

# Thermo-Scientific iS50 Raman-FTIR User's Guide

---

Version 1.0

Edited by Dr. Hartmut G. Hedderich

1/3/2024

---



The following guide describes the use of the Thermo-Scientific iS50 Raman-FTIR spectrometer. The guide is intended to assist instrument users after the initial training session.

## Thermo-Scientific iS50 Raman-FTIR – User's Guide, Ver.1.0

edited by Dr. Hartmut G. Hedderich

### 1. General Notice

Throughout this handout all **Raman-specific topics** are displayed in **blue**. General information and FTIR information are displayed in **black**.

### 1. Hardware

The iS50 Raman-FTIR is a compact bench unit. It consists of two units – **the Raman unit** with built-in diamond ATR FTIR (iS50 Raman, left side) and the transmission FTIR (iZ10, right side) unit.



The transmission FTIR unit has two detectors – a room temperature DTGS and a liquid nitrogen-cooled MCT/A. The ATR unit has a built-in DTGS detector. **The Raman attachment comes with an InGaAs detector.**

The system needs to be purged with nitrogen to suppress water and carbon dioxide bands. There is a valve marked FTIR on the manifold which will supply nitrogen gas from the liquid

nitrogen tank boil-off. This valve is always ON! There is a blue valve at the right side of the instrument besides the transmission unit:



OFF position

Turn it to the ON position to purge the FTIR. You want to run the purge for at least 30 minutes before starting your experiments. Do not forget to turn the valve OFF when you are done with your experiments! **The Raman setup does not need a purge!**

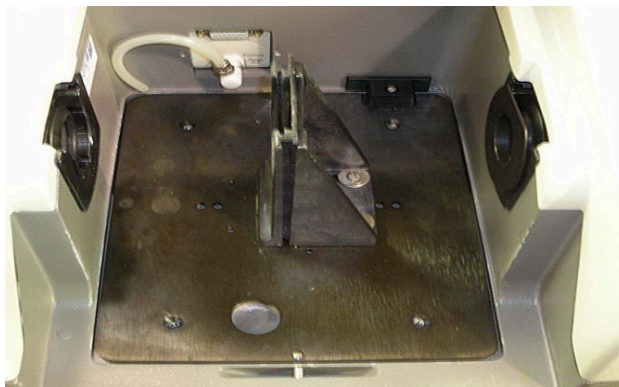
Hardware configuration and data collection is fully computer-controlled via OMNIC software (Version 9.13).

## 2. Samples

Thermo Nicolet FTIR spectrometers can measure gaseous, liquid and solid samples.

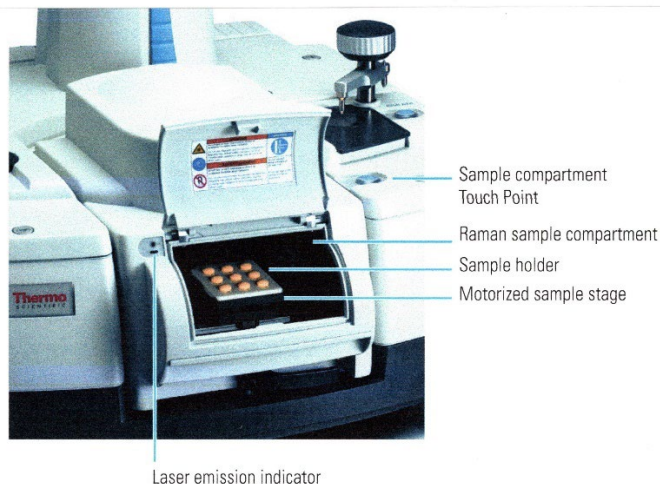
Solid and some liquid samples can be measured without further preparation by putting them directly on the ATR crystal. **Most liquids (corrosive, poisonous, BSL-II samples, most solvents) must be put in a liquid cell.** Ethanol, methanol, iso-propanol and hexane can be put directly on the ATR crystal. Please contact RIC staff if you don't know if your liquid sample should be put in a liquid cell.

The transmission sample chamber contains a standard holder (see picture). It fits standard IR pellet holders, thin film holders, salt plate holders and gas/liquid cells (user is responsible for supplying those).

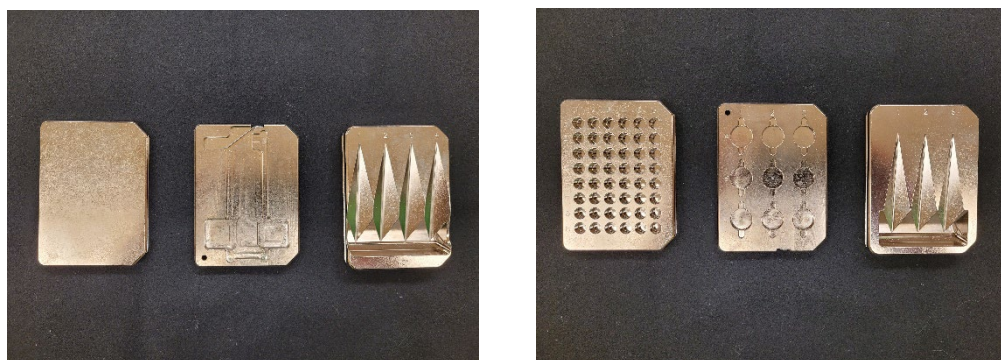


As a rule of thumb, I suggest purging for 1 minute every time you open the sample chamber to change samples. This will give you pretty equal conditions for every background and sample data collection. At the start of your experiment and when the chamber is open for longer times I suggest purging for several minutes.

The Raman attachment can be configured to measure solid and liquid samples. The microscope stage has an opening for a wide range of holders.



We provide holders for Raman tubes, microscope slides and cover slits, well plates (9 and 48 wells) and standard solid samples.



Front (left) and back (right) view of the Raman holders. From left to right the following sample holders are provided: standard solid, microscope slide and cover slits, 4 Raman tubes, 48 wells, 9 wells and 3 Raman tubes. **Please do NOT put samples directly in the well holders.** Either use an aluminum DSC pan or a piece of aluminum foil to cover the well and then put your sample on top. This will protect the wells from contamination.

**Important Notice:**

- Do NOT spill liquids directly onto the Raman module.
- Always place samples on Raman sample holder and then load the holder on the Raman sample stage. Do NOT put a sample directly on the stage!
- Before you install a sample holder on the sample stage, ensure that the height of the holder plus the sample (or the sample container) does not exceed 2.5cm (1.0"). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.

**3. OMNIC Software**

Start the OMNIC Software by double clicking on its icon from the desktop. **The program will start with the setup for the main chamber (Raman).** The setup can be switched to the ATR or the transmission box with either DTGS or MCT/A detector. Switching the setup can be done in 3 different ways:

- In the software click on the setup line and switch to the desired setup
- Push one of the 3 blue touch points on the instrument.
- Hit the quick setup button:



From left to right we have buttons for: iZ10/right AEM, diamond ATR, **main Raman** and touch button setup.

When the instrument switches setups you will see that one of the touch buttons will blink. This indicates that the instrument is changing beamsplitters and other optical components. **Please wait until the color of the touch button is solid again before using any software commands!**

The software interface is set up to make data collection and processing as easy as possible for the user. For that reason, the most common commands are put together on the tool bar in 5 groups:

- Experiment setup, collect background and sample,
- Save and print options,
- Data display, correction, manipulation, etc.,
- General commands,
- Libraries.



All commands that are displayed on the tool bar will be discussed in detail during the training session!

Start by pushing the *Experimental Setup* button to set up your experiment. There are a total of 7 tabs:

**Collect and Bench** tabs provide options for users to set up their experiments. They are described below in detail.

The **Quality** tab provides background checks for a wide range of variables and will give warnings or errors if the instrument performance is outside established boundaries.

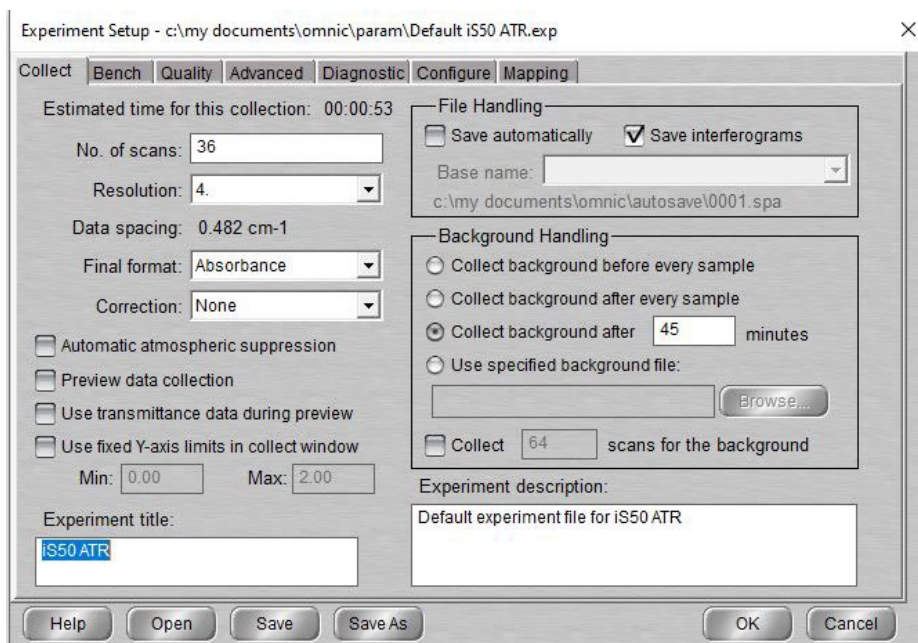
The **Advanced** tab shows the parameters used for the Fourier transformation.

The **Diagnostics** tab provides information about the status of the instrument.

There is a single rule for the **Configuration** tab: **do NOT touch!**

The **Mapping** tab contains some features for sample mapping which might be useful for Raman experiments.

## I. Experiment Setup – Collect Tab



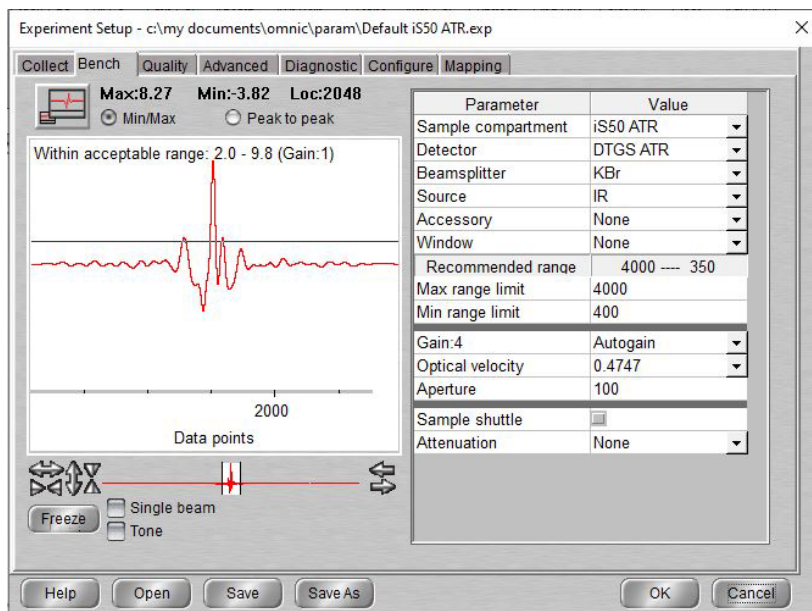
- *No. of scans*: set the number of scans to collect and co-add to obtain your spectrum (more scans will reduce noise but will add to the collection time). **Default = 36**.
- *Resolution*: set appropriately for your sample. Most solid and liquid samples are fine with a resolution of 4  $\text{cm}^{-1}$  but check your data to see if any sharp peaks are limited by this resolution setting in which case you may want to increase the resolution to 2 or 1  $\text{cm}^{-1}$ . The lower the resolution the faster the data collection time, so only use higher resolutions if you need to and are willing to spend more time collecting each scan. **Default = 4**.
- *Final format*: select the way the data will be processed and presented to you. Most users display their IR spectra in Absorbance (lines up) or %Transmittance (lines down). %Reflectance will automatically divide your sample spectra by a reference spectrum and present the results in percentage reflectivity. **Default = Absorbance. Always use absorbance for ATR measurements!** During the training session you will receive background information on why this is necessary to obtain best results. **For Raman measurements the final format is shifted wavenumber. The excitation laser wavelength is the  $0\text{cm}^{-1}$  reference!**
- *Correction*: most users leave this on 'None'. It will select a method to automatically correct for using an ATR (attenuated total reflectance) sampling technique and a few other corrections not typically used with the spectrometer.
- *Automatic atmospheric suppression*: check to have software remove any lines from residual  $\text{H}_2\text{O}$  and  $\text{CO}_2$ .
- *File Handling*: leave 'Save automatically' deselected and 'Save interferograms' selected. This way you will only save the files you choose to save. By saving the interferogram (the "raw data") you will have the ability to go back and reprocess your data later. If you choose to save all files automatically, you may type a four-character filename that will be incremented and saved in your autosave directory.
- *Background Handling*: 'Collect background before every sample' and 'Collect background after every sample' are usually impractical since they force you to take a new background before or after each sample. Most users have 'Collect background after xxx minutes' selected. This will remind you that after xxx minutes have passed that your background spectrum is getting old, and you should really acquire a new one. Pretty much any value between 10 and 120 minutes will work fine. You will need a new background when inverted water lines show up in your spectrum.
- Type in an *experiment description* and an *experiment title* that will be shown on the main OMNIC window.

Once all the parameters are set, you can click on the 'Save As...' button at the bottom of the window and save the experiment file. **WARNING: you need to give the experiment a new title**

and file name. If you don't make these changes, your file will be set as the new default setup file! When you want to go back to the saved experiment setup, just open the Experiment Setup window, click on the 'Open...' button and select the experiment file you want to open.

## II. Experiment Setup – Bench Tab

a) FTIR view:



This screen shows the real-time interferogram signal and allows the user to do some spectrometer configuration setup. Assuming you have a sample (which could also be just the air in the compartment) in the sample chamber and that the MCT/A detector is cooled with liquid nitrogen, you should see a signal like the one shown above.

The parameters in this window are:

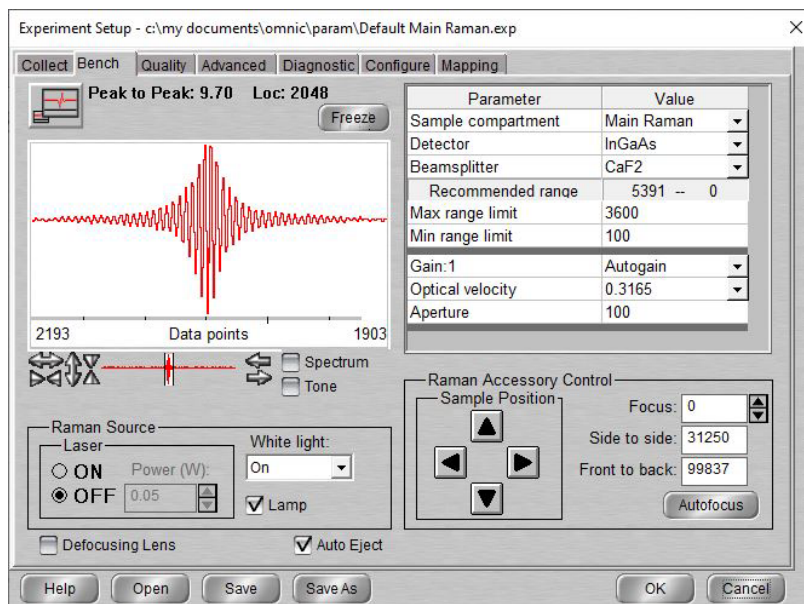
- *Sample compartment:* iS50 ATR, iZ10/Right AEM, and Main Raman
- *Detector:* The detector choices are DTGS ATR for ATR, DTGS KBr or MCT/A for transmission.
- *Beamsplitter:* The bench is equipped with a KBr beamsplitter for IR measurements.
- *Source:* Only the internal IR source (IR) is available.
- *Accessory:* Depending on setup.
- *Window:* With the ATR set to diamond, otherwise choose the window material of your cell if you use one, or KBr for pellets.



- *Spectral Range*: Using the IR source with the KBr beamsplitter, the maximum range is  $350\text{-}4,500\text{cm}^{-1}$ , the suggested range (best throughput) depends on the detector. The cut-off for the MCT/A detector is  $650\text{cm}^{-1}$  and  $100\text{cm}^{-1}$  for the DTGS detector, respectively. You can select a smaller range to match your sample interests.
- *Gain*: Set preamplifier gain to enhance small sample signals. The interferogram signal should be between 4 and 8 for best performance, depending on setup (the **max** value will indicate this on the interferogram display). The system will begin to saturate if the maximum peak signal is  $> 9.8$ , so make sure that you do not turn the gain up beyond these levels. Usually, it is best to leave the gain at the auto setting.
- *Velocity*: Set the mirror speed in the interferometer (measured in cm/sec).
- *Aperture*: Best around 100 when using the internal ATR unit. Using a transmission setup, the aperture range can be anywhere between 1 and 200 depending on the experimental setup used. When optimizing the signal, you can change the value to see which size gives you the best signal (shape and intensity). The smaller the aperture the higher the resolution but the signal strength will get weaker (less light reaching the detector).
- *Attenuation*: The attenuation is set to none for ATR experiments. Using the transmission setup, the screen wheel can be set to none, moderate or heavy attenuation. This might be necessary to avoid detector saturation.

Once again, now that you have all these parameters set, you can click on *Save As* to save this experiment to a file, so you won't have to reselect everything next time! The attenuation will always go back to open, independent of how the experiment file was saved.

## b) Raman view:

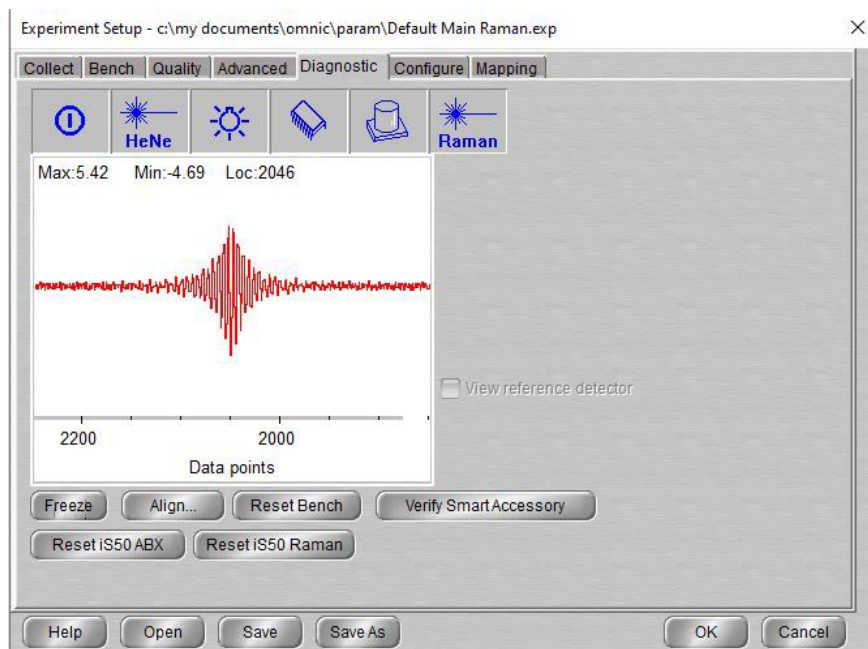


- *Sample compartment:* iS50 ATR, iZ10/Right AEM, and Main Raman
- *Detector:* The detector choice is InGaAs for Raman measurements.
- *Beamsplitter:* The bench is equipped with a CaF<sub>2</sub> beamsplitter for Raman measurements.

The bench tab for the Raman setup looks a bit different. This screen shot shows the interferogram with the white light on. There is a button to turn the excitation laser on and off. Laser power can be tuned between 50mW and 500mW. Please follow the table below (**Data Acquisition**) when adjusting the laser power.

The Raman accessory control allows the user to move the sample in x and y positions as well as adjust the focus. The complete guide for measuring Raman samples can be found under **Data Acquisition**.

### III. Experiment Setup – Diagnostics Tab



The '*Diagnostics*' tab window is where to go when something is not working. The six blue icons along the top of the window indicate the status of the power supplies, alignment laser, internal sources, spectrometer electronics, IR detectors, and Raman excitation laser respectively. If there is a problem with any of these, a diagonal red slash will appear over the appropriate icon. **The Raman laser has a red line when it is turned off!** If you see any of these indicators turn red, please call **Dr. Hartmut Hedderich** at 494-6543 ([hhedderi@purdue.edu](mailto:hhedderi@purdue.edu)). If nothing appears wrong, but you still do not see an interferogram signal, you may try pressing 'Reset Bench'. This will re-boot the bench electronics and will cause the spectrometer to re-initialize its zero positions. On occasion when the spectrometer has not had a signal in many hours, this button will make the spectrometer find the zero peak mirror displacement again and start functioning like normal. If you are using the MCT/A detector, please make sure that it is cooled with liquid nitrogen, or you will not see a signal! If you continue to have problems getting a signal, please contact the person-in-charge.

The '*Quality*', '*Advanced*', and '*Configure*' tabs in the Experimental Setup window are generally not changed for our experiments. The '*Quality*' tab allows you to set up hardware checks during data collection – i.e.: it checks if water and CO<sub>2</sub> levels are too high (no purge gas

running), if there is ice buildup on the detector and so on. Should a problem arise, you will get a “warning” during data collection. The ‘*Advanced*’ tab allows you to set some of the Fourier transform parameters. The default settings will work for just about everyone, but I would be happy to tell you more about them if you feel you want to get to this level of understanding with FTIR. The ‘*Configure*’ tab allows you to configure the FTIR bench. **Please do not change anything within the ‘*Configure*’ tab!**

#### 4. Data Acquisition - FTIR

Now that the Experiment Setup is complete, the data acquisition can begin!

- First you need to take a background spectrum. Once you are happy with the signal strength seen in the Bench Tab of the *Experimental Setup* window, close that window by pressing 'OK'. Hovering the mouse over the different icons will tell you which action the icon will perform. Push the **Collect Background** button. The system may prompt you again to make sure you are ready. Then it will acquire the number of scans you set in the *Experiment Setup* window. You can see the progress of the scans at the bottom left corner during data collection, and the screen will show you an occasionally updated average of the data you are collecting.
- Once the background is complete, you are ready to measure your sample.
  - Diamond ATR: Put solid sample over the window, use pressure tower to fasten down sample. After the tip touches the sample turn the knob until you hear a single “click”. For liquid samples just put a droplet of the liquid onto the diamond and collect a spectrum. Make sure to carefully clean the crystal with alcohol after use.
  - Transmission: Use the correct holder (liquid cell, thin film holder, pellet holder) for your sample! Put the sample in the sample chamber. Purge for about 1 minute with nitrogen. Then open the *Experimental Setup* window with the Bench tab selected and check the IR signal strength. Remember to adjust attenuation if the signal is too strong. When you are satisfied, close the *Experimental Setup* window by clicking 'OK'. Push the **Collect Sample** icon. Depending on the output format you have chosen you will get either a spectrum in absorbance or %transmittance.

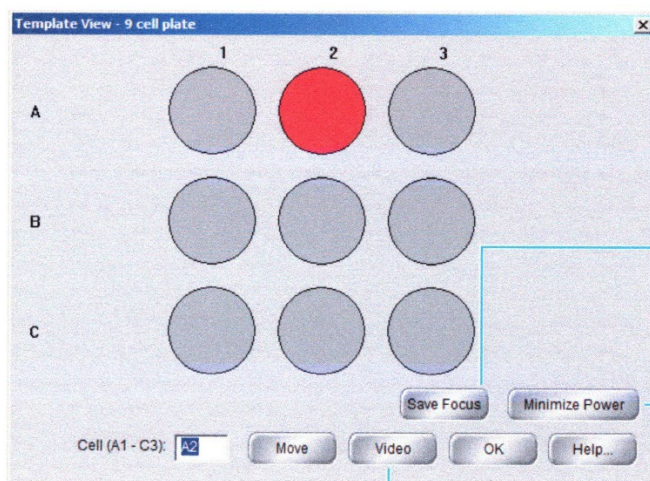
Now that you have an IR spectrum, you can do a lot of different types of data processing and visualization using the Process and Analyze menus. These functions will not be covered here but can be found in the extensive on-line help files for OMNIC and the OMNIC manuals.

**Online help is available when you move the mouse over a command or icon on the screen and press the right mouse button!**

## 5. Data Acquisition – Raman

Make sure that the white light on the *Bench* screen is turned on, the laser should be off.

Place your sample on the holder you want to use. Put the holder on the microscope stage. Click on the RAMAN pull-down menu icon. Then click on *Template* and choose the holder you want to use. A window will open showing the chosen template. Here we have the 9 well template:



For details, find Template View in Raman Help Topics, available from the Raman menu in OMNIC software.

Saves the current focus setting for this template; use only if you always measure samples that are the same thickness.

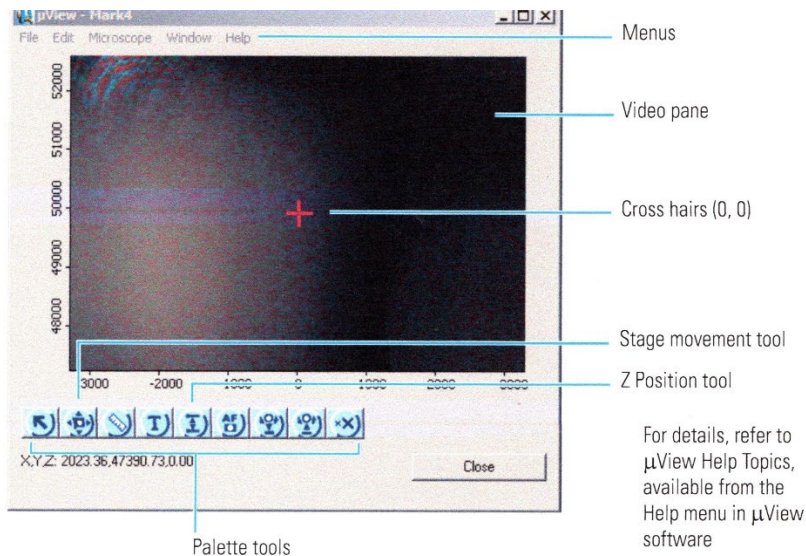
Quickly lowers the laser power to approximately 0.05 W

To select a cell, **double-click** it or enter the cell location and choose **Move**.

Opens  $\mu$ View software which shows a video image of the selected sample location

Choose the well you want to investigate and click on it with the mouse. The well will turn red, and the stage will move to the correct position. Click the video button. This will start the  $\mu$ View program. You will be able to see your sample:





The stage can be moved with the stage movement tool, the focus will be set by the Z positioning tool. The position with the cross hairs will be interrogated by the Raman laser. Once you are satisfied with the positioning, close the μView window.

Return to the *bench* screen. Turn the white light off and the laser on. Start with low laser power! The positioning should be good enough to get some signal. You can now either perform an Autofocus where the program tries to optimize the focus, or you can change the focus manually and check the signal strength by turning the display to Spectrum. After optimizing the signal, close the *Experiment Setup* window and push the **Collect Data** button.

### Compatible Samples for the Raman module:

The Raman module can be used for a wide variety of sample types. The following table shows some examples:

Types:	Characteristics:	Examples:
Solids <sup>1</sup>	Non-absorbing material	Minerals, Crystals, Sulfur
Tablets	Granular or crystalline powders	Pharmaceuticals
Powders, Granules, Pellets <sup>2</sup>	Non-absorbing material	Polymers, Pharmaceuticals
Liquids, Gels <sup>3</sup>	Non-absorbing material	Clear Solvents, Sol-Gels

In general, there are a few rules for sample preparation that should be followed:

1. Reduce the laser power for materials with dark inks or strong colorations such as printed tablets or carbon black rubber. These materials may absorb the laser light, resulting in extreme heating or destruction of the sample!

2. Sprinkle a few grains or crystals onto a Raman sample holder or a standard microscope slide. If a powder is packed loosely, you can improve the signal by pressing the powder into a pellet and then interrogate the pellet.
3. Use a Raman tube for your liquid samples. Alternatively, for small amounts of material, place a few drops of liquid directly into one of the wells in the 9 or 48 well sample holder. Use a pan or small cup (preferably aluminum) that fits into the well recess.

### Positioning Information:

This part will give some general information about positioning samples on the flat plate and the 3 and 4 Raman tube holders. This data should give a good starting point for finding and positioning samples correctly to get an initial signal. Each data set contains information for the focus. Side-to-side position and front-to-back position.

**Flat Plate** (solid sample holder, about center of plate):

**Focus:** 15750

**Side-to-side:** 35000

**Front-to-back:** 45000

### 3 Position Tube Holder:

<b>Position:</b>	<b>Focus:</b>	<b>Side-to-Side:</b>	<b>Front-to-back:</b>
1	14500 (solids), 15500 (liquids)	49000	13000
2	14500 (solids), 15500 (liquids)	31900	13000
3	14500 (solids), 15500 (liquids)	14600	13000

### 4 Position Tube Holder:

<b>Position:</b>	<b>Focus:</b>	<b>Side-to-side:</b>	<b>Front-to-back:</b>
1	17500 (solids), 19000 (liquids)	56500	14000
2	17500 (solids), 19000 (liquids)	40000	14000
3	17500 (solids), 19000 (liquids)	24000	14000
4	17500 (solids), 19000 (liquids)	6500	14000

## 6. Contacts

Dr. Ryan Hilger  
RIC Manager  
Office: BRWN 4135B  
Phone: 765-494-5234

[rhilger1@purdue.edu](mailto:rhilger1@purdue.edu)

Dr. Hartmut Hedderich  
Sr. Instrumentation Specialist, PIC  
Office: BRWN 4165B  
Phone: 765-494-6543  
Cell: 317-340-3149

[hhedderi@purdue.edu](mailto:hhedderi@purdue.edu)

**7. Notes:**