Raman Module with OMNIC Paradigm software



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WARNING

Avoid an explosion or fire hazard.

This instrument or accessory is not designed for use in an explosive atmosphere.

Raman Module

This section describes the optional Thermo Scientific Raman Module, which allows you to measure FT-Raman spectra with your Nicolet[™] iS[™]50 spectrometer. The module is installed in the spectrometer's sample compartment.



Figure 1-1: Nicolet iS50 spectrometer with the Raman Module installed

CONVENTIONS USED IN THIS DOCUMENT

DANGER



Avoid hazard. Indicates a hazardous situation which, if not avoided, will result in serious injury or death.

WARNING



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

CAUTION



Avoid hazard. 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 Raman Module

The 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

The Raman Module works with our security administration software and allows digital signatures and IQ-OQ-PQ-DQ certification. Please refer to your Security Suite software documentation for more information. For information about additional sampling modules for your spectrometer, contact our sales representative in your area.

Important features

Figure 1-2: Raman Module features



A	Raman sample compartment Touch Point. Configures the spectrometer for Raman sampling and shows the status of the Raman Module.
В	Raman sample compartment. Allows access to the Raman sampling area and sample stage. A laser emission indicator tells you when the laser beam is present in the sample compartment. Two independent safety interlocks ensure that the laser beam is blocked when the sample compartment door is open.

С	Sample holder. The stage accommodates a variety of sample holders that can be quickly interchanged.
D	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 a built-in video camera allow precise positioning for very small samples.
E	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 <u>Turning on the laser and adjusting the laser power</u> .

The Touch Point LED has three states, described below.

Table 1-1:

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

Note Two independent safety interlocks ensure that 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.

Back panel features

- 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 1-3: Raman Module back panel

Specifications

Feature	Specification
Excitation source	1064 nm laser Maximum power: 0.5 W

Feature	Specification
Reference interferogram source	White light
Beamsplitter	Calcium fluoride (CaF2) or extended range potassium bromide (XT-KBr), (located in the spectrometer)
Detector	Dedicated Indium Gallium Arsenide (InGaAs), (located in the spectrometer)

Compatible software

Table 1-3:

Software used with the Raman Module

Software	Use for
OMNIC Paradigm	Sample identification and processing your Raman spectra. For more information, see <u>Your first experiment</u> . You can also consult the OMNIC Paradigm software guide in our online <u>knowledge base</u> for additional and advanced instructions.
TQ Analyst	Creating quantitative and classification methods that can be used with OMNIC Paradigm software. TQ Analyst provides an extensive suite of chemometrics features you can use to identify raw materials, perform quantitative analysis and take spectral measurements. For more information, refer to the help system in TQ Analyst software.
Security Suite	Security Suite provides software for security administration, audit management, access control, and event logging.

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.

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 <u>Maintenance</u>.
- 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 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 <u>Cleaning, removing, and replacing the residue tray</u> 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.

FT-Raman sampling

Monochromatic light from the excitation laser in the 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 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.

Compatible samples

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

Table 1-4: Samples compa	tible with the Raman Module
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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

Several styles of sample holders are provided with the Raman Module for fast, reproducible sampling of solids, liquids, powders, and gels. For more information, see <u>Preparing samples</u>.

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 <u>Cleaning, removing, and replacing the residue tray</u> for instructions.

^aReduce 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.

^bSprinkle 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.

^cPlace 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.

Installing the Raman Module

This section explains how to install the Raman Module in the sample compartment of your spectrometer.

Note You will need to configure the Raman Module the first time it is used with OMNIC Paradigm 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).
- 2. Remove any optional filters or energy screens that may be installed on the left or right side of the spectrometer sample compartment.

NOTICE

Avoid scraping the electrical connector on the Raman Module back panel against the back wall of the sample compartment as you lower the 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 1-4: Installing the 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, then firmly press the handle toward the module.

Figure 1-5: Securing the Raman Module front latch



Note If the Microscope Configuration dialog box is displayed, the Raman Module is not configured. See <u>Configuring the Raman Module</u> for more information.

7. If beamsplitters must be installed manually for your spectrometer, install the CaF2 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 and select your measurement settings.

Note If your spectrometer is equipped with optional purge shutters, for best results, open the right purge shutter while the module is measuring data.

Configuring the Raman Module

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

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. Install the module in the spectrometer sample compartment. See Installing the Raman Module.
- 3. Start OMNIC Paradigm software.
- 4. If beamsplitters must be installed manually for your spectrometer, install the CaF2 or XT-KBr beamsplitter in the spectrometer beamsplitter compartment.

If you have the optional ABX (automatic beamsplitter exchanger), the correct beamsplitter installs automatically after you install the Raman Module and select your measurement settings.

5. 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 <u>Aligning the</u> <u>interferometer</u> for instructions and information about how often to perform this step.

Operation

This section covers general operation of the Raman Module, including sampling, adjusting laser power, adjusting the sample position, and setting a timer to turn off the laser.

- 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 spectrometer includes the 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.



OMNIC Paradigm software FT-Raman acquisition tab

To access the laser control and optics features, open OMNIC Paradigm software and select **Start Session**. This opens the acquisition tab opens and displays the Raman acquisition view, providing access to a number of settings on the left side of the screen:

Figure 1-6: Raman light and laser settings

	C Paradigm		
File	Acquire Data	Display	Process
М	icroscope	Measu	rement
Camera View	Capture Mosaic	Live Spectrum	Sample
Dashboard	iS50 Raman 🗙		
Defo Whi Lase	cocusing Lens te Light er On Power (mW) 0		

For a more thorough overview and understanding of the FT-Raman acquisition tab, consult the latest <u>OMNIC</u> Paradigm software user guide.

Α	Defocusing Lens: Check the box to activate the defocusing lens. See <u>Using the defo</u> - cusing lens to adjust the beam size.
В	White Light: Check the box to activate white light. See Using the white light source.
С	Laser On: Check the box to power on the laser. See <u>Turning on the laser and adjusting</u> the laser power.
D	Laser Power (mW): This shows the laser's power in milliwatts. You can adjust it by moving the accompanying slider in the software up or down as needed.

In the acquisition tab, you are able to capture mosaics and select sample points. You can also view your sample, optimize your signal, and begin measurements. See <u>Your first experiment</u> for more information.

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 maximum power level of 500 mW at the sample. The laser must be on when you are focusing a sample and measuring Raman data.

To turn on the laser, simply check the **Laser On** checkbox in the acquisition tab. It takes a few seconds for the laser to begin emitting radiation.

Note You can leave the White Light box checked, except when you are focusing a sample as it may obscure your spectrum. 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. In the acquisition tab, check the Laser On box to power the laser.
- 2. Select Live Spectrum to show the spectral data in the display.
- 3. Gradually adjust power by moving the **Laser Power** slider up or down as needed 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 turn off the laser. Move the sample so that the laser beam will strike an area that is not decomposed, 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 <u>Setting a timer to turn off the laser</u>.

Adjusting the sample position

You can adjust the position and focus of your sample by using the Camera View from the acquisition tab in OMNIC Paradigm software.

For basic information about using OMNIC Paradigm to position and bring a sample into focus as well as measure Raman data, see <u>Your first experiment</u>.

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.

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 Paradigm software closes). This will prevent the laser from being left on inadvertently and prolong its life.

- To set a timer for the laser
- 1. Go to the dashboard in OMNIC Paradigm software.
- 2. If needed, select More to see all additional settings.
- 3. Input the desired time (in minutes) in the Laser Timeout field.

Timeout only occurs if the software is left idle for the specified amount of time. The timer is suspended when you begin a measurement and resets once the measurement is complete.

You can check the **Disable Laser Timeout** box if you would like to prevent the laser from shutting off. However, the laser will automatically turn off if the session is ended regardless of any inputs.

Preparing samples

The Raman Module sample stage can be used with solids, liquids, and powders (see <u>Compatible samples</u> 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 sample cup holder or other sample holder.

Raman Module sample holders

The following sample holders are included:

- 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.

Installing a sample holder

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

These instructions show how to install any of the 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 stag.

To install a sample holder

- 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. If the sample stage does not move automatically, simply select **Eject Stage** from the dashboard in OMNIC Paradigm software.

3. Install the sample holder on the stage.

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

Figure 1-7: Standard sample holder



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 measurement optics and beam.

Removing a sample holder

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

- To remove a sample holder
- 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.

Sampling options

This section covers sampling options, such as using the white light source, defocusing lens, and preventing detector saturation.

Using the white light source

The Raman Module includes a white light source that is used for alignment and diagnostics. See <u>Aligning the</u> <u>interferometer</u> 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 bring your sample into focus. The white light must also be off during Raman data measurement, but the software does this automatically and turns it back on after data measurement is complete.

To step through a typical Raman analysis, see Your first experiment.

To manually control the white light source, simply check the **White Light** box in the OMNIC Paradigm software acquisition tab to turn it on, and uncheck it to turn it off as needed.

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 measurement 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 1-8: 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 that the laser strikes.

To defocus the laser, simply check the **Defocusing Lens** box in the OMNIC Paradigm software acquisition tab, and uncheck it to turn it off as needed.

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 measured 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 measure Raman data.

Your first experiment

The Raman Module is designed to illuminate a sample with laser radiation, measure 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.

Setting up the Raman Module to measure data

This section explains how to set up the Raman Module in OMNIC Paradigm software and how to use its functions. You can press the Touch Point to start the process and respond to software prompts while it is running, or simply use commands in the software to achieve the same result.

- To set up the Raman Module in OMNIC Paradigm software
- 1. See Installing the Raman Module and follow the steps.
- 2. Start OMNIC Paradigm software.

Note OMNIC Paradigm software automatically loads the default, commonly used settings for the Raman when it is installed, but you can also create your own settings for specific analysis needs.

To create new settings, select **New**, then enter a name for the new measurement settings. Modify the settings as needed, then select **Save**. The new measurement settings will be available for selection in the Settings dropdown menu.

3. Press the Raman sample compartment Touch Point or go to **Configure > Sample Location > Main Com**partment (Raman)in the software to establish the beampath if it isn't already selected.

Note Make sure that **Sampling accessory** shows as **Main Compartment (Raman)** in OMNIC Paradigm software. If it does not, a different instrument or accessory has been selected, or the instrument is not installed properly.

Measuring a tablet using the Raman Module

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

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 Paradigm software.
- Have your sample material at hand. For the default measurement settings, we recommend that you choose 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
- 1. Press the blue Raman Touch Point to configure the instrument for Raman sampling. Alternatively, go to **Configure > Sample Location > Main Compartment (Raman)** in OMNIC Paradigm software.

Figure 1-9: Sample compartment Touch Point



2. If your Raman Module is not configured, see Setting up the Raman Module to measure data.

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.

- 3. Prepare the sample.
 - a. Place the sample on one of the provided sample holders. See <u>Preparing samples</u> 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.

<image><image><image>

Figure 1-10: Sample holder on Raman sample stage

- d. Close the Raman sample compartment door.
- e. Position your sample under the measurement optics.
- 4. Select Start Session in the OMNIC Paradigm software dashboard.

If you would like to bring your sample into focus automatically, check **Autofocus before capture** before you start the session.

The software will open the acquisition tab and capture a mosaic for sampling. You can also capture a mosaic manually by selecting **Capture Mosaic**.

- 5. Select the point you want to analyze on your sample.
 - a. Bring the sample into focus.

Note This step is not as critical for defocused sampling. See <u>Using the defocusing lens to adjust</u> the beam size.

Select **Camera View**. Under Movement Speed, you may select **Autofocus** or focus manually using the navigational arrows in the software.

- b. Using the Point tool in the tool pane, click the desired sample points in the acquisition tab mosaic.
- 6. Turn on the laser, adjust the laser power, and focus the measurement optics.
 - a. Check the White Light box and select Live Spectrum to see a live interferogram. Uncheck the White Light box to turn the white light off.
 - b. Turn on the laser. To do this, check the Laser On box.

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

c. Adjust the laser power to an appropriate level for your sample. See <u>Turning on the laser and adjusting</u> the laser power.

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
 (see <u>Using the defocusing lens to adjust the beam size</u>). Be aware, however, 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.
- 7. Measure the spectrum.
 - a. Select Sample and wait for the process to complete.
 - b. The sample measurement will appear in the right-hand side Measure tab when complete.

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 measurement begins.



Figure 1-11: Report showing an Raman spectrum of a pharmaceutical tablet taken with the Raman Module

- 8. Turn off the laser. To do so, uncheck the Laser On box in the OMNIC Paradigm software acquisition tab.
- 9. If you are finished measuring Raman data, open the Raman sample compartment door, wait for the stage to stop moving, and remove the sample holder. Then close the software.

Tip For your convenience, you can set the laser to shut down automatically after a specified period of time. See <u>Setting a timer to turn off the laser</u>.

You can review your measurement history in OMNIC Paradigm software by going to the dashboard and selecting **Measurements** from the dropdown menu.

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, uncheck Laser On in the aqcuisition tab.

3. 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 its vertical position.

Figure 1-12: Release Raman latch handle



4. 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.

5. 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 1-13: Remove Raman Module



- 6. Install the sample compartment cover for the spectrometer and the Snap-In baseplate, if it was removed when you installed the Raman Module.
- 7. 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.
- 8. Remove the CaF2 or XT-KBr beamsplitter, if necessary, and install the appropriate beamsplitter for the next application.
- 9. If you installed a new beamsplitter, align the interferometer.

Consult your spectrometer user guide if needed.

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.

Maintenance

This section describes maintenance routines that you can perform on the 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, removing, and replacing 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, that the spectrometer has stabilized for at least 20 minutes, and that the proper beamsplitter is installed.

- To align the interferometer
- 1. Start OMNIC Paradigm software.
- 2. If the Module is installed, remove it.
- 3. Go to Acquire Data > Diagnostics and choose Align. Wait for the process to complete, then reinstall the Module.

Verifying Raman Module performance

A good way to verify the performance of your Raman Module is to regularly measure 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.

To check the Raman Module performance, first place the polystyrene sample disk provided with the Raman Module on the sample stage and close the sample compartment door, then run performance verification (PV) workflows.

Performance verification

Ensure that the Module is working well by running PV workflows in OMNIC Paradigm software and checking the system status.

PERFORMANCE VERIFICATION AND QUALIFICATION WORKFLOWS

You can check the performance of the Module by running qualification or performance verification workflows. These workflows use an established standard sample to check performance of the instrument. Each test follows different regulatory standards.

PV and qualification workflows use the polysterene standard disk to test the Module's performance.

Test	Description
Nicolet FT Raman Module - PHEUR Qualification	Executes qualification tests as defined in the European pharmacopoeia.
Nicolet FT-Raman Module - USP Qualification	Executes qualification tests as defined in the United States pharmacopoeia.
Nicolet FT-RAMAN Module - PV Test	Measures basic performance of the Module based on fact- ory-recommended settings.
Nicolet FT-RAMAN Module - Factory Qualification	Runs factory-recommended tests and all qualification tests.

- To run a qualification or performance verification workflow
- 1. Right-click the workflow and select Run.
- 2. Follow the on-screen prompts.

When the worflow is complete, the final reports are added to the **Reports** pane in the dashboard and can be printed.

System status

The system status icon displays information about your instrument and software services.

lcon	Icon with Security Suite installed	Description
\checkmark	V	The system is connected and all services are running correctly. You are ready to measure and save data. Click the system status icon to view details about the system.
	U	 A yellow icon means there may be a problem with the instrument, such as the following: The detector is warm The instrument is not scanning The instrument is not connected Click the system status icon for more details.
\bigotimes	8	There is a problem with one or more software services. Click the sys- tem status icon for details. If the service does not start automatically after a few minutes, restart the computer.

If you continue to have trouble with system status errors, contact customer support.

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 Paradigm software or initializing the sample stage.

To initialize the sample stage, you can briefly remove and reinsert the module.

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

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, removing, and replacing 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.

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.

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 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.

Cleaning, removing, and replacing the residue tray

A residue tray is located under the sample stage in the Raman Module sample compartment. The tray measures materials that may have fallen from the sample stage.

To clean the residue tray

Use a mild detergent and rinse the tray with water to prevent residue 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 Paradigm software, choose Start Session and check the Custom mosaic box.
- 4. Choose **Camera View**. Set the stage speed to **Fast** and input 20000 into the Focus field, then select the "up" arrow as many times as needed to move the sample stage up and out of the way until it is no longer moving. This gives you enough space to remove the tray.
- 5. 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 1-14: 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.

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.

Troubleshooting

The troubleshooting information will help you solve problems you may have with the 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 measured spectra.
Error conditions	How to deal with error messages you may see.

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 Paradigm software and choose **Help > Gather Log Files**. Wait for the process to complete and measure your logs. You can attach the file to an e-mail message to our technical support representative.

WARNING



Avoid hazard. Always follow the safety precautions included in this document and in any safety guides for your instrument or the Raman Module.

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 measurement, consider whether the <u>sample is</u> <u>compatible</u> with the Raman application or if the sample is not in focus. If you don't believe that is the problem and the issue persists, follow these steps:

- 1. Turn the white light on. If a white light interferogram appears on the left-hand menu in the acquisition tab, your sample does not scatter Raman energy from the laser. Try another application or sampling technique.
- 2. If no white light interferogram appears, remove the Raman module and align the beamsplitter.
- 3. If there is still no interferogram, contact us for assistance.

Applications problems

Noisy spectra

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

- 1. Make sure the Raman Module installed correctly. Confirm that the Module is fully inserted and the frontcentering pin is firmly seated into the latching mechanism.
- 2. Make sure that the sample is in focus. If it is not, bring the sample into focus and see it if improves your spectra.
- 3. Make sure the CaF2 or XT-KBr beamsplitter is installed.

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

4. Make sure the sample is Raman-compatible, such as a pharmaceutical, polymer, or plastic, and not a piece of glass, metal, or paper.

If the spectra are still noisy, install the standard polystyrene disk sample and run the PV workflow.

- 5. Remove the Module and align the interferometer, then reinsert the Module. If the problem persists after alignment is complete (or if no interferogram is present after you complete the steps above), proceed to the next step.
- 6. If an interferogram is present but the spectra are still noisy, contact our local service representative.

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 measure a Raman spectrum.
- 8. Run the qualification test.

Interferogram peak is not centered

1. In OMNIC Paradigm software, choose Acquire Data > Diagnostics > Reset Spectrometer.

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