NXR FT-Raman Spectrometer User's Guide

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Introduction

This manual explains how to use the NXR FT-Raman Module with a Nicolet™ Series spectrometer to collect FT-Raman data. The accessory contains a 976 nm or a 1064 nm excitation laser and a sample compartment in which you can install any of several sampling configurations.

Important The information and procedures in this manual refer to the NXR FT-Raman Module, but they also apply to the Nicolet NXR FT-Raman 9610 and the Nicolet NXR FT-Raman 9650 spectrometers. ▲

This manual provides a brief discussion of the theory of FT-Raman spectroscopy, a description of the main components of the NXR FT-Raman Module and how to use them, and a list of troubleshooting procedures. The software used to operate the accessory is described in the OMNIC™ and OMNIC For Raman on-line Help systems.

⚠ **Caution** The *Raman Accessory Safety Guide* that came with your system contains important safety information, which is presented in several languages. Before you use the NXR FT-Raman Module, read the entire portion of the guide that is in your language. To prevent personal injury and damage to equipment, follow the safety precautions contained in the guide whenever you use the accessory and other system components. ▲

⚠ **Caution** Follow all safety label precautions to avoid being injured while using the FT-Raman Module. ▲

Important If your are using a sampling accessory or configuration in a system with the 1064 nm laser, you should call Thermo Electron Technical Support before you attempt to use it in a system with the 976 nm laser. Your accessory or configuration may need to be refitted for use with the 976 nm laser. ▲

Conventions used in this manual

This manual includes safety precautions and other important information presented in the following format:

Note Notes contain helpful supplementary information. ▲

Important Follow instructions labeled "Important" to avoid damaging the system hardware or losing data. ▲

⚠ **Caution** Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices. ▲

⚠ **Warning** Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury. ▲

⚠ **Danger** Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. ▲

Questions or concerns

In case of emergency, follow the procedures established by your facility. If you have questions or concerns about safety or need assistance with operation, repairs or replacement parts, use the information below to contact Thermo Electron. Outside the U.S.A., contact the local Thermo Electron sales or service representative.

Phone: 1-800-642-6538 (U.S.A.) or
+1-608-273-5015 (worldwide)

Fax: +1-608-273-5045 (worldwide)

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World Wide Web: http://www.thermo.com/spectroscopy

Fundamentals of FT-Raman spectroscopy

The following sections provide a brief overview of FT-IR (Fourier transform infrared) spectroscopy to help you understand how your spectrometer collects data.

Raman spectrometers measure the light emitted by an experimental sample under laser illumination. Emission data has two main characteristics: the frequencies at which the sample emits the radiation, and the intensities of the emissions.

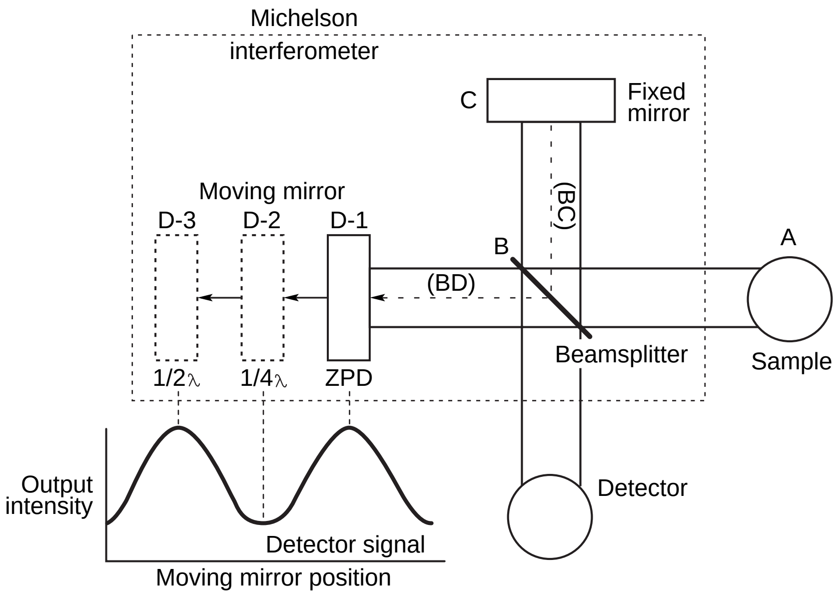
Determining the frequencies allows the sample's chemical makeup to be identified, since chemical functional groups are known to emit specific frequencies.

Intensity and frequency of sample emission are shown in a two-dimensional plot called a spectrum. Intensity is plotted along the Y-axis and frequency is plotted along the X-axis. 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 used by the exciting laser.

How the FT-Raman Module works

Light from the excitation laser 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 FT-Raman Module and bench optics to the detector. The laser light, on the other hand, does not travel past the sample compartment.

The following illustration shows schematically the path of the emitted radiation as it travels to the detector. Radiation from the sample (A) is directed into the Michelson interferometer and strikes the beamsplitter (B). About 50% of the radiation is reflected from the beamsplitter and is directed onto the fixed mirror (C). The remainder is transmitted through the beamsplitter and is directed onto the moving mirror (D).



The interferometer provides a means for the spectrometer to measure all frequencies simultaneously. The interferometer modulates the intensity of individual frequencies of radiation emitted by the sample.

The beams are reflected from the surfaces of the two mirrors and recombine at the beamsplitter. When the beams recombine, constructive or destructive interference occurs depending on the position of the moving mirror relative to the fixed mirror. The illustration shows the signal measured at the detector for a monochromatic, or single frequency, source.

When both mirrors are the same distance from the beamsplitter, the two beams travel exactly the same path length and, consequently, are totally in phase with each other. The resulting signal intensity is at its maximum, a point called the zero path difference (ZPD).

When the beams recombine to create a strong signal (at mirror positions D-1 and D-3), constructive interference is said to have occurred. When the beams recombine to create a weak signal (at mirror position D-2), destructive interference has occurred.

The recombined beam travels to the detector, which translates the beam into an electrical signal that can be processed by a computer.

The Fourier transformation

The product of an interferometer "scan" is called an interferogram, a plot of intensity versus mirror position (indicated by data point number). The interferogram in the following illustration is a summation of all the wavelengths (cosine waves) emitted by the sample; for all practical purposes it cannot be interpreted in its original form.

Using a mathematical process called Fourier Transformation (FT), the system computer converts the interferogram into a spectrum. The spectrum shows the sample emission at all the frequencies measured and thus can be used to identify the sample.

The Raman effect

The following sections briefly explain the nature of the Raman effect and describe the advantages of the Fourier Transform (FT) technique when applied to Raman spectroscopy.

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.

Some of the Raman radiation is slightly lower in frequency than the incident radiation and is called Stokes-shifted radiation. Another smaller portion of the Raman radiation is higher in frequency than the incident radiation and is called anti-Stokes radiation. The exact characteristics of Raman radiation depend on the chemical composition of the sample.

History of the Raman effect

When Sir Chandrasekhara Venkata Raman discovered the Raman effect in 1928, the instrumentation he used was rudimentary. The sun was his source, a telescope was the collector and his eyes were the detector. The effect he noted was so small that it is remarkable he noticed it at all.

His observations confirmed the theories developed by Smekal, Kramers, Heisenberg, Schrodinger and Dirac. They predicted in 1923 that radiation scattered by molecules would contain photons not only with the same frequency as the incident radiation, but also some with a changed frequency. Raman was awarded the Nobel prize for his work.

Over the next 30 years there were gradual improvements in all aspects of the instrumentation. For many years the preferred method of detection was the photographic plate. This technique was superseded by the photomultiplier tube, which was used in conjunction with a discharge lamp. The discharge lamp was finally superseded by the laser in the 1960s. The laser is the ideal source for a Raman experiment because it is both monochromatic and very intense.

FT-Raman spectroscopy

In 1986 the work of Tomas Hirschfeld and Bruce Chase showed that FT-Raman spectroscopy was a viable technique with performance comparable to that of conventional Raman spectroscopy. Their findings spurred activity at several academic laboratories as well as at most of the major manufacturers of FT-IR spectrometer systems.

One major advantage of FT-Raman over conventional Raman lies in the use of a near-IR laser as an excitation source. This source reduces or eliminates the fluorescence problem that has plagued Raman spectroscopy since its inception.

Fluorescence of the sample is commonly due to impurities that are present in most real-world samples. Generally, these impurities are compounds with extended double bonds or aromatic rings. Since there is a great difference between the intensity of Raman scattering and fluorescence, even trace sample impurities can cause serious difficulties in conventional Raman spectroscopy. This problem can make it difficult or impossible to measure the Raman spectrum against the fluorescent signal.

Many methods have been used to reduce this fluorescence, such as purifying the sample or bleaching it with the exciting laser beam; however, these techniques are not always successful.

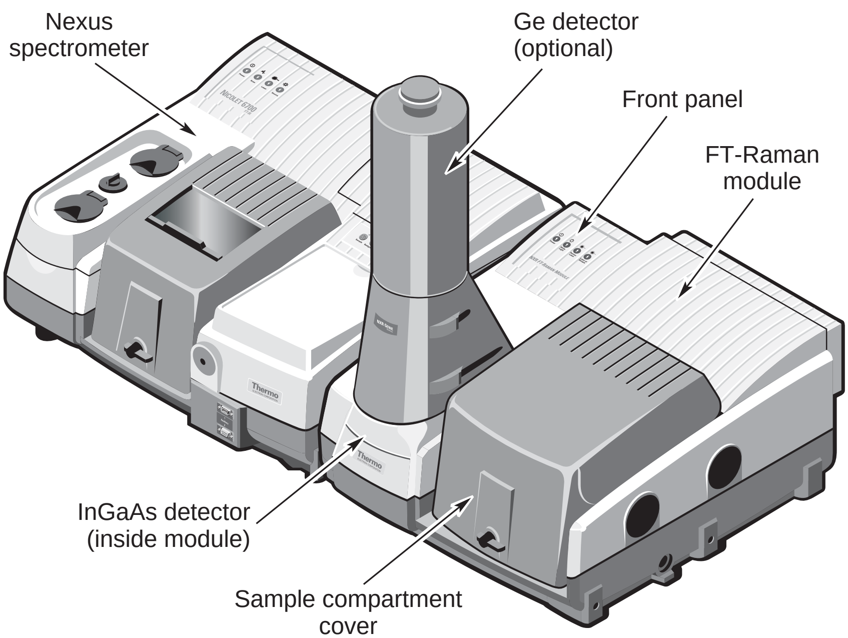
Using the established techniques of Fourier Transform spectroscopy for the Raman experiment results in several other advantages over dispersive Raman spectroscopy. These include:

- increased light throughput
- accurate wavenumber calibration of the instrument
- constant resolution of the spectrum over the range of Raman shifts
- the ability to measure high-resolution spectra in a routine manner

Description and Setup

This chapter describes the major features of the NXR FT-Raman Module. The 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.

The major parts of the FT-Raman system are shown in the following illustration:

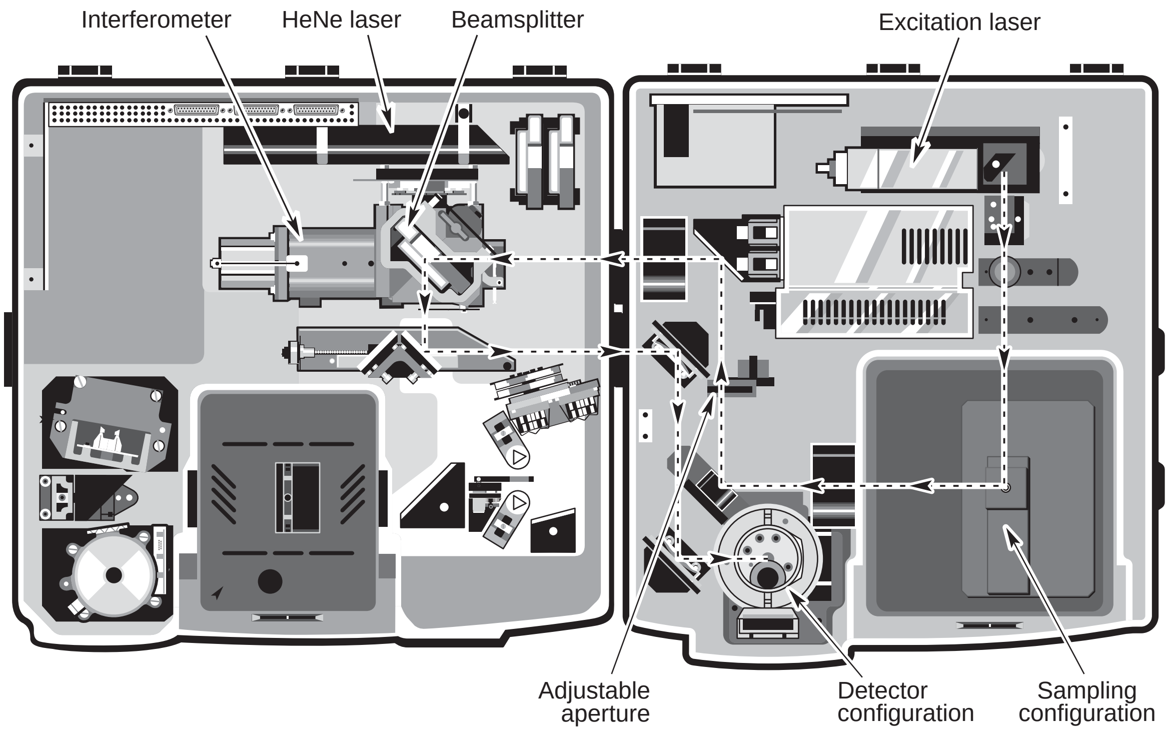


NXR FT-Raman Module

The NXR FT-Raman Module contains the excitation laser, sampling configuration, and the detector used to collect the Raman data. The interferometer is located in the spectrometer. The accessory components are described in detail in the following sections. For information on spectrometer components, see the user's guide and on-line help that came with the spectrometer.

Note Install the CaF_2 or XT-KBr beamsplitter in the spectrometer before using the NXR FT-Raman Module. After you install the beamsplitter, you will need to align the interferometer to optimize the detector signal. Use Spectrometer Help Topics in the Help menu to find details on changing beamsplitters. See "Aligning the Interferometer" in this manual for information on aligning the interferometer. ▲

The optical layout of the FT-Raman system is shown in the following illustration, which presents a top view of the FT-Raman Module and spectrometer with all covers removed:



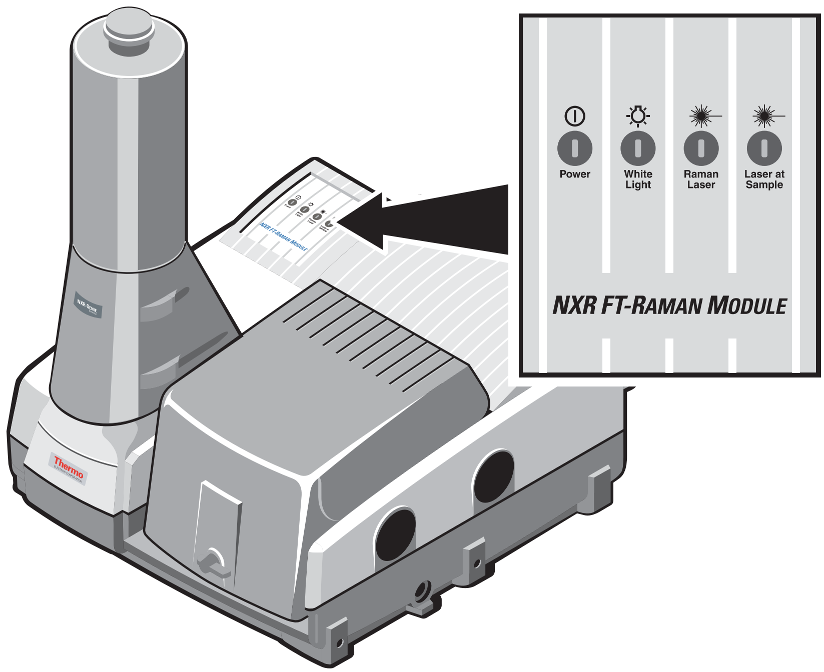
Optical layout with 1064 nm laser (top view)

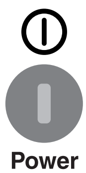
The sampling configuration shown in the optical layout is 180 degree reflective. See "Sampling configurations" for illustrations of other available configurations.

Note If you have a Nicolet NXR FT-Raman 9610 or a Nicolet NXR FT-Raman 9650, the left side of the spectrometer will not have all of the components shown above. ▲

Status indicators

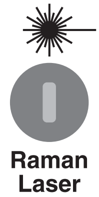
The top of the module contains indicator lights that show the current status of the accessory:



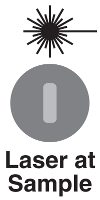


This indicator is lit when the NXR FT-Raman Module power is on. The module power switch is on the rear panel (see “Rear panel” for details).

This indicator is illuminated when the white light source is on. To turn on and adjust the intensity of the white light source, choose Experiment Setup from the Collect menu. Click the Bench tab and set the White Light parameter. See the OMNIC For Raman on-line Help system available in the Raman menu for more information on White Light.



This indicator is lit when the excitation laser is turned on from the software. The laser is emitting radiation when the indicator light is on. See “Turning on the laser and adjusting the laser power” for more information.



This indicator is lit when radiation from the excitation laser is present inside the sample compartment. Always make sure the light goes out when you open the sample compartment cover. Exposure to up to 2.0 watts of invisible laser radiation is possible if this indicator does not go out when you open the cover.

Covers

The NXR FT-Raman Module main cover serves as the laser protective housing required by law. Both the 1064 nm and the 976 nm excitation lasers used in the accessory are Class IV lasers.

The only cover on the NXR FT-Raman Module that you can open is the sample compartment cover (see the first illustration in this chapter). Open the sample compartment cover to change samples or sampling configurations. When you open the sample compartment cover, the Laser at Sample status light on the top of the module should go out and you should hear the safety interlock system click.

⚠ **Warning** Do not open the main cover of the NXR FT-Raman Module; this cover can be opened only by qualified Thermo Electron service personnel. ▲

The use of redundant, nondefeatable interlocks on the sample compartment cover allows the instrument to be designated as a Class II laser product. Therefore, the laser safety requirements required for a Class IV laser, such as room door interlocks, are not required during normal operation or maintenance.

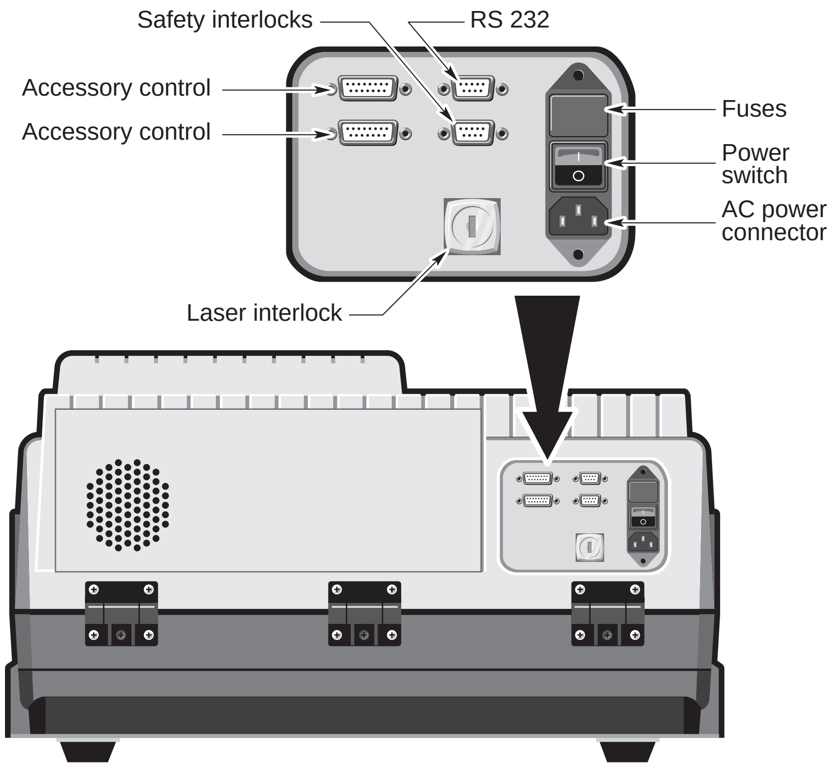
The sample compartment cover interlocks operate by blocking the excitation laser beam when activated instead of switching the laser off. This method results in more stable laser operation and better quality spectra.

⚠ **Caution** Keep all magnets and strong magnetic fields (such as those produced by a mass spectrometer, computer display monitors or various medical instruments) away from the interlock switches in the FT-Raman Module sample compartment while the sample compartment cover is open. ▲

Rear panel

The following illustration shows the rear panel of the NXR FT-Raman Module and identifies the connectors for the system cables.

The FT-Raman Module connects to the spectrometer via the Accessory connector. The module power cord plugs into the AC power receptacle next to the accessory power switch. Allow enough room behind the system for the power cord and other cables.



Important The illustration above shows the NXR FT-Raman Module with the 1064 nm laser, which is air-cooled by two fans, the one in the side of the instrument and one in the rear panel of the instrument. The 976 nm excitation laser is air-cooled by one fan in the side of the instrument. To avoid overheating the FT-Raman Module, do not block the free flow of air into or out of the rear panel or the flow of air under the module. There should be at least 15 cm (6 in) of clearance behind the accessory. ▲

Note The fans described in the preceding cautionary note produce a small amount of vibration and may generate noise spikes in infrared spectra. Before collecting infrared data in the far-IR region using the main optical bench, you may need to turn off the NXR FT-Raman Module power. ▲

Important If your instrument has the 1064 nm excitation laser, the laser control cable is connected to a COM port on the rear panel of the computer. COM port 2 can be used for the cable if COM port 1 is being used for a mouse. Whenever you install the OMNIC software, be sure to use the Configure Bench software, located on the Diagnostic tab of Experiment Setup in OMNIC, to select the correct COM port. See the OMNIC on-line Help system for details. ▲

Important If your instrument has the 1064 nm excitation laser, make sure that any optional plug-in cards are not configured for the same COM port as the laser. If an internal modem card is used, make sure it is configured for COM 3 IRQ 5. The laser should then use COM 2. ▲

Turning on the FT-Raman Module power

To turn on the NXR FT-Raman Module power, press the power switch on the rear panel to the I position; to turn the power off, press the switch to the O position. Turn the accessory power on before you turn on the spectrometer power.

Turning on the laser keyswitch

The laser interlock keyswitch on the rear panel allows you to turn on the excitation laser power from the OMNIC software. (See the illustration in the “Rear panel” section earlier in this chapter for the location of the laser interlock.) The key must be inserted into the keyswitch and turned clockwise to the vertical position to operate the laser.

You can turn off the laser by turning the laser interlock keyswitch to the horizontal position. The Laser indicator on the front panel will go out when the laser is off. To prevent the laser from being turned on, remove the key from the keyswitch when it is in the horizontal position.

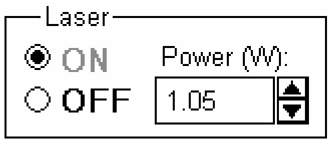
Excitation laser

The NXR FT-Raman Module has two different excitation laser options—a 1064 nm (9398 cm^{-1}) laser or a 976 nm (10246 cm^{-1}) laser. In either case, the laser is located at the rear of the accessory under the main cover, and both lasers emit continuous-wave laser energy that is invisible to the eye. The 1064 nm laser has a nonaccessible maximum power level of approximately 1.5 watts at the sample, and the 1064 nm laser beam is vertically polarized when it enters the sample compartment. The 976 nm laser has a nonaccessible maximum power level of approximately 1 watt at the sample. The polarization of the 976 nm laser beam varies with the power level, but it is vertically polarized at maximum power.

Make sure the safety interlock connector is installed and secured on the rear panel before attempting to turn on the laser.

Turning on the laser and adjusting the laser power

After you enable the excitation laser using the keyswitch on the rear panel, you can turn the laser on or off and adjust its power by choosing Experiment Setup from the Collect menu, clicking the Bench tab and setting the parameters. To turn the laser on, click the ON option button in the Laser box:



It takes a few seconds for the laser to begin emitting radiation. To adjust the laser power, set Power to the desired value in watts. This is the power at the laser head. The laser power in the sample compartment (which is lower than the power at the laser head) is shown along with the interferogram or spectrum in the live display of the Experiment Setup dialog box. See the OMNIC For Raman on-line Help system available in the Raman menu for complete information.

Note The laser switches itself off if the laser power is set to zero. To recover from this condition, quickly set the power to 0.2 watt or more to ensure that the laser stays on. ▲

Note If your system has the 1064 nm laser, you can reduce the laser power further by using an optional neutral density filter in the sample compartment. See “Sample compartment” for more information. ▲

Note If your system has the 976 nm laser, the minimum attenuation is 10 percent of the maximum power. This means that settings between 90 and 100 percent of the maximum power cannot be accepted in the Power drop-down list box. ▲

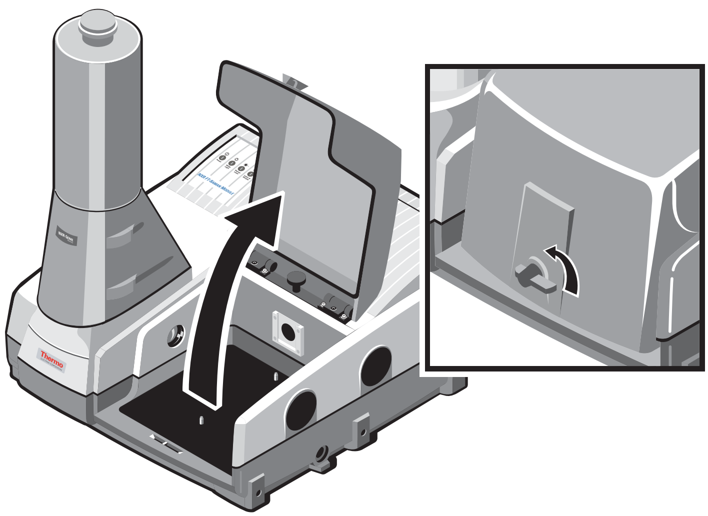
Sample compartment

The sample compartment of the NXR FT-Raman Module is large enough to accommodate a variety of sampling configurations that can be quickly interchanged. The optical components of each sampling configuration and accessory are mounted on a baseplate that is pinned in place. This allows you to easily change sampling configurations or accessories.

Important The NXR FT-Raman Module has the interlocks required by the safety regulations governing Class II laser systems. When you open the sample compartment cover, the laser beam is automatically blocked by a shutter to prevent exposure to laser radiation. The activation of the shutter is the source of the sound you hear as you open the cover. Always make sure the Laser at Sample status light on the top of the module goes out when you open the sample compartment cover. ▲

⚠ Caution Keep all magnets and strong magnetic fields (such as those produced by a mass spectrometer, computer display monitors or various medical instruments) away from the interlock switches while the sample compartment cover is open. ▲

To open the sample compartment cover, rotate the latch counterclockwise and lift up (see the following illustration).



Sampling configurations

The standard sampling configurations that are currently available for the NXR FT-Raman Module include:

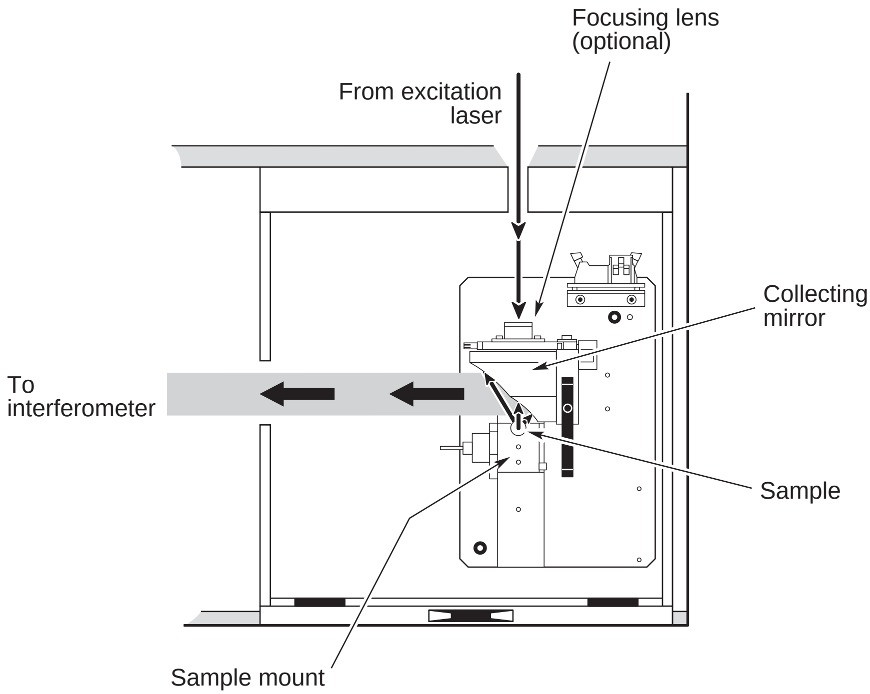
- A 180 degree reflective configuration with fully motorized sample position adjustment.
- A 90 degree refractive configuration with a removable aperture.
- A bottle holder configuration for measuring powder or liquid samples contained in glass bottles.

The next sections explain how to use each of the sampling configurations.

Important If your are using a sampling accessory or configuration in a system with the 1064 nm laser, you should call Thermo Electron Technical Support before you attempt to use it in a system with the 976 nm laser. Your accessory or configuration may need to be refitted for use with the 976 nm laser. ▲

180 degree reflective sampling configuration

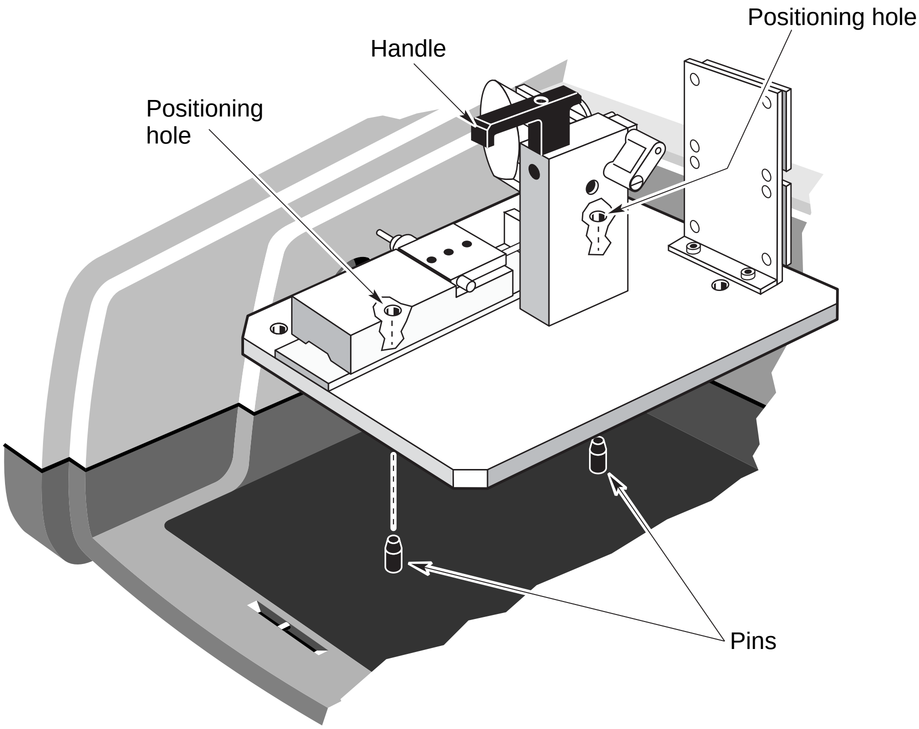
Use the fully motorized 180 degree reflective configuration (shown below) for samples that require maximum optical efficiency. This configuration collects the most energy.



Installing the configuration – Follow these steps to install the fully motorized 180 degree reflective configuration:

1. **Hold the optics by the handle and carefully position the baseplate so that the positioning holes fit over the pins.**

Important Do not touch any mirror or optical surfaces with your hands. Avoid hitting the connector on the back wall of the sample compartment. ▲



2. **After the configuration is in place, connect the cable to the connector on the back wall of the sample compartment.**

   The location of the connector is shown later in this section in the procedure describing how to remove the configuration.

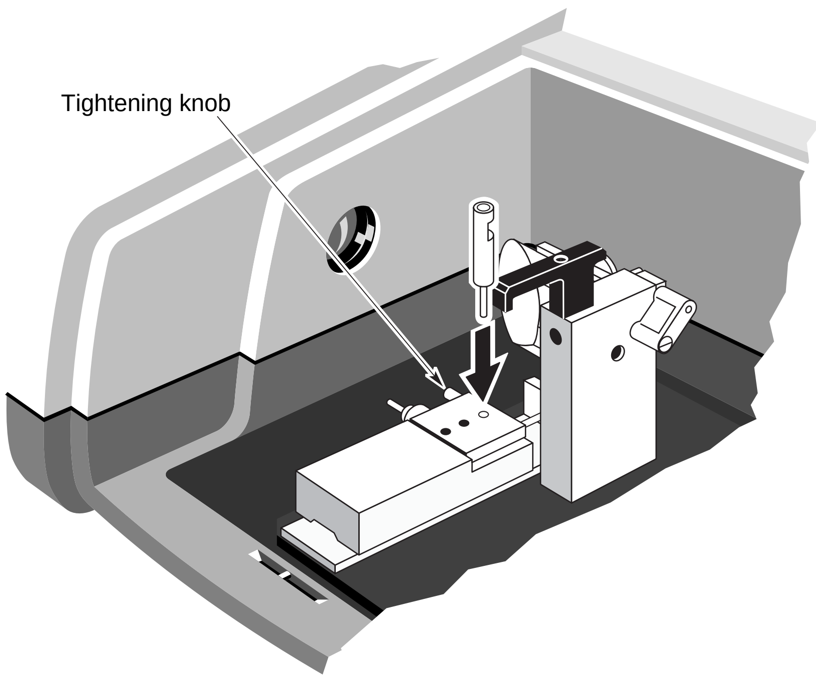
If desired, secure the configuration baseplate with the provided screws. Use a 5/32-inch hex wrench to tighten the screws. The location of the screw holes is shown later in this section in the procedure describing how to remove the configuration.

3. **Before using the configuration, exit and then restart OMNIC.**

   This allows the software to initialize the motorized sample position adjustments.

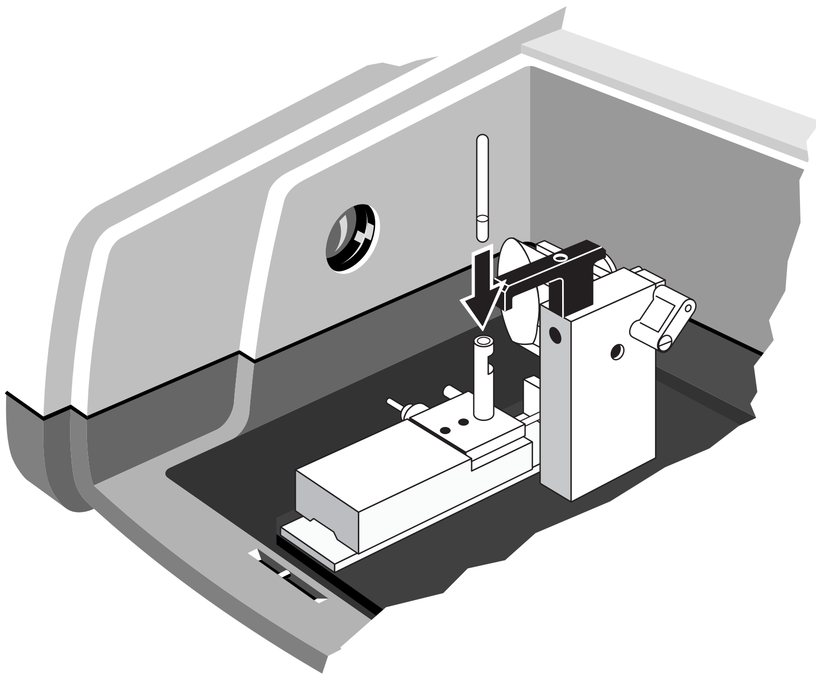
Installing a sample holder – The sample mount holds a variety of sample holders, each designed to position the sample at the focus of the excitation and collection optics. You can use the NMR tube holder to contain liquid or powder samples. The following illustration shows how to install an NMR tube holder in the sample mount.

A sample holder kit containing several other holders is available from Thermo Electron. An instruction manual is included.



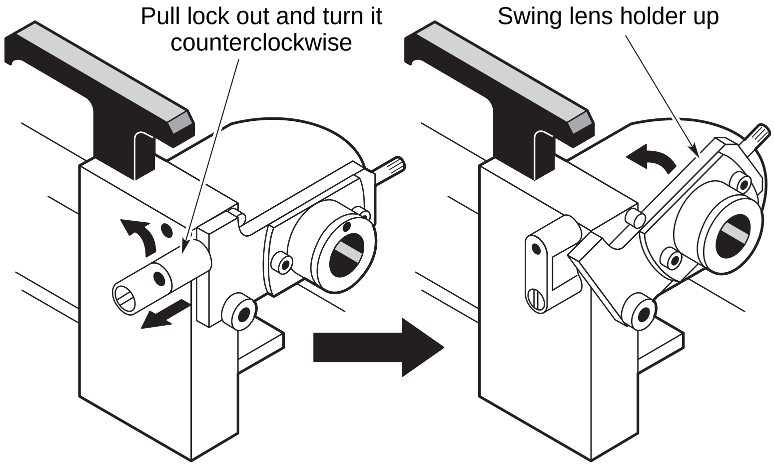
The shaft of the sample holder fits into any of three holes in the sample mount. The holder must be inserted all the way to ensure that the sample will be at the correct height. Insert the tightening knob (see the preceding illustration) into the appropriate hole on the side of the mount, and tighten the knob to keep the sample holder in place. (Two of the holes are on right side and one hole is on the left side as you face the sample compartment.)

Installing a sample tube – The NMR tube slides into the tube holder from the top as shown in the following illustration:



Using the beam focusing lens – The fully motorized 180 degree reflective configuration includes a beam focusing lens located in a movable holder at the rear of the configuration. This lens lets you focus the normally defocused laser beam onto a smaller area of the sample surface. See “Optional filters, lenses and accessories” for more information on how the lens works.

The lens is in the beam path when the holder is in the down position (see the first view of the holder in the illustration below). To move the lens out of the beam path, first pull the lock out enough to turn it counterclockwise, and position the lock vertically as shown below. Then swing the holder up as far as it will go.

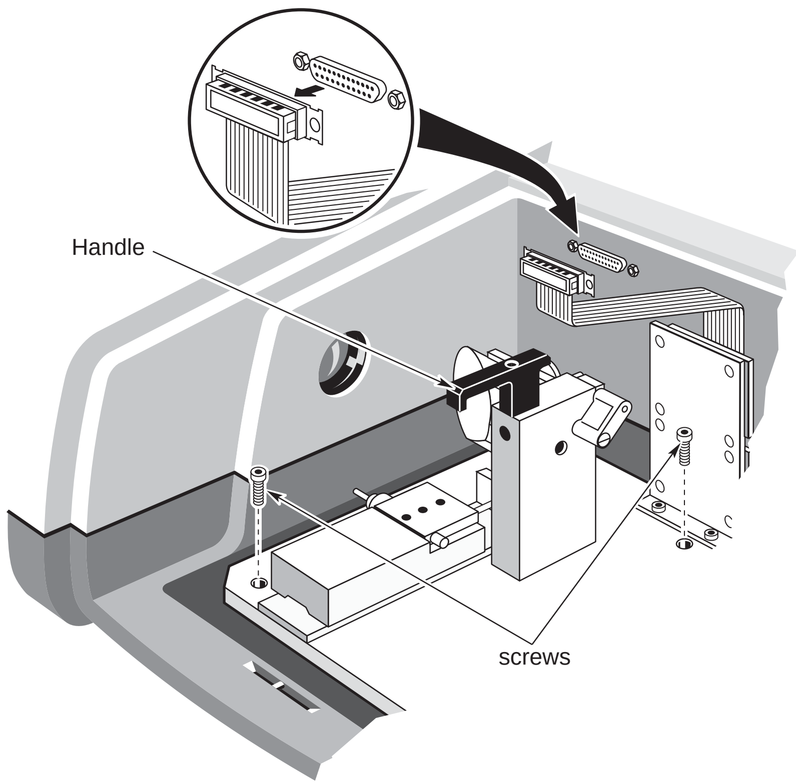


To move the lens back into the beam path, push the lens holder down until it stops. You can lock the holder in place by pulling the lock outward and then rotating it clockwise to return it to its former position (see the first view of the lens holder in the preceding illustration).

Removing the configuration – Follow these steps to remove the 180 degree reflective sampling configuration:

1. **Disconnect the cable from the connector on the back wall of the sample compartment, and remove the screws from the configuration baseplate (if they are installed).**

Important Do not touch any mirror or optical surfaces with your hands. Avoid hitting the connector on the back wall of the sample compartment. ▲



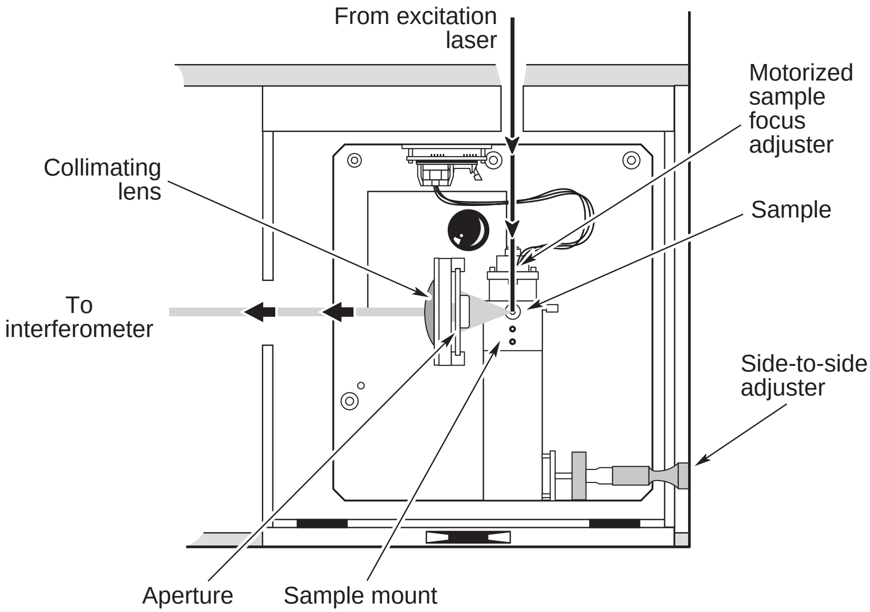
2. **Grasp the handle and carefully lift the configuration out of the sample compartment.**

   If the configuration baseplate is held down by screws, use a 5/32-inch hex wrench to loosen and remove the screws.

90 degree refractive sampling configuration

The 90 degree refractive sampling configuration comes with a removable aperture for limiting the angle of radiation of the beam that reaches the lens. Spectra collected using the aperture are closer to the theoretical results, which are based on an angle of radiation of 90 degrees.

The following illustration shows the optical layout of the configuration when installed.

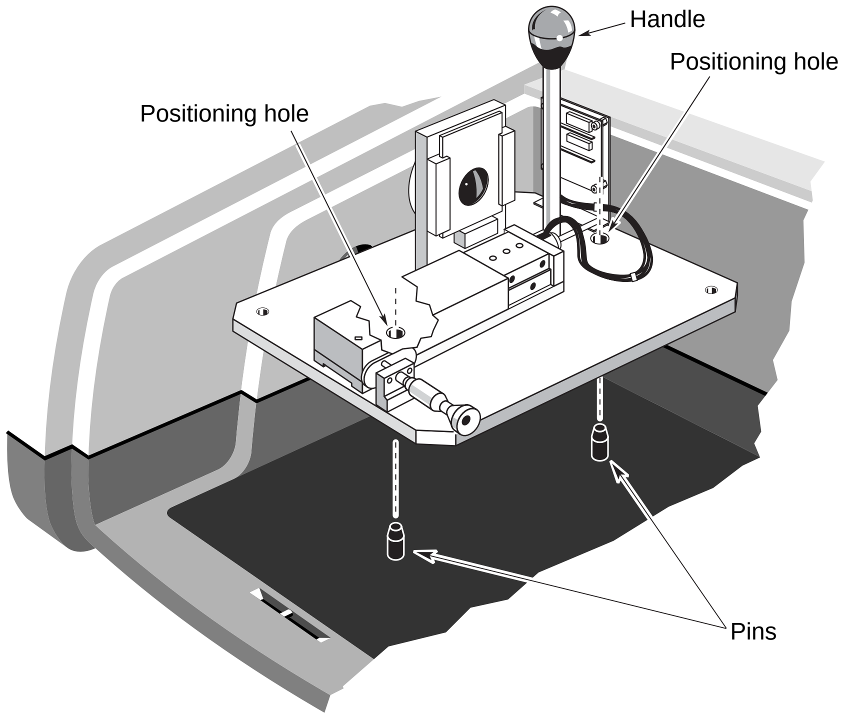


90 degree refractive sampling configuration (top view)

Installing the configuration – Follow these steps to install the 90 degree refractive configuration:

1. Hold the optics by the handle and carefully position the baseplate so that the positioning holes fit over the pins.

Important Do not touch any mirror or optical surfaces with your hands. Avoid hitting the connector on the back wall of the sample compartment. ▲



2. After the configuration is in place, connect the cable to the connector on the back wall of the sample compartment.

The location of the connector is shown later in this section in the procedure describing how to remove the configuration.

If desired, secure the configuration baseplate with the provided screws. Use a 5/32-inch hex wrench to tighten the screws. The location of the screw holes is shown later in this section in the procedure describing how to remove the configuration.

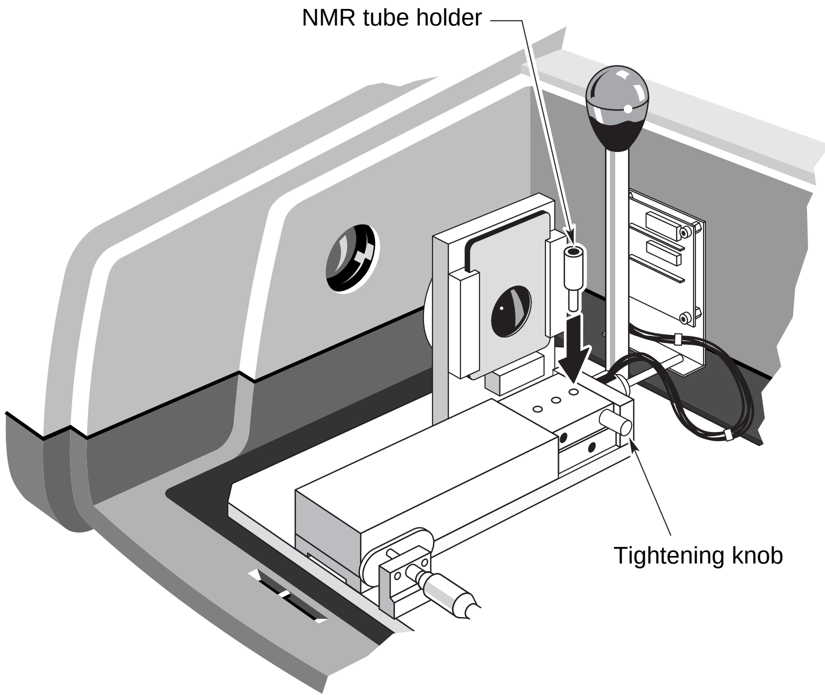
3. **Before using the configuration, exit and then restart OMNIC.**

   This allows the software to initialize the motorized sample focus.

Installing a sample holder – The sample mount holds a variety of sample holders, each designed to position the sample at the focus of the excitation and collection optics. You can use the NMR tube holder to contain liquid or powder samples. The following illustration shows how to install an NMR tube holder in the sample mount.

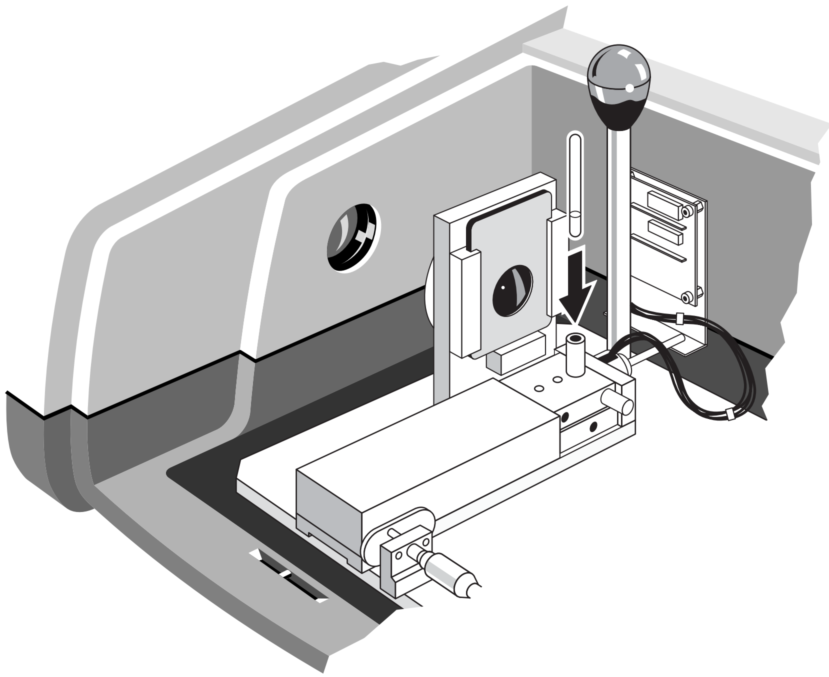
This configuration uses a special, shorter NMR tube holder.

A sample holder kit containing several other holders is available from Thermo Electron. An instruction manual is included.



The shaft of the sample holder fits into either of two holes in the sample mount. The holder must be inserted all the way to ensure that the sample will be at the correct height. Insert the tightening knob (see the preceding illustration) into the appropriate hole on the side of the mount, and tighten the knob to keep the sample holder in place.

Installing a sample tube – The NMR tube slides into the tube holder from the top as shown in the following illustration:



Important Proper alignment of the sample is critical for this configuration. The measured portion of the sample surface must be at the intersection of the laser beam and the collection axis (the axis passes through the center of the lens). Be sure to position the sample carefully, using the red spots formed by the HeNe laser. See "Adjusting the sample position in the standard configurations" for more information. ▲

Installing a beam focusing lens – The focusing lens on the 90 degree refractive configuration mounts behind the sample, toward the rear of the sample compartment.

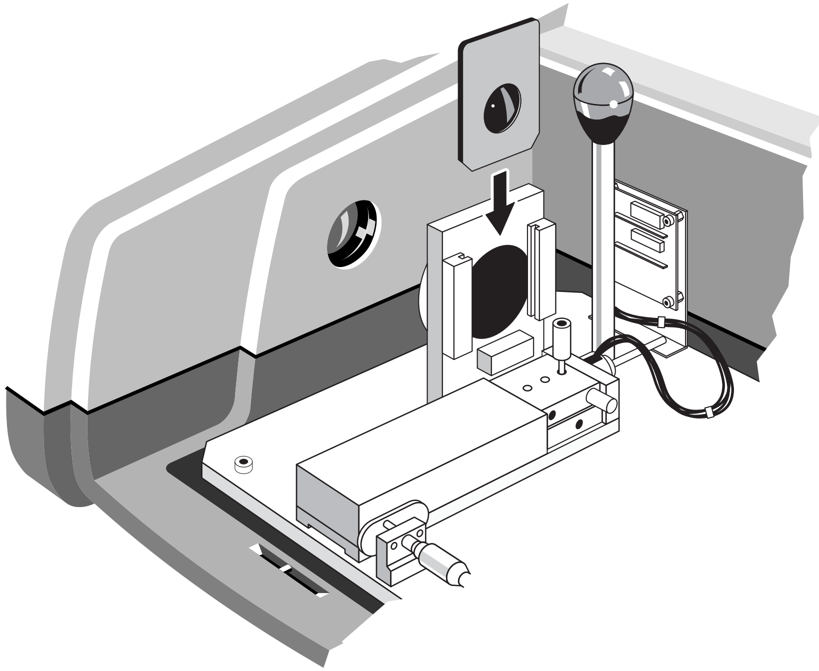
To install the lens in the holder on this configuration, grasp the lens handle and slide it down into the holder. The lens is held in place by leaf springs in the holder. To remove the lens, grasp the lens handle and pull it up and out of the holder.

See "Optional filters, lenses and accessories" for an explanation of what the lens does.

Installing the aperture – The 90 degree refractive configuration has an aperture holder between the sample mount and the collimating lens. By inserting the provided aperture in the holder, you can limit the angle of radiation reaching the lens (the collection angle) and thereby improve the precise measurement of the depolarization ratio when using the optional polarizer. This effect can be observed, for example, on a fully polarized band of benzene at approximately 1000 Raman shift wavenumbers.

Note Since using the aperture limits the collection angle, the signal intensity also decreases. ▲

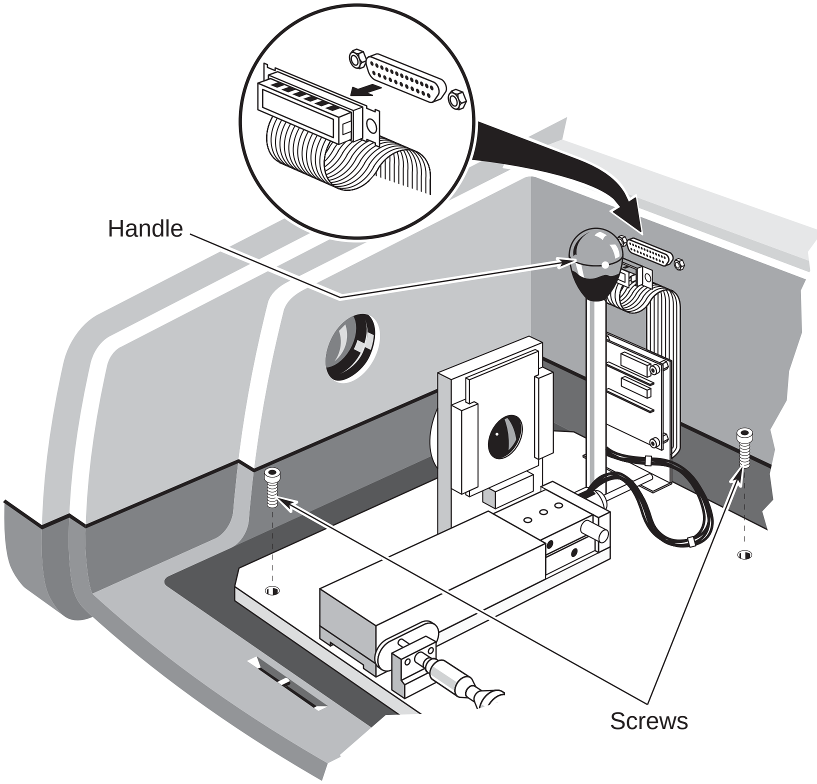
To install the aperture, slide it into the aperture holder as shown in the following illustration:



Removing the configuration – Follow these steps to remove the 90 degree refractive sampling configuration:

1. **Disconnect the cable from the connector on the back wall of the sample compartment, and remove the screws from the configuration baseplate (if they are installed).**

Important Do not touch any mirror or optical surfaces with your hands. Avoid hitting the connector on the back wall of the sample compartment. ▲



2. **Grasp the handle and carefully lift the configuration out of the sample compartment.**

   If the configuration baseplate is held down by screws, use a 5/32-inch hex wrench to loosen and remove the screws.

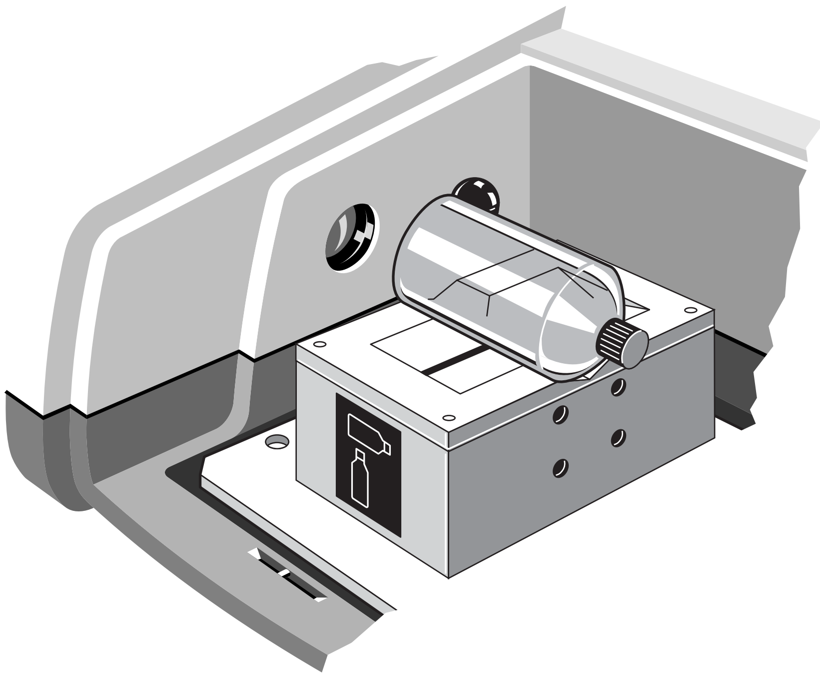
Bottle holder sampling configuration

A bottle holder is available for measuring powder or liquid samples contained in glass bottles. The holder fits over the two baseplate pins in the NXR FT-Raman Module sample compartment and can be secured with the same screws as used for the other sampling configurations. See the illustrations earlier in this chapter for the locations of the pins and screws.

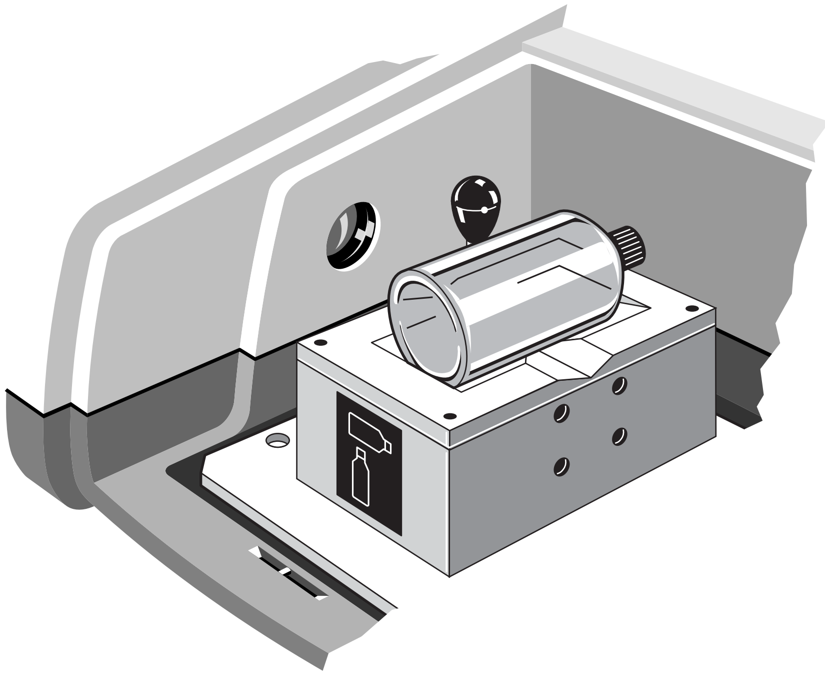
Because the optimum location of the focal point differs for solid and liquid samples contained in bottles, the top plate of the bottle holder has two grooves of different depths for holding the sample bottle.

The following illustrations show how to position the bottle in the grooves for solid and liquid samples.

Important The bottle holder is not recommended for use with the 976 nm laser due to the interference that may be caused by glass fluorescence. ▲



Bottle position for solid samples



Bottle position for liquid samples

The bottle holder is set at the factory for optimum performance and requires no further adjustments.

Adjusting the sample position

The position of the sample mount with respect to the excitation laser beam and the rest of the sampling optics can be adjusted in two directions:

- **Sample focus adjustment** moves the sample to the focal point of the collection optics (lens or mirror) for optimum collection efficiency. When you use a focused beam, it is focused at the same point.

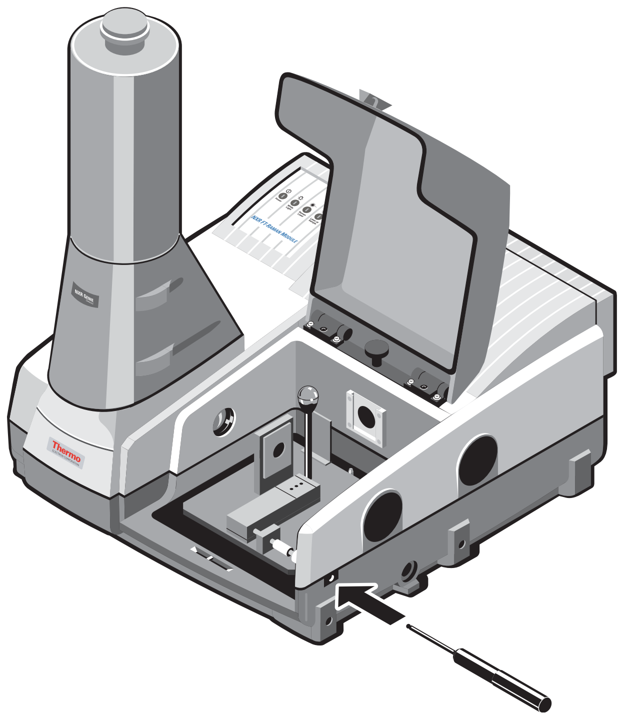
  The three standard sampling configurations have motorized sample focus adjustment. To adjust the focus on these configurations, choose Experiment Setup from the Collect menu and click the Bench tab. See the OMNIC For Raman on-line Help system available in the Raman menu for complete information.

- **Moving the sample from side to side** across the excitation laser beam allows you to position the desired portion of the sample in the beam.

  If your sampling configuration has motorized side-to-side adjustment, use the Experiment Setup dialog box to make the adjustment. See the OMNIC For Raman on-line Help system available in the Raman menu for complete information.

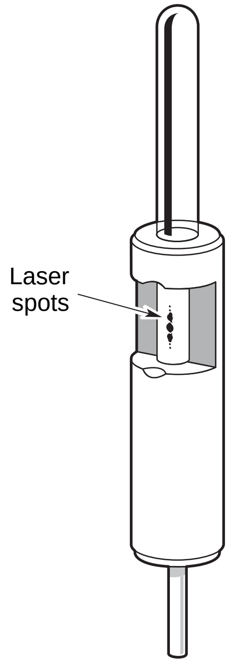
  To make a manual side-to-side adjustment on a 90 degree refractive sampling configuration, insert the provided tool (shown below) through the front adjustment slot on the right side of the NXR FT-Raman Module. When the adjustment tool engages the internal slot on the sampling configuration, rotate the tool in either direction.

The following illustration shows the location of the adjustment slot:



The correct sample position is closely approximated by the focal point of the *visible* HeNe laser beam. When the beam converges and strikes the sample, it forms red spots on the sample surface as shown in the following illustration.

Warning Do not stare into the HeNe laser beam or its reflections. ▲



These spots indicate the part of the sample that will be measured when data are collected. By positioning the sample at the center of the red spots, you can minimize the amount of fine adjustment that will be required later. You can make this rough position adjustment while the sample compartment cover is open. The spots are more visible when you use the KBr test sample.

Note For verifying instrument performance, the red spots should be in the center of the sample. ▲

Fine sample position adjustment can be made only while the excitation laser beam (or white light beam) is striking the sample. This means that the sample compartment cover must be closed. Use the Bench tab in the Experiment Setup dialog box (available through the Collect menu in OMNIC) to display a live interferogram while you adjust the sample position. The goal of the adjustment is to maximize the detector signal (interferogram or spectrum peak height) displayed on the screen. See the OMNIC For Raman on-line help system available in the Raman menu for details on using the live display during sample position adjustment.

Optional filters, lenses, and accessories

If your system has a 1064 nm laser, neutral density filters and a polarization rotator are available for use in the sample compartment. Beam focusing lenses are available for systems with either the 1064 nm laser or the 976 nm laser.

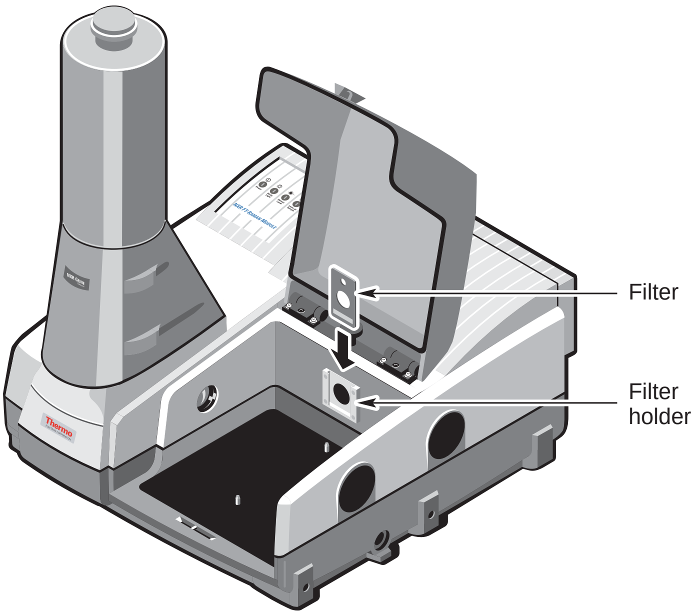
Neutral density filters allow you to reduce the laser power entering the sample compartment. This may be useful when you have a heat-sensitive (usually dark) sample. Use the following equation to determine what the actual laser power will be when using a neutral density filter:

Actual laser power = (Laser power setting) (10 $^{\text{-absorbance of filter}}$)

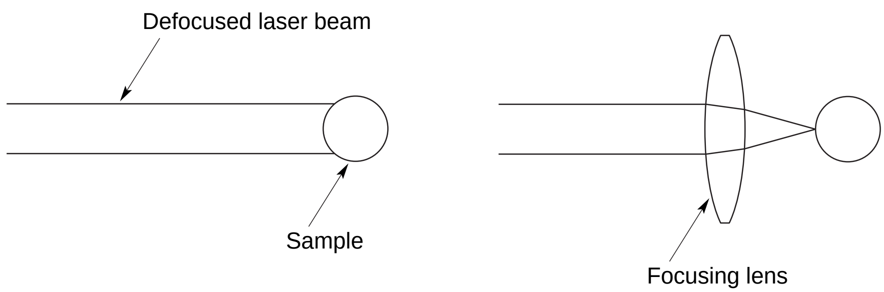
Important Reduce the laser power to less than 0.5 watt before installing a filter. Higher laser power can burn a hole in the filter. See the OMNIC For Raman on-line help system for details on setting the laser power. ▲

You can use the polarization rotator to rotate the polarized excitation laser beam from its perpendicular orientation to a horizontal one. A holder for the polarization rotator and the optional filters is located on the rear wall of the sample compartment. (See the following illustration.)

To place a filter or lens in the path of the excitation laser beam, slide the filter or lens into the holder. Leaf-springs in the holder keep the filter or lens in place.



You can use a beam focusing lens with a sampling configurations to focus the normally defocused laser beam onto a smaller area of the sample surface. (See the sections that describe the configurations for information on using the lens.)



Defocused laser beam ***Beam focused with focusing lens***

White light source

The white light source is located behind the sample compartment. To turn on this source and adjust its intensity, choose Experiment Setup from the Collect menu, click the Bench tab and set the White Light parameter.

When the white light source is on, the White Light indicator light on the front panel is lit. You can use the white light source to generate a reference spectrum for performing an instrument correction. Except for this purpose, the white light source should normally be off. See the OMNIC For Raman on-line Help system available in the Raman menu for details on setting White light.

Detectors

The NXR FT-Raman Module can be equipped with an indium-gallium arsenide (InGaAs) detector, a thermoelectrically cooled InGaAs detector, a germanium (Ge) detector, or a combination of both a Ge detector and a thermoelectrically cooled InGaAs detector.

Selecting the detector

Follow these steps to select the detector:

1. **Make sure Raman Accessory is turned on in the Raman menu.**

   A check mark appears next to the command name when the command is turned on. To turn on the command, choose it from the menu. This makes the needed OMNIC For Raman features available in the Collect menu.

2. **Choose Experiment Setup from the Collect menu.**

   The Experiment Setup dialog box appears.

   For more information about using Experiment Setup, see the OMNIC For Raman on-line Help system.

3. **Click the Bench tab.**

4. **Set Detector to InGaAs, TEC InGaAs, or Ge.**

The Sample Compartment parameter must be set to Raman to select the Raman detectors. See the OMNIC For Raman on-line Help system available in the Raman menu for more information on the Detector parameter.

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 if you are using the 1064 nm laser. If you are using the 976 nm laser, fluorescent lights can produce spikes at 3711, 1390, and 388 shifted wavenumbers. Make sure all instrument doors are closed before you collect Raman data.

Cooling the Ge detector

The germanium detector requires cooling with liquid nitrogen before you collect data. Cooling the Ge detector takes from 20 to 45 minutes when starting at room temperature, so it is advisable to cool the detector first and prepare your samples while you are waiting.

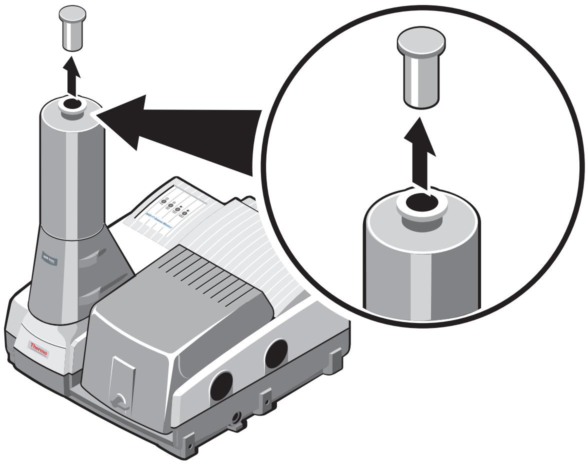
You will need the following items:

- A one-liter laboratory dewar.
- 4 liters of liquid nitrogen.
- Protective gloves
- Protective goggles

⚠ **Warning** Liquid nitrogen is extremely cold and therefore hazardous. When performing this procedure, be careful not to allow liquid nitrogen to come in contact with your skin. Wear protective gloves and goggles and follow standard laboratory safety practices. ▲

Use the following procedure to cool the detector:

1. **Remove the liquid nitrogen stopper from the top of the detector.**



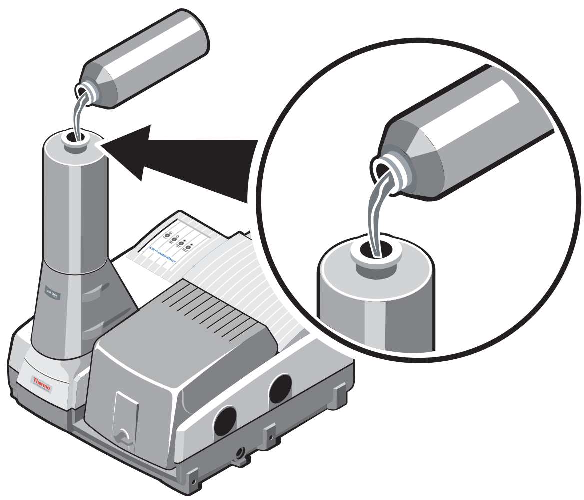
⚠ **Warning** Liquid nitrogen is extremely cold and therefore hazardous. When filling the laboratory and the detector dewars, be careful not to allow liquid nitrogen to come in contact with your skin. Wear protective gloves and goggles and follow standard laboratory safety practices. ▲

2. **Fill a laboratory dewar with liquid nitrogen.**

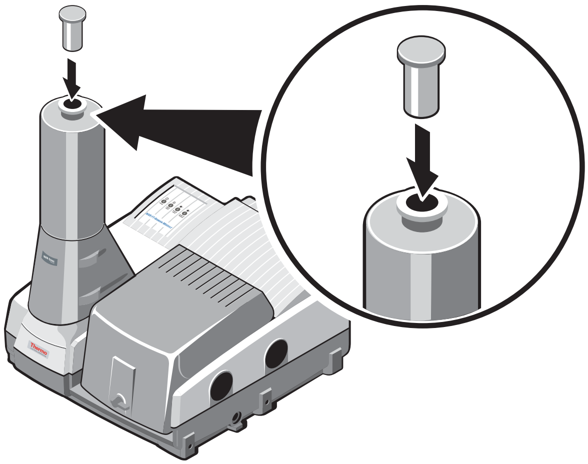
Important To prevent damage to system components, avoid spilling liquid nitrogen into the detector housing. Fill the detector dewar slowly; filling it too quickly can cause it to expel liquid nitrogen. ▲

3. **Slowly and carefully pour about one liter (approximately 1 quart) of liquid nitrogen from the laboratory dewar into the detector dewar.**

   Wait until the vapor plume disappears, and then repeat this step until the detector dewar has been filled. An empty detector dewar will hold about 2.5 liters of liquid nitrogen, but you will need 4 liters because approximately 1.5 liters of liquid nitrogen will boil off while you are filling the dewar.



4. **When the detector dewar is filled, reinsert the liquid nitrogen stopper.**



The stopper may wobble and bounce until the liquid nitrogen stops boiling vigorously. This is not a reason for concern.

It will take about 20 to 45 minutes for the detector to cool enough to produce a signal. During this time do not attempt to perform an automatic alignment of the system.

Note Because the Ge detector element is sensitive to room light, you should make sure the liquid nitrogen stopper is in place in the top of the detector dewar when you collect data. ▲

Polarizer option

If your NXR FT-Raman Module has a 1064 nm laser, a motorized polarizer option can be installed under the main cover of the module by qualified Thermo Electron service personnel. This allows you to polarize the beam emitted by the sample before it enters the interferometer. You can move the polarizer into or out of the beam and rotate it to either 0 or 90 degrees by setting the Polarizer parameter in the Experiment Setup dialog box.

⚠ **Warning** Do not open the FT-Raman Module main cover. Exposure to harmful radiation may result. ▲

Adjustable aperture

A motorized aperture is an option that can be installed under the FT-Raman Module main cover by qualified Thermo Electron service personnel. This allows you to control the beam's size (and thus limit its energy) before it enters the interferometer.

⚠ **Warning** Do not remove the FT-Raman Module main cover. Exposure to harmful radiation may result. ▲

To adjust the size of the aperture opening, choose Experiment Setup from the Collect menu, click the Bench tab and set the Aperture parameter. The allowed values of Aperture are 0 (almost totally closed) to approximately 150 (totally open). The aperture should normally be totally open except when you are collecting a high-resolution spectrum. See the OMNIC For Raman on-line Help system available for details on setting the aperture.

Note If your aperture setting is too large to achieve the currently specified resolution, a message will appear when you close the Experiment Setup dialog box stating that the optimum aperture setting will be used instead. Choose OK to use the optimum aperture setting, or choose Cancel to use the setting you selected. If you choose Cancel, data you collect may not have the specified resolution.

Also, to avoid unnecessary attenuation of the Raman signal, do not use an aperture setting smaller than is required to achieve the specified resolution. ▲

Aligning the Interferometer

To ensure that the system is capable of producing the strongest possible signal, you need to align the interferometer periodically; once a week is usually sufficient. Use the following procedure to perform an automatic alignment of the spectrometer's interferometer.

1. **Turn on the system components.**

   Turn on the NXR FT-Raman Module power before you turn on the spectrometer power, and make sure the computer is turned on.

Important The computer must be turned on before you start OMNIC. ▲

2. **Start the OMNIC software.**

3. **Install the CaF_2 or XT-KBr beamsplitter in the spectrometer.**

   Use Spectrometer Help Topics in the Help menu to find details on installing the beamsplitter.

4. **Select the detector to use.**

   Use Spectrometer Help Topics in the Help menu to find details on selecting the detector.

5. **Enable the excitation laser by turning the keyswitch on the rear panel clockwise to the vertical (ON) position.**

6. **Set the OMNIC software for FT-Raman operation by choosing Use Raman Accessory from the Raman menu.**

   A check mark appears next to the command name when the command is on:

7. **Set the optical bench parameters.**

   To set the parameters, choose Experiment Setup from the Collect menu and click the Bench tab.

   Set the parameters as shown below. Set Focus to the value that you typically use for samples contained in an NMR tube. Set Aperture to the maximum allowed value, approximately 150. If the excitation laser is on, turn it off by clicking the OFF button in the Laser box. The Spectral Range values default to a setting based on the Final Format setting you selected on the Collect tab in the Experiment Setup dialog box. The Sample Compartment must be set to Raman to use the Raman detectors. See the OMNIC For Raman on-line Help system available in the Raman menu if you need more information on setting the parameters and turning off the laser.

   Raman Source
   - Laser: OFF
   - White Light: Low (or Med)

   Detector: InGaAs (or Ge if installed)
   Aperture: 150.00
   Polarizer: Out
   Velocity: 0.3165 for InGaAs (or TEC InGaAs) or 0.4747 for Ge
   Gain: 1.0

   Spectrum: Off
   Tone: Off

   Raman Module Control
   - Focus: 150 (or as required)
   - Side To Side: 150 (or as required)
   - Sample Compartment: Raman

Note Do not close the Experiment Setup dialog box. If you inadvertently close the dialog box, choose Experiment Setup again and make sure the parameters are set correctly. ▲

8. **Install the KBr test sample in the sample compartment.**

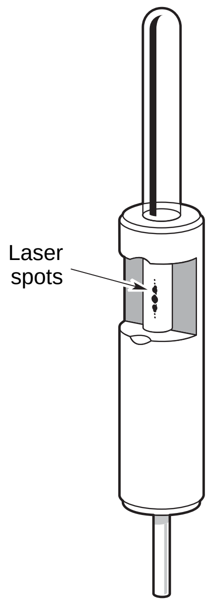
   A test sample of KBr is provided with the system. The sample is contained in an NMR tube or a vial.

⚠ **Warning** Do not stare into the HeNe laser beam or its reflections. ▲

Note The drawing below does not show the KBr sample in a vial, but the instructions for positioning the sample at the laser focus are equally applicable when you are using a vial. ▲

9. **Position the sample at the focus of the HeNe laser beam.**

   Leave the sample compartment cover open while you adjust the sample position. Position the sample so that HeNe laser beam shines red spots at approximately the center of the sample surface. (See “Adjusting the sample position in the standard configurations” in the “Description and Setup” chapter for details on adjusting the sample position.)



10. **Close the sample compartment cover.**

11. **If an interferogram is visible, click the Diagnostic tab in the Experiment Setup dialog box, and then click the Align button to perform an automatic alignment of the interferometer.**

If no interferogram is visible, see “Accessory scans but no interferogram displayed” in the “Troubleshooting” chapter for instructions.

When the alignment is finished, choose OK to close the Experiment Setup dialog box.

Note 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. ▲

Turning Off the System

Use the following procedure to turn off the system components.

1. **Turn off the Raman laser.**

   To do this, click the OFF button in the Laser box on the Bench tab in the Experiment Setup dialog box and then turn the keyswitch on the rear panel to the horizontal position.

Important *For best stability, the FT-Raman Module power and the spectrometer power should be left on at all times.* Turn off the excitation laser power if the system will not be used for several hours. s

Note The fans in the FT-Raman Module produce a small amount of vibration and may generate noise spikes in infrared spectra below 600 cm^{-1}. Before collecting infrared data in the far-IR region using the main optical bench, you may need to turn off the NXR FT-Raman Module power. ▲

2. **Exit OMNIC.**

3. **Turn off the FT-Raman Module power.**

   The power switch is on the rear panel.

4. **Turn off the spectrometer power.**

   The power switch is on the rear panel.

5. **Use Shut Down (available through the Windows Start button) to exit Windows.**

6. **Turn off the display monitor and computer if they were not shut off automatically when you exited Windows.**

Note When you turn the system back on, the power to the NXR FT-Raman Module should be turned on before you turn on the power to the spectrometer. You can turn on the computer before or after you turn on the module and the spectrometer. ▲

Checking the Detector Cool-Down Time

The optional germanium detector dewar can lose vacuum over time. As this happens, the time it takes the detector to become cool gradually increases. Frost can start to form on the outside of the dewar, possibly damaging the instrument.

It is a good idea to check the cool-down time periodically and keep a record of it. If the cool-down time exceeds 45 minutes, call your Thermo Electron service representative to have the detector dewar reevacuated.

Follow these steps to check the detector cool-down time.

1. **Wait until the detector reaches room temperature.**

   If the detector has been cooled recently, this can take up to eight hours.

⚠ **Warning** Liquid nitrogen is extremely cold and therefore hazardous. Avoid contact with skin. Wear protective gloves and goggles and follow standard laboratory safety practices. ▲

2. **Note the time and fill the detector dewar with liquid nitrogen.**

   Close the detector fill cover. The preceding section explains how to fill the detector dewar.

3. **Mount the supplied KBr sample in the sample compartment.**

   Your sample may be contained in an NMR tube or a vial. Position the sample as well as possible in the HeNe laser beam, and then shut the sample compartment cover.

4. **Make sure Use Raman Accessory is turned on in the Raman menu. Then choose Experiment Setup from the Collect menu and click the Bench tab.**

5. **Make sure the excitation laser is on and adjust the laser power to about 0.5 watt in the sample compartment.**

   The laser is on when the ON option button is selected in the Laser box on the Bench tab. To adjust the laser power, click the up and down arrows to the right of the text box.

6. **When the interferogram peak becomes visible in the live display, maximize it by adjusting the sample focus and side-to-side position.**

7. **Note the time when the detector signal is strong enough to allow you to achieve a normal interferogram intensity.**

   The normal intensity (Peak To Peak value) is what you have achieved in the past when verifying instrument performance. Record the elapsed time. This is the detector cool-down time.

Replacing Fuses

The main fuses in the NXR FT-Raman Module power supply are accessible from the rear panel. You may need to replace these fuses if the Power indicator light does not light when you turn on the accessory power switch. You must also change fuses if you change the input line voltage. Use replacement fuses with the specifications shown in the following table:

For this input line voltage...	*Use this fuse type...*
120 VAC	2A, 250V, 5 by 20 mm, slow-blow
240 VAC	1A, 250V, 5 by 20 mm, slow-blow

⚠ **Warning** Avoid shock hazard. Always turn off the FT-Raman Module and disconnect the power cord before you check or change the fuses. ▲

Follow these steps to check or change the fuses:

1. **Turn off the FT-Raman Module power by pressing the power switch on the rear panel.**

2. **Disconnect the power cord from the rear panel of the accessory.**

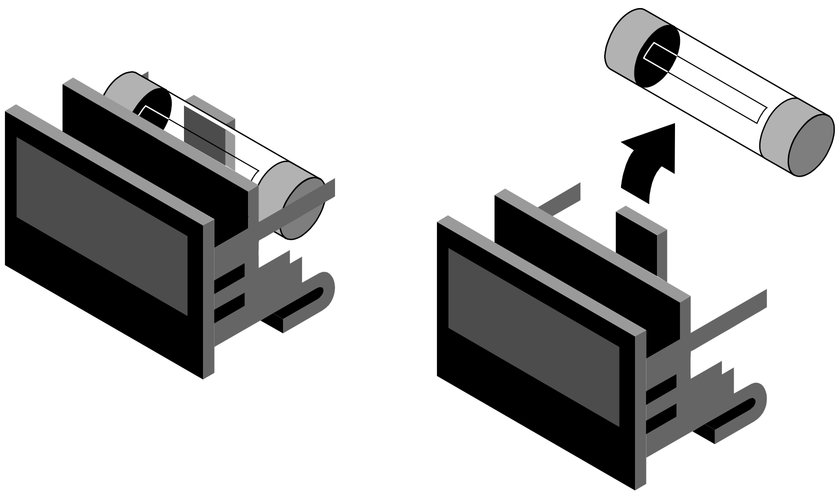
3. **Push the fuse holder latch to the left.**

   Use a small screwdriver to pry the holder away from the rear panel, and then pull the holder out of the power supply.

4. **Slide the fuse holder out of the rear panel.**

5. **Pull the fuses out of the holder.**

   To remove a fuse, pull the latch away from the fuse and then lift the fuse out of the holder.



Warning Avoid shock hazard. Always use an exact replacement for the fuses. s

6. **Check the fuses and replace them if necessary.**

   Use fuses with the specifications shown in the table at the beginning of this chapter.

7. **Slide the fuses into the holder.**

8. **Slide the fuse holder into the rear panel.**

   Be sure to slide the holder all the way into the rear panel. You should hear the latch click into place.

9. **Reconnect the power cord and turn on the module power.**

Troubleshooting

This chapter describes some problems that may occur infrequently while you operate the NXR FT-Raman Module and explains the corrective action you should take in each case. If a problem persists, contact Thermo Electron Technical Support for assistance. Outside the United States, contact your local service representative.

In addition, a diagnostics program is provided with your system for checking the performance of accessory components in case a problem occurs. See “Using the Raman Bench Diagnostics software” at the end of this chapter for details.

⚠ **Warning** To avoid exposure to high voltages and laser radiation, do not open the accessory main cover. This cover can be opened only by qualified Thermo Electron service personnel. ▲

Interferogram peak is not centered

If the interferogram peak is not centered in the live display of the Experiment Setup dialog box, click the Reset Bench button on the Diagnostics tab window to center it.

FT-Raman Module scans but no interferogram is displayed

If the system does not display an interferogram during data collection, follow this troubleshooting procedure:

1. **Make sure all the accessory covers are closed and tightened down.**

   The covers must be closed for safety and to prevent room light from saturating the detector. Make sure the NXR FT-Raman Module sample compartment cover is latched shut. When the laser is on, you should hear the safety interlock system click when you close the sample compartment cover.

⚠ **Warning** To avoid exposure to high voltages and laser radiation, *never* open the accessory main cover. This cover can be opened only by qualified Thermo Electron service personnel. ▲

2. **Set the optical bench parameters.**

   To set the parameters, choose Experiment Setup from the Collect menu, and then click the Bench tab. Make sure Detector is set to the appropriate detector type. Set White Light to Low or Medium. If you are using the optional Ge detector, make sure it is cooled with liquid nitrogen.

3. **Insert the provided KBr test sample (white powder) and close the sample compartment cover.**

   If your sampling configuration or accessory will not accept the KBr sample provided, load your own KBr powder (or some other white powder) into your configuration or accessory.

4. **If an interferogram is visible, click the Align button to start automatic alignment.**

   If the system still does not display an interferogram during data collection, you should:

   - Contact Thermo Electron Technical Support for assistance, if you have a Nicolet NXR FT-Raman 9610 or a Nicolet NXR FT-Raman 9650 spectrometer.
   - Check the main bench as described in the next step, if you have an NXR FT-Raman Module.

5. **Check the performance of the spectrometer.**

   Make sure Use Raman Accessory is turned off in the Raman menu. (To do this, click Use Raman Accessory so that there is not a check mark next to the command.)

   Install the KBr beamsplitter and set Beamsplitter to KBr in the Experiment Setup dialog box.

   Set Detector to DTGS.

   Check the performance of the main bench. See the OMNIC on-line help system for complete information.

   Switch the system back to FT-Raman operation. If there is still no interferogram, contact Thermo Electron Technical Support for assistance.

Noisy spectra

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

Important If you are using a Ge detector, make sure you install a neutral density filter to prevent detector saturation. ▲

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. **Choose Experiment Setup from the Collect menu, and then click the Bench tab.**

3. **Select Low from the White Light drop-down list box, and set the Power box to 0.5 (watts).**

4. **Install the provided KBr test sample and close the sample compartment cover.**

5. **If an interferogram is present, click the Diagnostic tab in the Experiment Setup dialog box, and then click the Align button.**

   If an interferogram is not is not present, proceed to step 8. If an interferogram is present and the problem persists after the alignment is finished, proceed to the next step.

6. **If the optional polarizer is installed, make sure the polarizer is not in the beam path.**

   Polarizer should be set to Out. (This applies only to systems with the 1064 nm laser.)

7. **Make sure the adjustable aperture is fully open.**

   Aperture should be set to the maximum value (approximately 150).

8. **If an interferogram is not present after you complete step 4, select High in the White Light drop-down list box.**

   Complete steps 4 through 7 again. If there is still no interferogram, click reset Bench on the Diagnostic tab, then click Align. If the problem continues, contact Thermo Electron Technical Support for assistance.

Spikes or excessive noise in mid-IR spectra

If you have an NXR FT-Raman Module, and the mid-IR spectra you collect with your spectrometer contain spikes or excessive noise in the region below 600 cm^{-1}, the cause may be vibrations from the fans in the module. You can eliminate these spikes and the noise by turning the FT-Raman Module off before collecting FT-IR data with the main bench. Follow these steps:

1. **Turn off the excitation laser.**

   To do this, click the OFF button in the Laser box on the Bench tab in the Experiment Setup dialog box and then turn the keyswitch on the rear panel to the horizontal position. (It is possible to turn off the laser by just turning the keyswitch, but we recommend that you first click the OFF button.)

2. **Turn off the FT-Raman Module power.**

   The power switch is on the rear panel. You can begin collecting data now.

To turn the FT-Raman Module back on, follow these steps:

1. **Turn off the spectrometer power.**

2. **Turn on the FT-Raman Module power.**

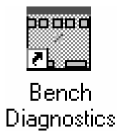
3. **Turn on the spectrometer power.**

No laser power in the sample compartment

If there is no laser power in the sample compartment when you attempt to collect data, check the rear panel of the module. (For a description of this panel, see “Rear panel” in the “Description and Setup” chapter.) If the laser interlock keyswitch is in the horizontal position, the laser is turned off. Turn the laser on by turning the key clockwise to the vertical position.

If there is still no laser power in the sample compartment, the safety interlock jumper plug may be missing from a connector on the rear panel. (For the location of this plug, see “Rear panel” in the “Description and Setup” chapter.) To reinstall the plug, push it onto the connector. If you cannot locate the plug and need a new one, contact your Thermo Electron Technical Support.

Using the Raman Bench Diagnostics software



If your system is not performing properly, you can use the Raman Bench Diagnostics software to check the performance of NXR FT-Raman Module components. To use the software, start the Raman Bench Diagnostics program and then follow the instructions that appear on the screen.

Preparing for a service call

Whenever your FT-Raman Module requires service, you will need to set up a temporary controlled area. During FT-Raman servicing, dangerous exposure to invisible laser radiation produced by the excitation laser is possible. Allow only essential personnel to enter the work area.

Specifications

The NXR FT-Raman Module specifications are shown in the following table:

Characteristic	*Specification*	
Spectral range	with 1064 nm laser	with 976 nm laser
Stokes	3500-100 cm^{-1}	4350-100 cm^{-1}
anti-Stokes	3100-200 cm^{-1}*	
Sampling configurations	180° reflective 90° refractive bottle holder	
Instrument correction	built-in calibrated white light source	
Sample compartment	29 cm (11.4 in) wide 30 cm (11.8 in) deep 22 cm (8.7 in) high	
Dimensions	with Ge detector	without Ge detector
width	61 cm (24 in)	61 cm (24 in)
depth	71 cm (28 in)	71 cm (28 in)
height	88 cm (35 in)	38 cm (15 in)
Power requirement	2 A at 120 VAC 1 A at 230 VAC	
Weight	with 1064 nm laser	with 976 nm laser
with Ge detector	51 kg (112 lb)	41 kg (89 lb)
with other detector	45 kg (100 lb)	35 kg (77 lb)

* With optional notch filter

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