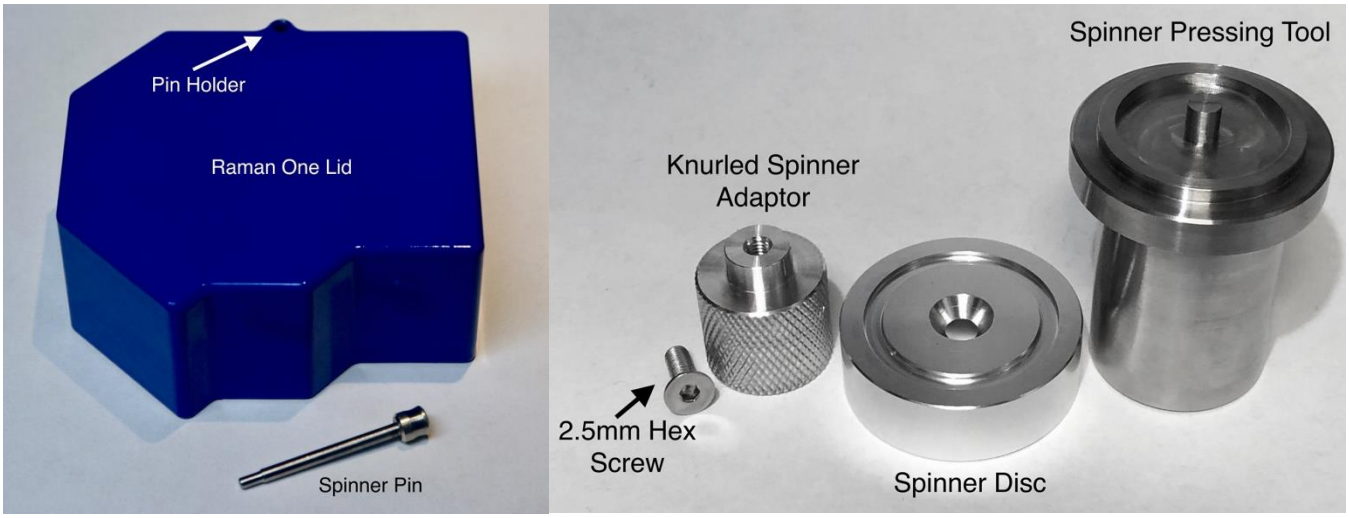

Figure 3. Left: the Raman One stainless steel spinner pin, lid, and pin holder. Right: the 2.5 mm hex screw (which holds the spinner adaptor to the spinner disc), the knurled spinner adaptor, the spinner disc, and the spinner pressing tool.

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Introduction to the Raman One

The Raman One is a uniquely designed Raman spectrometer that provides laboratory-quality analytical data. The system is built with the rugged high-quality construction you expect from Wilson Analytical and features a variety of sampling accessories that makes using it super easy. Choose your sample holder based on your sample type: liquids, pellets, powders, capillary tubes, or solid sample spinner.

Multiple unique features make the Raman One a valued addition to any lab or process-control facility:

- Unit externals are made entirely of anodized or powder-coated aluminum to resist spills and damage
- Class 1 laser enclosure is designed for safety, with a double-magnetic interlocked lid that turns the laser off when opened
- Two main sampling techniques are available within the sample chamber:
    - The Sample Spinner allows the user to run solid samples without fear of damaging coloured materials when using high laser power to obtain good quality spectra.
    - The Rotating Stage allows the user to run pressed pellets, microscope slides, capillary tubes and liquids at adjustable angles.
- Safe viewing of the samples and aligning of laser beam are made easy *with the laser on* via a microscope imbedded into the design of the system (60x magnification). The built-in 2 MP microscope camera can also record video, take photos, or measure the samples being tested.
- An integrated neon lamp allows the user to perform a spectrometer wavelength calibration at any time.
- LED indicators ensure user safety and provide status information on the system.
- Integrated IPS 785 nm laser (20–435 mW output power) is included. Other laser wavelengths are also available.
- Integrated Ibsen Freedom mini-Raman FCT-101 spectrometer 475 –1100 nm wavelength, 0.6 nm resolution, Hamamatsu S11639-01 detector with an integrated cylindrical collection lens for enhanced sensitivity is included. Other spectrometers are also available.
- IPS 785 nm laser excitation together with Ibsen Freedom spectrometer detection and Iridian optical filtration give a measurement range of 100 to 3650 cm^{-1} with good spectral resolution and excellent sensitivity.

The Raman One can be controlled using *SpectraGryph* software or via other software programs associated with the spectrometer of your choice. *Digital Viewer* and *Meazure It* software packages are supplied to run the microscope.

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Laser Operation Safety Precautions

The Raman One uses an invisible, high-powered laser which could cause permanent damage to the retina of the eye if laser light were to escape from the system. To ensure safe operation and prevent operator harm, the Raman One is equipped with a lid interlock system that does not allow the laser to operate unless the lid is correctly in place. An Interlock LED indicator shows the state of the interlock system during operation.

When using the Raman One, suggested mandatory added laser safety precautions are the following:

- **the laser should never be switched on without the lid properly in place, and**
- **the laser should always be switched off before removing the lid.**

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Quick Start

Running a Sample

Figure 4. Image of the back of the Raman One showing (from left to right): the Laser Caution label, the Wilson Analytical tag, the USB data port, the 5 A Fuse, and the 12 V power port.

1. Using the provided power supply system, connect the round 12 V DC power plug to the back of the Raman One (Figure 4), and the mains power cord from the power supply brick to any convenient power socket.

2. Connect the computer to the USB port on the rear of the system (see Figure 4) using the supplied USB cable, and turn on the computer.

3. Power up the Raman One by turning on the System Power switch at the top left of the system (Figure 1). A green light on this switch turns on when the system powers up, and the display screen to the right of the System Power switch will also turn on.

4. Open the *Digital Viewer, Meazure it,* and *SpectraGryph* software (or the software associated with the alternative spectrometer you have chosen).

5. The interlock system helps to ensure the safe operation of the laser, and it has an Interlock LED to indicate what is happening with the system. The green Interlock LED turns on when the lid is on and correctly seated on the unit, and it turns off when the lid is removed or is not correctly positioned. If the interlock has been tripped (and the LED turns off), the laser and the sample spinner will not turn on again until the lid has been repositioned properly on the chamber.

6. If you are using the Raman One Sample Spinner, take off the lid from the sample chamber, connect the sample disc to the threaded shaft, and put the lid back on the system. When the lid is on properly, the green Interlock LED will turn on. Turn on the Sample Spinner power switch (Figure 1). The spinner percent speed will be displayed above the spinner power switch and can be controlled using the Speed dial to the right.

    - To simplify putting the disc on the threaded shaft of the spinner, a pin (Figure 3) is supplied with the system. The pin can be inserted into the edge of the sample chamber to lock the shaft in place (further details are provided in “Using the Sample Spinner” on p. 17 of this manual). Remember to remove the pin before putting the lid back on and to put the pin in the holder provided in the lid (Figure 3).

7. If you are using the Sample Holder Rotating Stage, take off the lid to the sample chamber, put your sample holder onto the rotating stage, and then put the lid back onto the system. When the lid is on properly, the green Interlock LED will turn on.

8. Once your sample is in the sample chamber and the lid is on properly, you can turn on the Microscope Light switch (Figure 1) to illuminate your sample.

    - Remember that the microscope light can interfere with your Raman spectra. To remove this interference, turn off the microscope light while you are collecting data. If this is not desired, take a background measurement with the light on to enable you to later reduce the effect on the Raman data obtained.

9. To turn on the laser beam, flip the Laser Enable switch located above the indicator LEDs (Figure 1). When the laser is on, the red Laser Enable LED will turn on, the Interlock LED will be green, the Laser On LED will be blue, and the laser output screen will display the drive setting, laser power, and status of the instrument (Figure 5). (See the section on “Power Switches and LED Status Indicators” on p. 12 of this manual for more information.)

Figure 5. Image of the Laser Display Screen which indicates 100% Drive setting and 421 mW Laser power. The Interlock is satisfied, and the laser is on as indicated by the indicator LEDs.

10. To change the laser intensity (drive) setting, simply adjust the Laser Intensity dial under the laser display screen. You can also fine tune or lock the percent laser intensity by pushing down on the dial. The settings will be displayed in the top right-hand corner of the screen as Coarse or Fine along with an image of a lock.

    - The Coarse function allows for rapid movement of the drive setting, and the Fine function allows for smaller changes to the drive setting. The lock will ensure the drive setting does not change while you are testing.

    - Please note: at the 100% drive setting, it is normal for the indicated laser power to appear to drift by up to 10%. However, the actual value of the laser power drift is more like 5%. This disparity in the indication arises from temperature effects in the coupling of the laser to the internal photodiode.

11. Just below and slightly to the right of the Laser Intensity dial is a fine adjustment dial. Different sample geometries have the different optimal focal points for maximum signal sensitivity. You can use the fine adjustment dial to optimize the position of the incident laser beam on the sample to ensure the most efficient collection of the Raman scatter into the spectrometer.

    - For your convenience, the fine adjustment dial displays a number that shows the current focal position when testing, which allows you to return to known focal points easily. This capability is useful when optimizing the position and focus of a sample. To begin, simply note the initial fine adjustment dial position and the intensity of the Raman peaks obtained with this setting. The signals at this focal point can now be compared with signals at other focal points without fear of not being able to return to the original setting.

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Raman One and SpectraGryph Software

1. Ensure that the Raman One is powered up and properly connected to the computer before opening SpectraGryph. If you open the software before powering up the system, or if you have to turn the system power off, the software will then be unable to connect to the Raman One. To fix the problem, close the software and restart it after the system has fully powered up.

2. Once the Raman One is powered up and the SpectraGryph software is open, click on the *Acquire* tab at the top right of the program. When you are in the *Acquire* tab, you will be able to click on *Device Type*. Select the spectrometer *Ibsen Photonics* from the drop-down menu. Click on *Connect,* and you are now ready to run a sample.

3. To run a sample, click on the "Play" button labelled *Acquire,* which will generate a spectrum. From here, many tools are available on SpectraGryph to process your spectrum. You can save your process settings (e.g., Raman Shift, Advanced Baseline Correction, etc.) by clicking on the *Settings* buttons located under the *Connect* button. For future tests, you can then just upload your saved settings.

4. Setup tip: when trying to determine the best setting for the fine adjustment dial on the Raman One, switch to *Additive* on SpectraGryph. This function is located under the *Acquire* button; you can click on the drop-down menu to select modes such as single shot, continuous, additive, burst, and loop modes. The *Additive* mode allows you to take multiple measurements and compare them with each other right away; this is particularly beneficial when trying to determine the best fine adjustment for your sample.

5. For more information on how to use SpectraGryph, visit the website at https://www.effemm2.de/spectragryph/about_help.html .

Power Switches and LED Status Indicators

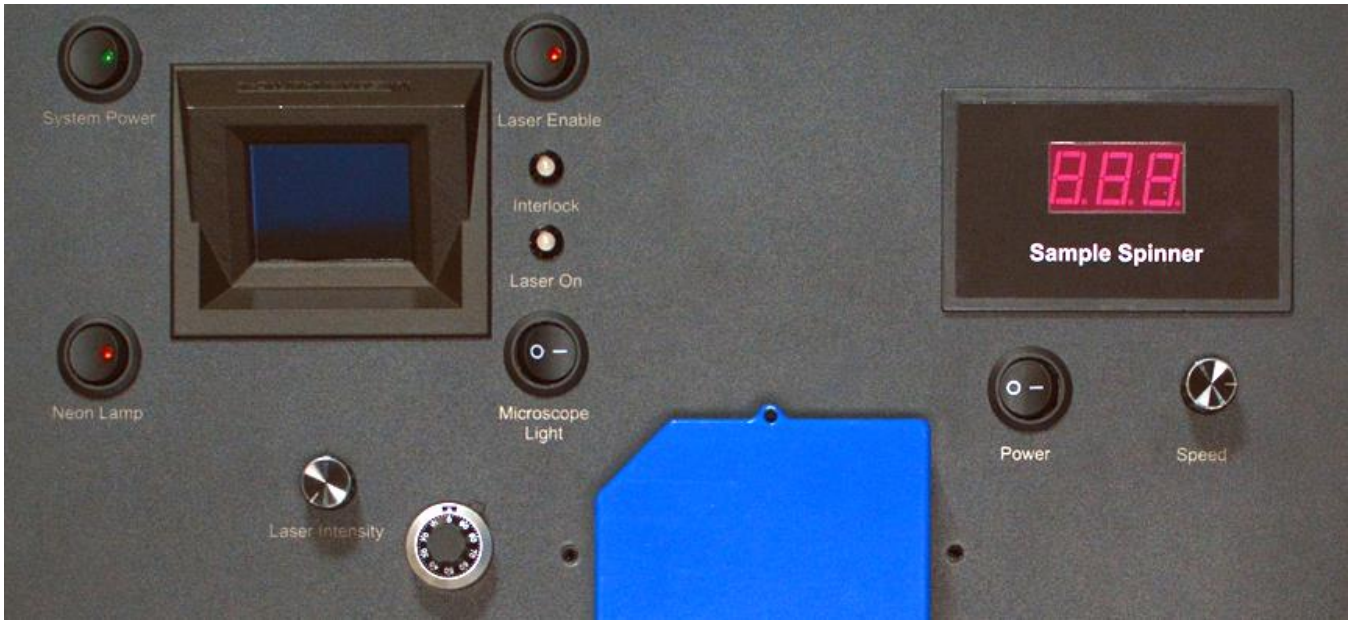

Figure 6. Image of the top of the Raman One, showing the power switches, the LED indicators, and the sample lid on the top of the unit (which covers the sample spinner and the sample holder rotating stage).

Please refer to the following tables for more information on the power switches and the LED indicators.

Table 1. Switch Functions and Conditions.

Switch Name and Function	Power-On LED Colour	Conditions
System Power: Turns the power to the System on/off.	Green	● 12 V DC power supply is plugged into the system
Neon Lamp: Turns the neon lamp on/off.	Red	● 12 V DC power supply is plugged into the back of the system ● the System Power switch is on ● the lid is on correctly (Interlock LED shows green)
Laser Enable: Turns the laser beam on/off.	Red	● 12 V DC power supply is plugged into the system ● the System Power switch is on ● the lid is on correctly (Interlock LED shows green)
Microscope Light: Turns the microscope light on/off.	None	● 12 V DC power supply is plugged into the system ● the USB cable is plugged into the system ● the System Power switch is on
Sample Spinner Power: Turns the sample spinner on/off.	None	● 12 V DC power supply is plugged into the system ● the System Power switch is on ● the lid is on correctly (Interlock LED shows green)

Table 2. LED Indicators and Conditions.

LED Indicator Name	LED State	System Status	Conditions
Interlock	Green	Laser and spinner operation is allowed	● 12 V DC power supply is plugged into the system ● the System Power switch is on ● the lid is on correctly (Interlock LED shows green)
	Unlit	Laser and spinner operation is not allowed	● Lid is off or not positioned correctly (Interlock LED is unlit)
Laser On	Blue	Laser is on	● 12 V DC power supply is plugged into the system ● the System Power switch is on ● the lid is on correctly (Interlock LED shows green) ● the Laser Enable switch is on
	Unlit	Laser is off	● the Laser Enable switch is off, or ● the laser has been disabled by a tripped interlock (Interlock LED is unlit)

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Raman One Sample Preparation

When preparing a sample, you must first decide how you wish to test your sample.

Using the Sample Spinner in the sample chamber allows you to obtain a more homogeneous reading on your solid sample while also protecting the sample. The focal point of the laser does not continuously rest on one spot of the sample during analysis, so there is less likelihood of burning or melting the sample (especially for coloured materials).

Using the sample mounts in the sampling chamber allow you to run a variety of sample types: surface samples, pressed powder solid pellets, pastes, surface-coated discs, microscope slides, capillary tubes, and liquids.

Pressed Powders in the Sample Spinner

1. To press a powder into the sample spinner disc, first ensure that the powder is finely ground. Then detach the disc from the knurled spinner adapter using the supplied 2.5 mm Allen key (see Figure 7 below).

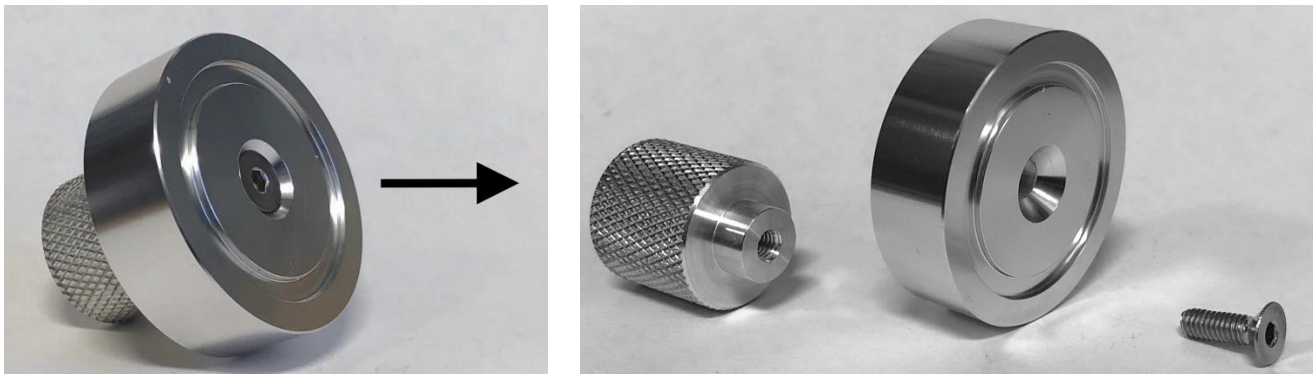


Figure 7. Image of dismantling the disc from the knurled spinner adaptor.

2. Fill the moat (the groove) around the sample spinner disc with the powder and tap it down until it appears level with the rest of the disc. At this point, we recommend using the sample spinner pressing tool to press the powder down into the moat by hand (Figure 8). Repeat this process as needed, filling the moat up and pressing the powder down until the moat is at least 90% filled. Finally, fill the moat one more time until the powder is level with (or slightly higher than) the rest of the disc. Then secure the powder in place using the Raman spinner pressing tool with sufficient force in a mechanical press to secure the powder in place so that it does not come out of the moat when you are spinning the sample.

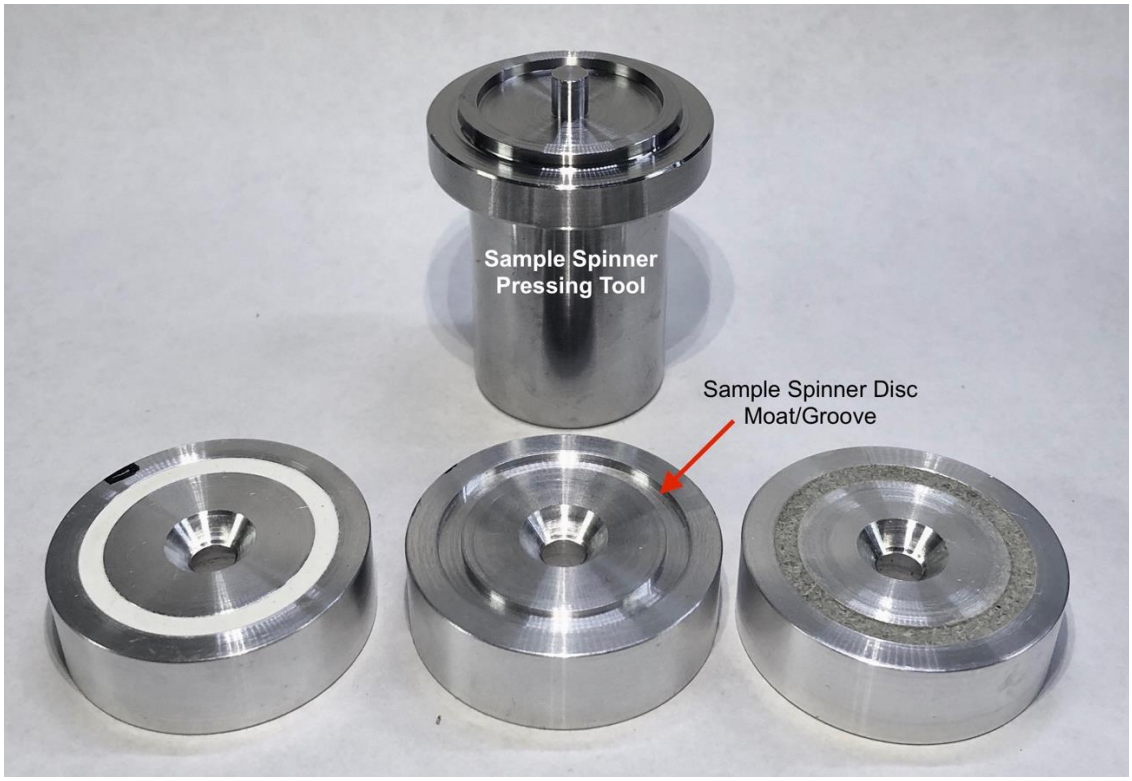


Figure 8. Three Sample Spinner Discs with the Sample Spinner Pressing Tool. The discs on the left and right contain sample material, whereas the centre disc shows the empty Sample Spinner Disc moat (or groove).

3. The best results are achieved with the spinner when the level of the sample is even with the rest of the disc. However, this method can require a significant amount of sample. If the amount of sample is limited, an alternate method can be used:

    - Perform step 2 above using infrared-grade potassium bromide (KBr) powder to fill the moat, and then sprinkle a small amount of sample onto the KBr already secured in place in the moat in step 2.

    - Distribute the sample evenly by gently rotating the spinner pressing tool on the sample, and then use the mechanical press with sufficient force to secure the sample in place so that no sample material detaches from the KBr support bed when the sample is spinning in the Raman One. (Infrared-grade KBr is used as a base here, because it has no significant vibrational bands above 220 cm^{-1}.)

Pressed Powder Solid Pellets

1. To press a pellet, first ensure the sample is ground into a fine powder. Then use a 12–13 mm die set and fill the die with the finely ground powder. We recommend rotating the plunger on top of the sample and then pressing the powder down by hand to help gauge the thickness of the pellet before using the mechanical press to form the final pellet. A final thickness of 3–7 mm is recommended. We recommend using the press to push the pellet out of the die, because this technique ensures that the pressure is distributed evenly and the pellet is not destroyed.

    - If you do not have enough sample for a 3 mm thick pellet, we recommend using infrared-grade potassium bromide (KBr) powder as the base of your pellet to provide added thickness. The KBr can be pre-pressed and left in the die before adding in the sample on top. The pellet can then be pressed for a final time after the sample is added on top. (Infrared-grade KBr is used as a base, because it has no significant vibrational bands above 220 cm^{-1}.) Care should be taken when obtaining the Raman spectrum to ensure that the top (sample) side of the pellet is measured rather than the bottom (KBr) side.

    - We recommend that you consult the instructions on your die set for more information.

13 mm Discs or Microscope Slides

1. If you wish to mount your sample on a 13 mm round disc or on a microscope slide, ensure that your sample will not slide off when it is standing upright in the instrument. If using a slide, the sample must also be positioned to one end of the slide rather than in the middle, so that the laser beam focuses on the sample during measurement (see Figure 13).

Cuvettes or Melting Point Capillary Tubes

1. If you have a liquid you wish to sample, simply fill a cuvette or capillary tube with your sample. For cuvettes, ensure that your sample fills the cuvette halfway or more, whereas capillary tubes need only a few mm of sample. The same methods can be used for the analysis of fine powders. See Figure 14.

Running Samples

Using the Sample Spinner

1. Follow steps 1 through 4 on page 8 of this manual to get the system ready for analysis.

2. To run a solid sample on the sample spinner, remove the lid of the Raman One to access the sample chamber (see Figure 9). The green Interlock LED on the top of the system will turn off while the lid is off.

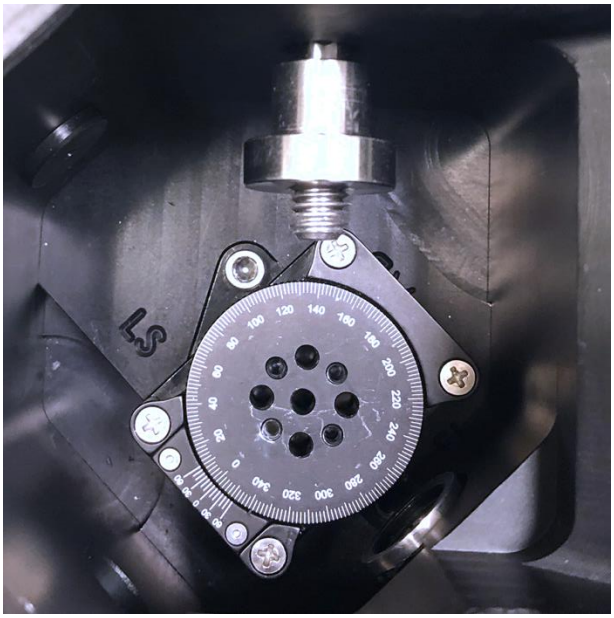


Figure 9. Image of the Raman One sample chamber.

3. Take the assembled sample spinner disc (the left side of Figure 7) and screw it onto the threaded shaft coming down from the top of Figure 9. To keep the threaded shaft from rotating while attaching the disc, place the spinner pin into the shaft through the sampling chamber as shown in Figure 10a. Once the disc has been successfully mounted (Figure 10b), remove the pin. For convenience, the lid contains a Pin Holder (storage hole) to hold the spinner pin when it is not in use (Figure 3).

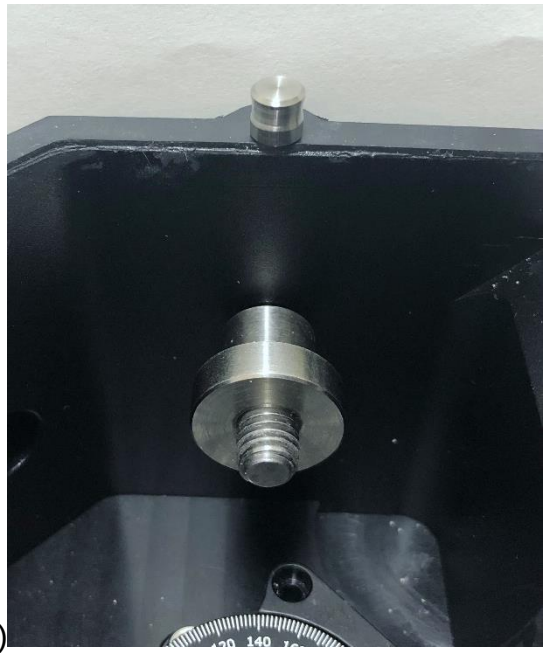
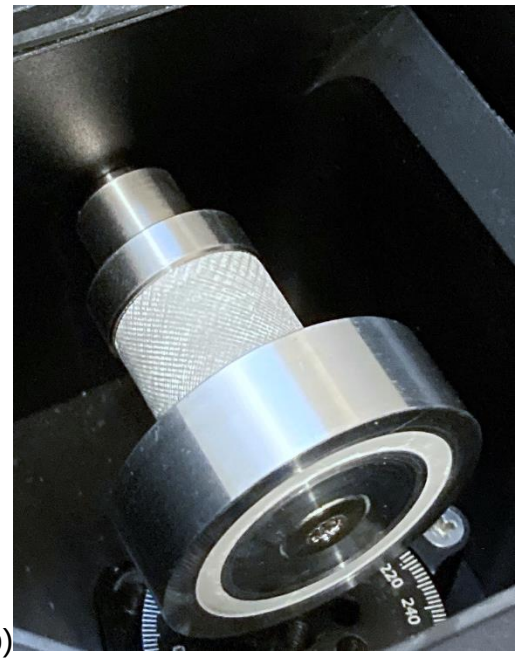
a)  b) 

Figure 10. a) Image of the Raman One spinner pin placed into the spinner shaft through the sampling chamber to secure the shaft in place while mounting the spinner disc. b) Image of the Raman One spinner properly mounted with a sample in the disc ready to run.

4. Place the lid back onto the Raman One to cover the sample chamber. If the lid is on correctly, the Interlock LED will turn green. If you have forgotten to remove the spinner pin, the cover cannot be properly replaced and the Interlock LED will not light.

5. Once your sample is in the sample chamber and the lid is on, you can turn on the Microscope Light switch to see your sample.

    - Remember that the microscope light can interfere with your sample results. To remove the light interference, turn off the microscope light while you are collecting data or take a background measurement with the light on (to subtract later).

6. Turn the Sample Spinner power switch on. The sample spinner screen displays the percent speed of the spinner. To change this speed, turn the dial located to the right of the sample spinner power switch.

7. Ensure the Interlock LED is green, indicating you are ready to run your sample. If the indicator LED is off, check that the lid is on securely.

8. Turn on the laser beam using the Laser Enable switch located above the indicator LEDs. When the laser is on, the Laser Enable switch will illuminate red and the Laser On LED will be blue. In addition, the laser output screen will display the drive setting, the laser power, and the status of the instrument (Figure 5).

9. To change the laser intensity, simply turn the dial under the laser display screen. You can also fine tune or lock the percent laser intensity by pushing down on the dial. The settings will be displayed in the top right-hand corner of the screen as Coarse or Fine along with an image of a lock.

    - The Coarse function allows for rapid movement of the drive setting, and the Fine function allows for smaller changes to the drive setting. The lock will ensure that the drive setting does not change while you are testing.

10. Just below and slightly to the right of the Laser Intensity dial is a fine adjustment dial. Different sample geometries have the different optimal focal points for maximum signal sensitivity. You can use the fine adjustment dial to optimize the position of the incident laser beam on the sample to ensure the most efficient collection of the Raman scatter into the spectrometer.

    - The fine adjustment dial displays a number that shows the current focal position when testing, which allows an easy return to known focal points. This is useful when optimizing the position and focus of a sample. To begin, simply note the initial fine adjustment dial position and the intensity of the Raman peaks obtained with this setting. These signals can now be compared with signals at other focal points without fear of not being able to return to the original setting.

Using the Rotating Stage

1. Follow steps 1 through 4 on page 8 of this manual to get the system ready for analysis.

2. To run a sample on the rotating stage, remove the lid of the Raman One to access the sample chamber (Figure 9). The green Interlock LED on the top of the system will turn off while the lid is off.

3. If a sample mount is already in the sample holder rotating stage, first remove the mount. The sample mount fits into the stage with the four pegs on the bottom of the mount (Figure 11).

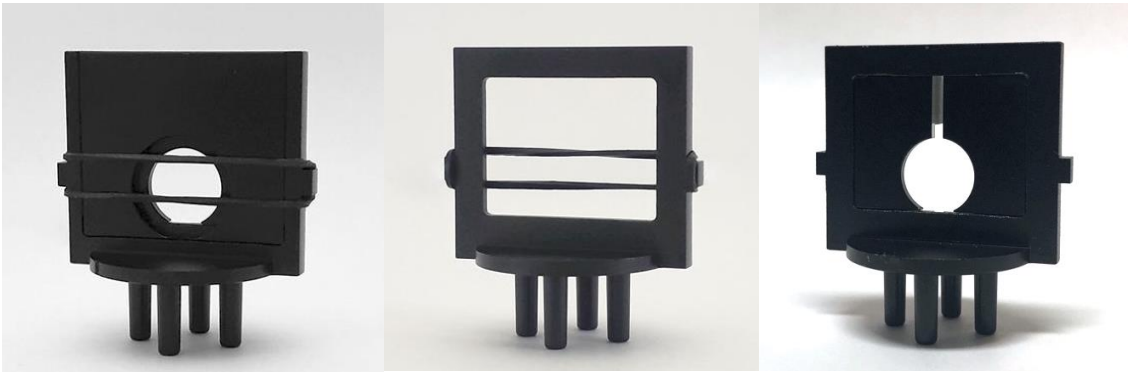


Figure 11. Image of the four pegs on the bottom of the pellet holder mount (left), which easily converts to a microscope slide holder mount (centre) or a capillary tube mount (right).

4. The sample mount is designed to accommodate surface samples, pressed pellets, pastes, surface-coated discs, capillary tubes, and cuvettes.

    - To test sticky powders and pastes, smear the sample onto a blank 13 mm pellet, a disc, or a 1-inch x 3-inch rectangular microscope slide. The pellet holder can easily convert to a microscope slide holder by simply removing the 1-inch insert (Figure 11). Once your sample is ready, place the pellet, disc, or slide into the back of the recess of the sample mount. Use an elastic band around the back of the sample to hold the sample securely in place (Figures 12 and 13).

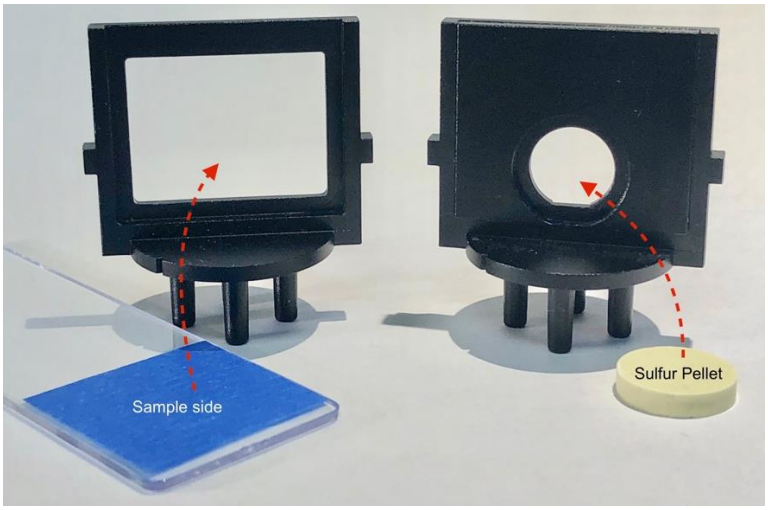


Figure 12. When putting your sample into the microscope slide holder and pellet holder, ensure that the front of the sample goes into the recess of the sample holder. In this image, we see the back sides of the sample holders before the samples have been mounted. Note, the sample is placed on one end of the slide represented by the blue tape in the photograph.

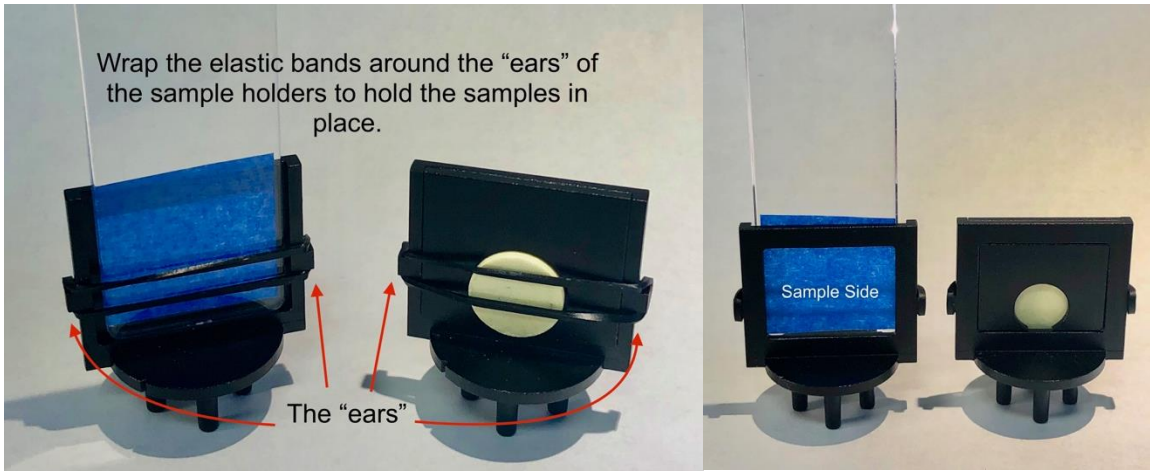
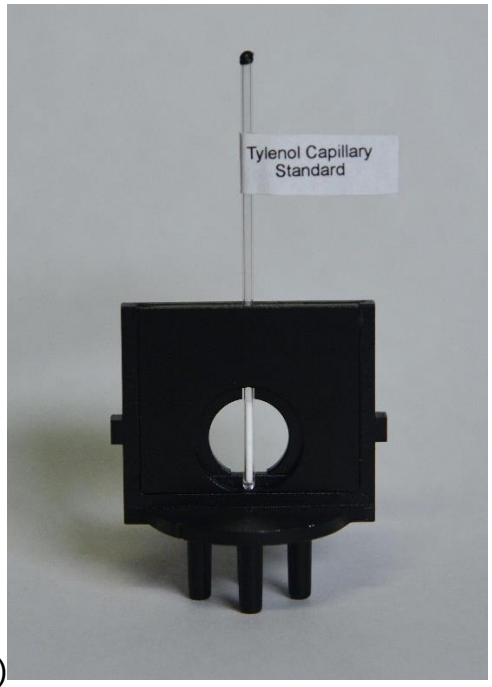


Figure 13. Left: sample holders with elastic bands wrapped around the backs of the samples to keep them in place. Right: the front view of the samples in the sample holders. Note, the sample is placed on one end of the slide represented by the blue tape in the photograph.

- To run a sample in a capillary tube, simply fill your capillary tube to around 5 mm or more and place it into the capillary tube holder mount. Note that not all pellet holder mounts can be used for capillary tubes, because the mount must have a specific slot for the capillary tube (Figures 11 and 14).

- The centred cuvette holder can be used for transparent sample solution analysis (Figure 14). The solution is held in a regular rectangular 10 mm pathlength fluorescence cuvette (4 sides clear), and the cuvette is held by the cuvette holder.

a) 

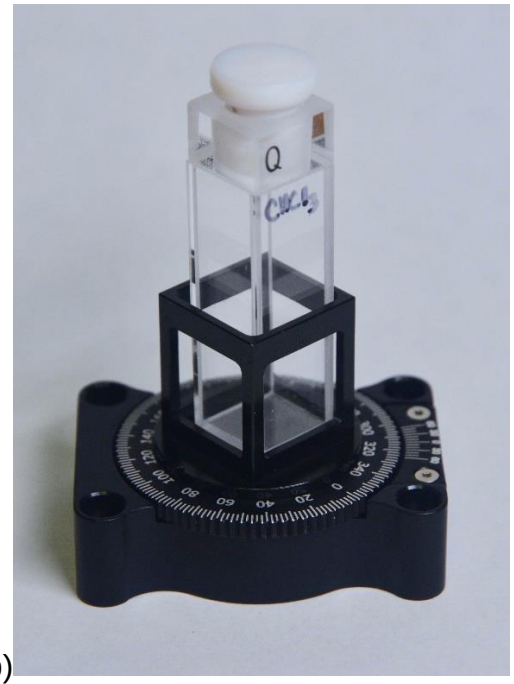
b) 

Figure 14. a) Tylenol sample in the capillary tube holder. b) Cuvette in liquid cuvette holder.

5. Next, place the sample mount on to the rotation stage.

    - Rotate the stage so that the zero degrees mark on the circular rotating part of the stage aligns with the zero degrees mark on the top non-rotating part of the stage (Figure 15). Insert the four pegs of the sample holder into the holes in the rotation stage such that the flat sample holder points to 45 degrees on one side of the stage and to 225 degrees on the other side. Make sure that the holder is inserted all the way into the stage holes so that the circular holder base sits flat on the rotation stage and that the back of the sample is pointed towards the sample spinner shaft (Figure 15). The sample should now be set at a 45° angle to both the laser and the spectrometer for a 90° geometry (between laser and spectrometer) measurement. Note that a 90° geometry measurement means that the sample is facing the microscope at right angles.

    - The rotation stage can be set to any angle between the sample and the light source.

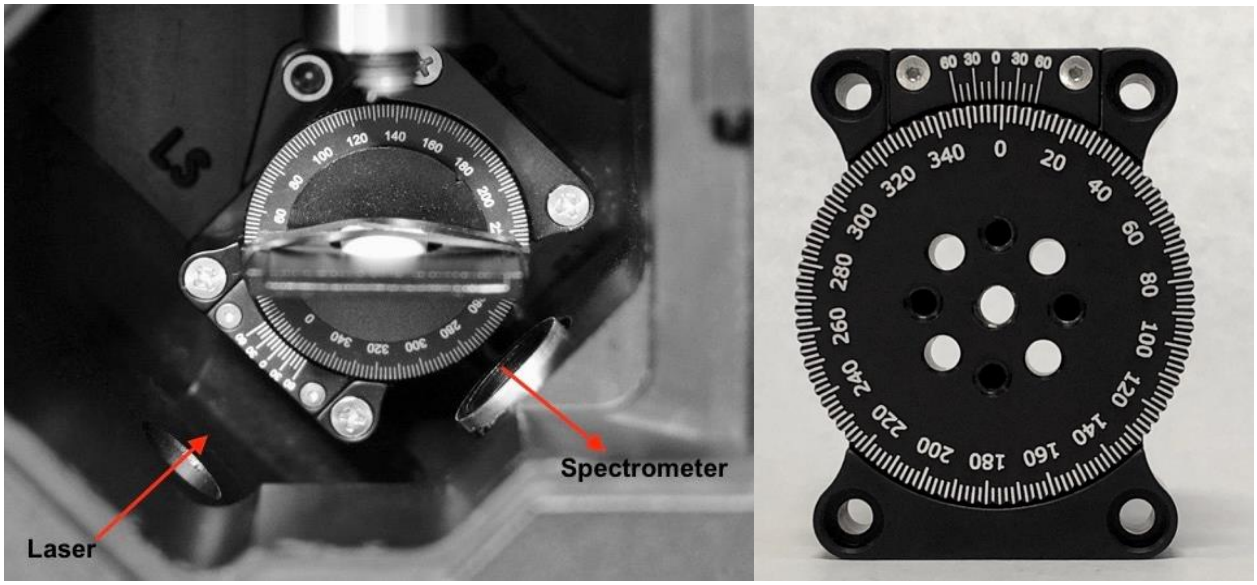


Figure 15. Lining up the rotation stage to 0° so that the sample mount sits at a 45° angle to both the light source and the spectrometer (left image). Note that the four pegs on the bottom of the sample holders will fit only into the four open holes on the rotating stage (right image).

6. Place the lid back onto the Raman One to cover the sample chamber. If the Interlock LED is green, this indicates that you are ready to run your sample. If the indicator LED is off, reposition the lid until the indicator LED shows green, thereby ensuring that the lid is securely in place.

7. Once your sample is in the sample chamber and the lid is on, you can turn on the Microscope Light switch to see your sample.

    - Remember that the microscope light can interfere with your sample results. To remove the light interference, turn off the microscope light while you are collecting data or take a background measurement with the light on.

8. To turn on the laser beam with the Laser Enable switch located above the indicator LEDs. When the laser is on, the Laser Enable switch will illuminate red and the Laser On LED will be blue. In addition, the laser output screen will display the drive setting, the laser power, and the status of the instrument (Figure 5).

9. To change the laser intensity, simply turn the dial under the laser display screen. You can also fine tune or lock the percent laser intensity by pushing down on the dial. The settings will be displayed in the top right-hand corner of the screen as Coarse or Fine, along with an image of a lock.

    - The Coarse function allows for rapid movement of the drive setting, and the Fine function allows for smaller changes to the drive setting. The lock ensures that the drive setting does not change while you are testing.

10. Just below and slightly to the right of the Laser Intensity dial is a fine adjustment dial. Different sample geometries have the different optimal focal points for maximum signal sensitivity. Use the fine adjustment dial to optimize the position of the incident laser beam on the sample to ensure the most efficient collection of the Raman scatter into the spectrometer.

    - For your convenience, the fine adjustment dial displays a number that shows the current focal position when testing, which allows an easy return to known focal points. This is useful when optimizing the position and focus of a sample. Simply note the initial fine adjustment dial position and the intensity of the Raman peaks obtained with this setting. These signals can now be compared with signals at other focal points without fear of not being able to return to the original setting.

    - When using the sample holder rotating stage, you might also have to change the angle at which your sample rests on the rotating stage to receive the best results. An angle slightly offset from 45 degrees is often useful if the sample is reflecting too much laser scatter into the spectrometer, leading to elevated baselines in the Raman spectra. Changing the sample angle by a few degrees in one direction or the other can often improve the baseline without greatly affecting the Raman signal intensities.

Standards and Calibration

Running the Neon Standard Test Lamp

A neon standard test lamp is embedded within the Raman One. The neon lamp allows the user to ensure the spectrometer meets quality control standards. Please note that the Neon Lamp switch cannot be turned on when the Raman lid is off and the interlock has been tripped. Though the neon light is not harmful to the eye, it is impossible to run the neon standard successfully with the lid off. This interlock ensures that you do not have interfering emission spectra from the lights in the room, which can be detected by the spectrometer and cause problems with your quality control check.

The test lamp is intended to be used as a quick check to ensure that the system is working correctly and to verify that the wavelength calibration of the spectrometer is accurate.

1. Follow steps 1 through 4 on page 8 of this manual to get the system ready for analysis.

2. Next, make sure the lid is on and that the Interlock LED is green. Finally, turn on the Neon Lamp switch (red) and capture the neon light's emission spectrum to perform wavelength calibration. (Figure 16 provides a reference spectrum.)

3. If you do not see a spectrum, ensure you do not have a sample in the sample chamber that is blocking the neon light from reaching the spectrometer.

4. For the NIST listing of the strong emission lines of neon, see the following link: https://physics.nist.gov/PhysRefData/Handbook/Tables/neontable2_a.htm

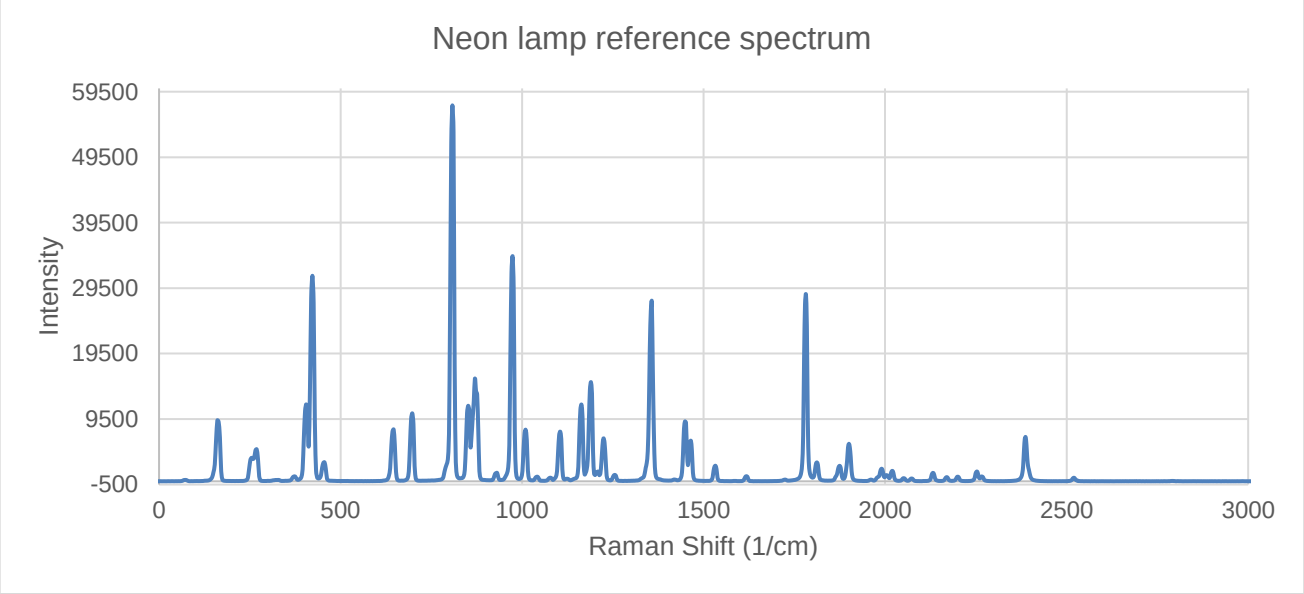


Figure 16. Neon light reference spectrum plotted in Raman wavenumbers (not wavelengths).

Running the Reference Standards

1. Reference standards can be used to ensure that the Raman One is running properly and that the fine adjustment dial setting has been optimized. Please refer to the section “Fine Adjustment Dial Calibration” if you believe your fine adjustment dial is off.

2. Two commonly used solid reference standards for Raman spectroscopy are sulfur and acetaminophen (Tylenol). Either can be used with the Raman One to test the pellet holder, the spinner, or the capillary tube holder.

3. Each sampling technique will require a different position of the Fine Adjustment Dial for the best results from user samples, so it is beneficial to pre-optimize the system using a reference standard that can provide high-intensity signals. Settings obtained using the standards provide good starting points for optimizing real samples.

4. The Tylenol Pellet Reference Standard was created for the pellet holder (Figure 17). The test sample is intended to be used as a quick check to ensure that the system is working correctly and is properly calibrated for that particular sample type. When the Raman One is running the Tylenol reference standard, the spectrum obtained should be similar to the one provided in Figure 18.

5. As an additional source of reference information, the McCreery research group at the University of Alberta has published a series of carefully verified Raman spectra of several reference standards, including Tylenol, sulfur, and several solvents (available at https://www.chem.ualberta.ca/~mccreery/ramanmaterials.html).

Figure 17. Tylenol Pellet Reference Standard for checking the Raman One and ensuring that the fine adjustment dial is centred.

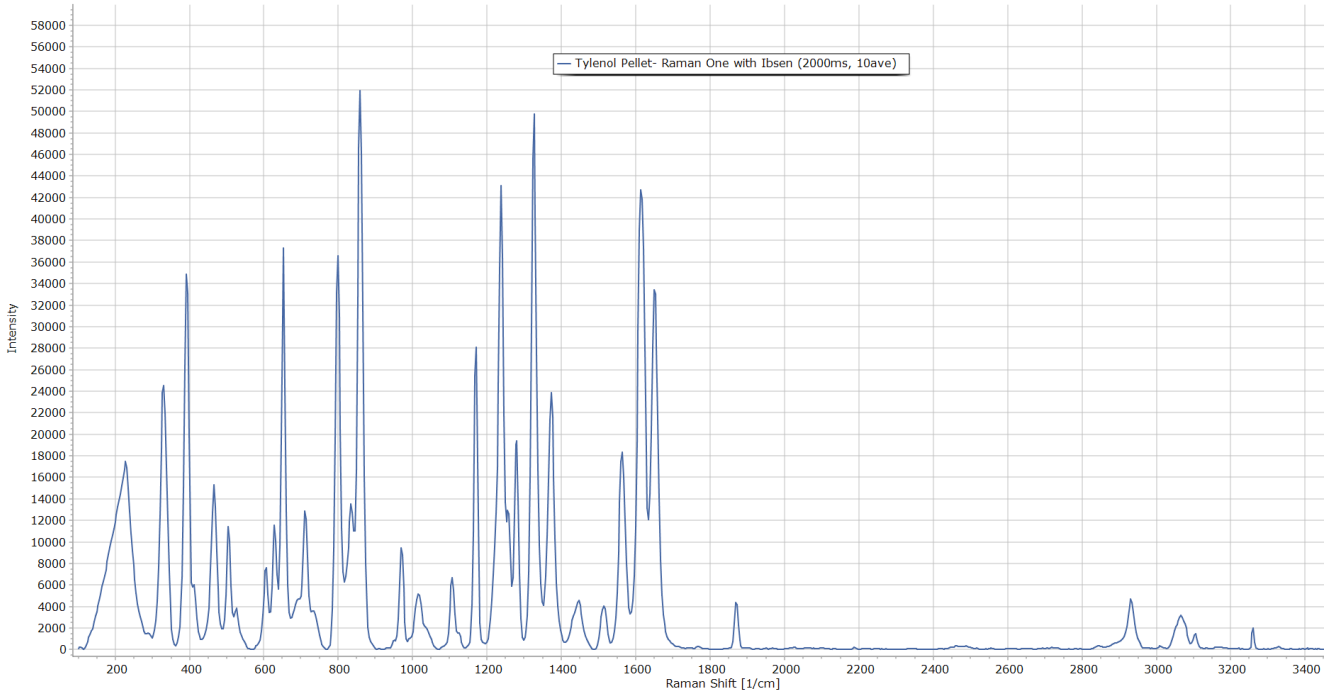


Figure 18. Reference spectrum for the Tylenol Pellet Reference Standard.

Fine Adjustment Dial Calibration

1. The fine adjustment dial allows the user to adjust the horizontal position of the laser spot on the sample. It is used to optimize the spectral results for different types of samples.

2. When running different samples, we recommend taking note of the fine adjustment dial reading to use for future testing with the same sampling device and for calibration purposes.

3. If you are running a pellet sample and the optimal position for the fine adjustment dial is not between the 7 and 9 positions (seen in Figure 19), we recommend recalibrating the fine adjustment dial.

4. Why is recalibration important?

    - The span-range on the fine adjustment dial goes from 0 to 15. To optimize the functionality of the fine adjustment dial, we recommend having a setting of close to 8.0 on the fine adjustment dial when at the optimal position for good spectral readings with the pellet standard sample. This will provide the maximum *adjustment range* for laser focusing on all Raman samples.

Figure 19. The fine adjustment dial in the 8.0 position.

5. To re-calibrate, first insert the pellet standard and then determine the optimal signal-to-noise ratio by comparing the intensity of the spectral peaks at different dial readings.

6. Once you have found the best intensity, loosen both set screws on the fine adjustment dial (Figure 20). Then remove the fine adjustment dial by lifting it straight up. After the fine adjustment dial has been detached from the Raman One, turn the dial to the 8.0 position.

Figure 20. The fine adjustment dial set screws.

7. Then reinstall the fine adjustment dial by lining up the small pin on the bottom of the fine adjustment dial to the small hole on the Raman One case (Figure 21).

Figure 21. Align the small pin on the bottom of the fine adjustment dial to the small hole on the Raman One case.

8. Finally, retighten both set screws to ensure the proper calibration on the fine adjustment dial is retained.

Troubleshooting

Interlock and System Operation

The light-tight seal on the Raman One sample chamber ensures that no harmful light escapes while the laser is on and that accurate measurements can be made free of external room light during testing. Therefore, the Raman One laser and the neon lamp will not turn on when the Interlock LED is off (indicating that the instrument is not light tight). To turn the Interlock LED on, try the following:

1. Ensure that the sampling lid is on correctly. If the lid for the sample chamber is already on, try removing the lid and reseating it again to get the Interlock LED to turn green. The lid must be positioned properly so that the interlock switch in the Raman One is satisfied before the Interlock LED will turn green.

If the system does not illuminate on the start-up, try the following:

1. Ensure that one end of the power brick mains cord is plugged into a power outlet and that the other end is securely pushed into the 12 V power brick.

2. Ensure that the 12 V power cord from the power brick is plugged into the back of the Raman One.

3. Next, try unplugging and replunging the 12 V cord into the Raman One, and then turn off and on the System Power switch.

4. Check to make sure that the 5A fuse in the fuse holder on the back of the unit is still good. (see Figure 4)

System Communication

1. If the Raman One does not connect to the computer software, ensure that the USB cable is connected to the back of the Raman One and to the computer. You must also remember to turn on the Raman One *before* opening the SpectraGryph software on the computer.

2. If the laser switch has been turned on but the display screen reads 0 mW; turn the laser off, then turn the system power switch off, and then restart the Raman One and computer software.

3. If the system loses power during operation, turn the main power off and then on again.

4. Remember that the laser display screen reports the status of the instrument at the bottom of the screen. If you are uncertain about what is going, on you can read the message at the bottom of the display screen to help you to troubleshoot. If troubles persist, please contact us at Wilson Analytical.

Laser Safety

1. Remember that when the Laser On LED is blue, the laser is operating and a high-power invisible laser beam is shining inside the sample chamber. DO NOT remove the sampling lid while the laser beam is on. If you wish to remove the lid, ***turn the laser off first.*** This action will be confirmed by the Laser On LED turning off and the laser display screen reading the laser power at zero.

If for any reason troubles persist, please contact us at Wilson Analytical:

Email: info@wilsonanalytical.com
Phone: (780) 702-0610
Toll free: 1-866-3wilson (394-5766)
Website: www.wilsonanalytical.com

Figure 22. The Wilson Analytical Raman One.

www.wilsonanalytical.com