Nicolet iS50 FT-IR Spectrometers

iS50 Raman Module
User Guide

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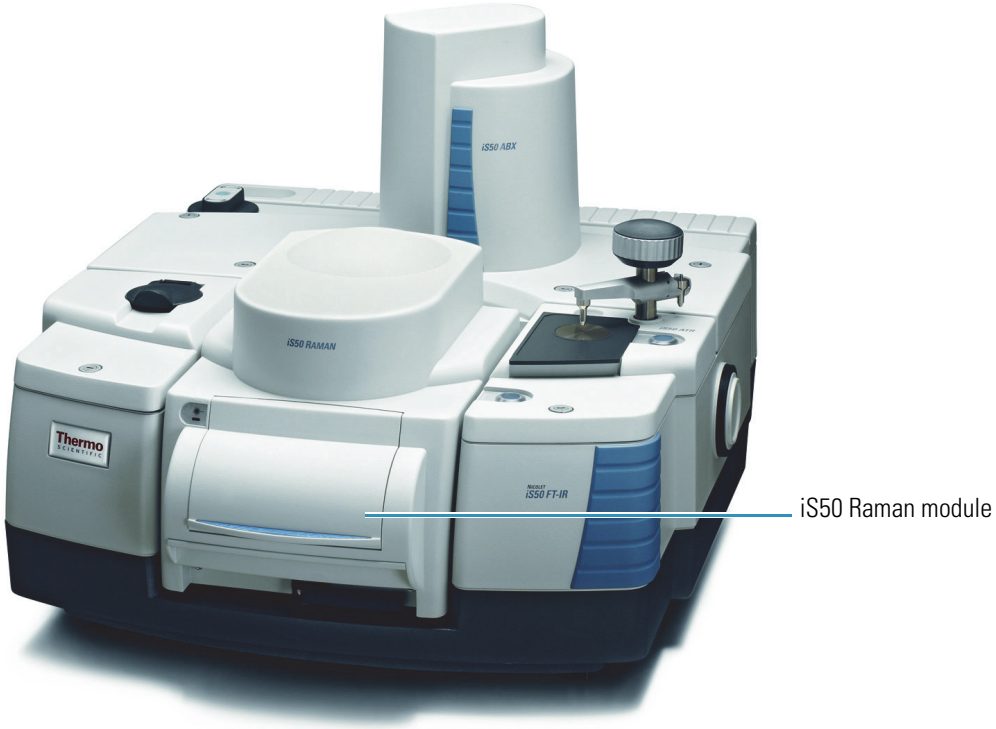
WARNING Avoid an explosion or fire hazard. This instrument or accessory is not designed for use in an explosive atmosphere.

iS50 Raman Module

This section describes the optional Thermo Scientific iS50 Raman module which allows you to collect FT-Raman spectra with your Nicolet™ iS™50 spectrometer.

Figure 1. Nicolet iS50 spectrometer with iS50 Raman module installed



The iS50 Raman module mounts in the spectrometer sample compartment.

Contents

NOTICE Be sure that all persons operating this system read the site and safety manual first.

Conventions Used in this Document

This manual uses these conventions for providing safety and other special information:

WARNING Indicates a hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

NOTICE Follow instructions with this label to avoid damaging the system hardware or losing data.

Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

About the iS50 Raman module

The iS50 Raman module provides these enhanced features:

- Compact, integrated design
- Push button configuration with the optional ABX (Automatic Beamsplitter eXchanger)
- Long wavelength (low energy) excitation laser
- Dedicated detector
- Motorized sample stage
- Built-in video camera
- Superior Raman sensitivity
- Small beam spot size
- Sample screening capabilities using our sampling templates and cluster analysis software.

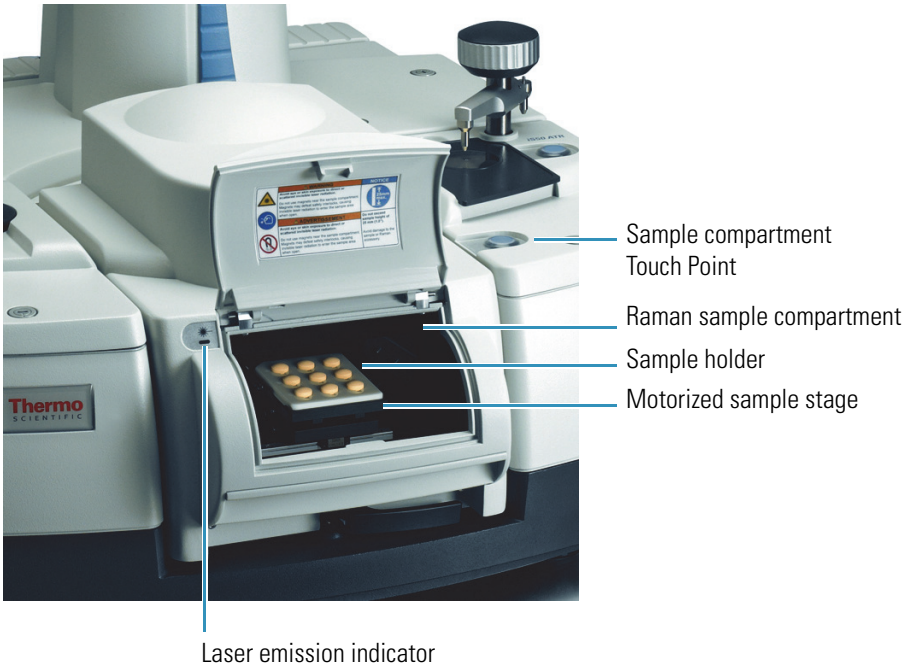
The Raman module works with our ValPro™ qualification package and security administration software and allows digital signatures and IQ-OQ-PQ-DQ certification. Please refer to your ValPro and OMNIC™ DS documentation for more information. For information about additional sampling modules for the Nicolet iS50 spectrometer, contact our sales representative in your area.

Related Topics

- Important Features

Important Features

Figure 2. iS50 Raman module features



The iS50 Raman module features include:

- **Raman sample compartment.** Allows access to the Raman sampling area and sample stage. The stage accommodates a variety of sample holders that can be quickly interchanged.

  A laser emission indicator (see below) tells you when the laser beam is present in the sample compartment. Two independent safety interlocks ensure the laser beam is blocked when the sample compartment door is open.

- **Motorized sample stage.** Used to mount a sample or sample tray in the Raman module and to position the area of interest under the laser for analysis. Fine motor control in all directions and built-in video camera allow precise positioning for very small samples.

- **Sample compartment Touch Point**. Configures the spectrometer for Raman sampling and shows the status of the Raman module. The Touch Point LED has three states described below.

  Table 1. Sample compartment Touch Point indicator states

LED status	Meaning
Off	Raman module is not installed or connected properly
Blinking	Optics are configuring for Raman sampling
On	Raman module is ready for use

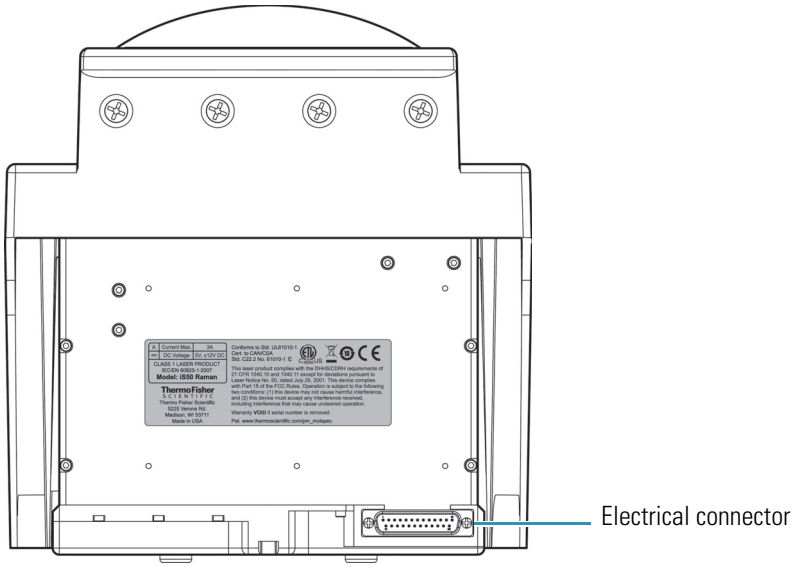
- **Laser emission indicator**. Shows the status of the excitation laser inside the Raman module. When this LED is lit, the laser is on. For more information, see "Turning on the Laser and Adjusting the Laser Power."

> **Note** Two independent safety interlocks ensure the laser beam is blocked when the sample compartment door is open.

> **NOTICE** Do not leave samples inside the sample compartment for extended periods when the laser is on.

- **Video USB cable**. Allows the software to communicate with the video camera in the Raman module. One end of the cable plugs into the Sample Compartment Accessory port on the back of the spectrometer. The other end connects to any type A USB port on the system computer.
- **Electrical connector**. Provides power to the Raman module and communication with the spectrometer. For more information, see "Installing the Raman Module."

Figure 3. iS50 Raman module back panel



Related Topics

- About the iS50 Raman module
- Installing the Raman Module
- Turning on the Laser and Adjusting the Laser Power

Specifications

Table 2. iS50 Raman module specifications

Feature	Specification
Excitation source	1064 nm laser Maximum power: 0.5 W
Reference interferogram source	White light
Beamsplitter	Calcium fluoride (CaF_2) or extended range potassium bromide (XT-KBr), (located in the spectrometer)
Detector	Dedicated Indium Gallium Arsenide (InGaAs), (located in the spectrometer)

Related Topics

- About the iS50 Raman module
- FT-Raman Sampling
- Turning on the Laser and Adjusting the Laser Power

Compatible Software

Table 3. Software used to run the iS50 Raman module

Software	Use for...
OMNIC	Configuring the Raman workflow, including quantitative analysis and spectral search. Also, selecting sampling templates and processing Raman spectra. For more information, see "Your First Experiment" or open OMNIC Help Topics from the Help menu in OMNIC software.
OMNIC Raman	Collecting and processing Raman data. For more information, open the Raman menu in OMNIC software and select Raman Help Topics.
µView™	Enhanced sample viewing and capturing and annotating video images. The software works with a microscope video system and video image to allow the image from the camera to be displayed on the computer monitor and recorded.
TQ Analyst Professional Edition	Creating quantitative and classification methods that can be used with OMNIC software. This software provides an extensive suite of chemometrics features including Partial Least Squares and other advanced algorithms for quantitative analysis and material identification. For more information, open the Help menu in TQ Analyst software and select TQ Analyst Help Topics.

Table 4. Optional Software for the iS50 Raman module

Software	Use for...
Atlμs™	Performing automated data collection and spectral mapping experiments as well as image analysis with the iS50 Raman module. You can automatically collect different types of compositional maps and display the collected data in a variety of formats. You can also create reports containing map data and print contour maps, 3-D displays, and comprehensive video images of the sample. For more information, open the Atlμs menu in OMNIC software and select Atlμs Help Topics.
Array Automation	Collecting and processing data from groups of samples that are arranged in an ordered array. The array may be rectangular as provided by a micro-titer well plate or linear if capillary tubes are being used. For more information, open the Array menu in OMNIC software and select Array Help Topics.
OMNIC Specta™	Analyzing unknowns. OMNIC Specta software includes our unique multi-component search feature for identifying the spectra of mixtures, a 9,000 compound spectral database, and features for using your computer's hard drive as a spectral library. For more information, open the Help menu in OMNIC Specta software and select the Software Help Topics item.

Related Topics

- Your First Experiment

Safety Considerations

The use of redundant safety interlocks on the main cover of the Raman module and on the Raman sample compartment cover prevent accidental exposure to invisible laser radiation. The presence of a strong magnetic field in the area may interfere with or defeat the safety interlocks.

WARNING Avoid laser hazard. Keep all magnets and strong magnetic fields (such as those produced by mass spectrometers and some kinds of medical equipment) away from the Raman module, and do not attempt to defeat the safety interlocks on the module housing and sample compartment cover—hazardous exposure to laser radiation could occur.

Related Topics

- Turning on the Laser and Adjusting the Laser Power
- Your First Experiment

Operating Precautions

- Do not pour liquids directly onto the Raman module.
- Do not use harsh detergents, chemicals or abrasives to clean the surface of the Raman module; these can damage the finish. For cleaning instructions and recommended solvents, see "Maintenance."
- Always place samples on a sample holder and then load the holder on the sample stage. Do not place a sample directly on the stage.
- Before you install a sample holder on the sample stage, ensure the height of the holder plus the sample (or the sample container) does not exceed 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located behind or above the stage. If those components appear soiled or damaged, contact our local sales or service representative for assistance.
- Avoid spilling samples or solvents in the Raman sample compartment. Clean the residue tray regularly; see "Cleaning the Residue Tray" for instructions.
- Do not allow moisture or cleaning products to come into contact with any part of the interior of your sample stage. Do not attempt to clean any internal components, and do not touch any optical surface inside the Raman sample compartment.
- Store the Raman module right side up.

Related Topics

- Raman Sample Holders
- Installing a Sample Holder
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

FT-Raman Sampling

Monochromatic light from the excitation laser in the iS50 Raman module is directed to the sample. When the light strikes the sample, the sample emits Raman radiation; in effect, the sample becomes the "source" of radiation. This radiation travels through the Raman module and spectrometer optics to a dedicated detector located in the spectrometer. The laser light, on the other hand, does not travel past the Raman sample compartment.

Raman Spectrum

The Raman spectrum shows the sample emission at all the frequencies measured and thus can be used to identify the sample. Intensity is expressed in emittance units, which measure the amount of light emitted by the sample. Frequency is expressed in terms of wavenumber, which is a measurement of number of waves per centimeter (cm^{-1}). In Raman spectroscopy the X-axis is converted to Raman shift, which is a measure of the difference between the observed spectral bands and the wavelength of the excitation laser.

The Raman Effect

When a laser beam strikes a sample, a very small portion of the laser light, less than 0.0001%, is scattered by the sample at a frequency different from that of the excitation laser. This shifting of the frequency is called the Raman effect, and the frequency-shifted light is called Raman radiation. For more information, open Raman Help Topics from the Raman menu in OMNIC software and find "Raman Shift."

Related Topics

- Important Features
- Specifications

Compatible Samples

The Raman module can be used to measure a wide variety of samples types. Some examples are provided below.

Table 5. Samples compatible with the Raman module

Types	Characteristics	Examples
Solids[a]	Non-absorbing materials	Minerals, crystals, sulfur
Tablets	Non-absorbing materials	Aspirin, other pharmaceuticals
Powders, granules, pellets[b]	Granular or crystalline powders	Active and inactive pharmaceutical ingredients, polymer pellets
Liquids, gels[c]	Non-absorbing materials	Clear solvents, sol-gels

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

[b] Sprinkle a few grains or crystals onto a Raman sample holder or a standard microscope slide (metal is best but glass works too) attached to a Raman slide holder. If a powder is loosely packed, you can improve the Raman signal by pressing the powder into a pellet and then placing the pellet on a Raman sample holder. We offer an optional powder press for this purpose. Contact our representative for details.

[c] Place a few drops of liquid directly into one of the wells in the 9 or 48 well sample holder, or into a small cup or pan that fits the sample holder recesses, or into a vial that fits the Raman vial holder.

Several styles of sample holders are provided with the iS50 Raman module for fast, reproducible sampling of solids, liquids, powders and gels. For more information, see "Preparing Samples."

NOTICE

- Do not spill liquids directly onto the Raman module.
- Always place samples on a Raman sample holder and then load the holder on the Raman sample stage. Do not place 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 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.
- Avoid spilling samples or solvents in the Raman sample compartment. Clean the residue tray regularly; see "Cleaning the Residue Tray" for instructions.

Related Topics

- Raman Sample Holders
- Installing a Sample Holder
- Removing a Sample Holder
- Using the Defocusing Lens to Adjust the Beam Size
- Cleaning the Residue Tray

Configuring the Raman Module

This section explains how to configure the iS50 Raman module the first time it is installed on a Nicolet iS50 spectrometer.

Note You do not need to configure the iS50 Raman module each time you install OMNIC software; only the first time OMNIC software is used with the Raman module.

Before you begin, check that the following software is installed on the computer connected to the Nicolet iS50 spectrometer:

- Thermo Scientific OMNIC, version 9, SP1 or higher
- Thermo Scientific µView version 9, SP1 or higher, or
  Thermo Scientific Atlµs, version 9, SP1 or higher

❖ **To configure the Raman module**

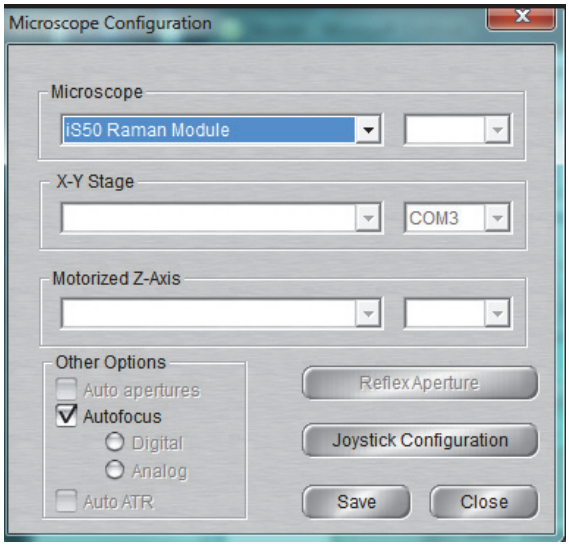
1. Make sure the spectrometer and computer are powered on (allow at least 20 minutes for the spectrometer to stabilize).

2. Close OMNIC, µView and Atlµs software if they are open.
3. Install the Raman module in the spectrometer sample compartment.
4. Start OMNIC software.

   The Microscope Configuration dialog box is displayed.

   Note If the Microscope Configuration dialog box does not appear after you start OMNIC software, the Raman module has already been configured.

   Figure 4. Microscope Configuration dialog box



5. Set **Microscope** to iS50 Raman Module and choose **Save**.
6. If beamsplitters must be installed manually for your spectrometer, install the CaF_2 or XT-KBr beamsplitter in the spectrometer beamsplitter compartment. For more information, find "Using Beamsplitters" in Spectrometer Help Topics.

   Note If you have the optional ABX (automatic beamsplitter exchanger), the correct beamsplitter installs automatically after you install the Raman module.

7. Align the interferometer.

   The system remembers the alignment settings for each beamsplitter. This means the next time you use the Raman module, you won't necessarily need to align the spectrometer. See "Aligning the Interferometer" for instructions and information about how often to perform this step.

Note To see the iS50 Raman module configuration settings at any time, open µView software and choose **Microscope > System Configuration.**

Related Topics

- Installing the Raman Module

Installing the Raman Module

This section explains how to install the iS50 Raman module in the sample compartment of your Nicolet iS50 spectrometer.

Note You will need to configure the iS50 Raman module the first time it is used with OMNIC software. See "Configuring the Raman Module" for more information.

❖ To install the Raman module

1. Remove the sample compartment cover from the spectrometer, any connectors at the back of the sample compartment, and the Snap-In™ baseplate (if installed).

   For more information, find "Using Your Spectrometer" in the Spectrometer Help Topics or the *Nicolet iS50 User Guide*.

2. Remove any optional filters or energy screens that may be installed on the left or right side of the spectrometer sample compartment.

   For more information, find "Installing or Removing Optional Hardware" in the Spectrometer Help Topics or the *Nicolet iS50 User Guide*.

   NOTICE Avoid scraping the electrical connector on the Raman module back panel against the back wall of the sample compartment as you lower the Raman module into the sample compartment.

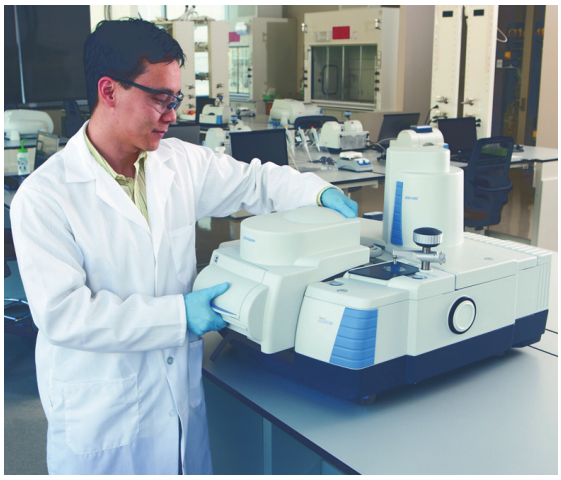
3. Connect the Video USB cable that came with the Raman module.

   Plug one end of the cable into the Sample Compartment Accessory port which is located at the bottom of the spectrometer back panel near the purge connector.

   Connect the other end of the cable to any type A USB port on the computer.

4. Grasp the hand holds at the front and back edges of the Raman module and carefully place the module at the front of the sample compartment.

   Figure 5. Installing the iS50 Raman module



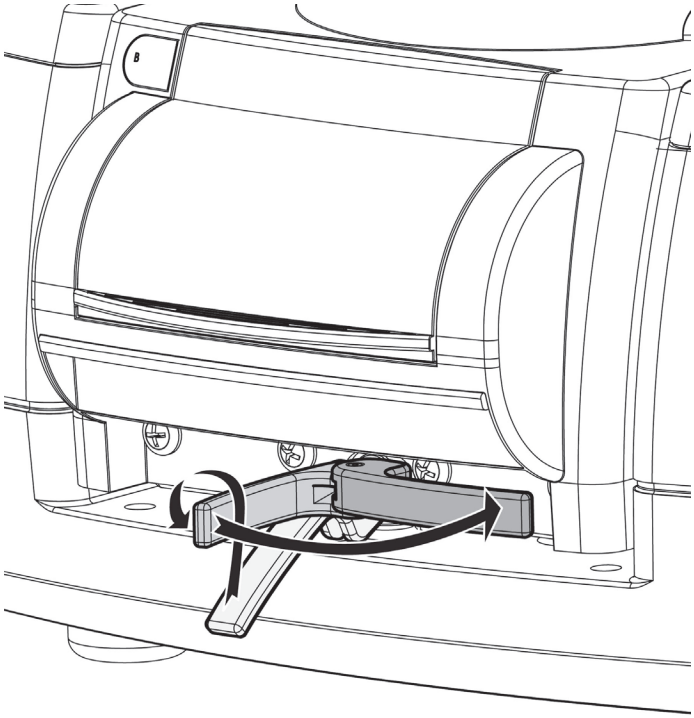
5. Slide the Raman module back to secure the electrical connection.

   The motors inside the Raman module will activate when the electrical connector is secured.

6. Secure the front latch.

   To do this, lift and rotate the handle 90 degrees counterclockwise and then firmly press the handle toward the module.

   Figure 6. Securing the iS50 Raman module front latch



   Note If the Microscope Configuration dialog box is displayed, the Raman module is not configured. See "Configuring the Raman Module" for more information.

7. If beamsplitters must be installed manually for your spectrometer, install the CaF_2 or XT-KBr beamsplitter in the spectrometer beamsplitter compartment. For more information, find "Using Beamsplitters" in Spectrometer Help Topics.

   If you have the optional ABX (automatic beamsplitter exchanger), the correct beamsplitter installs automatically after you install the Raman module.

Note If your spectrometer is equipped with optional purge shutters, for best results, open the right purge shutter while the Raman module is collecting data. For more information, find "Controlling the Purge Shutters" in OMNIC Help Topics. (If Shutter Mode is set to Automatic on the Bench tab in Experiment Setup, the purge shutters will open automatically for Raman data collection.)

Related Topics

- Configuring the Raman Module
- Aligning the Interferometer

Operation

This section covers these topics:

- Configuring the System for Raman sampling
- Turning on the Laser and Adjusting the Laser Power
- Adjusting the Sample Position
- Setting a Timer to Turn Off the Laser

Configuring the System for Raman sampling

If your Nicolet iS50 spectrometer includes the iS50 ABX (Automatic Beamsplitter eXchanger), after you install the Raman module, the spectrometer automatically configures its optics for Raman sampling. (If you don't have an ABX, the software will prompt you to install the appropriate beamsplitter.)

To configure the spectrometer for Raman sampling when the Raman module is already installed, for example after using the optional NIR or ATR module, press the sample compartment Touch Point.

Related Topics

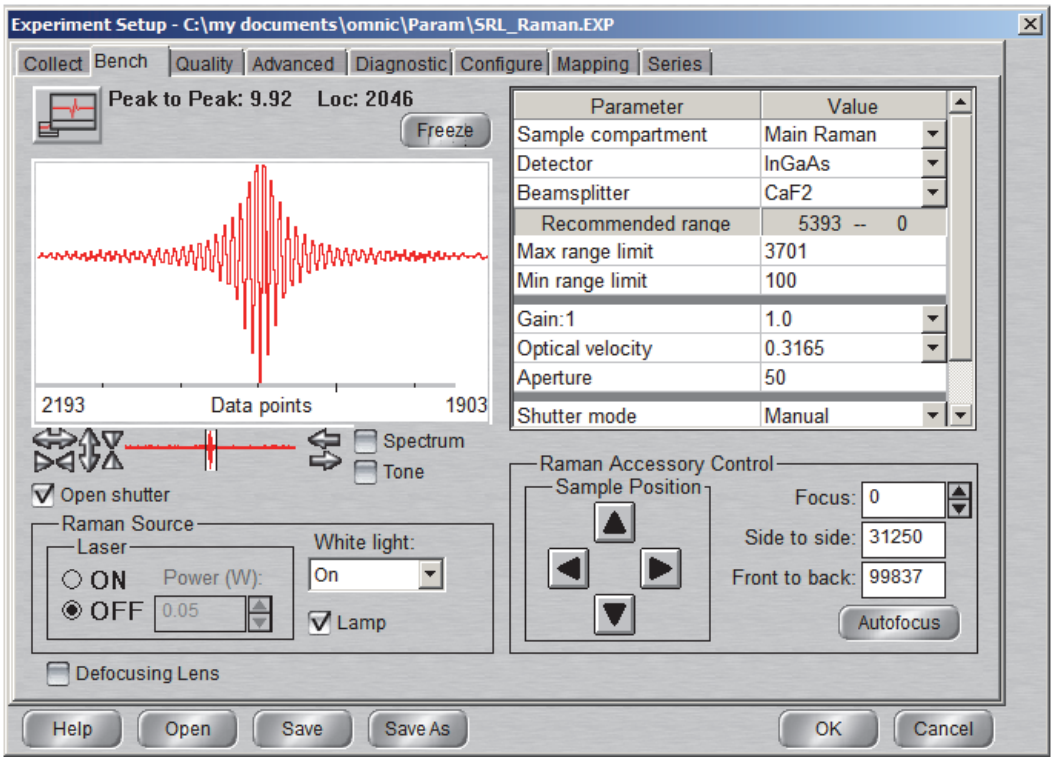
- Turning on the Laser and Adjusting the Laser Power
- Adjusting the Sample Position
- Setting a Timer to Turn Off the Laser

Turning on the Laser and Adjusting the Laser Power

The Raman module has a 1064 nm excitation laser. The laser is located at the rear of the module under the main cover, and emits continuous-wave laser energy that is invisible to the eye. The laser has a nonaccessible maximum power level of 0.5 W at the sample. The laser must be on when you are focusing a sample and collecting Raman data.

To access the features to control the laser, open OMNIC software, choose **Collect > Experiment Setup** and select the **Bench** tab.

Figure 7. Bench tab settings for the iS50 Raman module



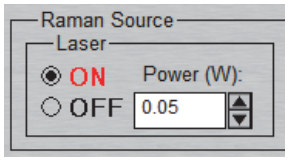
To turn on the laser, click the ON button in the Laser box. It takes a few seconds for the laser to begin emitting radiation.

> **Note** You can leave the white light set to On except when you are focusing a sample. See "Using the White Light Source" for details.

Neutral density filters are used to control the power of the laser that reaches the sample compartment. Reducing the power at the sample can be useful when you have a heat-sensitive (usually dark) sample.

❖ To adjust the laser power

1. Set **Power** to 0.25 W.



2. Select **Spectrum** to show the spectral data in the display pane on the Bench tab.
3. Adjust the power up or down until you see adequate sample peaks in the spectral display.

   While you adjust the power, monitor the video image of your sample for signs of decomposition, especially if the sample is a dark color.

NOTICE

- If your sample starts to decompose, reduce the laser power and then turn off the laser. Move the sample so that the laser beam will strike an area that is not decomposed, and then turn the laser on. If the sample starts to decompose again, repeat these steps, but be aware that some samples may be too sensitive to laser energy to be suitable for analysis.
- To maximize the life of your laser, do not leave the laser on for extended periods when not in active use. To set a timer for the laser, see "Setting a Timer to Turn Off the Laser."

Related Topics

- Important Features
- Specifications
- Configuring the System for Raman sampling
- Adjusting the Sample Position
- Setting a Timer to Turn Off the Laser
- Using the White Light Source

Adjusting the Sample Position

You can adjust the sample position and focus using Template View, available from the Raman menu in OMNIC software, along with the video pane in either µView or Atlµs software. For basic information about using OMNIC and µView software to position and focus a sample and collect Raman data, see "Your First Experiment."

Note The laser must be on and the white light off while you adjust the sample position and focus. See "Turning on the Laser and Adjusting the Laser Power" and "Using the White Light Source" for details.

For complete information about controlling the sample stage with µView, double-click the µView icon on your computer desktop and choose **Help > µView Help Topics**.

Complete information about using Atlµs to control the sample stage is available by choosing Atlµs Help Topics from the help menu in OMNIC software.

> **Note** Position settings in OMNIC, Atlµs and µView software are calibrated in micrometers.

Related Topics

- Turning on the Laser and Adjusting the Laser Power
- Setting a Timer to Turn Off the Laser

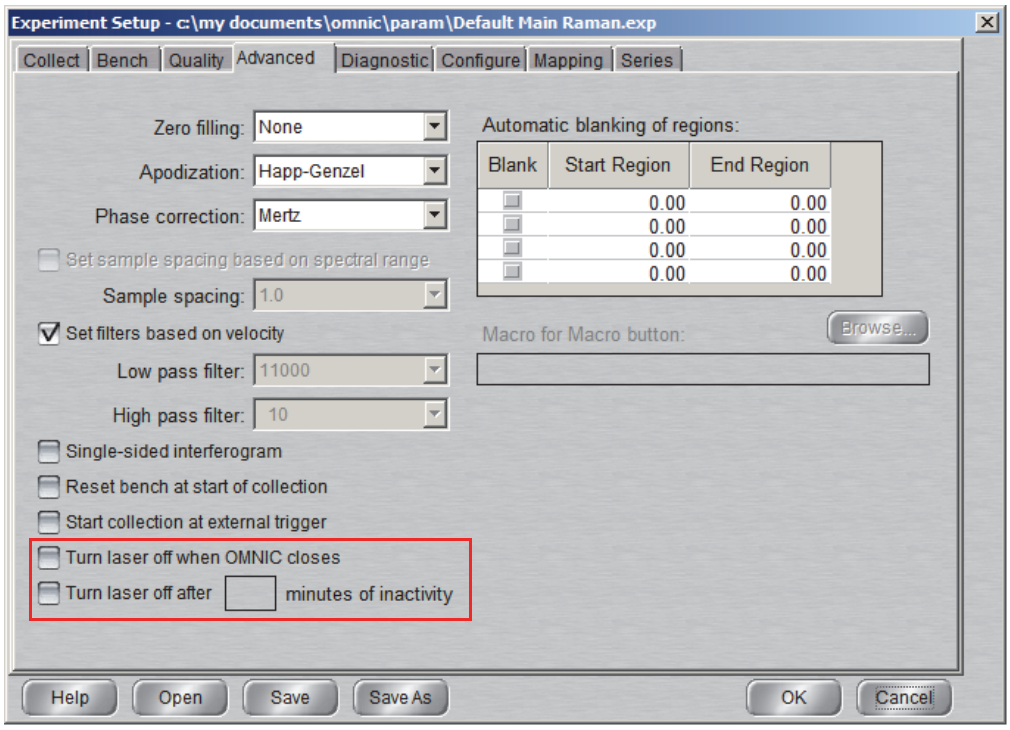
Setting a Timer to Turn Off the Laser

You can set up the Raman laser to turn itself off after a specified period or after OMNIC software closes. This will prevent the laser from being left on inadvertently and prolong its life.

❖ To set a timer for the laser

1. In OMNIC software, choose **Collect > Experiment Setup**.
2. Select the **Advanced** tab.

Figure 8. Advanced tab parameters in Experiment Setup



3. Set one of these options:
   - Select **Turn Laser Off When OMNIC Closes**
   - Select **Turn Laser Off After ___ Minutes Of Inactivity** and then enter the number of minutes.
4. Choose **OK**.

Related Topics

- Turning on the Laser and Adjusting the Laser Power
- Adjusting the Sample Position
- Your First Experiment

Preparing Samples

The Raman sample stage can be used with solids, liquids and powders (see "Compatible Samples" for details). They can be held in metal, glass or transparent plastic containers or metal or glass slides, and can also be placed in reusable or disposable sample cups or slides, or directly in the recess of a Raman sample cup holder or other Raman sample holder.

These topics are covered:

- Raman Sample Holders
- Installing a Sample Holder

Raman Sample Holders

The following sample holders are included with the iS50 Raman module:

- 9 well. Contains nine 13 mm wells.
- Slide holder (on the reverse side of the 9 well sample holder). Accepts a standard microscope slide (metal, clear plastic or glass).
- 48 well. Contains 48 7 mm wells.
- Vial holder. One side has three recesses to hold glass or clear plastic vials; the other side has four.

NOTICE

- Always place samples on a Raman sample holder and then load the holder on the Raman sample stage. Do not place 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 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.

Related Topics

- Installing a Sample Holder
- Removing a Sample Holder
- Your First Experiment

Installing a Sample Holder

The sample stage holds a variety of sample holders, each designed to position the sample at the focus of the excitation and collection optics (except when the defocusing lens is used).

These instructions show how to install any of the iS50 Raman module sample holders on the sample stage.

NOTICE

- Always place samples on a Raman sample holder and then load the holder on the Raman sample stage. Do not place 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 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.

❖ To install a sample holder (watch video)

1. Load your samples into the sample holder (see "Preparing Samples" for more information).
2. Open the Raman sample compartment door.

   The sample stage moves down and forward. Wait until it stops moving before you proceed.
3. Install the sample holder on the stage.

   Position the sample holder so that the angled corners are on the right side.

Figure 9. Standard sample holder

Position the sample holder with the angled corners on the right side.

Gently slide the sample holder onto the stage, pressing down on the front edge of the holder so it snaps in place.

4. Close the sample compartment cover.

   The sample stage moves the sample holder to the focus point of the collection optics and beam.

Related Topics

- Raman Sample Holders
- Removing a Sample Holder
- Your First Experiment
- Using the Defocusing Lens to Adjust the Beam Size

Removing a Sample Holder

Always wait until the stage stops moving before you remove the sample holder.

❖ **To remove a sample holder (watch video)**

1. Open the Raman sample compartment door.

   Wait until the stage stops moving before you proceed.

2. Lift the sample holder off the stage.
3. Close the Raman sample compartment door.

Related Topics

- Raman Sample Holders
- Installing a Sample Holder
- Your First Experiment

Sampling Options

This section covers these topics:

- Using the White Light Source
- Using the Defocusing Lens to Adjust the Beam Size
- Preventing Detector Saturation

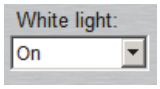
Using the White Light Source

The iS50 Raman module includes a white light source that is used for alignment and diagnostics. See "Aligning the Interferometer" for more information.

The white light source is normally left on to ensure that the spectrometer always has a strong enough signal to maintain system alignment. You will need to turn off the white light in order to focus your sample. The white light must also be off during Raman data collection but the software does this automatically and turns it back on after data collection is completed. To step through a typical Raman analysis, see "Your First Experiment."

❖ To control the white light source manually

1. Choose **Collect > Experiment Setup** and select the **Bench** tab.
2. Set **White Light** to On or Off as needed.



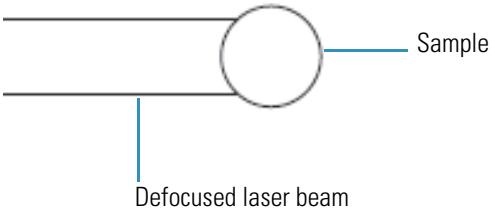
Related Topics

- Using the Defocusing Lens to Adjust the Beam Size
- Preventing Detector Saturation
- Your First Experiment
- Aligning the Interferometer

Using the Defocusing Lens to Adjust the Beam Size

There are two modes of operation for the Raman laser, focused and defocused. The focused (default) setting causes the laser beam to have the same focal point as the collection optics and produces a 50 micrometer diameter beam at the sample. The defocused setting creates an area with a diameter of approximately 1 mm at the sample by inserting a diffusing lens in the path of the laser beam.

Figure 10. Defocused laser beam

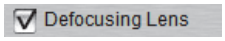


There are two reasons to use the defocusing lens:

- The focused laser beam can burn some samples.
- If a sample is non-homogeneous, defocusing the laser beam causes the spectrum you obtain to be an average of all the different sample components in the area the laser strikes.

❖ To defocus the laser

1. Choose **Collect > Experiment Setup** and select the **Bench** tab.
2. Select **Defocusing Lens**.

   The defocusing lens moves into the path of the laser beam and remains there until you clear this option.

Related Topics

- Using the White Light Source
- Preventing Detector Saturation
- Your First Experiment
- Aligning the Interferometer

Preventing Detector Saturation

Raman detectors are very sensitive to stray light. If light from ceiling light fixtures or other sources enters the spectrometer sample compartment, the detector can become saturated, resulting in little or no output from the detector. Stray light can also add noise to collected spectra. Fluorescent lights can produce spikes at 2860, 2085, and 540 shifted wavenumbers. Make sure all instrument covers and beam ports are closed before you collect Raman data.

Related Topics

- Using the White Light Source
- Using the Defocusing Lens to Adjust the Beam Size
- Your First Experiment

Your First Experiment

The Raman module is designed to illuminate a sample with laser radiation, collect as much of the radiation scattered by the sample as possible and filter this radiation so that only the Raman-shifted radiation is left. This radiation travels to the interferometer and then to the detector.

This section steps you through a typical Raman analysis. These topics are covered:

- Configuring Your Raman Workflow
- Measuring a Tablet using the iS50 Raman Module

Configuring Your Raman Workflow

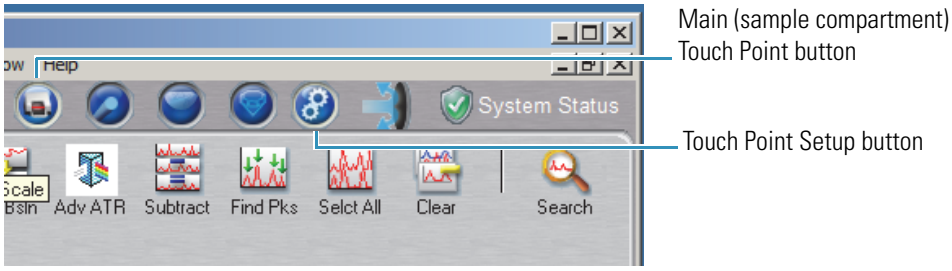
The sample compartment Touch Point has an associated Raman workflow that can define everything from background and sample collection to quantitative analysis and spectral search. You simply press the Touch Point to start the workflow. The Touch Point can also be used to respond to prompts in the software while the workflow is running.

❖ To configure your Raman workflow

1. Install the Raman module.
2. Start OMNIC software.

   The OMNIC window is displayed. For information about the OMNIC window, choose Help > OMNIC Help Topics.
3. Choose the Main (sample compartment) **Touch Point button** in the software to configure the instrument for Raman sampling.

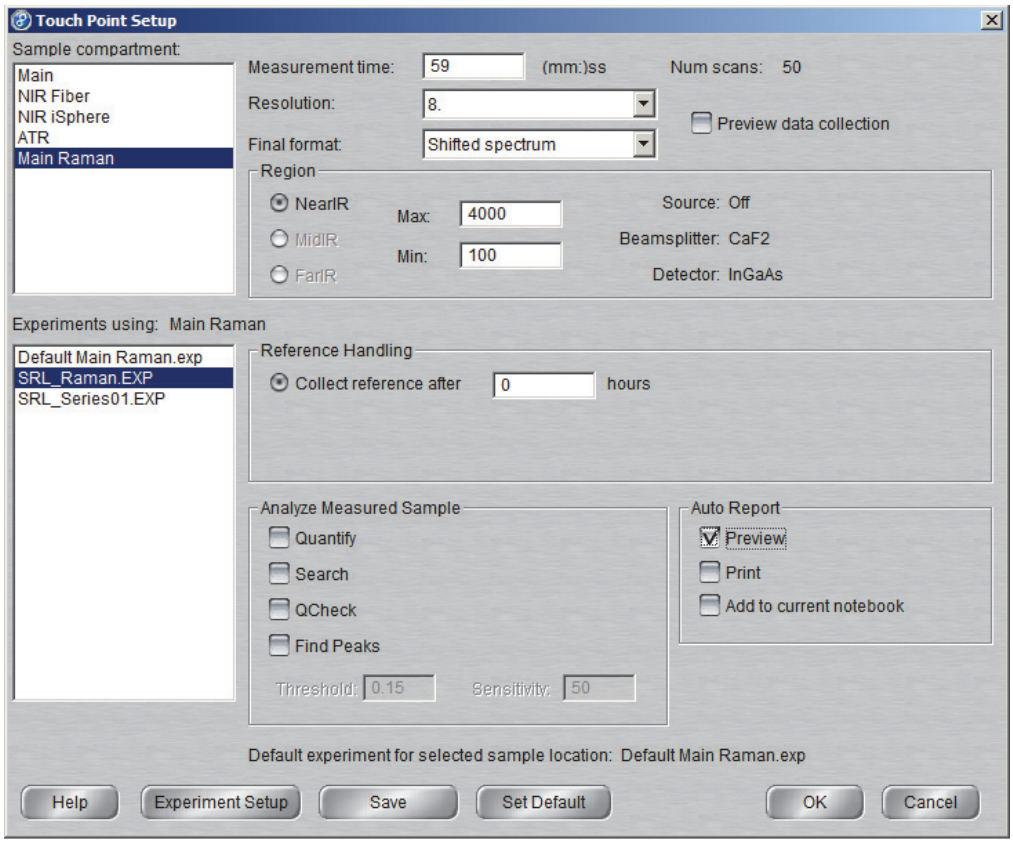
   Figure 11. Touch Point buttons in OMNIC software



4. Choose the **Touch Point Setup button** in the software.

A dialog box is displayed.

Figure 12. Touch Point settings for the iS50 Raman module



Notice that Sample Location (upper left pane) is set to Main Raman, and below that pane is a list of iS50 experiments for the iS50 Raman module. The selected experiment is highlighted and its associated settings are visible in the dialog box.

The Touch Point Setup dialog box provides commonly used settings that define how your Raman data will be collected, displayed, measured and reported. Brief descriptions are provided below.

Table 6. iS50 Raman Touch Point settings

Option	Description
Measurement time	Determines the duration of scanning for sample data collection.
Number of scans	Shows the scans to be collected for the selected measurement time. Multiple scans are averaged.
Resolution	Sets the spectral resolution of the data you collect. Typically 8 cm^{-1} or 4 cm^{-1} is appropriate for most Raman applications. The smaller the resolution value, the higher (better) is the resolution. The resolution value, along with the number of scans, affects the collection time.

Table 6. iS50 Raman Touch Point settings

Option	Description
Final format	Determines the Y-axis unit used for the collected data. These options are available: • **Interferogram**. Shows the raw data from the spectrometer. The Y-axis is in volts and the X-axis is in data points. • **Raman spectrum**. Converts the raw data to an emission spectrum (peaks will point up). Shows the amount of light emitted by the sample at each frequency that is measured. • **Shifted spectrum** (default). Converts the X-axis to Raman shift, which is a measure of the difference between the observed spectral bands and the laser excitation wavelength. This is the typical setting for Raman data and is independent of the laser excitation wavelength. • **Corrected spectrum**. Converts the X-axis to Raman shift and corrects for instrument effects. Selecting this option will improve the accuracy of band intensities for experiments where relative intensities of spectral information is important (for example when creating a spectral library). Requires a white light reference spectrum (refer to "Reference Handling" in Raman Help Topics for details).
Preview data collection	Select this option if you want to collect and view (but not save) preliminary data before the start of a sample data collection. This lets you verify that your experimental conditions are correct before collecting the spectrum.
Region	Determines the spectral range of the spectrometer that will be used to collect the data. The spectral range is defined by the type of source, beamsplitter and detector used. The iS50 Raman module collects emission data in the region between 9,500 and 5,000 cm^{-1}, which is in the near-IR range. If Final Format is set to "Shifted Spectrum," the software subtracts the wavelength of the excitation laser from each point in the spectrum. The "shifted" spectrum is displayed in the range between 4,000 and 100 cm^{-1} where the peaks align with the mid-infrared spectrum.

Table 6. iS50 Raman Touch Point settings

Option	Description
Max/Min	Shows the default limits of the Raman module detector's spectral range. The Max/Min limits can be reduced to eliminate spectral regions that provide no additional information about the sample. If **Final Format** is set to Raman Spectrum, start with **Max/Min** set to 9294 cm^{-1} and 5693 cm^{-1}, respectively. If **Final Format** is set to Shifted Spectrum, start with **Max/Min** set to 4,000 cm^{-1} and 100 cm^{-1}, respectively.
Reference handling	Lets you specify a reference spectrum to be used for correcting the Raman sample spectra you collect. Refer to "Reference Handling" in Raman Help Topics for more information. If Final Format is set to "Corrected Spectrum," you will be prompted to select a reference the very first time you collect a Raman sample spectrum.
Analyze Measured Sample	Allows you to select and run analyses on the collected sample spectrum. The analysis results are attached to the spectrum and saved in the spectral (.spa or .spg) file. The following analyses are available: • **Quantify**. Finds the concentrations of components in a sample spectrum. For more information, open OMNIC Help Topics and find "Quantifying a Spectrum." To select the quantitative analysis method you want to use to quantify a spectrum, choose **Analyze > Quant Setup**.
	• **Search**. Identifies an unknown material by comparing the unknown sample spectrum with each reference spectrum in the selected Raman libraries to find the spectra that most closely match the unknown. For more information, open OMNIC Help Topics and find "Searching a Spectral Library." Use **Analyze > Library Setup** to select the Raman reference libraries to be searched.
	• **QCheck**. Compares spectra to determine their degree of similarity, expressed as a correlation value from 0.0 (no similarity) to 1.0 (the spectra are identical). For more information, find "Comparing Spectra with QCheck" in OMNIC Help Topics. Use **Analyze > QCheck Setup** to set up the comparison.

Table 6. iS50 Raman Touch Point settings

Option	Description

	• **Find Peaks**. Identifies peak locations in a spectrum by finding peaks whose Y values exceed a specified threshold value. The peaks are labeled with their X values. For more information, find "Finding Peaks Above a Specified Height" in OMNIC Help Topics.
	  • **Threshold**. The Y value above which peaks can be found.
	  • **Sensitivity**. Determines how readily shoulders on peaks and small peaks in the baseline will be found.

Auto Report	Can be used to automatically display, print or save an auto report of the analysis results. For more information, find "Working with Auto Reports" in OMNIC Help Topics. • **Preview.** Displays the auto report after the analysis is completed. • **Print.** Prints the auto report after the analysis is completed. • **Add to current notebook.** Adds the auto report to the current report notebook. For more information, find "Adding a Report to a Notebook" in OMNIC Help Topics.
Default experiment	Shows the file name of the default experiment for the selected sample location.

5. Select an appropriate experiment in the "Experiments Using: Main Raman" pane.
6. Set the Touch Point Setup options as desired.
7. If you want to overwrite the default settings, choose **Set Default**.
8. Choose **Save**.

Note

- If you need to specify other experiment settings, choose **Experiment Setup**, set the options as desired, choose **Save** and then choose **OK** to close Experiment Setup. For more information, choose Help from Experiment Setup.
- Some settings appear in Experiment Setup and in Touch Point Setup. Setting these options in one location automatically changes them in the other.

9. Choose **OK** to close Touch Point Setup.

Related Topics

- Installing the Raman Module
- Installing a Sample Holder
- Measuring a Tablet using the iS50 Raman Module
- Aligning the Interferometer
- Removing a Sample Holder
- Setting a Timer to Turn Off the Laser

Measuring a Tablet using the iS50 Raman Module

The following example is a basic Raman experiment that analyzes a common pharmaceutical tablet.

Note For the purposes of this example experiment, the µView software and tools are used to display and interact with the video image of the sample and the OMNIC software is used to control the laser, focus the sample and collect the sample data. You may choose to use the Atlµs software to display a video image of the sample and control the sample stage.

Before You Begin

- Power on the spectrometer and allow the instrument to stabilize. If your spectrometer has been powered off for more than 20 minutes, this will take at least an hour.
- Install the Raman module.
- Start OMNIC software if it is not already open.
- Have your sample material at hand. For the collection settings in the default workflow, we recommend that you choose one of the following items for your sample:
  - a coated or uncoated over-the-counter pharmaceutical tablet such as a pain medication
- Remove any previous sample from the Raman sample compartment.

WARNING Avoid laser hazard. Keep all magnets and strong magnetic fields (such as those produced by mass spectrometers and some kinds of medical equipment) away from the Raman module, and do not attempt to defeat the safety interlocks on the module housing and sample compartment cover—hazardous exposure to laser radiation could occur.

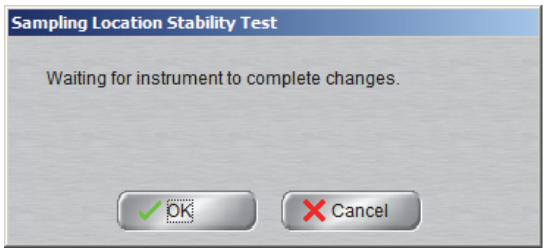
❖ To measure a tablet sample by Raman (watch video)

1. Press the Raman Touch Point to configure the instrument for Raman sampling and load the default Raman workflow.

   Figure 13. Sample compartment Touch Point

   A message may be displayed while the instrument collects scans to determine stability with the new configuration.

2. Wait until the stability test is completed, or choose **OK** to skip that test or **Cancel** to stop all instrument tests.

3. If your Raman workflow is not configured, follow the steps in "Configuring Your Raman Workflow" to configure it.

   NOTICE 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 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.

4. Prepare the sample.

   a. Place the sample on one of the provided sample holders. See "Preparing Samples" for more information.

   b. Open the Raman sample compartment door.

c. Wait until the motors stop moving and then place the sample holder on the sample stage. See "Installing a Sample Holder" for details.

Figure 14. Sample holder on iS50 Raman sample stage

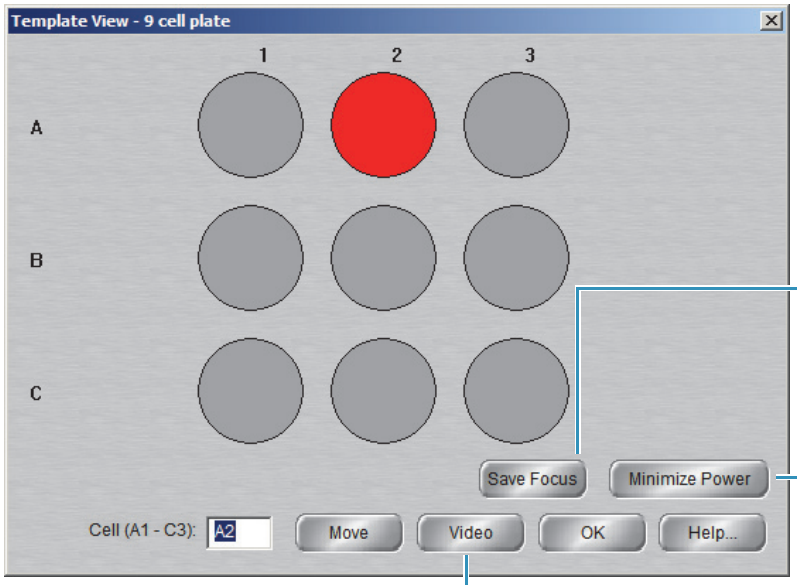
d. Close the Raman sample compartment door.

5. Select a template that matches your sample holder.

In OMNIC software, choose **Raman > Select Template** and select a template. For example, select "9 cell plate" in the software for the sample holder shown above.

The Template View window shows you an image of the selected template.

Figure 15. Template View window showing 9 Cell Plate



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.

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

Opens µView software which shows a video image of the selected sample location

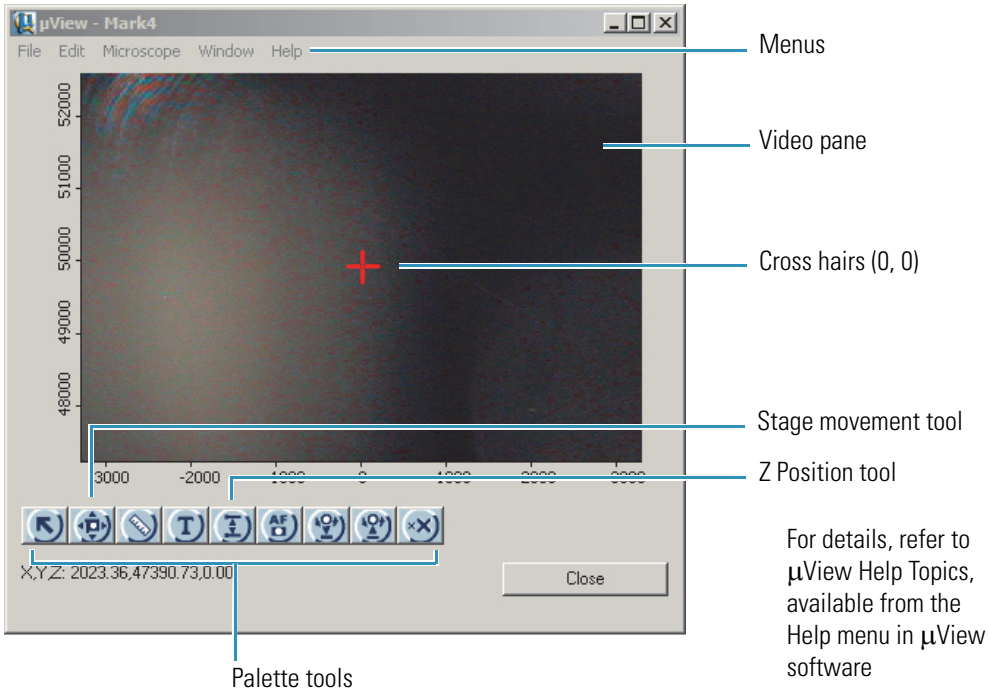
Quickly lowers the laser power to approximately 0.05 W

NOTICE Using Save Focus programs the sample stage to return to the previous focus setting regardless of the installed sample or sample holder. To prevent automatic focusing for a thick sample, open the Raman sample compartment door, install your sample holder, display the correct template and choose **Save Focus** while the stage is still in the loading position. This resets the focus to accommodate the thickest sample (2.5 cm or 1 in).

6. Position your sample under the collection optics.
   a. In Template view, double-click a location on the template that matches the location in the sample holder of the first sample you plan to analyze. You will hear the motors move the sample stage to the proper position.
7. Open µView software by choosing **Video** in the Template View window.

   The µView window has menus, a video pane, and a tool palette with features for controlling the sample stage.

Figure 16. µView window



The video pane displays a live image from the Raman camera.

Note If you can't see an image in the video pane, in OMNIC software choose **Collect > Experiment Setup**, select the **Bench** tab and make sure **Lamp** is selected. This turns on the light in the Raman sample compartment.

Cross hairs mark the center of the pane. The X-axis and Y-axis represent the video field of view in micrometers. When the stage is at its origin point, the axes are oriented such that point 0,0 is in the center of the video pane, the point indicated by the cross hairs.

The palette provides tools for adjusting the Z axis and for annotating your video image.

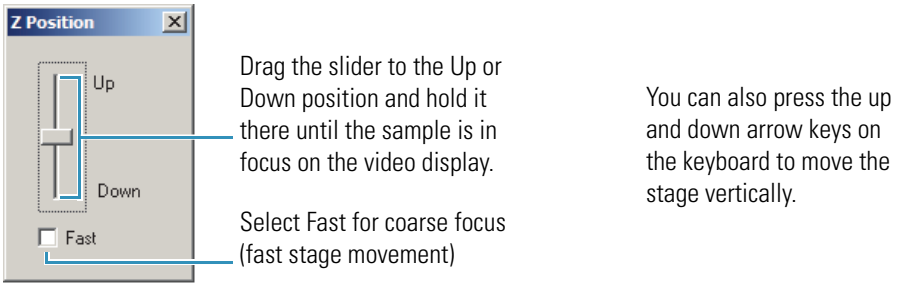
8. Close the Template View window and arrange the OMNIC and µView windows so you can see both windows with minimal overlap.

9. Select the point you want to analyze on your sample.

   a. Focus the sample.

   Note This step is not as critical for defocused sampling.

   In the µView window, choose the Z position tool (up and down arrows in the previous image). A slide control with a speed check box opens.



   Watch the image in the video pane while you use the Z Position tool to bring your sample into visual focus.

   b. Click a point in the video image in the µView window.

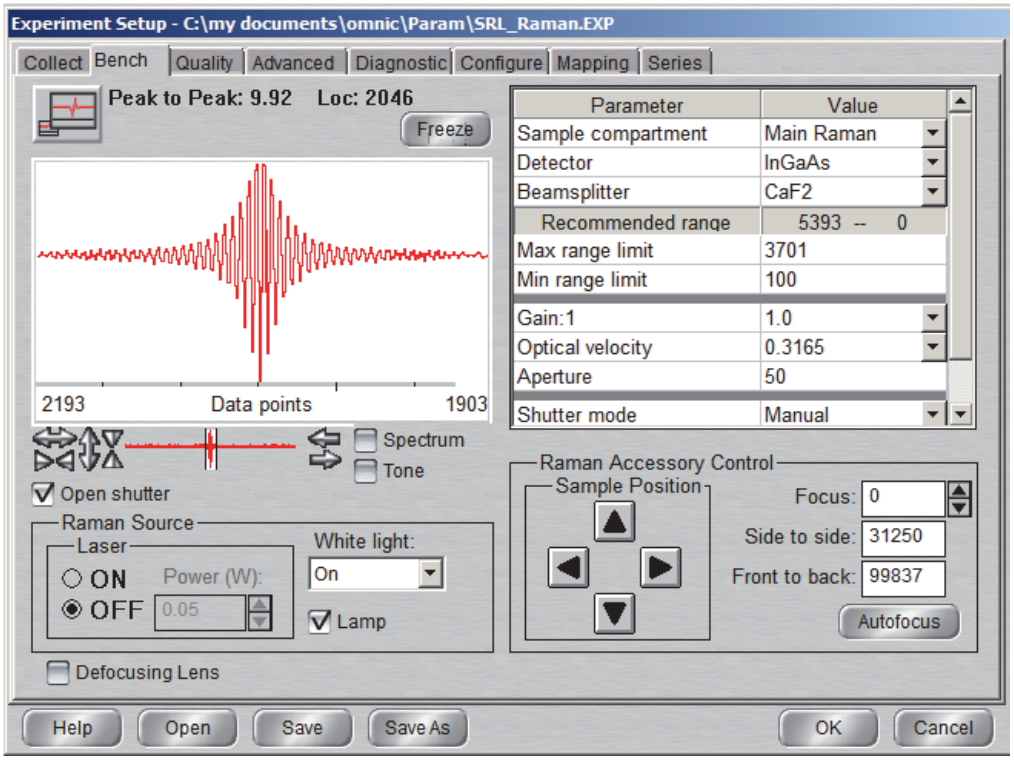
   Select the stage movement tool (cursor is shaped like a target; see the previous image). Each time you click a point in the video image, the stage moves to position that point under the collection optics, the video image is redisplayed with the point at the center, and the video pane axes are adjusted accordingly.

   c. Close the Z Position window (leave the µView window open).

10. Turn on the laser, adjust the laser power and focus the collection optics.

    a. In OMNIC software, choose **Collect > Experiment Setup** and select the **Bench** tab.

Figure 17. Bench tab settings for the iS50 Raman module



After a few seconds, an interferogram resembling the one above should appear in the live display (White Light must be set to On). If you don't see an interferogram, select the **Diagnostic** tab and choose **Reset iS50 Raman**.

> **Note** If Shutter Mode appears in the Parameter list, set it to Automatic (causes the purge shutters to open automatically for Raman data collection).

    b. Set **White Light** to Off.

    c. Set **Laser** to On.

    After a few seconds a bright spot created by the laser should be visible on your sample.

    d. Adjust the laser power to an appropriate level for your sample.

    While you adjust the power, monitor the video image of your sample for signs of decomposition, especially if the sample is a dark color.

NOTICE

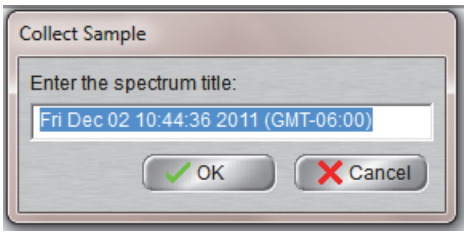
- If your sample starts to decompose, reduce the laser power and then turn off the laser. Move the sample so that the laser beam will strike an area that is not decomposed and then turn the laser on. If the sample starts to decompose again, repeat these steps or use a defocusing lens, but be aware that some samples may be too sensitive to laser energy to be suitable for analysis.
- To maximize the life of your laser, do not leave the laser on for extended periods when not in active use.

e. Choose **Autofocus**.

f. When Autofocus is completed, choose **OK** to close Experiment Setup.

11. Collect the spectrum.

a. In OMNIC software, choose **Collect > Collect Raman**. A message asks you to enter a spectrum title.



The white light is turned off during dat collection and back on when data collection is completed

b. Enter a title for your sample spectrum and choose **OK** (or choose OK to accept the default title).

A message tells you to prepare for the sample collection.

c. Choose **OK** to collect the sample data.

If your spectrometer has the purge shutter option, a message may appear asking you to wait a few seconds for the purge to stabilize. After the delay period has passed, data collection begins. The collected data appear in the OMNIC main window.

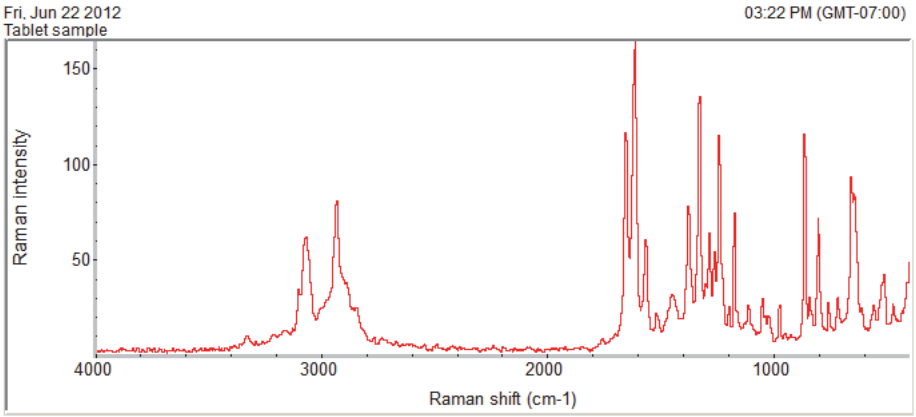
When the spectrum is completed, a message asks whether to add it to a window.

d. Choose **Yes** to add the spectrum to the selected window and complete the workflow.

If your workflow specifies an analysis such as quantify or search, those steps occur automatically. To see the analysis results, choose the "r" button to the left of the title box above the OMNIC spectral window.

The sample spectrum and its associated analysis results are stored in a virtual auto report. If your workflow is set up to preview or print auto reports, the report is displayed or printed using the default printer. Below is an example report showing a Raman spectrum of a pharmaceutical tablet taken with the iS50 Raman module.

Figure 18. Auto report showing an Raman spectrum of a pharmaceutical tablet taken with the iS50 Raman module



12. Turn off the laser.
    a. Choose **Collect > Experiment Setup** and select the **Bench** tab.
    b. Set **Laser** to Off (or minimize the laser power if you plan to collect another spectrum).
13. To analyze another point on the same sample, repeat steps 9 through 12.

    To analyze another sample in the same sample holder, repeat steps 6 through 12.
14. If you are finished collecting Raman data, follow these steps:
    a. On the **Bench** tab, set **Laser** to Off.
    b. Open the Raman sample compartment door, wait for the stage to stop moving and remove the sample holder.
    c. Close Experiment Setup and µView software.

Tip For convenience, set up the laser to turn itself off after a specified period of inactivity or after OMNIC software closes.

Related Topics

- Installing the Raman Module
- Verifying Raman Module Performance
- Configuring Your Raman Workflow
- Installing a Sample Holder
- Adjusting the Sample Position
- Turning on the Laser and Adjusting the Laser Power
- Using the Defocusing Lens to Adjust the Beam Size

- Preventing Detector Saturation
- Aligning the Interferometer
- Removing a Sample Holder
- Setting a Timer to Turn Off the Laser

Removing the Raman Module

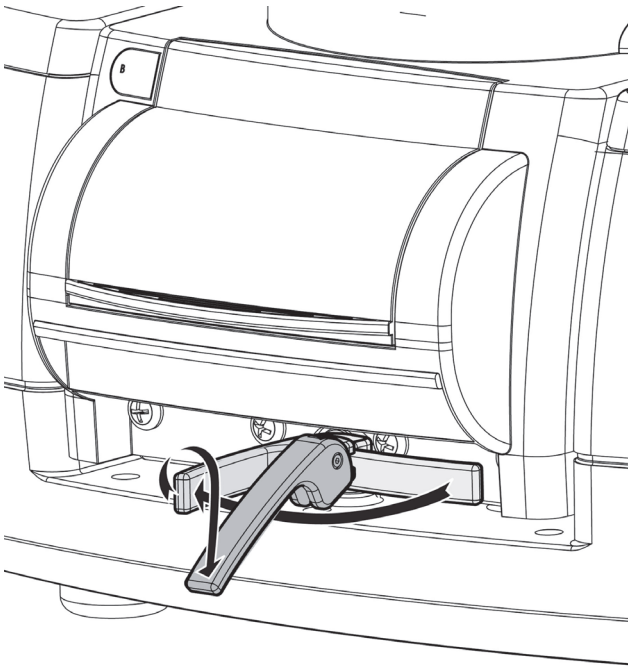
❖ **To remove the Raman module**

1. Open the Raman sample compartment door and remove any sample holder or sample from the sample stage.
2. Turn off the excitation laser.

   To do this, in OMNIC software, choose **Collect > Experiment Setup**, select the **Bench** tab and set **Laser** to Off.
3. Exit OMNIC software.
4. Release the spring-loaded latch handle at the base of the Raman module by pulling the handle towards you.

   This disengages the electrical connector at the back of the Raman module. After you release the latch handle, it snaps to it's vertical position.

   Figure 19. Release iS50 Raman latch handle

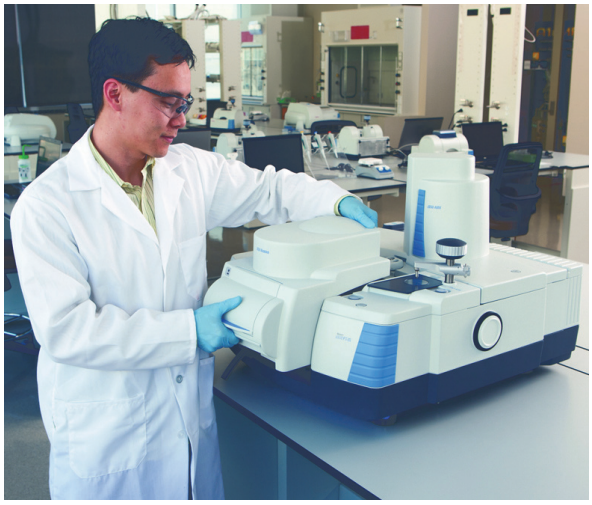


5. Gently slide the module forward.

NOTICE Avoid scraping the electrical connector on the Raman module back panel against the back wall of the sample compartment as you lift the Raman module out of the sample compartment.

6. Grasp the hand holds at the front and back edges of the Raman module and lift the module up and out of the sample compartment.

   Figure 20. Remove iS50 Raman module

7. Install the sample compartment cover for the spectrometer and the Snap-In baseplate, if it was removed when you installed the Raman module.

   Refer to "Using Your Spectrometer" in Spectrometer Help Topics or the *Nicolet iS50 User Guide* for instructions.

8. Install any optional filters or energy screens that were removed from the left or right side of the spectrometer sample compartment when you installed the Raman module.

   For more information, find "Installing or Removing Optional Hardware" in Spectrometer Help Topics or your *Nicolet iS50 User Guide*.

9. Remove the CaF_2 or XT-KBr beamsplitter, if necessary, and install the appropriate beamsplitter for the next application.

   For more information, find "Using Beamsplitters" in Spectrometer Help Topics or in your *Nicolet iS50 User Guide*.

10. If you installed a new beamsplitter, align the interferometer.

    See "Aligning the Interferometer" in Spectrometer Help Topics or your *Nicolet iS50 User Guide* for instructions.

Note The video USB cable can be left connected or removed. The cable connects the Video port on the back of the spectrometer to a USB port on the computer.

Related Topics

- Removing a Sample Holder
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

Doing Experiments with Macros

Note If your spectrometer is equipped with optional purge shutters, for best results, open the right purge shutter while the Raman module is collecting data. For more information, find "Controlling the Purge Shutters" in OMNIC Help Topics. (If Shutter Mode is set to Automatic on the Bench tab in Experiment Setup, the purge shutters will open automatically for Raman data collection.)

A macro is small computer program that allows you to automate operations and functions such as performing sample stage experiments.

To write and use macros, you must have either the Macros\Basic™ or Macros\Pro™ software. Macros\Basic automates many of the tasks involved in building macros, while Macros\Pro is a more powerful program that allows advanced users more flexibility and choices when building macros.

To order Macros\Basic or Macros\Pro, contact our local sales representative. For information about building macros and using Macros\Basic or Macros\Pro, refer to the user documentation provided with the software.

Related Topics

- Compatible Software

Maintenance

This section describes maintenance routines that you can perform on the iS50 Raman module to keep it running efficiently. These topics are covered:

- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage

- Cleaning the Residue Tray
- Storing the Raman Module

WARNING Avoid a laser hazard. Do not remove the main cover of the Raman module. Exposure to harmful radiation may result. There are no user serviceable parts inside.

Aligning the Interferometer

Align the interferometer after you install the Raman module for the first time, and once a week during continuous use. Alignment ensures that the system is capable of producing the strongest possible signal from the sample. We recommend that you keep a weekly log of the signal intensity produced by your system under the same conditions. This allows you to track the performance of the system over time.

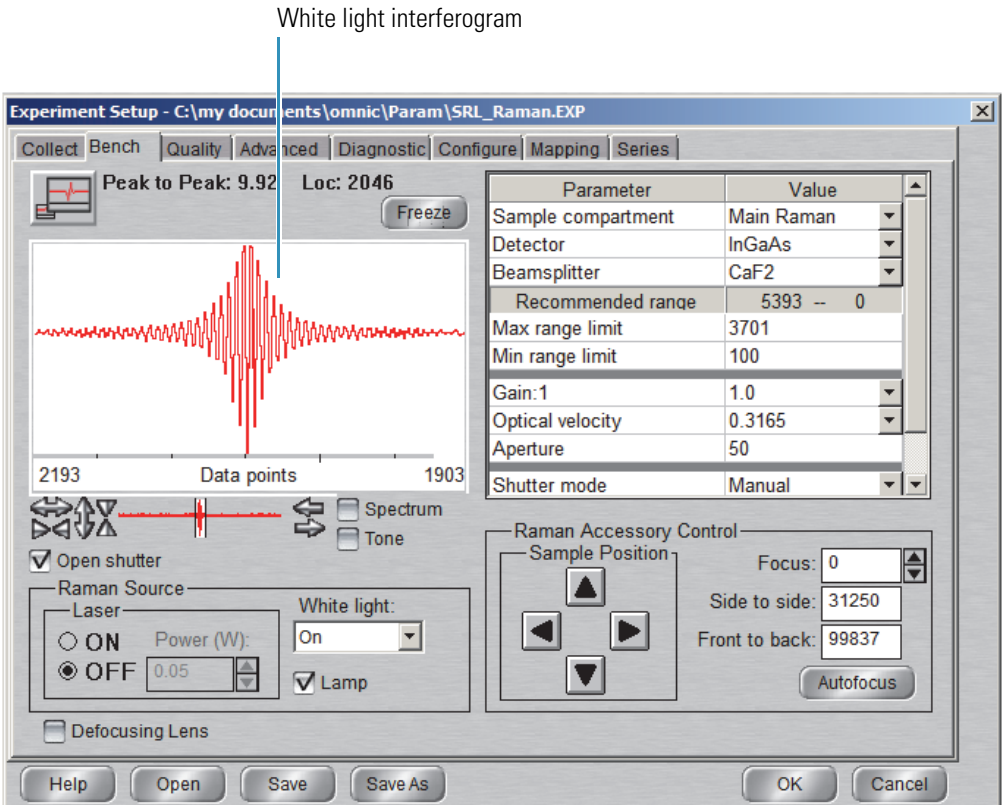
Before you perform an alignment, make sure the spectrometer and computer are powered on, the spectrometer has stabilized for at least 20 minutes, and the Raman module and the proper beamsplitter are installed. For detailed instructions, see "Installing the Raman Module."

❖ To align the interferometer

1. Start the OMNIC software.
2. Press the sample compartment Touch Point to configure the system for Raman sampling.

3. Choose **Collect > Experiment Setup** and select the **Bench** tab.

Figure 21. Bench tab settings in Experiment Setup



4. Set the following features.

Table 7. Spectrometer alignment settings

Feature name	Setting
Gain	1.0
Velocity	0.3165
Aperture	50
Laser	Off
White light	On
Spectrum	Off
Tone	Off

For more information, find “Experiment Setup” in OMNIC Help Topics.

Note Do not close Experiment Setup. If you inadvertently close it, choose Experiment Setup again and make sure the features are set correctly.

5. If an interferogram is visible on the Bench tab (see the previous image), select the **Diagnostic** tab and choose **Align** to perform an automatic alignment of the interferometer.

   If no interferogram is visible on the Bench tab, select the Diagnostic tab, choose **Reset iS50 Raman** and then choose **Align**.

6. When the alignment is finished, note the signal intensity in your Raman alignment log.
7. Choose **OK** to close Experiment Setup.

Related Topics

- Configuring the Raman Module
- Installing the Raman Module
- Verifying Raman Module Performance
- Initializing the Sample Stage
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

Verifying Raman Module Performance

A good way to verify the performance of your iS50 Raman module is to regularly collect the spectrum of a known material and compare the spectra over time. We suggest running a performance test each time you install the Raman module or once a week. We provide a thick polystyrene sample with the Raman module for this purpose. The sample is a clear plastic disk about 2.5 cm (1 in) in diameter and about 0.5 cm (3/16 in) thick.

Make sure you use the proper settings for the collection parameters each time you measure the polystyrene sample.

❖ To check the Raman module performance

1. Place the polystyrene sample disk provided with the Raman module on the sample stage.
2. Close the sample compartment door.
3. In OMNIC software, choose **Collect > Experiment Setup** and set the following features:

   On the **Collect** tab:

   - Number of Scans: 50

- Resolution: 8 cm^{-1}
- Final Format: Shifted Spectrum

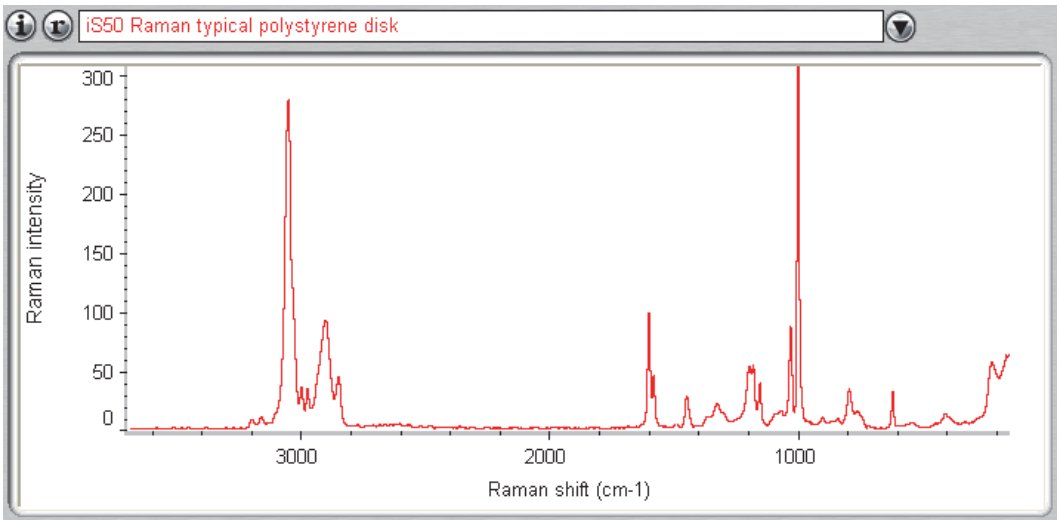
On the **Bench** tab:

- Sample compartment: Main Raman
- Detector: InGaAs
- Beamsplitter: CaF_2 or XT-KBr
- Max range limit: 3702 cm^{-1}
- Min range limit: 100 cm^{-1}
- Gain: 1.0
- Optical velocity: 0.3165 cm/s
- Aperture: 50
- Shutter mode: Automatic (if your spectrometer has purge shutters)
- Lamp: On
- Defocusing Lens: Cleared (off)

4. Follow the instructions in "Measuring a Tablet using the iS50 Raman Module" to select a sampling point, focus the optics and collect the sample spectrum (set Laser Power to 0.5 W).
5. When you are finished, turn off the laser and remove the sample from the Raman sample compartment.
6. Store the polystyrene sample in its original shipping container.

Here is an example of a typical Raman spectrum of a typical polystyrene disk. The relative shape and peak intensities of your spectrum should look similar to the one below, but they will not look exactly like this one due to differences in the spectrometer and Raman module components and other factors.

Figure 22. iS50 Raman module typical polystyrene disk spectrum



Related Topics

- Configuring the Raman Module
- Installing the Raman Module
- Aligning the Interferometer
- Initializing the Sample Stage
- Measuring a Tablet using the iS50 Raman Module
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

Initializing the Sample Stage

The sample stage will automatically initialize when you install the Raman module or press the sample compartment Touch Point when the Raman module is installed. If the movement of the stage is blocked, remove the blockage and then reset the stage.

NOTICE To avoid damaging delicate optics, always remove any samples from the sample stage before starting OMNIC software or initializing the sample stage.

To initialize the sample stage, you can either exit and restart OMNIC software or follow the steps below.

❖ To initialize the sample stage

1. Choose **Collect > Experiment Setup**.
2. Select the **Diagnostic** tab.
3. Choose **Reset iS50 Raman**.

   This initializes the motors for the sample stage, diffuser and neutral density filter and confirms or resets the Raman signal.

Related Topics

- Aligning the Interferometer
- Verifying Raman Module Performance
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

Cleaning the Raman Module Housing

NOTICE

- Do not pour liquids directly onto the Raman module or the quartz window on the right side of the module.
- Do not use harsh detergents, chemicals or abrasives to clean the surface of the Raman module; these can damage the finish.
- Avoid spilling samples or solvents in the Raman sample compartment. Clean the sample stage and residue tray regularly; see "Cleaning the Residue Tray" for instructions.

❖ To clean the Raman module housing

Use a damp (not wet), soft cloth and a mild soap to clean the cover of the Raman module. Dry it with a clean cloth.

Related Topics

- Aligning the Interferometer
- Verifying Raman Module Performance

- Initializing the Sample Stage
- Cleaning the Sample Stage
- Cleaning the Residue Tray
- Storing the Raman Module

Cleaning the Sample Stage

The sample stage moves samples into and out of the laser beam and is close to sensitive motors and optics. If the stage assembly becomes clogged with debris, or the mirrors in the Raman sample compartment become soiled or damaged, they will affect your Raman results.

Follow the precautions below when working with the sample stage. If the stage platform is soiled, following the instructions below to clean it. If a stage component or the stage mirror appears soiled or damaged, contact our sales or service representative in your area, or use the information at the beginning of this document or help system to contact us.

NOTICE

- Always place samples on a sample holder and then load the holder on the sample stage. Do not place 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 2.5 cm (1.0 in). Materials above the maximum height may break inside the module or damage delicate mirrors and motors located above or behind the stage.

❖ To clean the sample stage

Wipe the surface of the stage platform with a soft, damp (not wet) cloth and a mild detergent. Do not use harsh abrasives or detergents.

NOTICE Do not allow moisture or cleaning products to come into contact with any part of the interior of your Raman sample compartment. Do not attempt to clean any internal components besides the sample stage, and do not touch any optical surface inside the Raman sample compartment.

Related Topics

- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage
- Cleaning the Raman Module Housing
- Cleaning the Residue Tray
- Storing the Raman Module

Cleaning the Residue Tray

A residue tray is located under the sample stage in the Raman module sample compartment. The tray collects materials they may have fallen from the sample stage. Clean the tray regularly with a mild detergent and rinse with water to prevent these materials from interfering with the sample stage movement.

❖ To remove the residue tray

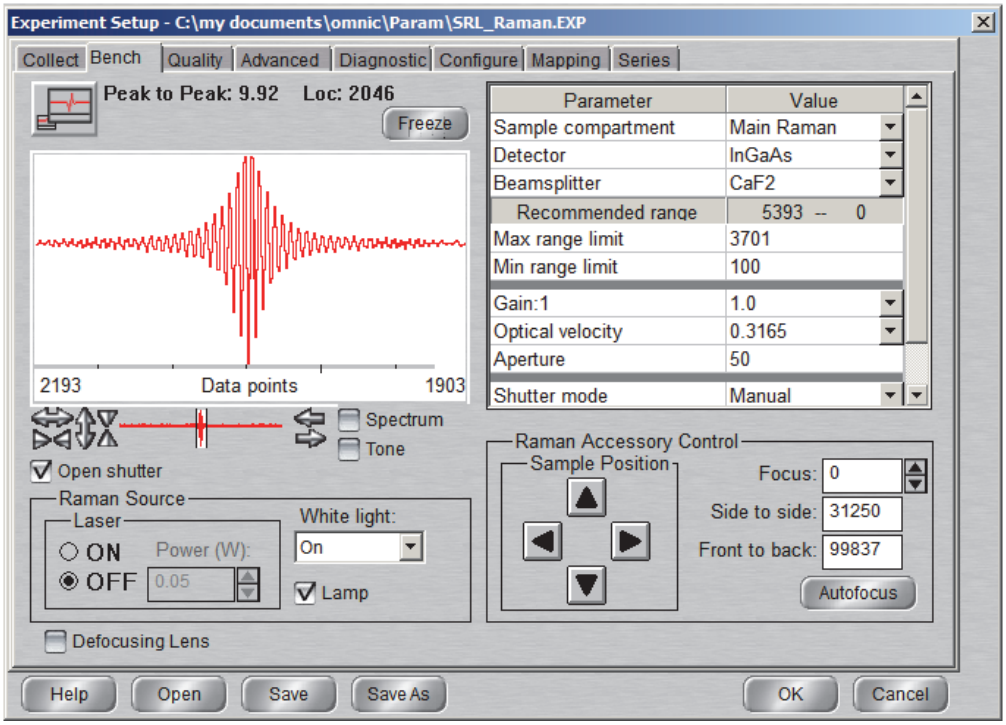
1. Open the Raman sample compartment door.

   The sample stage automatically moves all the way down and forward.

2. Remove any sample holder or sample from the sample stage.

3. In OMNIC software, choose **Collect > Experiment Setup** and select the **Bench** tab.

   Figure 23. Bench tab features of Experiment Setup

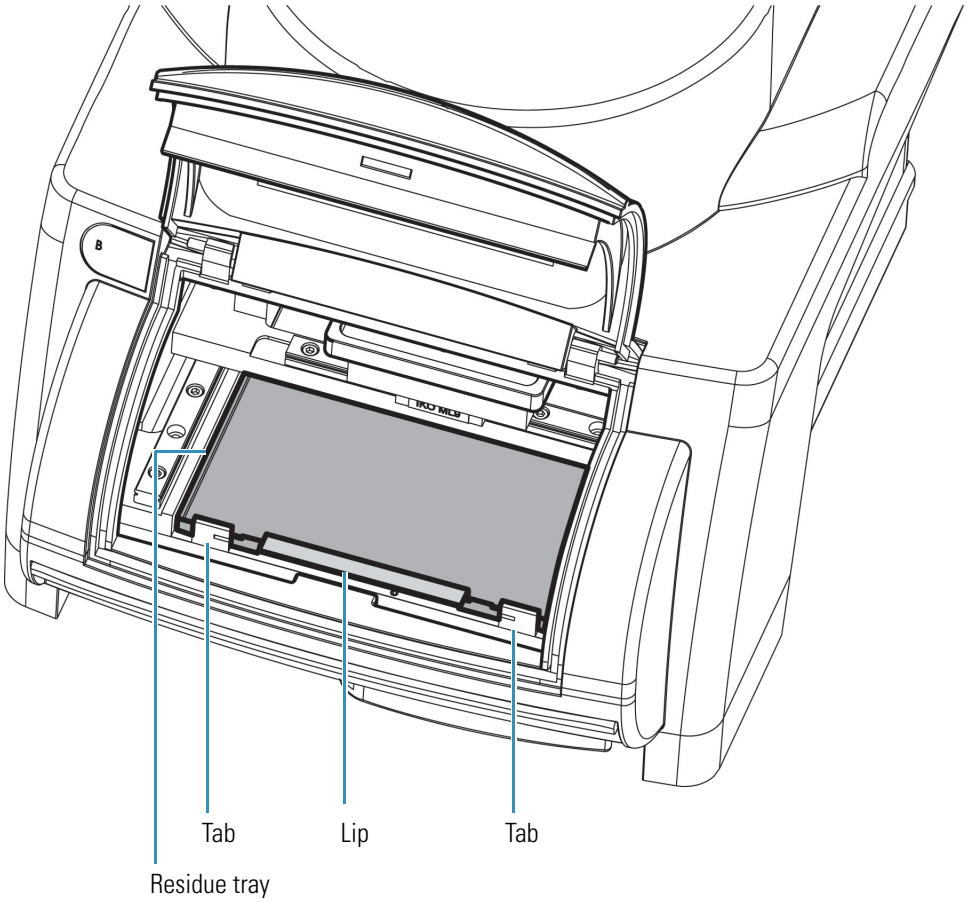


4. Set **Front To Back** to 0 and press **Enter** to move the stage all the way back.

5. Set **Focus** to 20,000 and press **Enter** to move the stage up and out of the way.

6. Press the lip of the tray back firmly to release it from the both tabs, and then lift the tray up and slide it out of the sample compartment.

Figure 24. Remove residue tray



❖ To replace the residue tray

1. Hold the residue tray by the front edge (it has a generous lip).
2. Slide the tray under the sample stage and press the lip down firmly until the tray snaps in place.

> **NOTICE** May sure the tray is firmly seated and lays flat against the bottom of the sample compartment before you close the sample compartment door or move the sample stage.

3. Close the Raman sample compartment door.
4. Initialize the sample stage.

   Choose **Collect > Experiment Setup**, select the **Diagnostic** tab and choose **Reset iS50 Raman**.

Related Topics

- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Storing the Raman Module

Storing the Raman Module

When the Raman module is not in use, protect the electrical connector on the back panel. Store the module right side up.

Related Topics

- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage
- Cleaning the Raman Module Housing
- Cleaning the Sample Stage
- Cleaning the Residue Tray

Troubleshooting

The troubleshooting information will help you solve problems you may have with the iS50 Raman module. The information is divided as follows:

Hardware Problems	Steps you can take to resolve problems with the Raman module.
Applications Problems	How to fix problems in the collected spectra.
Error Conditions	How to deal with error messages you may see.
Advanced Diagnostics	Runs a standard performance test

To expedite and assist in problem diagnosis, please be prepared to send us a copy of your diagnostics log file. The diagnostics log includes a history of important system parameters and events that occur during system operation. To copy the diagnostics log to your Windows® desktop, start OMNIC software and choose **File > Copy Diagnostics File to Desktop**. You can attach the file to an e-mail message to our technical support representative.

WARNING When performing any of the procedures in the troubleshooting section, always follow the safety precautions included in this document or Help system and in any safety guides that came with your instrument or the iS50 Raman module.

In addition, a diagnostics program is provided with your system for checking the performance of accessory components in case a problem occurs (see "Advanced Diagnostics" for details).

WARNING Avoid laser hazard. Do not remove the main cover of the Raman module. Exposure to harmful radiation may result. There are no user serviceable parts inside.

Hardware Problems

This section lists common problems with Raman module components and how to resolve them.

Raman Module scans but no sample interferogram is displayed

If the system does not display an interferogram during data collection, consider whether the sample is compatible with the Raman application. If you don't believe that is the problem, follow these steps:

1. Choose **Collect > Experiment Setup**.
2. Select the **Diagnostic** tab and choose **Reset iS50 Raman**.
3. If the interferogram doesn't appear on the Diagnostic tab, open µView or Atlµs software and verify that the sample is in focus.
4. If an interferogram still doesn't appear on the Diagnostic tab, select the **Bench** tab in Experiment Setup and make sure **Aperture** is set to 80 or higher.
5. If an interferogram doesn't appear on the Bench tab, set **Laser** to Off and set **White Light** to On.
6. If a white light interferogram appears on the Bench tab, your sample does not scatter Raman energy from the laser. Try another application or sampling technique.

   If no white light interferogram appears, remove the Raman module and align the spectrometer.

   a. On the Bench tab, set **Beamsplitter** to CaF2.
   b. Set **Detector** to Top DTGS.
   c. Select the **Diagnostic** tab.
   d. Choose **Align**.
7. If no interferogram appears, select the **Diagnostic** tab and choose **Reset Bench.**

8. If there is still no interferogram, contact our local service representative.

Related Topics

- Applications Problems
- Error Conditions
- Advanced Diagnostics
- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage

Applications Problems

Noisy spectra

If your spectra are abnormally noisy, follow these steps to troubleshoot the problem:

1. Make sure the CaF_2 or XT-KBr beamsplitter is installed.

   The standard KBr beamsplitter will produce a signal, but the signal will be low in amplitude and collected spectra will be noisy.

2. If the spectra are still noisy, complete these steps:

   a. Choose **Collect > Experiment Setup** and select the **Bench** tab.

   b. Set **White Light** to On.

   c. Set **Power** to 0.5 W.

   d. Install the provided polystyrene test sample and close the sample compartment door.

   e. Focus the sample.

   f. If an interferogram is present on the Bench tab, select the **Diagnostic** tab and choose **Align**.

3. If the problem persists after alignment is completed (or if no interferogram is present after you complete step d above), proceed to the next step.

4. Make sure the adjustable aperture is fully open by selecting the **Bench** tab and setting **Aperture** to its maximum value (approximately 150).

5. If an interferogram is present but the spectra are still noisy, contact our local service representative.

   If an interferogram is still not present, repeat step 2.

If there is still no interferogram, select the **Diagnostic** tab and do the following in this order:

a. Choose **Reset Bench**.

b. Choose **Align**.

If the problem continues, contact our local service representative.

Related Topics

- Hardware Problems
- Error Conditions
- Advanced Diagnostics
- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage

Error Conditions

Loss of power

The Raman module delivers 0.5 W of power to the sample position.

❖ **To recover after loss of power**

1. Remove the Raman module from the spectrometer sample compartment.
2. Select the main Top DTGS detector.
3. Verify that the interferogram is the usual amplitude.
4. Align the spectrometer if necessary.
5. Reinstall the Raman module.
6. Insert the standard polystyrene sample and focus it.
7. Turn on the laser and collect a Raman spectrum.
8. If you have ValPro, run the qualification test.

Interferogram peak is not centered

1. Choose **Collect > Experiment Setup**.
2. If the interferogram peak is not centered in the live display, select the **Diagnostic** tab and choose **Reset Bench**.

   If the peak is still not centered, contact our local service representative.

Related Topics

- Hardware Problems
- Applications Problems
- Advanced Diagnostics
- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage

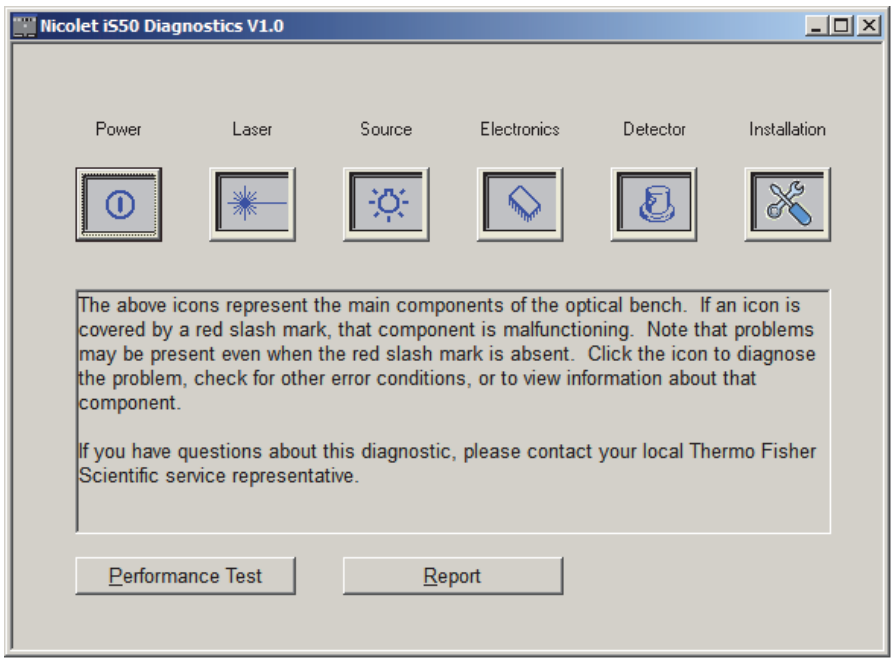
Advanced Diagnostics

If your system is not performing properly, run the following test:

1. Place the thick polystyrene sample provided with the Raman module on one of the provided (metal) sample holders and install the sample holder in the Raman sample stage.
2. In OMNIC software, choose **Collect > Advanced Diagnostics**.

   A diagnostics window is displayed.

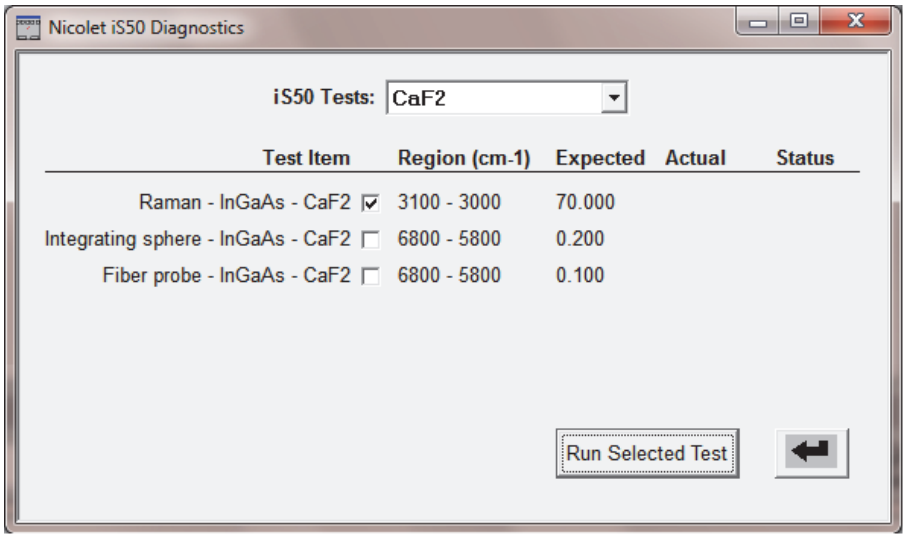
   Figure 25. OMNIC Diagnostics window



3. Choose **Performance Test.**

   A performance test window is displayed.

   Figure 26. Nicolet iS50 Diagnostics window



4. Set **iS50 Tests** to CaF2.
5. Select **Raman - InGaAs - CaF2**.
6. Choose **Run Selected Test**.

   The software collects a spectrum from the polystyrene sample and calculates the signal-to-noise ratio. The test result appears in the Actual column and a pass or fail result appears in the Status column.

   If your Raman module fails this test, contact our local service representative for assistance.

Related Topics

- Hardware Problems
- Applications Problems
- Error Conditions
- Aligning the Interferometer
- Verifying Raman Module Performance
- Initializing the Sample Stage