Nicolet NIR with OMNIC Paradigm software



User Guide

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For technical support, please contact: www.thermofisher.com

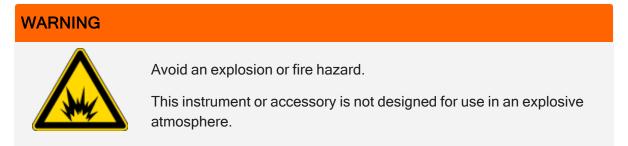
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Chapter 1

NIR Module

The optional Thermo Scientific NIR Module offers convenient, reproducible diffuse reflection sampling with the Nicolet[™] iS[™]50 spectrometer using the Integrating Sphere or a fiber optic accessory.

Figure 1-1: NIR Module



A Thermo Scientific SabIR Probe

- B Fiber optic sampling area (optional)
- C Integrating Sphere sampling area
- D Thermo Scientific NIR module (mounts on the left side of the spectrometer)

The NIR Module provides these enhanced features:

- Integrated design
- Push button configuration
- Two sampling options: local (Integrating Sphere) and remote (fiber optic, optional)
- Up to two dedicated detectors
- Automated internal reference
- Standards wheel (contains 2%, 10%, 20%, 40%, and 80% glass filters and a polystyrene sample)

The optional fiber optic sampling area can be used with the Thermo Scientific SabIR diffuse reflection probe, or with probes from other manufactures if they have SMA connectors. For information about additional sampling modules for the Nicolet iS50 spectrometer, contact our sales representative in your area.



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

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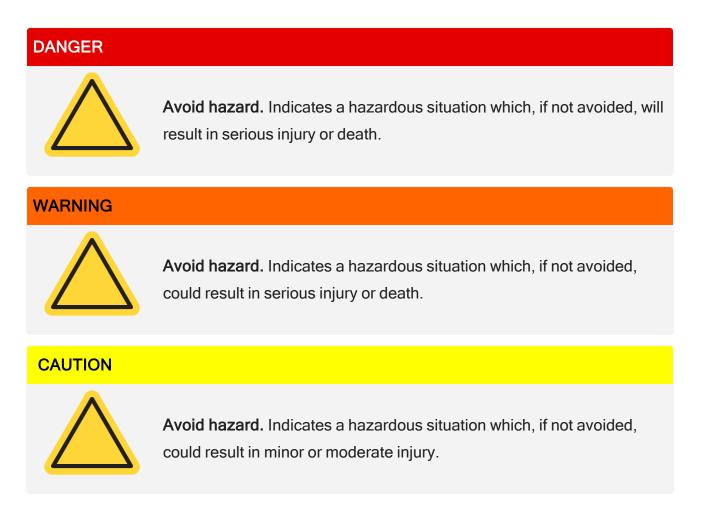
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Conventions used in this manual



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.

Operating precautions

Read these operating precautions before using the NIR Module to avoid damaging your equipment.

Precautions for the Integrating Sphere:

- Do not pour liquids directly onto the Integrating Sphere.
- Do not use harsh detergents, chemicals, or abrasives to clean the surface of the Integrating Sphere; these can damage the finish.
- Make sure your sample materials will not react with the sapphire crystal sampling window.
- Use solvents that do not react with sapphire crystals when cleaning the sampling window.
- Avoid scraping the sampling window with samples or sample containers.
- Protect the electrical port on the surface of the Integrating Sphere. If the connector is clogged or jammed, contact a Thermo Scientific service representative.

Precautions for fiber optic cables:

- Do not drop the cables or knock them against a hard surface.
- Do not bend the cables or tightly coil them. The radius of the coils for the SabIR Probe should be at least 30 cm. For other fiber optic accessories, refer to their user guides.
- Avoid twisting the cables when attaching the connectors to the ports.
- Do not over-tighten or use tools to tighten the connectors when attaching the cables to the ports.
- Do not touch the end of a fiber optic cable connector or use solvents to clean it.
- Keep a protective cap on each end of the cable when it is not in use.

Precautions for the SabIR Probe:

- Do not drop the probe or knock it against a hard surface.
- Do not subject the probe to rapid or excessive changes in temperature.

- Wipe off spilled chemicals immediately.
- Make sure your sample material will not react with the sapphire sampling window.
- Do not allow hard materials (glass, metal, ceramic, etc.) to contact the sampling window.
- Use solvents that do not react with sapphire crystals when cleaning the sampling window.
- Power off the instrument before connecting the SabIR Probe.
- Make sure the probe is clean before you insert it into the holster.
- Do not use the probe by itself to measure liquids. When sampling liquids, use the transflectance adapter available for the probe.

Chapter 3

Specifications

Feature	Specification
Crystal material	Sapphire
Internal reference	Diffuse gold
Detector	Dedicated Indium Gallium Arsenide (InGaAs)

Table 3-1: Specifications for the Integrating Sphere sampling area

Table 3-2: Specifications for the optional fiber optic sampling area

Feature	Specification
Fiber optic connectors	SMA type
Detector	InGaAs
Probe (optional)	Thermo Scientific SabIR diffuse reflection probe
SabIR Probe internal reference	Spectralon®

The NIR Module spectral range is dependent upon the spectrometer configuration. The table below shows the available configurations and their corresponding spectral ranges.

Source	Beamsplitter	Detector	Spectral range
White light	CaF2 ¹	InGaAs ²	12,000 - 3,800 cm-1
White light	XT-KBr ³	InGaAsb	11,000 - 3,800 cm-1

Table 3-3: NIR Module available spectral ranges

Note The NIR Module requires a CaF2 window installed in the external beam port on the left side of the spectrometer.

¹Calcium Fluoride

²Indium Gallium Arsenide

³Extended range Potassium Bromide

Compatible software

Table 4-1: Software compatible with the NIR Module

Software	Use for
OMNIC Paradigm	Collecting and analyzing NIR spectra.
TQ Analyst™	Creating quantitative and classification methods for use with OMNIC Paradigm software or RESULT [™] software. TQ Analyst software provides an extensive suite of chemometrics features that you can use to identify raw materials, perform quantitative analysis, and take spectral measurements. For more information, refer to the help system in your TQ Analyst software.
RESULT	Running Integrating Sphere or SabIR quantitative methods that have been transferred to any Thermo Scientific Antaris [™] near-infrared analyzer that uses RESULT version 3.0 or higher. For information about adding TQ Analyst methods to a RESULT workflow, please refer to your RESULT Integration Software User Guide. Note: Only methods created in TQ Analyst software version 8.4 or higher can be transferred from OMNIC Paradigm to RESULT software.

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Chapter 5

Integrating Sphere sampling

The Integrating Sphere is a fast and easy-to-use tool for diffuse reflection sampling of solids and powders.

Integrating Sphere features

The major features of the NIR Module Integrating Sphere are highlighted below.

Figure 5-1: NIR Integrating Sphere sampling area features



А	Touch Point
В	Sample accessory holder

C Sampling window D Gold reference

• **Sampling window.** Light from the spectrometer passes through this transparent sapphire window to the sample.

CAUTION



Avoid eye hazard. Do not stare at the beam that exits the Integrating Sphere sampling window when the spectrometer is powered on and the Integrating Sphere beam path is selected.

- **Sample accessory holder.** Mounts over the sampling window and holds Thermo Scientific sample cups and tablet holder accessories.
- Internal gold reference. Can be used to collect background spectra. When enabled, the reference automatically moves into the beam path during background data collection and out of the beam path during sample collection.
- Touch Point. Automatically configures the spectrometer for Integrating Sphere sampling.

Samples compatible with the Integrating Sphere

The Integrating Sphere can be used to measure a wide variety of samples. Some examples are provided below. Samples may be measured directly or in clear glass or plastic containers.

Types	Characteristics	Examples
Solids ¹	Must have a rough or diffuse surface	Coated textiles, paper, wood, polymers, and plastics (especially plastics that have a milky, opaque appearance)
Tablets ²	Must have a reflective surface	White or colored doses of aspirin, acetaminophen, or other drugs
Powders, granules, pellets ³	Particles can be any size, from fine to rough ground up to large pellets	Polyethylene, excipients, flour
Liquids, gels ⁴	Must be a suspension or opaque, or use a dip probe	Milk, colloidal suspensions, dispersed solids

Table 5-1: Samples compatible with the Integrating Sphere

NOTICE

- Do not pour liquids directly onto the sampling area of the Integrating Sphere, especially the area around the electrical connector for the Sample Spinner accessory.
- Make sure your sample materials and cleaning solvents will not react with the sapphire sampling window. Refer to the <u>Cleaning the Integrating Sphere sampling window</u> section for more information.
- Avoid scraping the sampling window with samples or sample containers.

¹Use enough sample to completely cover the Integrating Sphere window. Place abrasive solids in a clear glass or plastic container.

²May be placed directly on the Integrating Sphere window or in one of the tablet holder accessories.

³May be measured in clear glass, flat-bottom vials, clear plastic bags, or in Thermo Scientific sample cups. Fill the sample cup to a depth of at least 5 mm. To improve reproducibility, fill the cup with the powder until it is overflowing and then level it with a spatula. If possible, use a closed sample cup and cover.

⁴Must be measured in clear glass, flat-bottom vials or clear plastic bags. Do not use Thermo Scientific sample cups for liquids. If the liquid is clear, you can analyze it using the SabIR Probe with a transflectance adapter. See <u>Using the optional SabIR Probe</u> and <u>Sampling accessories for the</u> <u>SabIR Probe</u> for more information.

A variety of accessories are available for fast, reproducible sampling of tablets, pellets, powders, and gels. For more information, please refer to the <u>Sampling accessories for the Integrating Sphere</u> section.

Sample thickness, or the amount of sample, should be taken into consideration when using the Integrating Sphere. If a solid sample is too thin or if there is not enough of a powder sample, you may encounter problems with the spectra.

Your first experiment with the NIR Integrating Sphere

This section explains how to configure your NIR Integrating Sphere and demonstrates how to use the it to analyze a sample. You can press the Touch Point to start the process and respond to prompts in the software while it is running, or simply use software commands to achieve the same result.

SETTING UP THE INTEGRATING SPHERE

1. Start OMNIC Paradigm software. The software automatically loads the default, commonly used settings, but you can create your own settings for your specific analysis needs.

If you need to create new settings, select **New**, then enter a name for the new collection settings. Modify the settings as needed, then select **Save**. The new collection settings will be available for selection in the **Settings** dropdown menu.

NOTICE

Make sure that Sampling accessory shows as **NIR Integrating Sphere**. If it does not, a different instrument or accessory has been selected or the instrument is not installed properly.

2. Ensure that the Integrating Sphere is properly recognized. If not, go to **Configure** and select **Sample Location > NIR Integrating Sphere**.

MEASURING A SAMPLE WITH THE INTEGRATING SPHERE

CAUTION



Avoid hazard to the eye. Do not stare at the beam that exits the Integrating Sphere sampling window when the spectrometer is powered on and the Integrating Sphere beam path is selected.

Before you begin

- If the spectrometer has been powered off for more than 20 minutes, power it on and allow it to stabilize for at least an hour.
- Make sure the sampling window is clean. Please refer to the <u>Cleaning the Integrating Sphere</u> <u>sampling window</u> section for instructions and recommended solvents.
- Have your sample material on hand. For your first analysis, we recommend that you use a 325g aspirin tablet.

NOTICE

- Do not pour liquids directly onto the sampling area of the Integrating Sphere, especially not the area around the electrical connector for the sample spinner.
- Make sure your sample materials and cleaning solvents will not react with the sapphire window. Refer to the Cleaning the Integrating Sphere sampling window section for more information.
- Avoid scraping the sampling window with samples or sample containers.



Figure 5-2: Solid sample ready for measurement with the Integrating Sphere

- To measure a tablet with the Integrating Sphere
- 1. If you have not already done so, go to **Configure> Sample Location > NIR Integrating Sphere** (or press the Integrating Sphere Touch Point) to configure the instrument for Integrated Sphere sampling.

Figure 5-3: Integrating Sphere Touch Point



- 2. In the Dashboard, select **Preview and Measure Background**. This will create a new spectra tab and open a background preview. Select **Start Background Measurement** to continue, then wait for the process to complete. For more information, see <u>Collecting backgrounds with the</u> Integrating Sphere
- 3. When the background measurement is done, place the sample in the center of the Integrating Sphere sampling window. See <u>Samples compatible with the Integrating Sphere</u> for more information.
- 4. After loading the sample, you can preview the sample and start the measurement. Select **Start Sample Measurement** and wait for the process to complete.

Note If you do not enter a name for your measurement (when prompted or in the Dashboard settings), the measurement name will automatically default to the current time and date. If desired, you can rename the measurement later.

5. Remove the sample and clean the Integrating Sphere window.

Moisten a clean white paper napkin with isopropyl alcohol and use it to wipe the window. Dry the window with a clean napkin.

Collecting backgrounds with the Integrating Sphere

A background is a reference spectrum that accounts for the unique optics of the sampling module and the instrument. Each sample spectrum is ratioed against a background so that the final spectrum is free of these features.

You can collect backgrounds using an internal or external reference (or using no reference). The most recent background spectrum remains in memory and is compared against sample data until a new background spectrum is collected.

When the internal reference is used, a gold mirror located underneath the Integrating Sphere window closes during background collection, causing all of the light beam to be reflected back into the Integrating Sphere. A typical background spectrum using the internal reference should resemble the following.

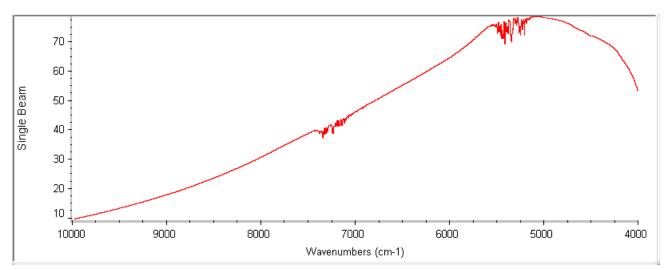


Figure 5-4: Typical gold reference background spectrum taken with the Integrating Sphere

Tip Once a material is selected for a background reference, make sure to use the same material each time you run your experiment.

Center the reference over the Integrating Sphere window and then respond to the prompt by choosing OK or pressing the Integrating Sphere Touch Point. Do not move the reference until the instrument has finished collecting the data.

Sampling accessories for the Integrating Sphere

Optional Thermo Scientific accessories for the Integrating Sphere are described in the table below, followed by additional information. To order an accessory, contact our local sales or service representative or see our contact information at the beginning of this document or help system.

Table 5-2: Optional Thermo Scientific accessories for the Integrating Sphere



Does this...

Holds Thermo Scientific sample cups and tablet holder accessories.

The sample accessory holder attaches to the top of the NIR Module. The kit includes two sample cup rings: a concentric (centering) ring and an eccentric (offset) ring. The concentric ring can be used to achieve consistent positioning with the sample cups and the sample accessory holder. The eccentric ring works with the sample accessory holder and the 5 cm sample cups to allow sampling over a wider area. Refer to the Using the Sample Cups and Rings section for more information.

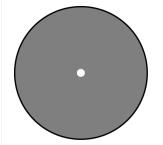
These come in packages of three sample cups. Each cup has a 4.78 mm low OH quartz window and can be used with the sample accessory holder and sample rings to measure powders. The open sample cups are not recommended for liquids and gels.

This accessory...

Tablet holder kit



Universal tablet holder



Custom tablet holder

Viscous Liquid Sampler (VLS)



Does this...

The tablet holder kit contains one universal tablet holder, a tablet holder ring, and two custom tablet holders. The tablet holder accessories fit in the sample accessory holder.

The universal tablet holder adjusts to fit snugly around a round tablet for consistent sampling. Please refer to the<u>Using the Universal Tablet Holder</u> section for more information.

The custom tablet holders can be cut to fit the exact dimensions of a tablet. The custom tablet holders require the tablet holder ring. See the <u>Using the Custom</u> <u>Tablet Holders</u> section for details.

The VLS allows you to run transflection experiments with viscous liquids. The VLS fits in the sample accessory holder. For detailed information about the VLS, please refer to the document or topic with that title.

This accessory...

Does this...

5 cm sample spinner



The 5 cm sample spinner allows multipoint sampling for large particles such as pellets and grains. The Sample Spinner fits the 5 cm open sample cups. For detailed information about the Sample Spinner, please refer to the document or topic with that title.

USING THE SAMPLE ACCESSORY HOLDER

- To install the sample accessory holder
- 1. Insert the tab into the slot.



Α	Tab
В	Slot

2. Lower the holder onto the NIR Module (a magnet holds it in place).



- To remove the sample accessory holder
- 1. Remove all sampling materials and accessories from the sample accessory holder.
- 2. Lift the accessory holder until it is perpendicular with the NIR Module, and then carefully pull up on the accessory holder.

USING THE SAMPLE CUPS AND RINGS

When used with the sample accessory holder and the sample cup rings, the sample cups allow you to run multiple collections of a powder in different sampling positions. The concentric ring opening is centered around the Integrating Sphere window and can be used with all sizes of the open and closed sample cups. The eccentric ring is offset around the Integrating Sphere window, and can be used with the 5 cm sample cup to conduct experiments covering all areas of the sample surface.

The sample cup rings have an indicator that can be aligned with indentations at 60° increments around the circumference of the sample cups. When performing multiple data collections on a sample, you can use these indicators, along with the indentations in the sample cups, to rotate the sample to repeatable positions.

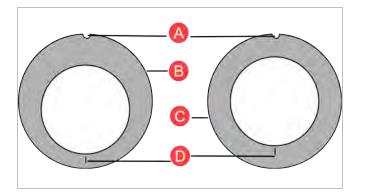


Figure 5-5: Repeatable positioning using sample cups and rings

Α	Notches
В	Eccentric ring
С	Concentric ring
D	Indicators

To install a ring, install the sample accessory holder and align the notch in the ring with the pin in the accessory holder.

To fill the sample cups

- 1. Fill the cup with the powder until it is overflowing.
- 2. Use a flat edge to level off the top of the sample cup.



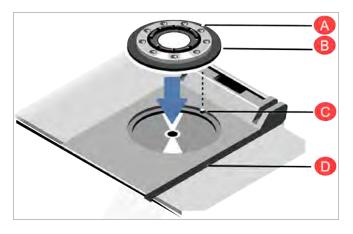
Α	Spatula
В	Powder
С	Sample cup

USING THE UNIVERSAL TABLET HOLDER

The universal tablet holder can be used along with the sample accessory holder to position round tablets of many sizes and thicknesses on the Integrating Sphere.

To install the Universal Tablet Holder

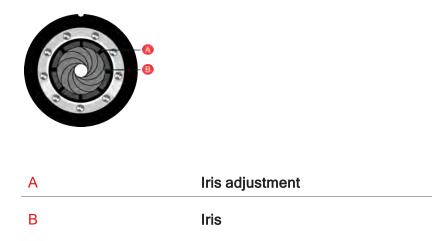
1. Align the notch with the pin.



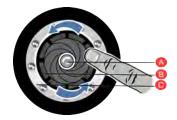
Α	Notch
В	Universal tablet pin holder
С	Pin
D	Sample accessory

✤ To adjust the iris

1. Use your finger to gently turn the black ring to open the iris.



- 2. Place the tablet in the center of the tablet holder.
- 3. Adjust the black ring until the iris is almost touching the tablet (see the image below).
- 4. Use the silver ring to adjust the height of the iris so it is approximately at the center of the tablet's width.



Α	Tablet
В	Iris adjustment
С	Height adjustment

5. Gently turn the black ring to close the iris around the tablet.

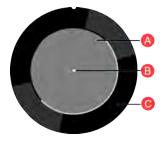
NOTICE Do not overtighten the iris or you may damage it.

А	Iris
В	Tablet
С	Iris adjustment ring

USING THE CUSTOM TABLET HOLDERS

The custom tablet holders are designed to be used with the tablet holder ring, which has a lip around the inside opening (it does not have the position indicators like the sample cup rings).

The custom tablet holders contain a small hole to reference the center of the holder. The hole aligns with the center of the Integrating Sphere when the custom tablet holder is used with the sample accessory holder and tablet holder ring.



А	Custom tablet holder
В	Centering hole
С	Tablet holder ring

Use a utility knife or die to cut the tablet holder to the precise size of the tablet.

Tip If your experiments require a great deal of precision, repeatability, and consistency, consider machining a metal tablet holder that fits the exact dimensions of the tablet and tablet holder ring.

Fiber optic sampling

The optional fiber optic sampling area adds mirrors and ports to direct the near-infrared beam to a pair of input and output optical fibers for remote sampling with a fiber optic accessory. The ports are compatible with fiber optic sampling accessories that use various sampling techniques and have standard SMA connectors, including the optional Thermo Scientific SabIR diffuse reflection probe.

Figure 5-1: NIR fiber optic sampling area features



Α	Fiber optic Touch Point
В	Fiber optic ports
С	SabIR Probe (optional)

The fiber optic sampling area includes these features:

- SMA type connectors.
- **Dedicated InGaAs detector**. The detector offers the same spectral range options as the Integrating Sphere. Please refer to the <u>Specifications</u> section for details.
- **Touch Point**. Automatically configures the spectrometer for NIR sampling with a fiber optic accessory. The Touch Point LED shows the status of the NIR Module.

Using the optional SabIR Probe

The fiber optic sampling area is optimized for use with the Thermo Scientific SabIR diffuse reflection probe to provide easy, remote sampling of solids and powders.

Figure 5-1: Optional SabIR diffuse reflection probe



SabIR Probe features

The major features of the SabIR diffuse reflection probe are highlighted below.

Figure 5-2: SabIR Probe



- **Probe and sheath**. The stainless-steel shaft has graduated rings to indicate the sampling depth. The tip contains an angled sapphire sampling window. The probe start button can be used to start or continue sample collection from a remote location. Keep the sheath on the probe shaft when the probe is not connected to the NIR Module.
- Holster. The holster is used for taking background collections with the internal reference. Make sure the probe is clean before you insert it into the holster to avoid contaminating the reference standard.
- Fiber optic cables. Cables are available in two- and three-meter lengths. The cable connectors are color-coded to correlate with the In and Out ports on the NIR Module.
- Electrical connector. Attaches to the electrical port on the NIR Module and is the power source to the probe.
- LED indicators. Three LEDs on the probe handle tell you the probe status. The LED states are described below.

Table 5-1: SabIR Probe LED states

LED status	Meaning
Green - steady Green - blinking	Instrument is ready to begin collecting data. Instrument is waiting for a response from a software prompt.
Yellow - steady	Probe is collecting data (do not move the probe when the yellow light is on).
Red	An error occurred in the software.

Connecting the Strain Relief Mechanism

The NIR Module includes a strain relief mechanism that prevents the SabIR fiber optic and electrical cables from bending excessively at the connectors. It is easiest to attach the strain relief to the spectrometer before you plug in the connectors for the SabIR Probe.

NOTICE

Make sure you use the strain relief with the SabIR Probe to protect the optical fibers when the probe is used for remote sampling.

To attach the strain relief mechanism to the NIR Module

1. Align the two pegs and the center thumbscrew on the end of the strain relief mechanism with the holes below the fiber optic connectors.



А	Thumbscrew
В	Strain relief mechanism

2. Hand tighten the thumbscrew until the surface of the strain relief mechanism presses firmly against the NIR Module.

Connecting the SabIR Probe to the NIR Module

NOTICE

- Do not drop the fiber optic cables or the probe or knock them against a hard surface.
- Do not bend the cables or touch the ends, and avoid twisting them when attaching the connectors to the ports or you may contaminate or damage the optical fibers.
- Power off the instrument before connecting the SabIR Probe to avoid damaging the electronics.
- Do not use the probe in an explosive environment.
- Save all protective caps so they can be put back on the probe shaft and cable connectors and the fiber optic ports when they are not being used.

- To connect the SabIR Probe to the NIR Module
- 1. Power off the instrument.
- 2. If the strain relief mechanism is not installed, follow the instructions in the <u>Connecting the Strain</u> Relief Mechanism section to install it.

NOTICE

Make sure you use the strain relief with the SabIR Probe to protect the optical fibers when the probe is used for remote sampling.

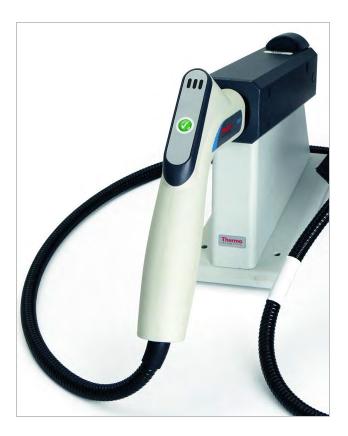
3. Remove the cable cover from the strain relief mechanism by loosening the two thumbscrews.



Α	Cable cover
В	Thumbscrews (2)

4. Remove the sheath from the SabIR Probe tip and insert the probe into the holster.

The probe is in position when you hear it "click" into place.



5. Remove the protective caps from the fiber optic cables and from the red and blue ports on the NIR Module. Save the caps.

NOTICE

Do not overtighten or use tools to tighten the connectors or you may damage the connectors on the probe or the NIR Module.

6. Plug the red connector from the SabIR Probe into the red In port on the NIR Module, and the blue connector into the blue Out port.

Hand-tighten the connectors until you feel a small amount of resistance, i.e., until they are fingertight. Attach the cables to the correct ports to achieve consistent, repeatable results. Do not discard the protective caps; replace them when the ports are not in use.



Α	In port (red)
В	Out port (blue)
С	Electrical port

7. Connect the electrical cable from the SabIR Probe to the electrical port on the NIR Module (see the previous image).

NOTICE

- Do not bend the cables when fitting them into the strain relief mechanism.
- Make sure the cables are resting in the groove and are not being compressed by the cable cover.
- 8. Gently maneuver the cables so the silver ring fits in the groove in the strain relief mechanism (see the previous image).
- 9. Replace the strain relief cover and secure it with the thumbscrews. Make sure the silver ring is completely hidden by the cover.



CAUTION



Avoid eye hazard. Do not stare at the beam that exits the SabIR sampling window when the SabIR Probe is connected to the NIR Module and the spectrometer is powered on and the Fiber Optic beam path is selected.

10. Power on the instrument.

If the instrument has been powered off for more than 20 minutes, allow it to stabilize for approximately one hour before collecting data.

Samples compatible with the SabIR Probe

The SabIR Probe can be used to measure powders and solids that have a rough surface. Some examples are provided below.

NOTICE

- Make sure your sample materials and cleaning solvents will not react with the sapphire window. Refer to the <u>Cleaning the SabIR Probe</u> section more information.
- Do not allow hard materials (i.e., glass, metal, ceramic, etc.) to contact the sapphire sampling window.
- Do not use the probe by itself to measure liquids. When sampling liquids, use the transflectance adapter. Refer to the <u>Sampling accessories for the SabIR Probe</u> section for more information.

Table 5-2: Samples compatible with the SabIR Probe

Sample	Туре	Examples
Powders ¹	Powders, crystals, granules	Pharmaceutical precursors, sugars, grains, polymer beads
Solids ²	Must have a rough or diffuse surface	Coated textiles, tablets, paper, wood, polymers, and plastics (especially plastics with a milky, opaque appearance).

Sample thickness or the amount of sample should be taken into consideration when using the SabIR Probe. If a solid sample is too thin or if there is not enough of a powder sample, you may encounter problems with the spectra.

Tip For repeatable sampling with a homogeneous material, collect the data three times and hold the SabIR in the same position on the sample for each collection.

SAMPLING SOLIDS

NOTICE

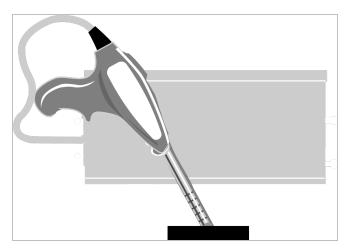
Place abrasive solids in a clear glass container or plastic bag before collecting data to avoid damaging the sapphire window.

When sampling a nonabrasive solid, hold the probe tip flush against the surface of the solid. Use enough sample to completely cover the probe window.

¹May be measured directly in small or large bins, or indirectly through clear packaging such as glass or plastic.

²May be measured directly or indirectly through a clear glass or plastic container.

Figure 5-3: Sampling a nonabrasive solid with a diffuse reflection probe



You can also sample solids through clear packaging materials such as plastic bags, plastic wrap, or glass. Sampling through colored packaging materials may affect spectral data.

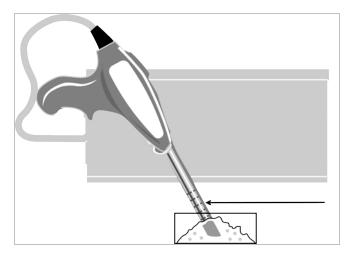
Note Polymeric materials such as plastics have spectral features which may affect analysis results.

SAMPLING POWDERS

You can sample powders directly, or indirectly through clear packaging materials such as glass vials or plastic bags. When sampling a powder through packaging materials, follow the same sampling instructions used for sampling solids.

When sampling powders directly, make sure the sample is thick enough so the probe window is fully covered by the sample material but that enough of the sample material remains underneath the probe window to obtain an accurate reading.

Figure 5-4: Sampling a powder with a diffuse reflection probe



Use the depth indicator rings on the probe shaft to maintain a consistent insertion depth when collecting multiple samples.

Your first experiment with the NIR SabIR Probe

This section explains how to configure your Thermo Scientific optic SabIR Probe and demonstrates how to use it to analyze a sample. You can press the Touch Point to start the process and respond to prompts in the software while it is running, or simply use commands in the software to achieve the same result.

SETTING UP THE SABIR PROBE

1. Start OMNIC Paradigm software. The software automatically loads the default, commonly used settings, but you can create your own settings for your specific analysis needs.

If you need to create new settings, select **New**, then enter a name for the new collection settings. Modify the settings as needed, then select **Save**. The new collection settings will be available for selection in the **Settings** dropdown menu.

NOTICE

Make sure that Sampling accessory shows as **NIR Fiber Optic Probe**. If it does not, a different instrument or accessory has been selected or the instrument is not installed properly.

2. If you need to work with the NIR SabIR Probe, ensure that it is recognized properly. If not, go to **Configure** and select **Sample Location**, then **Fiber Optic Probe**.

MEASURING A SAMPLE WITH THE SABIR PROBE

CAUTION



Avoid eye hazard. Do not stare at the beam that exits the SabIR sampling window when the SabIR Probe is connected to the NIR Module and the spectrometer is powered on and the Fiber Optic beam path is selected.

Before you begin

- Power on the spectrometer and allow the instrument to stabilize for at least an hour (if the instrument has been powered off for more than 20 minutes).
- Make sure the probe window is clean. Refer to the <u>Cleaning the SabIR Probe</u> section for instructions and recommended solvents.

- Have your sample material at hand. For your first analysis, choose one of the following items for your sample:
 - $^\circ~$ a piece of fabric, or
 - a small, clear, glass or plastic container filled with sugar or another granular solid (we use polymer beads for this demonstration)
- To measure a sample with the Thermo Scientific SabIR Probe
- 1. If you have not already done so, go to **Configure > Sample Location > Fiber Optic SabIR Probe** (or press the Fiber Optic Touch Point) to configure the instrument for fiber optic sampling.

Figure 5-5: Fiber Optic Touch Point



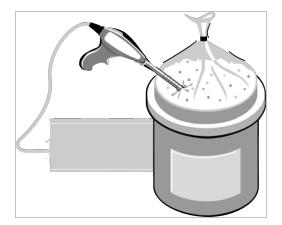
- Ensure that the probe is in the holster and collect a background. In the Dashboard, select Preview and Measure Background. This will create a new spectra tab and open a background preview. Select Start Background Measurement to continue, then wait for the process to complete. For more information, see Collecting backgrounds with the SabIR Probe.
- 3. Remove the probe from the holster (do not press the probe start trigger) and dip the tip in the sample so the window is completely covered. There must be a thick layer of sample below the window to get an accurate reading.

If the sample is in a container, hold the probe tip flush against the container surface. Use enough sample to completely cover the probe window. For more information, refer to the <u>Sampling Solids</u> and <u>Sampling Powders</u> sections.

Figure 5-6: Sample ready for measurement with the SabIR Probe



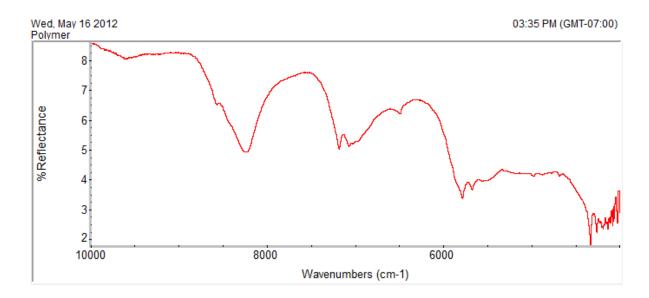
Figure 5-7: Powder sample ready for measurement with a diffuse reflection probe



4. When the probe is in position, press and release the probe trigger (or press the Fiber Optic Touch Point) to collect the sample data.

Do not move the probe until the yellow light on the probe handle has turned off. Wait for the process to complete in the software and the spectrum will be collected.

Figure 5-8: Report showing a diffuse reflection spectrum of polymer taken with the iS50 NIR Module and Thermo Scientific SabIR Probe



5. Clean the probe tip.

Pour a small amount of cleaning solution (one part isopropyl alcohol with nine parts water) onto a soft cloth and gently rub the cloth on the probe shaft.

Using an eye-dropper, place a small amount of the cleaning solution on a clean piece of lens paper and then gently rub the paper on the optical window.

6. Insert the shaft into the holster until you hear it click into place.

Collecting backgrounds with the SabIR Probe

A background is a reference spectrum that accounts for the unique optics of the sampling accessory and the instrument. Each sample spectrum is ratioed against a background so that the final spectrum is free of these features.

You can collect a background with the SabIR Probe using the internal Spectralon reference or an external reference. The most recent background spectrum remains in memory and is compared against sample data until a new background spectrum is collected.

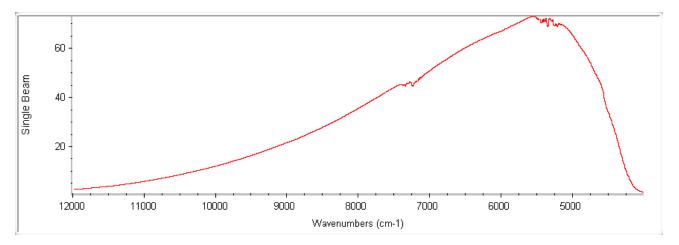
The SabIR Probe holster contains a Spectralon reference that can be used to collect backgrounds. Spectralon is a very diffuse substance with high reflectance, but it is a soft, porous material that must be handled carefully. It can be cleaned if it becomes dirty. Please refer to the <u>Cleaning the spectralon</u> <u>background reference</u> section for instructions on how to handle and clean the reference.

Make sure the probe sheath and window are clean before you insert the probe into the holster.

While the instrument is collecting data, the yellow indicator light on the probe handle will be on. Do not remove the probe from the holster until the yellow light turns off.

A typical background spectrum using the Spectralon reference should resemble the following.

Figure 5-9: Typical Spectralon background spectrum taken with the NIR Module and Thermo Scientific SabIR Probe



Tip Once a material is selected for a background reference, make sure to use the same material each time you run your experiment.

Hold the probe to the reference material if the reference is a solid (or dip the probe in the reference if it is a powder) and then press and release the probe trigger (or press the fiber optic Touch Point) to start collecting the data. While the instrument is collecting data, the yellow indicator light on the probe handle will be on. Do not move the probe until the yellow light turns off.

Sampling accessories for the SabIR Probe

Optional Thermo Scientific accessories for the SabIR Probe are described briefly below. To order an accessory, contact our local sales or service representative or see our contact information at the beginning of this document.

Table 5-3: Optional Thermo Scientific accessories for the SabIR Probe

This accessory	Does this
SabIR Transflectance Adapter	
<text><text></text></text>	Slides on the SabIR Probe shaft and allows you to analyze liquids using the transflectance sampling technique. For detailed information about the SabIR Transflectance Adapter, see the document or help topic with that title.

This accessory...

Does this...

Probe stand



SabIR Probe properly fitted in probe stand



Secures the SabIR Probe for repeatable measurements where the tip angle and pathlength are important such as measurements with an external standard.

Disconnecting the SabIR Probe

It is not necessary to disconnect the SabIR Probe from the NIR Module after each use. However, if you are not planning to use the probe for a long period of time, or if you need to remove it to attach a different accessory, follow these steps to disconnect the probe.

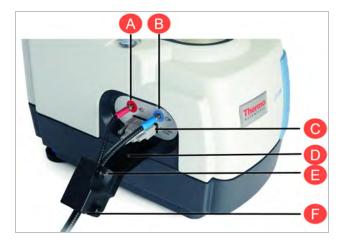
NOTICE

Power off the instrument before you disconnect the SabIR Probe to avoid damaging the instrument's electronics.

- To disconnect the Thermo Scientific SabIR Probe
- 1. Power off the instrument.

To avoid contaminating the internal reference, make sure the probe shaft and tip are clean before you insert the probe into the holster.

- 2. Insert the SabIR Probe into the holster to avoid causing strain on the fiber optic cables from the weight of the probe.
- 3. Remove the strain relief cable cover by loosening the two captive thumbscrews.



Α	Fiber optic port (blue)
В	Fiber optic port (red)
С	SabIR electrical connector
D	Strain relief base
Е	Thumbscrews (2)
F	Cable cover

4. Gently remove the cables from the groove in the strain relief base. It is not necessary to remove the strain relief base.

- Do not bend the fiber optic cables or touch the ends, and avoid twisting them when detaching the connectors from the ports or you may contaminate or damage the optical fibers.
- Do not tightly coil the cables. The radius of the coils for the SabIR Probe should be at least 30 cm.
- 5. Disconnect the SabIR fiber optic cables from the ports (see the previous image).
- 6. Disconnect the SabIR electrical cable (see the previous image).
- 7. Replace the sheath on the probe shaft.
- 8. Replace the protective caps on the ends of the cables and the fiber optic ports.
- 9. Replace the strain relief cable cover.

Storing the SabIR Probe

Store the SabIR Probe in a dust-free environment such as a cabinet or its original shipping box.

NOTICE

- Do not bend the fiber optic cables or tightly coil them to avoid damaging the optical fibers. The radius of the coils for the SabIR Probe should be at least 30 cm.
- Place the sheath on the probe shaft and the protective caps on the fiber optic cable connectors and the ports on the NIR Module when they are not being used.

Using fiber optic accessories from other manufacturers

This section covers usage of your fiber optic accessories from other manufacturers besides Thermo Fisher.

Connecting a fiber optic sampling accessory

- To connect a fiber optic sampling accessory to the NIR Module
- 1. Remove any protective caps from the sampling accessory connectors.

Save the caps so they can be put back onto the accessory before it is stored.

CAUTION



Avoid eye hazard. Do not stare at the beam that exits the fiber optic ports, or the fiber optic cables when they are connected to the ports, when the spectrometer is powered on and the Fiber Optic beam path is selected.

- 2. Remove the protective caps from the In and Out fiber optic ports on the NIR Module. Save the caps.
- 3. Connect the SMA connector from your sampling accessory to each fiber optic port.



The near-infrared beam travels out the blue "out" port and into the fiber optic accessory. The light leaves the accessory and travels back into the module through the red "in" port. Use the color coding to assist you in consistently connecting the cables on your sampling accessory to the same ports on the module.

Tip It is important to consistently attach the fiber optic cables to the same ports when connecting sampling accessories. If there are differences between the fiber optic cables, and they are not always attached to the same ports, your data may be affected.

Do not over tighten the connectors or use tools to tighten them or you may damage the connectors on the accessory or the NIR Module.

4. Hand-tighten the connectors until you feel a small amount of resistance, that is, until they are finger-tight.

Collecting data

Select a background material and sample that is compatible with your fiber optic accessory. Refer to the documentation that came with the accessory for information about compatible samples.

To collect the data, follow the steps in the <u>Your first experiment with the NIR SabIR Probe</u> section using the fiber optic Touch Point or the software to continue to the next step. Do not move the sampling accessory until the instrument has completed collecting data.

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Chapter 6

Maintenance

This section describes maintenance routines that you can perform on the NIR Module to keep it running efficiently.

Cleaning the NIR Module housing

NOTICE

- Do not pour liquids directly onto the sampling area of the Integrating Sphere, especially the area around the electrical port for the Sample Spinner.
- Do not use harsh detergents, chemicals or abrasives to clean the surface of the Integrating Sphere; these can damage the finish

Use a damp (not wet), soft cloth and a mild soap to clean the Integrating Sphere cover and other metal parts, including the sample accessory holder. Dry them with a clean cloth.

Figure 6-1: Sample accessory holder



A Sample accessory holder

B Integrating Sphere cover

Cleaning the Integrating Sphere sampling window

NOTICE

- The cleaning fluid should be at or close to room temperature (or the temperature of the previous sample). Applying extremely hot or cold liquids to the sampling window may crack it.
- Some chemicals including acetone, chlorine, fluorine, and amyl alcohol can attack the epoxy seal around the sampling window. Do not allow these chemicals to come into contact with the window.

CAUTION



Avoid eye hazard. Do not stare at the beam that exits the Integrating Sphere sampling window when the spectrometer is powered on and the Integrating Sphere beam path is selected.

Cleaning the Integrating Sphere sampling window

- 1. Remove any sample, sample cup or accessory from the Integrating Sphere.
- 2. If the sample came in contact with the sampling window, gently clean the window with a clean, white paper napkin or cotton ball moistened with methanol, ethanol, isopropyl alcohol, or water.
- 3. Dry the window with a clean napkin or cotton ball. Avoid materials that have been treated with aloe or other additives.

Тір

Chemical wipes are usually not recommended for cleaning the Integrating Sphere sampling window as embedded clays can make these slightly abrasive.

Use of soaps or other surfactants for cleaning the sampling window is discouraged as these can leave a residue bound to the window surface.

Cleaning Integrating Sphere Sampling Accessories

To clean this accessory	Do this
Sample cups	Use a mild soap solution and then rinse with distilled water. Dry them with a jet of clean air, a non-abrasive cloth, or a paper napkin. Notice Do not use harsh solvents to clean the sample cups or you may damage them.
Custom tablet holder	Use a mild soap solution and then rinse with distilled water. Dry them with a jet of clean air or allow it to air dry.
•	Notice Do not use harsh solvents to clean the custom tablet holders or you may damage them.
Sample rings	Use a dry, soft cloth or dampen it with isopropyl alcohol, distilled water, or a mild soap solution. Dry them with a clean cloth or allow it to air dry.
UUU	Notice Do not use harsh solvents to clean the sample rings or you may damage them.

To clean this accessory	Do this
Universal tablet holder	Use a jet of clean air to remove any particles. If this is not sufficient, then gently rub the tablet holder with a cloth dampened with isopropyl alcohol and let it air dry.
	Notice Do not use water to clean the universal tablet holder or it may rust. The iris is delicate; handle it gently.

Cleaning the SabIR Probe

If the tip or shaft of the SabIR Probe becomes dirty or contaminated with sample or other material, prepare a cleaning solution of one part isopropyl alcohol and nine parts water in a clean container and follow these instructions.

NOTICE

The cleaning fluid should be at or close to room temperature (or the temperature of the previous sample). Applying extremely hot or cold liquids may crack the sapphire sampling window.

Some chemicals including acetone, chlorine, fluorine and amyl alcohol can attack the epoxy seal around the sapphire window. Do not allow these chemicals to come into contact with the window.

CAUTION



Avoid eye hazard. Do not stare at the beam that exits the SabIR sampling window when the SabIR Probe is connected to the NIR Module and the spectrometer is powered on and the Fiber Optic beam path is selected.

To clean the Thermo Scientific SabIR Probe

- 1. Remove the SabIR Probe from the holster.
- 2. Pour a small amount of the cleaning solution onto a soft cloth and use it to gently clean the probe shaft. Allow the shaft to air dry.
- 3. Using an eye-dropper, place a small amount of the cleaning solution on a fresh piece of lens paper.
- 4. Gently rub the lens paper on the optical window for 10 to 20 seconds.

Allow the window to air dry for at least two minutes.

Tip

Chemical wipes are usually not recommended for cleaning the SabIR Probe sampling window as embedded clays can make these slightly abrasive.

Use of soaps or other surfactants for cleaning the window is discouraged as these can leave a residue bound to the window surface.

Cleaning the spectralon background reference

Spectralon is an optical standard and should be handled with care. Keep the Spectralon reference in the SabIR Probe holster at all times unless you need to clean or replace it.

NOTICE

Avoid touching the surface of the Spectralon reference. Dirt and oils from your fingers can leave residue which will affect background spectra quality.

To clean the Spectralon reference

- 1. Put on a pair of clean gloves or finger cots.
- 2. Remove the SabIR Probe from the holster and place it securely on a table.
- 3. Firmly grasp both sides of the reference holder with your thumb and forefinger and pull it out of the holster. Remove the SabIR Probe before you attempt to remove the reference holder.

Figure 6-2: Removing the Spectralon reference



- 4. Clean the Spectralon surface with an air brush, using clean dry air or nitrogen.
- 5. If this is insufficient, gently sand the surface with a 220-240 grit waterproof emery cloth.
- 6. If the surface is still not sufficiently clean, sand it under running water with a 220-240 grit waterproof emery cloth until water beads and immediately runs off the surface. Dry the surface with a jet of clean dry air or nitrogen, or allow the material to air dry.

If the background quality does not improve after thoroughly cleaning the Spectralon reference, the reference may need to be replaced. Contact our sales or service representative in your area for more information.