



Nicolet iS50 FT-IR Spectrometers

iS50 GC-IR Module User Guide

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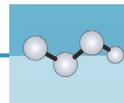
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WARNING Avoid an explosion or fire hazard. This instrument or accessory is not designed for use in an explosive atmosphere.



iS50 GC-IR Module

This section describes the Thermo Scientific iS50 GC-IR module which is an optional sampling module for your Nicolet™ iS™50 spectrometer.

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- [About the GC-IR Module](#)
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NOTICE Be sure that all persons operating this system read the site and safety manual first.

Conventions Used in This Document or Help System

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



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



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

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

Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

About the GC-IR Module

The iS50 GC-IR module allows you to combine the separating power of a gas chromatograph (GC) with the selectivity of an infrared spectrometer. The GC-IR module is compatible with the Nicolet™ iS™50 FT-IR spectrometer and the following gas chromatographs and autosamplers:

- Thermo Scientific TRACE™ 1300 series gas chromatographs with autosamplers
- Agilent™ 7890 gas chromatograph

Figure 1. iS50 GC-IR system



The GC-IR module mounts on the right side of the Nicolet iS50 spectrometer

Before operating the GC-IR module, you should be familiar with the fundamentals of capillary gas chromatography and the operating characteristics of your FT-IR spectrometer and GC.

A Thermo Scientific TRACE 1310 GC is shown in the illustrations in this document. Installation procedures for injectors, detectors and columns are explained in the operating guides provided with the GC and will not be reproduced here. For instructions to connect your column to the GC-IR module transfer line, see [“Setting Up the GC-IR System.”](#)

The iS50 GC-IR module provides these enhanced features:

- Integrated design
- Push button configuration
- High efficiency optics
- Dedicated, highly sensitive detector
- Software-controlled transfer line and flow cell temperature
- Support for GC autosamplers

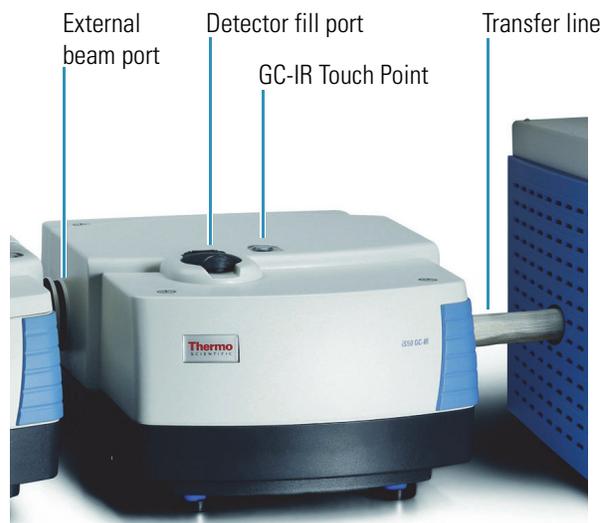
For information about additional sampling modules and accessories for your Nicolet iS50 spectrometer, contact our sales representative in your area.

Important Features

This section describes the major features of the iS50 GC-IR module.

From the Front

Figure 2. iS50 GC-IR module front view



These features are visible from the front and sides of the GC-IR module:

- **External beam port.** Light from the spectrometer passes through this port to the flow cell in the GC-IR module. Your iS50 GC-IR module may have an optional KBr window installed in the external beam port.

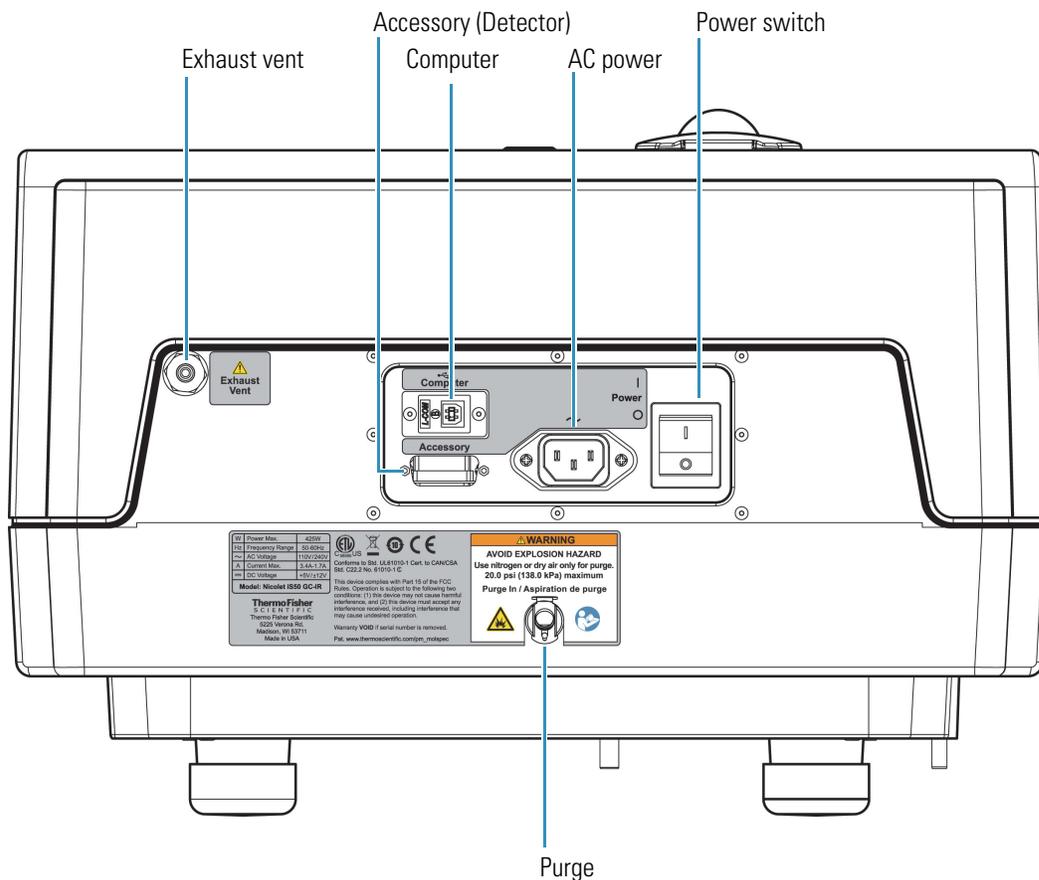
- **Transfer line.** Vaporized sample from the GC passes through this precisely heated line to the heated flow cell inside the GC-IR module. The flow cell is where the sample interacts with the infrared beam to produce the signal for the GC-IR detector.
- **Detector fill port.** Used to add liquid nitrogen to the dewar to cool the GC-IR detector before each use. For more information, see “[Cooling the GC-IR Module Detector.](#)”
- **Touch Point.** Automatically configures the spectrometer for GC-IR sampling. The Touch Point LED shows the status of the GC-IR module. The LED has three states described below.

Table 1. GC-IR status indicator states

LED status	Meaning
On	Ready for use
Off	System is not configured for GC-IR sampling
Blinking	Optics are reconfiguring

Back Panel Features

Figure 3. iS50 GC-IR module back panel



Feature	Description
Purge	Connects to the purge gas supply. For more information, see “Connecting a Purge Source.”
Accessory	Connector for the detector cable. See “Cabling the GC-IR Module” for details.
Computer	Connector for the USB communications cable
AC power	Connector for the power cord.
Power switch	Powers the GC-IR module on and off. A built-in circuit breaker automatically shuts off power to the instrument if an over-current condition occurs. For information on recovering from an over current condition, see “Resetting the Circuit Breaker.”
Exhaust vent	Effluent leaving the flow cell travels to a room temperature carbon filter and then out this vent. The vent can be routed to a fume hood if needed; see “Venting the GC-IR Module Exhaust” for details.



WARNING Your GC accessory is preset to work with the correct AC line voltage in your country. To avoid shock and fire hazard, do not attempt to change the setting. Contact our customer support if your system does not meet your local power standards.

Related Topics

- [Connecting a Purge Source](#)
- [Cabling the GC-IR Module](#)
- [Resetting the Circuit Breaker](#)
- [Venting the GC-IR Module Exhaust](#)
- [Maintaining the Carbon Filter](#)

Specifications

Table 2. Specifications for the iS50 GC-IR module

Feature	Specification
Compatible gas chromatographs	<ul style="list-style-type: none"> • Thermo Scientific TRACE 1300 series • Agilent 7890
Maximum temperature of transfer/exhaust lines and flow cell	300 °C
Temperature status output	Continuous
Detector	Dedicated nitrogen-cooled MCT-A

Operating Precautions

- Make sure the spectrometer is fully purged before you turn on the GC-IR transfer line and flow cell heaters to prevent damage to the KBr windows in the flow cell. For more information, choose Help > Spectrometer Help Topics and search for the “Checking the Purge” topic.
- To prevent the flow cell windows from becoming contaminated, do not turn on the flow cell or transfer line heaters unless a GC column is installed and the helium make-up gas is flowing through the flow cell.
- Pieces of graphite, column or dirt in the transfer line or connecting Tee union may enter the flow cell and block the infrared beam. Keep the Tee union and transfer line clean and free of debris.
- Do not attempt to slide a wide-bore (0.53 mm I.D.) column through the transfer line. These columns may become lodged in the Tee union and block the transfer line.
- Avoid spilling excessive amounts of liquid nitrogen on the outer housing of the GC-IR detector dewar. Repeatedly cooling the MCT detector window and the seals in the vacuum valve can cause the dewar to lose its vacuum.

Compatible Software

Table 3. Software compatible with the GC-IR module

Software	Use for...
OMNIC Series	Collecting, processing and searching time series data including GC-IR, TGA-IR, rapid scan and kinetics data. Includes Mercury GC , a dedicated tool for GC-IR analysis (automated profile peak finder and multi-component search). For more information, find Mercury GC in the Series Help Topics.
OMNIC™	Configuring your GC-IR workflow, including quantitative analysis and spectral search, and collecting and analyzing GC-IR spectra. For more information, see the “ Your First Experiment ” section.
OMNIC Spectra™	Analyzing unknowns. This software includes our unique multi-component search feature for identifying the spectra of mixtures, a 9,000 compound spectral database, and features for using your computer’s hard drive as a library. For more information, refer to the Help system in OMNIC Spectra software.
TQ Analyst	Creating quantitative and classification methods that can be used with OMNIC. This software provides an extensive suite of chemometrics features you can use to identify raw materials, perform quantitative analysis, and take spectral measurements. For more information, refer to the help system in your TQ Analyst software.

GC-IR Sampling

In a GC-IR experiment the sample is injected into the column of a gas chromatograph which separates the sample components. The **GC column** is routed through a heated glass-lined, stainless-steel **transfer line** to the GC-IR module. Effluent leaving the GC column immediately enters the **flow cell**, where it is scanned by the infrared beam. In effect, the infrared spectrometer is a chemically specific detector for the gas chromatograph, sensitive only to compounds that absorb infrared radiation.

The flow cell and its fittings are coated with **gold**, making them inert to materials entering from the GC column. Dedicated **controllers** maintain the temperature of the flow cell and transfer line.

When a standard capillary column is used, a **make-up gas** is added to the transfer line. This gas flows around the GC column in the transfer line to prevent backflow. The make-up gas also carries the effluent through the larger diameter flow cell with minimal loss of chromatographic resolution. The stream exits the flow cell and travels through an exhaust line to an unheated **carbon filter**, which vents out the back of the GC-IR module.

The GC-IR module performs best with **standard capillary chromatography columns**. However, you can also use it with **wide-bore capillary columns**. In this case, the column is connected directly to the transfer line. No make-up gas is required because of the higher flow rates used in wide-bore column gas chromatography. For instructions on connecting columns to the GC-IR module, see [“Setting Up the GC-IR System.”](#)

Related Topics

- [Setting Up the GC-IR System](#)
- [Setting Flow Cell and Transfer Line Temperature](#)
- [Maintaining the Carbon Filter](#)

Setting Up the GC-IR System

After the GC-IR module has been installed by our representative, you are ready to connect a purge source and the transfer line, make-up gas line, column and GC detector, if desired. This section explains how to connect these items and also describes how to prepare the system for running GC-IR experiments.

- [Cabling the GC-IR Module](#)
- [Connecting a Purge Source](#)
- [Connecting the Transfer and Make-up Gas Lines](#)
- [Installing a Standard Capillary GC Column](#)
- [Installing a Wide-Bore Capillary GC Column](#)

- [Connecting the GC Detector](#)
- [Venting the GC-IR Module Exhaust](#)
- [Measuring and Adjusting the Make-up Gas Flow Rate](#)
- [Cooling the GC-IR Module Detector](#)
- [Setting Flow Cell and Transfer Line Temperature](#)
- [Recommended Column Flow Rates](#)



CAUTION Avoid burn hazard. Do not touch the bare transfer line, flow cell or vent line when the insulation has been removed and the heaters are on. Temperatures can exceed 300 °C (572 °F).

Cabling the GC-IR Module

This section describes the electrical, purge and exhaust connections for the iS50 GC-IR module.



WARNING Avoid inhaling GC-IR module exhaust. Keep the work area properly ventilated or connect the exhaust vent to a fume hood.



WARNING Avoid explosion hazard. Never use a flammable, combustible, or toxic gas to purge this instrument. The purge gas must be free of oil and other reactive materials. Heat from the source or from laser absorption may ignite flammable gases or reactive materials in purge gas. Use only dried air or nitrogen to purge your instrument.

Table 4. iS50 GC-IR module electrical, purge and exhaust connections

Connection	Description
Purge	Connects to the purge gas supply. For more information, see “Connecting a Purge Source.”
AC power	Plug the AC power cord into this receptacle and then into an AC wall outlet.
Detector	Plug one end of the detector cable into the Accessory port on the back of the GC-IR module. The other end goes to the Accessory port on the back of the spectrometer. (The spectrometer has a right and left Accessory port. Use the one that is closest to the GC-IR module.)

Connection	Description
Exhaust	<p>Connect a length of 1/8 in diameter plastic tubing to the Exhaust port on the back of the GC-IR module. Route the other end to a fume hood if necessary. For more information, see “Venting the GC-IR Module Exhaust.”</p> <p>Note: You can connect a flow meter to the exhaust vent to measure the carrier gas flow rate through the flow cell.</p>
Remote trigger (optional)	<p>Connect the 37-pin D-type connector of the remote start cable to the Auxiliary Signals port on the back of the spectrometer. Connect the BNC connector of the cable to the data collection trigger signal for your GC. For more information, see the documentation that came with your GC.</p> <p>To set up the remote start cable in OMNIC software:</p> <ol style="list-style-type: none"> 1. Start OMNIC software. 2. Choose Collect > Experiment Setup, select the Series tab and select External Trigger.
Communications ^a	<p>This USB cable runs the temperature controllers in the GC-IR module. Plug one end of the cable into a USB connector on the computer. Connect the other end to the Computer USB port on the back of the GC-IR module.</p>

^aThe Agilent 7890 GC requires an Ethernet cable instead of a USB cable.

Related Topics

- [Back Panel Features](#)
- [Venting the GC-IR Module Exhaust](#)
- [Connecting a Purge Source](#)

Connecting a Purge Source

The GC-IR module must be purged with dry air or nitrogen. The spectrometer window and beamsplitter configuration needed for the GC-IR module also require that the spectrometer is purged (using only desiccant is insufficient). The purge gas requirements for the GC-IR module are the same as for the spectrometer.

We recommend using dry air supplied by a purge gas generator or pure air generator (available from us), or nitrogen. Please refer to your *Site and Safety Information* guide for specific purge gas requirements.



WARNING Avoid explosion hazard. Never use a flammable, combustible, or toxic gas to purge this instrument. The purge gas must be free of oil and other reactive materials. Heat from the source or from laser absorption may ignite flammable gases or reactive materials in purge gas. Use only dried air or nitrogen to purge your instrument.

NOTICE Do not use argon as a purge gas. Argon is an insulator and prevents the system from cooling properly.

The purge input connector is on the back of the GC-IR module (see the illustration in “[Back Panel Features](#)”). For information on how to install the purge equipment, set the controls, and inspect and clean the filter, find “Installing a Purge Kit” in Spectrometer Help Topics.

Adjust the GC-IR module purge gas supply pressure and flow rate as described in the table below.

Table 5. Recommended purge pressure and flow rate for the iS50 GC-IR module

Purge Pressure	Purge Flow
20 psig	10 SCFH

We recommend that you leave the purge on at all times. This keeps the GC-IR module free of undesirable gases, protects the optics and improves the system’s thermal stability.

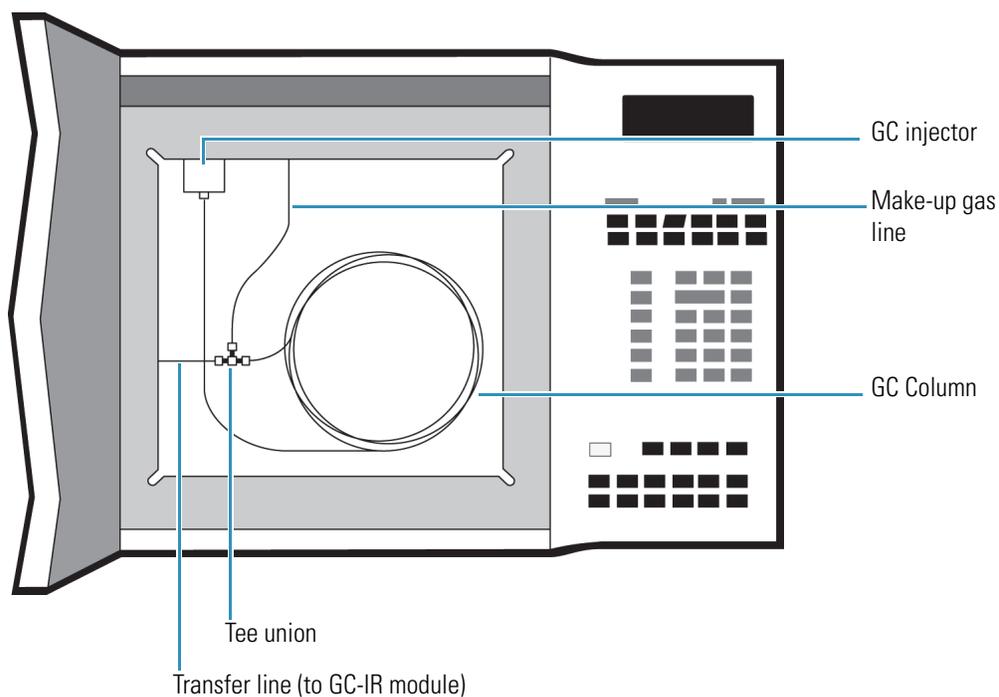
NOTICE To protect flow cell windows from contamination, make sure a GC column is installed and helium make-up gas is flowing through the flow cell before you turn on the flow cell or transfer line heaters.

Related Topics

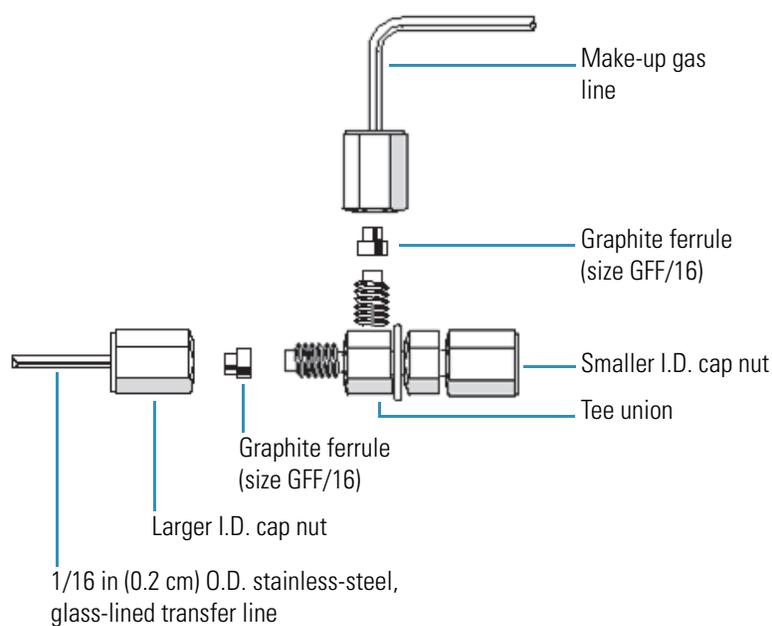
- [Back Panel Features](#)

Connecting the Transfer and Make-up Gas Lines

The transfer line and the make-up gas line are connected by a stainless-steel Tee union. The third opening on the Tee union connects to either the GC column or a splitter if the GC column is routed to both the IR detector and the GC detector.

Figure 4. iS50 GC-IR module column connections (without the splitter)

This union is used for all capillary GC applications. We connect the Tee union during installation. You will need these instructions only if you are converting from wide-bore capillary columns to standard capillary columns.

Figure 5. Transfer line and make-up gas line connections

❖ To connect the transfer line and the make-up gas line

1. Locate a Tee union in your GC-IR accessory kit.

You should also find some graphite ferrules. Look at the cap nut on each end of the straight-through portion of the Tee union. The nut with the larger internal diameter (I.D.) secures the transfer line. The smaller I.D. nut attaches the column (or a splitter if one is used). The make-up gas line attaches to the perpendicular stem of the Tee union.

2. Connect the transfer line to the Tee union.
 - a. Remove the larger I.D. cap nut from the straight-through portion of the Tee union.
 - b. Slide the cap nut over the transfer line.
 - c. Carefully slide a size GFF/16 graphite ferrule onto the transfer line, taking care not to plug the transfer line with graphite from the ferrule after you slide it over the end.

Tip If you have a scribe tool with a narrow tapered end, you can slide the ferrule onto the scribe and then insert the point of the scribe into the transfer line and slide the ferrule off the scribe and onto the transfer line. This technique prevents graphite from the ferrule from getting inside the transfer line.

- d. Feed the transfer line through the Tee union until the line stops, and then back out the line about 1 mm.

The transfer line should go almost all the way through the union before it stops.
 - e. Tighten the cap nut finger-tight.
3. Connect the make-up gas line to the Tee union.
 - a. Remove the cap nut from the perpendicular arm of the Tee union.
 - b. Slide the cap nut over the make-up gas line.
 - c. Carefully slide a size GFF/16 graphite ferrule onto the make-up gas line, taking care not to plug the line with graphite after you slide the ferrule over the end (use the same technique as in step 2 above).
 - d. Feed the make-up gas line into the Tee union until the line stops and then tighten the nut finger-tight.

4. Use a 5/16-in and 1/4-in wrench to tighten the cap nuts for the transfer and make-up gas lines.

Turn the nuts about one full turn past finger-tight.

Related Topics

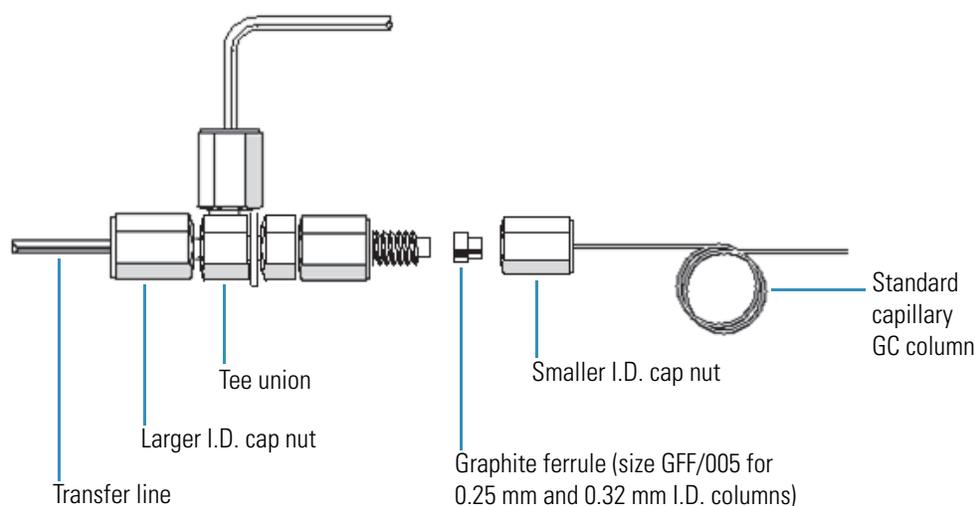
- [Installing a Standard Capillary GC Column](#)
- [Installing a Wide-Bore Capillary GC Column](#)
- [Measuring and Adjusting the Make-up Gas Flow Rate](#)

Installing a Standard Capillary GC Column

This section explains how to install a standard 0.25 mm or 0.32 mm I.D. fused silica capillary column into your iS50 GC-IR system. These types of columns connect to the GC injection port at one end. The other end must be fed through the Tee union and transfer line and into the flow cell of the GC-IR module.

Note If a splitter is used to connect the GC detector, the GC column attaches to the GC injection port as described above. The other end of the column attaches to the open end of the splitter instead of the Tee union. For details, see “[Connecting the GC Detector](#).”

Figure 6. Connections for a standard capillary GC column



❖ To install a standard (0.25 mm or 0.32 mm I.D.) capillary GC column

1. Attach the column inlet to the injection port on the GC.

Refer to the operating guide that came with your GC for specific instructions.

Note If a splitter is used to connect the GC detector, stop here and follow the instructions in “[Connecting the GC Detector](#)” to connect the other end of the GC column to the splitter.

2. Start the OMNIC software.

You will watch a live interferogram while attaching the other end of the column. This will help you determine how far to insert the column into the flow cell.

3. Choose **Collect > Experiment Setup** and select the **Bench** tab.
4. On the Bench tab, set **Sample Compartment** to GC-IR.

5. Locate the Tee union that joins the transfer and make-up gas lines. Remove the cap nut and ferrule from the free arm of the straight through portion of the Tee union.
6. Thread the free end of your GC column through the cap nut and a ferrule that fits onto the column.
7. After you have fed the column through the graphite ferrule, remove 2 to 3 mm from the end of the column to create a clean end with no burrs or jagged edges.

To do this, score the column with a tool or scribe intended for this purpose and snap off the end piece.

NOTICE Pieces of graphite, column or dirt in the transfer line or Tee union may enter the flow cell and block the infrared beam. Keep the Tee union and transfer lines clean and free of debris.

8. While watching the live display at the left side of the Bench tab, carefully feed the column through the Tee union and transfer line and into the flow cell.

The live display shows the detector signal. The signal is represented both as an interferogram (if the Single Beam check box is cleared) and numerically as minimum and maximum voltage values (if the Min/Max check box is selected). Refer to the OMNIC Help system if you need more information on Experiment Setup and the live display.

9. When the column enters the flow cell, it will block the infrared beam, causing the height of the interferogram in the live display to decrease markedly.

You may have trouble guiding the column through the opening in the end cap of the flow cell. Do not force it, as this may damage or break the end of the column. Instead, rotate the column gently between your fingers while slowly pushing it further into the transfer line.

Note If you still cannot get the column to block the beam, just feed in the column as far as it will go. Some capillary columns are too large to fit through the end cap.

10. Slowly withdraw just enough column so that the interferogram regains its original height.
11. Use a 5/16-in wrench and a 1/4-in wrench to tighten the column nut on the Tee union until you feel the nut start to offer resistance. Then tighten the nut another one-quarter turn.
12. Choose OK to close Experiment Setup.

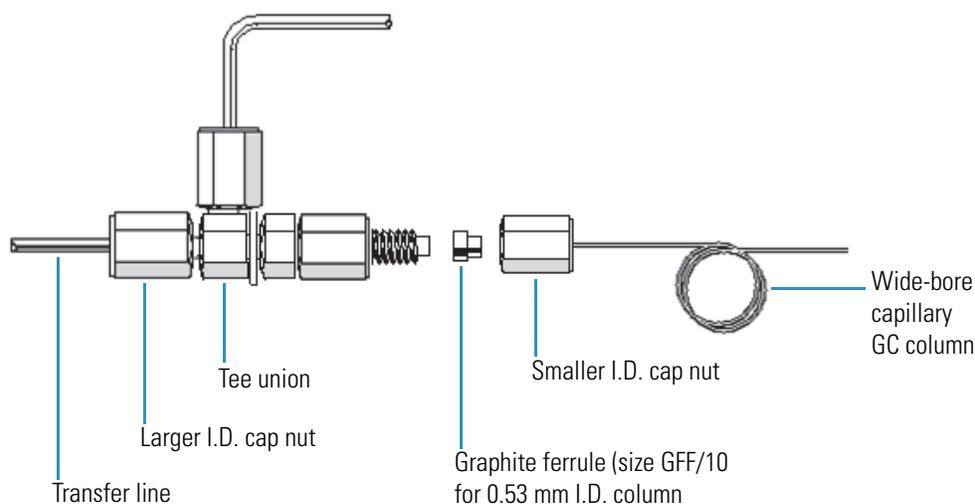
Related Topics

- [Installing a Wide-Bore Capillary GC Column](#)
- [Connecting the Transfer and Make-up Gas Lines](#)
- [Connecting the GC Detector](#)

Installing a Wide-Bore Capillary GC Column

These columns have an outside diameter that is nearly the same size as the inside diameter of the transfer line. To prevent scratching or breakage inside the transfer line, do not try to feed these columns into the flow cell as described in the previous section. Instead, thread the end of the column through the Tee union and a few millimeters into the transfer line as described below. Since the transfer lines are lined with glass, connecting the column this way does not adversely affect GC-IR performance.

Figure 7. Connections for a wide-bore capillary GC column



❖ To install a wide-bore (0.53 mm I.D.) capillary GC column

1. Attach the column inlet to the injection port of the GC.

Refer to the operating guide that came with your GC for specific instructions.

Note If a splitter is used to connect the GC detector, stop here and follow the instructions in “[Connecting the GC Detector](#)” to connect the other end of the GC column to the splitter.

2. Locate the Tee union that joins the transfer and make-up gas lines. Remove the cap nut and ferrule from the free arm of the straight-through portion of the Tee union.
3. Thread the free end of your GC column through the cap nut and a ferrule that fits onto the column.
4. Remove 2 to 3 mm from the end of the column to create a clean end with no burrs or jagged edges.

To do this, score the column with a tool or scribe intended for this purpose and snap off the end piece.

NOTICE Pieces of graphite, column or dirt in the transfer line or Tee union may enter the flow cell and block the infrared beam. Keep the Tee union and transfer lines clean and free of debris.

5. Feed the column through the Tee union and a few millimeters into the transfer line.

NOTICE Do not attempt to feed the wide-bore column through to the flow cell. The column may become lodged in the Tee and cause blockage of the transfer line.

6. Use a 5/16-in and a 1/4-in wrench to tighten the column nut on the Tee union one-half turn past finger-tight.

Related Topics

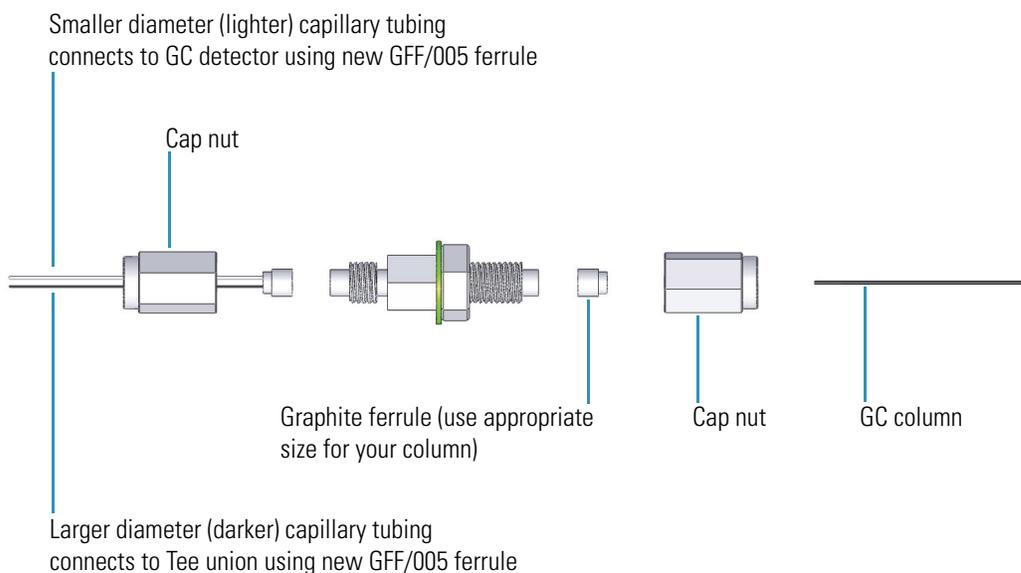
- [Installing a Standard Capillary GC Column](#)
- [Connecting the Transfer and Make-up Gas Lines](#)
- [Connecting the GC Detector](#)

Connecting the GC Detector

You can connect the GC column to any detector installed on the GC by redirecting a portion of the column effluent through a splitter. Although this is not required for GC-IR operation, it allows you to monitor the GC chromatography during GC-IR experiments to make sure you are getting the expected separation. You may omit this step if desired and connect the GC column directly to the Tee union as described in the previous sections.

To connect the column to the GC detector, you will need a 10:1 splitter kit (see our local sales or service representative to order the correct splitter). The kit includes a splitter body, extra ferrules and two lengths of capillary tubing that are joined at one end by a special ferrule. The internal diameter of the slightly darker tubing is 10 times larger than that of the lighter one, which produces the 10:1 split ratio. In the package, the smaller diameter tubing is coiled more tightly than the larger diameter tubing. Before you remove the coils from the package, make sure you mark one of the coils so you can tell them apart during assembly.

The GC column attaches to the short end of the splitter (see the diagram below). The joined end of the two lengths of capillary tubing connects to the long end of the splitter. The larger diameter tubing from the splitter connects through the Tee union and transfer line to the flow cell. The smaller diameter tubing connects to the GC detector.

Figure 8. GC detector connection using a 10:1 splitter

❖ To connect the GC detector

1. Connect the joined end of the splitter tubing to the splitter body.
 - a. Remove the cap nut from the long end of the splitter body.
 - b. Thread the free ends of the two lengths of capillary tubing from the splitter through the cap nut (the lines are joined by a ferrule so no additional ferrule is needed).
 - c. Screw the cap nut back onto the splitter body and tighten it with two 1/4-in wrenches.
2. Connect the GC column to the splitter.
 - a. Remove the cap nut from the short end of the splitter body.
 - b. Thread the free end of your GC column through the cap nut and a ferrule that fits onto the column.
 - c. Remove 2 to 3 mm from the end of the column by scoring it with a column cutter and snapping off the end piece. This creates a clean end with no burrs or jagged edges.

NOTICE Pieces of graphite, column or dirt in the splitter, transfer line or Tee union may enter the flow cell and block the infrared beam. Keep these connectors and lines clean and free of debris.

3. Connect the larger diameter (darker) capillary tubing from the splitter body to the Tee union that joins the transfer line and make-up gas line.
 - a. Remove the cap nut from the free arm of the Tee union.

- b. If there is a ferrule inside the cap nut (from a previous connection), make sure you remove the ferrule.
 - c. Thread the darker capillary tubing from the splitter through the cap nut and then add a new graphite ferrule that fits onto the tubing (several GFF/005 ferrules are provided in the splitter kit).
 - d. Remove 2 to 3 mm from the end of the tubing as described above to create a clean edge.
 - e. Follow steps 2 through 12 in the “[Installing a Standard Capillary GC Column](#)” section to feed the tubing through the transfer line and into the flow cell.
4. Connect the smaller diameter (lighter) capillary tubing from the splitter body to the GC detector using a new graphite ferrule from your splitter kit and removing 2 to 3 mm from the end of the tubing to create a clean edge.

Follow the instructions in the operation manual for your gas chromatograph.

Related Topics

- [Connecting the Transfer and Make-up Gas Lines](#)
- [Installing a Standard Capillary GC Column](#)
- [Installing a Wide-Bore Capillary GC Column](#)

Venting the GC-IR Module Exhaust

Effluent leaving the flow cell travels through a heated transfer line to a room temperature carbon filter and then to the exhaust vent. The carbon filter absorbs the GC components in the effluent before they reach the vent.



WARNING Avoid inhaling GC-IR module exhaust. Keep the work area properly ventilated or connect the exhaust vent to a fume hood.

❖ To connect an exhaust line to the GC-IR module

1. Connect a length of 1/8-in diameter plastic tubing to the exhaust vent on the back of the GC-IR module.

The other end can be routed to a room vent or fume hood if necessary.

Related Topics

- [Back Panel Features](#)
- [Maintaining the Carbon Filter](#)

Measuring and Adjusting the Make-up Gas Flow Rate

Skip this section if you are using a wide-bore (0.53 mm) capillary GC column, since those columns do not require make-up gas. Before continuing, make sure that the valve from the make-up gas source is closed by turning the knob fully clockwise.

If you are using a standard (0.25 mm or 0.32 mm I.D.) capillary column, you need to supply enough make-up gas to purge the transfer line around the capillary column. This requires a flow rate of only 0.3 to 0.5 mL/min. Higher make-up gas flow rates improve chromatographic flow through the flow cell but also dilute the sample, causing the IR sensitivity to decrease. Flow rates greater than 1.5 mL/min do not further improve chromatographic peak shapes.

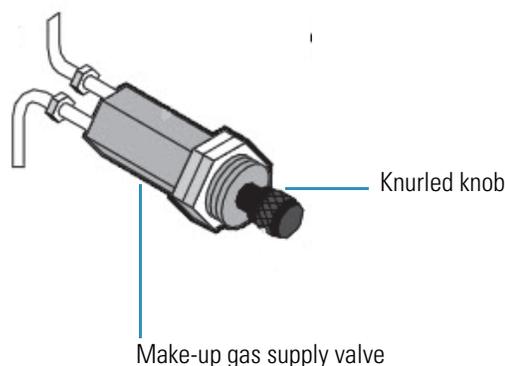
❖ To measure and adjust the make-up gas flow rate

1. Locate the make-up gas supply valve.

For the TRACE 1310 GC, the valve is installed on the top of the GC under the hinged cover for the rear detector module.

2. Open the flow cell make-up gas supply valve by turning the knurled knob counterclockwise as far as it will go.

Figure 9. Make-up gas supply valve



3. Turn off the GC detector gas flow.
4. Attach the flow meter tube to the exhaust vent on the back of the GC-IR module.
5. Measure and record the total flow rate.

This is the column flow rate plus the make-up gas flow rate.

6. Turn off the GC column flow.

You can do this either by setting the column flow rate to zero (if your GC has an electronic flow control) or by reducing the column head pressure until the column flow reaches zero.

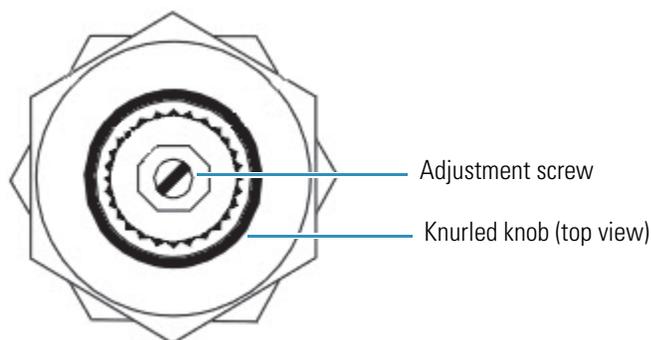
7. Measure the flow rate again.

This is the make-up gas flow rate.

- Adjust the make-up gas valve so that the make-up gas flow rate is between 0.3 and 0.5 mL/min.

To adjust the valve, turn the slotted adjustment screw inside the knurled knob counterclockwise to increase the flow or clockwise to decrease the flow.

Figure 10. Make-up gas flow rate adjustment screw



Note The outer knurled knob is not used for adjustment; it is used only for turning the flow on or off. Make sure the outer knob is in the full counterclockwise (open) position before you adjust the inner flow control screw.

Related Topics

- [Connecting the Transfer and Make-up Gas Lines](#)

Cooling the GC-IR Module Detector

GC-IR applications require a highly sensitive mercury-cadmium telluride (MCT) detector. This detector is sensitive to infrared radiation between 5000 cm^{-1} and 600 cm^{-1} .

An MCT detector must be cooled with liquid nitrogen before you collect data. The detector dewar is located under the dewar cover on the GC-IR module.

Figure 11. GC-IR Module detector dewar cover

Equipment needed:

- small plastic laboratory funnel
- one-quart, glass lined or stainless-steel lined laboratory vacuum bottle
- a supply of liquid nitrogen



WARNING Avoid freeze burns. Liquid nitrogen is extremely cold and therefore potentially hazardous.

- Wear protective equipment and follow standard laboratory safety practices.
- To avoid hazardous contact with liquid nitrogen, make sure any dewar or container used to hold liquid nitrogen can do so safely without breaking.
- When filling the dewar, be careful not to contact the liquid nitrogen with your skin. Fill the dewar slowly. Cooling the detector too quickly may cause the dewar to rapidly boil off liquid nitrogen.

❖ **To cool the GC-IR module detector**

1. Open the detector dewar cover and remove the plastic stopper.
2. Insert a funnel into the liquid nitrogen fill port of the detector dewar.
3. Slowly fill the detector dewar with liquid nitrogen through the funnel.

Cool the funnel and funnel stem first: fill the funnel and let it drain completely into the detector two or three times. Always let the liquid nitrogen flow into the detector before adding more liquid nitrogen.

A small amount of liquid nitrogen spillage will not harm the instrument, but do not overfill the detector or pour too quickly. Cooling the detector too quickly may cause the dewar to rapidly boil off or spill out excess amounts of liquid nitrogen.

NOTICE Do not spill liquid nitrogen onto or near the detector window. Rapid cooling of the window's o-ring seal can cause the dewar to lose vacuum. Prolonged exposure to atmospheric pressure can damage the detector element. Always fill the dewar with the instrument cover in place.

When the detector is nearly full, a small amount of liquid nitrogen will boil off. Approximately one liter of liquid nitrogen will be consumed in this process.

4. Let the vapor plume dissipate, replace the plastic stopper, and close the dewar cover.

Allow at least 20 minutes for the detector to cool before use.

Setting Flow Cell and Transfer Line Temperature

Two software-driven controllers located inside the GC-IR module heat the flow cell and transfer line. The temperature of the flow cell and transfer line should be stable before you start collecting data.

Table 6. Flow cell and transfer line recommended temperature

Normal Operating Range ^a	Acceptable Operating Range
200 to 280 °C	30 to 300 °C

^aWe generally recommend a flow cell and transfer line temperature 10 °C hotter than the maximum GC oven temperature program setting being used for the run. Infrared throughput, and therefore sensitivity, decreases with temperature, so do not set the controllers unnecessarily high.

NOTICE

- To prevent the flow cell windows from becoming contaminated, do not turn on the flow cell or transfer line heaters unless a GC column is installed and the helium make-up gas is flowing through the flow cell.
- Do not attempt to set the GC-IR temperature controllers manually or you may affect proper operation of the GC-IR module.

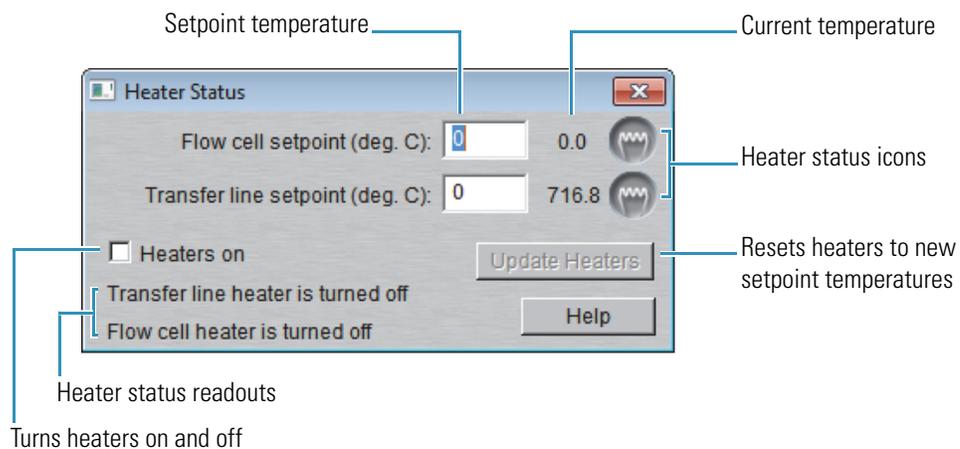
❖ To set the flow cell and transfer line temperature

1. Make sure a GC column is connected to the GC-IR module and the helium make-up gas is flowing through the flow cell. For more information, see [“Measuring and Adjusting the Make-up Gas Flow Rate.”](#)
2. Turn on the power switch for the GC-IR module.
3. Press the Touch Point on the top of the GC-IR Module.

4. Open OMNIC software.
5. Choose **Series > Heater Status**.

A dialog box is displayed.

Figure 12. Heater Status dialog box in OMNIC software



6. Select **Heaters On**.
7. Enter a value between 30 and 300 °C for each heater and then choose **Update Heaters**.

The table below describes the functions of the status indicators:

Table 7. Heater Status LED status and failure modes

Indicator	Function
Heater status	<p>LED states are:</p> <ul style="list-style-type: none"> • Gray - GC-IR module is powered off or the USB communications cable is not connected (GC-IR module to computer) • Yellow - Heating • Green - At setpoint temperature • Red - Failed (a message describes the error)
Possible failure errors	<ul style="list-style-type: none"> • Flow cell/transfer line input failure • Flow cell/transfer line data failure • Flow cell/transfer line heater failure • Flow cell/transfer line over temperature failure <p>If an error condition occurs, turn off the GC-IR module power switch, wait 1 minute and then turn the power switch back on. If that doesn't correct the problem, call our local service representative.</p>



CAUTION Avoid burn hazard. If the temperature indicator is red and an “over temperature failure” message appears, turn off the GC-IR module power switch and do not touch the transfer line, flow cell or vent line. Contact us for assistance.

Note After the heaters have reached the specified operating temperature, they will remain at that temperature until you clear the Heaters On option in Heater Status or turn off the power to the GC-IR module.

Related Topics

- [Cabling the GC-IR Module](#)

Recommended Column Flow Rates

GC-IR sensitivity is best when your sample elutes with sharp, narrow peaks. This level of performance is usually obtained by operating your chromatograph in its optimum efficiency range. For best results with the GC-IR application, use these recommended flow rates.

Table 8. Recommended flow rates for capillary GC columns

Column type	Flow rate (mL/min)
Standard (0.25 to 0.32 mm I.D.)	1 to 3
Wide-bore (0.53 mm I.D. or greater)	2 to 3

Related Topics

- [Installing a Standard Capillary GC Column](#)
- [Installing a Wide-Bore Capillary GC Column](#)
- [Measuring and Adjusting the Make-up Gas Flow Rate](#)

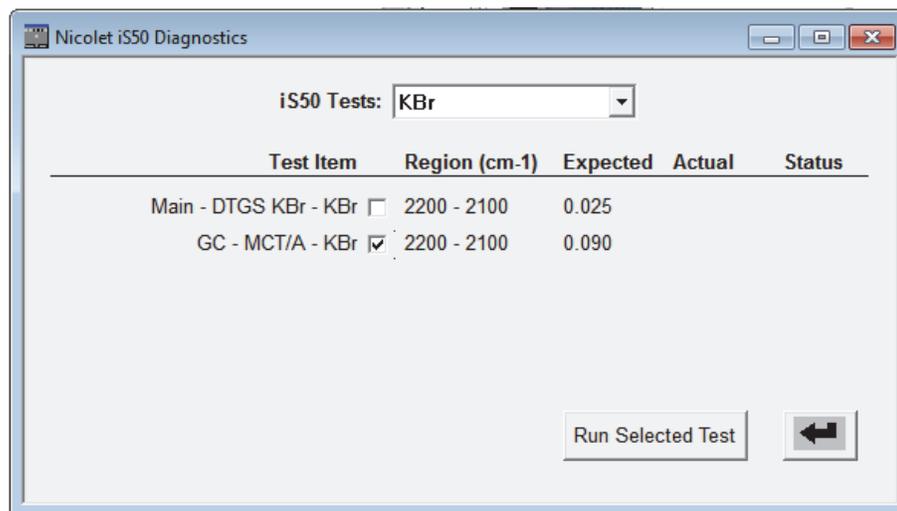
Checking Performance

You should periodically check the performance of the GC-IR module by using the Bench Diagnostics software. Keep a log of the date each test is run and the test result. You should also record the flow cell temperature, the number of scans and the resolution.

❖ To run the performance test

1. Open OMNIC software.
2. Choose **Collect > Advanced Diagnostics**.
3. Choose **Performance Test**.

The Nicolet iS50 Diagnostics window lists the available performance tests.

Figure 13. Nicolet iS50 Diagnostics window

4. Select **GC - MCT/A**.
5. Make sure the check boxes for all the other tests are cleared.
6. Choose **Run Selected Test**.

The software collects a few background and sample scans to generate a 100% line spectrum and calculates the spectrum's signal to noise ratio. The numerical result is added to the Actual column in the diagnostics window and a Pass or Fail result appears in the Status column. If the Actual noise value is less than the Expected (or maximum allowable) value, the test will report a pass result.

7. Close the Bench Diagnostics window.

Note If the GC-IR module fails the performance test, consult the [Troubleshooting](#) information or contact our local service representative for assistance.

Related Topics

- [Troubleshooting](#)

Samples Compatible with GC-IR

Any sample that can be measured by gas chromatography will work with the GC-IR application. GC-IR is especially useful for differentiating configurational stereoisomers such as diastereomers and enantiomers.

Your First Experiment

This section explains how to configure your GC-IR workflow and demonstrates how to use the GC-IR system to analyze a sample. These topics are covered:

- [Configuring Your GC-IR Workflow](#)
- [Preparing the GC-IR System for Sampling](#)
- [Measuring a Sample by GC-IR](#)

Configuring Your GC-IR Workflow

The GC-IR Touch Point has an associated user-configurable workflow that can define everything from background and sample collection to quantitative analysis and spectral search. You simply press the Touch Point to start the workflow.

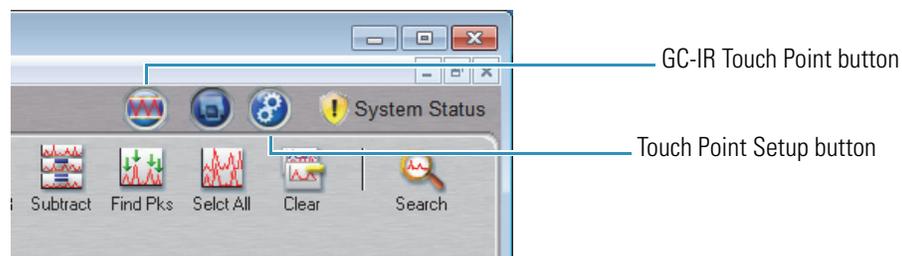
❖ To configure your GC-IR workflow

1. Start OMNIC software.

The OMNIC window is displayed. For information about the OMNIC window, choose Help > OMNIC Help Topics.

- Choose the **GC-IR Touch Point button** in the software to configure the instrument for GC-IR sampling.

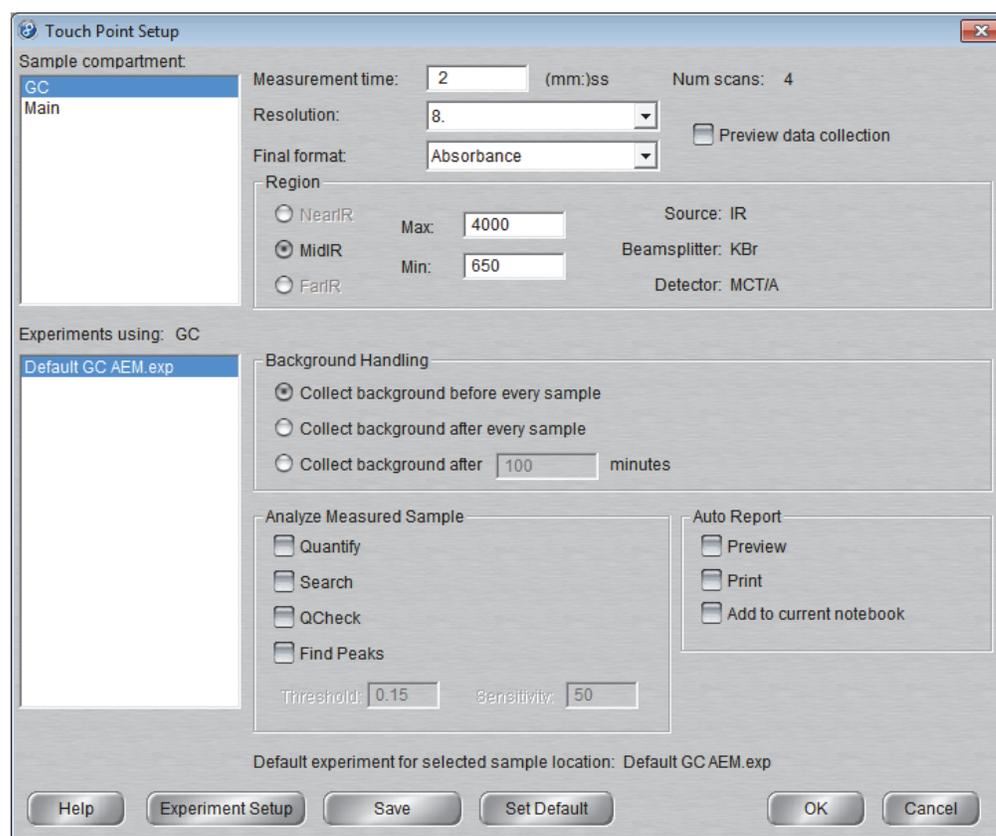
Figure 14. GC-IR Touch Point buttons



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

A dialog box is displayed.

Figure 15. Touch Point Setup for iS50 GC-IR module



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

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

Table 9. iS50 GC-IR Touch Point settings

Option	Description
Measurement time	Determines the duration of scanning for each collected spectrum.
Number of scans	Shows the scans collected for the selected measurement time. Multiple scans are averaged.
Resolution	Sets the spectral resolution of the data you collect. Typically 8 cm^{-1} or 4 cm^{-1} is a good starting point for most GC-IR applications. The smaller the resolution value, the higher (better) is the resolution. The resolution value, along with the measurement time, affects the number of scans.
Final format	Determines the Y-axis unit used for the collected data. Typically set to Absorbance for GC-IR applications.
Preview data collection	Select this option if you want to collect and view (but not save) preliminary data before the start of a sample or background data collection. This lets you verify that your experimental conditions are correct before you start collecting GC-IR data. This option is typically off for GC-IR applications since the start of data collection is usually coordinated with the injection time.
Region	Determines the spectral range of the spectrometer that will be used to collect the data. The spectral range is defined by the type of source, beamsplitter and detector used. Mid-IR is the only available Region option for the GC-IR module.
Max/min	Shows the default limits of the GC-IR detector's spectral range. The Max/Min limits can be reduced to eliminate spectral regions that provide no additional information about the sample.
Background handling	Lets you specify when to collect a background spectrum to be used for ratioing sample spectra you collect. Collecting a background after a specified number of minutes is a good choice for many applications. You will be prompted to collect a background the very first time you collect sample data.

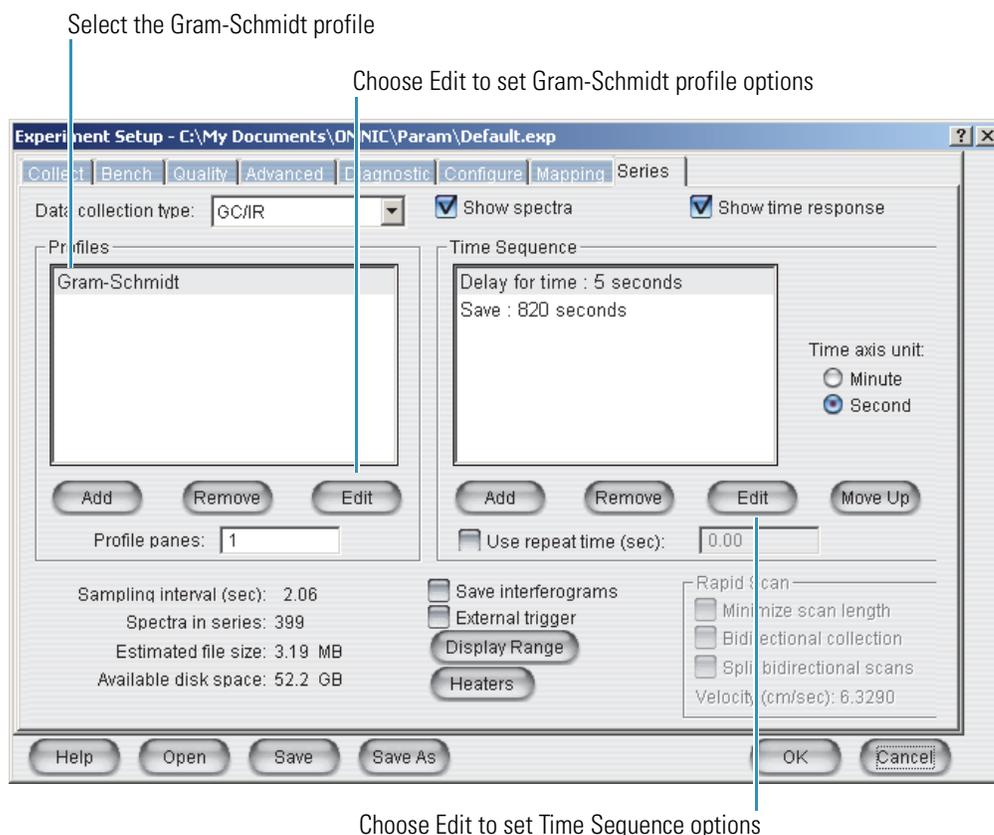
Table 9. iS50 GC-IR Touch Point settings

Option	Description
Analyze Measured Sample	<p>Allows you to select and run analyses on a collected sample spectrum. The analysis results are attached to the spectrum and saved in the spectral (.spa or .spg) file.</p>
	<p>Note: These settings apply only to single spectra collected as an .spa or .spg file. The settings are available for GC-IR applications but not typically used since most GC-IR experiments collect a series of spectra over a period of time and save them in a series (.srs) file.</p>
	<p>The following analyses are available:</p>
	<ul style="list-style-type: none"> • Quantify. Finds the concentrations of components in a sample spectrum. For more information, find “Quantifying a Spectrum” in OMNIC Help Topics. To select the quantitative analysis method you want to use to quantify a spectrum, choose Analyze > Quant Setup.
	<ul style="list-style-type: none"> • Search. Identifies an unknown material by comparing the unknown sample spectrum with each reference spectrum in the selected libraries to find the spectra that most closely match the unknown. For more information, find “Searching a Spectral Library” in OMNIC Help Topics. Use Analyze > Library Setup to select the reference libraries to be searched.
	<ul style="list-style-type: none"> • QCheck. Compares spectra to determine their degree of similarity, expressed as a correlation value from 0.0 (no similarity) to 1.0 (the spectra are identical). For more information, find “Comparing Spectra with QCheck” in OMNIC Help Topics. Use Analyze > QCheck Setup to set up the comparison.
	<ul style="list-style-type: none"> • Find Peaks. Identifies peak locations in a spectrum by finding peaks whose Y values exceed a specified threshold value. The peaks are labeled with their X values. For more information, locate “Finding Peaks Above a Specified Height” in OMNIC Help Topics. <ul style="list-style-type: none"> • Threshold. The Y value above which peaks can be found. • Sensitivity. Determines how readily shoulders on peaks and small peaks in the baseline will be found.

Table 9. iS50 GC-IR Touch Point settings

Option	Description
Auto Report	<p>Can be used to automatically display, print or save an auto report of the analysis results. For more information, find “Working with Auto Reports” in OMNIC Help Topics.</p> <p>Note: These settings apply only to single spectra collected as an .spa or .spg file. The settings are available for GC-IR applications but not typically used since most GC-IR experiments collect a series of spectra over a period of time and save them in a series (.srs) file.</p> <p>The following options are available:</p> <ul style="list-style-type: none"> • Preview. Displays the auto report after the analysis is completed. • Print. Prints the auto report after the analysis is completed. • Add to current notebook. Adds the report to the current notebook. For more information, find “Adding a Report to a Notebook” in OMNIC Help Topics.
Default experiment	Shows the file name of the default experiment for the selected sample location.

4. Select an appropriate experiment in the “Experiments Using: GC” pane.
5. Set the Touch Point Setup options as desired.
6. If you want to overwrite the default settings without overwriting the default experiment, choose **Set Default**.
7. Choose **Save**.
8. Choose **Experiment Setup** at the bottom of Touch Point Setup.
9. In Experiment Setup, select the **Series** tab.

Figure 16. Series tab in Experiment Setup

10. In the Profiles box, select **Gram-Schmidt** to create a Gram-Schmidt profile from the collected data.
11. To see the options available for the Gram-Schmidt profile, choose **Edit** below the Profiles box. For more information, choose a **Help** button in the software.
12. Choose **Edit** below the Time Sequence box and set **Segment Type** to **Save** and **Save Time** to match the total collection time for the GC and choose **OK**. This will save all the data collected during your GC run.
13. Choose **Save** at the bottom of Experiment Setup and then choose **OK**.

For an introduction to Series data collection including setting up profiles and time segments, refer to your *Series Getting Started* guide. For details about the features on the Series tab, open Series Help Topics from the OMNIC Help menu. For information about features on other tabs in Experiment Setup, choose Help from Experiment Setup.

Note Some settings appear in Experiment Setup and Touch Point Setup. Setting these options in one location automatically changes them in the other.

Related Topics

- [Preparing the GC-IR System for Sampling](#)

- [Measuring a Sample by GC-IR](#)

Preparing the GC-IR System for Sampling

Follow the steps in this section to prepare the GC-IR system and measure a sample.

❖ To prepare the GC-IR system

1. 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).
2. Power on the GC-IR module.
3. Open OMNIC software.
4. Press the GC-IR Touch Point to configure the instrument optics for GC-IR sampling and load the associated workflow.
5. Choose the **Touch Point Setup** button in the software and adjust the Touch Point Setup and Experiment Setup settings as needed. See “[Configuring Your GC-IR Workflow](#)” for more information.
6. Power on the GC and set the oven temperature. Refer to the operating manual that came with your GC for more information.
7. Set the make-up gas pressure and flow; see “[Measuring and Adjusting the Make-up Gas Flow Rate](#)” for more information.

NOTICE To prevent the flow cell windows from becoming contaminated, make sure make-up gas is flowing through the flow cell before you turn on the flow cell and transfer line heaters.

8. In OMNIC software, choose **Series > Heater Status**. Turn on **Heater** and enter a setpoint temperature for the GC-IR transfer line and flow cell (should be 10 °C hotter than the maximum temperature for the GC oven). For more information, see “[Setting Flow Cell and Transfer Line Temperature](#).”



CAUTION Avoid burn hazard. If the temperature indicator is red and an “over temperature failure” message appears, turn off the GC-IR module power switch and do not touch the transfer line, flow cell or vent line. Contact us for assistance.

9. Wait for the spectrometer and heaters to stabilize.

Related Topics

- [Measuring and Adjusting the Make-up Gas Flow Rate](#)
- [Setting Flow Cell and Transfer Line Temperature](#)
- [Configuring Your GC-IR Workflow](#)

- [Measuring a Sample by GC-IR](#)

Measuring a Sample by GC-IR

Before you begin

Have your sample material at hand. For this first experiment, we recommend that you choose a known material that produces a GC spectrum with distinct, sharp peaks.

Figure 17. GC-IR system ready for sampling



❖ To measure a sample with your GC-IR system

1. Follow the steps in [“Preparing the GC-IR System for Sampling.”](#)
2. If your GC-IR workflow is not configured, follow the steps in [“Configuring Your GC-IR Workflow”](#) to configure it.
3. Choose **Collect > Collect Series**.

If no infrared background spectrum is available, a message asks you to prepare for the background collection. The background serves as a reference to compare with the sample data that is collected over time.

4. Choose **OK** to start collecting a background spectrum.

When the background collection is completed, another message asks you to prepare to collect a set of basis vectors which are used to calculate the Gram-Schmidt profile.

5. Choose **OK** to start collecting the basis vectors.

The Collect Series Basis Vectors window appears showing the progress of the collection. When data collection is finished, the Save Group box appears.

6. Specify a file name and directory location and choose **OK**.

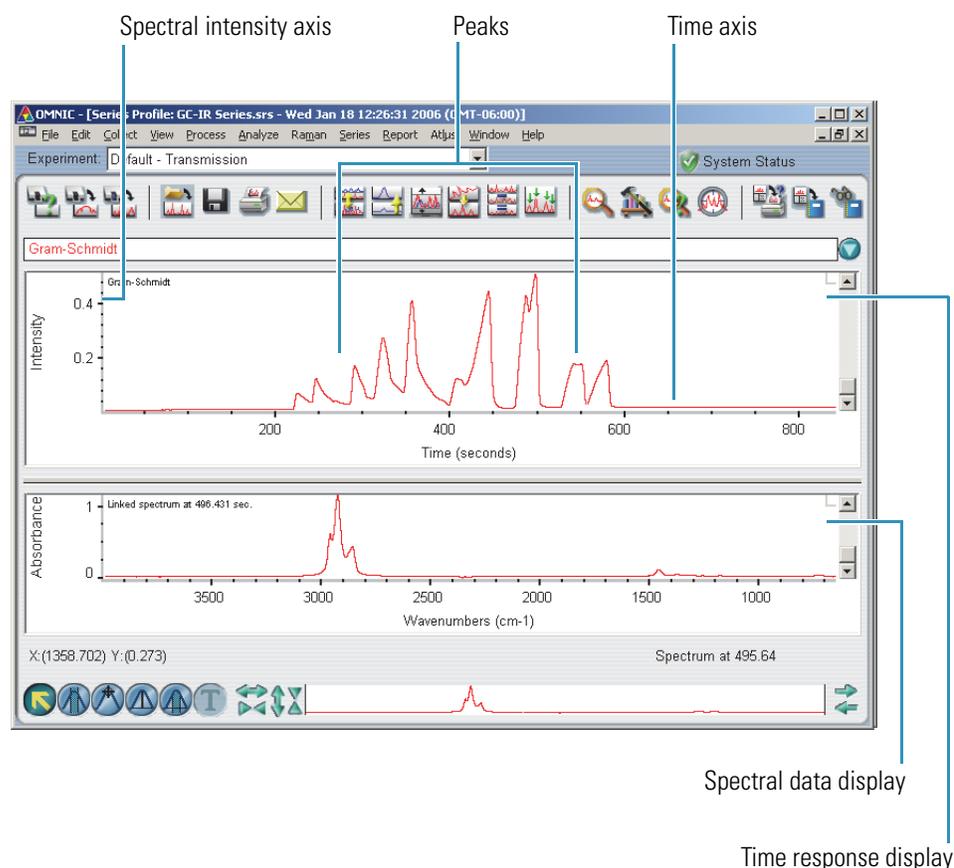
After the basis vectors have been collected, another message tells you to choose OK when you are ready to begin sample data collection.

7. Inject a sample into the GC and start the GC run.
8. Choose **OK** in the GC-IR system software (or press the space bar on the computer keyboard) to start GC-IR data collection.

Note If you are using an external trigger, GC-IR data collection will start automatically when you start the GC run. For more information, refer to the document that came with the external trigger cable.

As the experiment proceeds, the Gram-Schmidt profile gradually appears, left to right, in a time response display. The Gram-Schmidt profile is a plot showing how overall spectral intensity changed during the experiment. In the GC-IR example below, the peaks in the Gram-Schmidt plot indicate when different compounds in the sample eluted from the GC column, passed through the spectrometer beam path and absorbed infrared energy.

Figure 18. Gram-Schmidt reconstruction



The spectrum currently being collected appears in the spectral data display.

Note If you don't see spectral features in this window, inject a sample that is at a higher concentration (2 to 10 times higher than the previous one) and collect the data again. If you are using a split injection technique, you can also decrease the split ratio so a higher concentration of the sample goes onto the GC column. For example, if you were using a 100:1 split ratio, try changing it to 20:1.

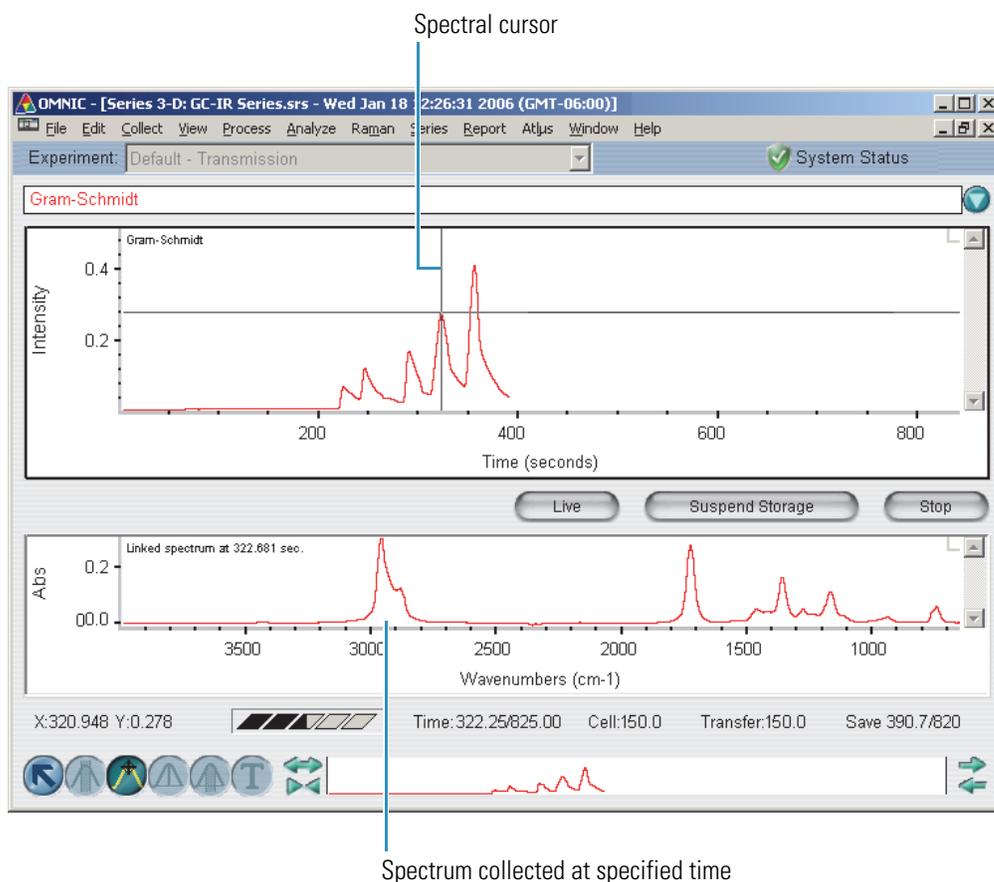
9. To display a spectrum collected at a particular time, first select the spectral cursor tool...



...and then click the desired time location in the profile in the time response display.

Here is an example showing the cursor located at the top of a peak in the time response display and the corresponding spectrum in the spectral data display.

Figure 19. Using the spectral cursor tool to display the spectrum collected at a specific time in the series data

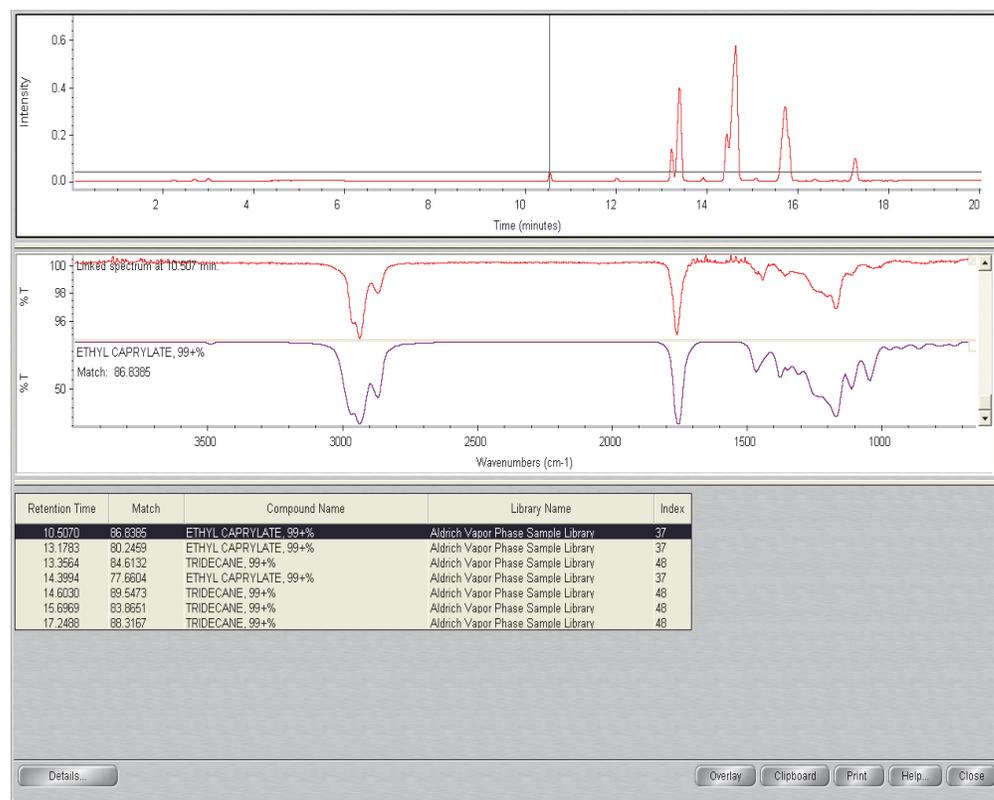


Note To return to a “live” display that shows the spectrum currently being collected, click the Live button.

When the series experiment is completed, the collected data appears in a series profile window.

10. If you want to search the selected GC-IR spectrum, choose **Series > Mercury GC > GC Setup**. In the Mercury GC Setup box, select a vapor phase library that contains compounds that are similar to your sample material.
11. Choose **GC Identify** to perform the search. For more information about Mercury GC features, choose the Help button in the Mercury GC results window.

Figure 20. Mercury GC search results



12. If you are finished analyzing GC-IR samples, lower the flow cell and transfer line heater settings to 125 °C (257 °F) but leave the make-up gas flow on.

If you don't plan to use the GC-IR module for an extended period, lower the flow cell and transfer line heater settings as described above. When the flow cell and transfer lines have cooled to room temperature, turn off the make-up gas flow and then turn off the power switch on the GC-IR module. There should still be purge gas flowing through the GC-IR module.

Related Topics

- [Connecting a Purge Source](#)
- [Setting Flow Cell and Transfer Line Temperature](#)

- [Configuring Your GC-IR Workflow](#)
- [Preparing the GC-IR System for Sampling](#)

Maintenance

This section covers these topics:

- [Resetting the Circuit Breaker](#)
- [Maintaining the Carbon Filter](#)



CAUTION Avoid burn hazard. Do not touch the bare transfer line, flow cell or vent line when the insulation has been removed and the heaters are on. Temperatures can exceed 300 °C (572 °F).

Resetting the Circuit Breaker

If the GC-IR module power indicator does not light when you turn on the power switch, the circuit breaker may need to be reset. This may occur after a ground fault or a short in the power line.

❖ To reset the breaker switch

1. Turn off the GC-IR module power by pressing the power switch on the back panel.
2. Wait 1 to 3 minutes and then turn the power switch back on.

If the power indicator still does not light, contact our service representative in your area.

Related Topics

- [Troubleshooting](#)

Maintaining the Carbon Filter

The carbon filter does not require maintenance or replacement. It is expected to last the lifetime of your instrument.

Troubleshooting

This section explains how to troubleshoot problems with the GC-IR system. These topics are covered.

- [Chromatographic Hardware Problems](#)
- [Spectrometer or GC-IR Module Hardware Problems](#)
- [Other Problems](#)

Chromatographic Hardware Problems

The best way to determine if there is a problem with the GC hardware is to look at the same data from the GC detector. If your GC-IR module has the 10:1 splitter (see [“Connecting the GC Detector”](#)), the GC detector is already connected. If your GC-IR module does not have a splitter, disconnect the GC column from the GC-IR module and connect it directly to the GC detector. The GC can then be used by itself.

Run your sample on the GC and check to see if the separation and results are what you expect. If the problem persists when the GC is used by itself, refer to the troubleshooting section of the GC reference manual. If the GC results look normal, you can assume the problem is not with the GC hardware. See [“Spectrometer or GC-IR Module Hardware Problems”](#) for more information.

Related Topics

- [Connecting the GC Detector](#)
- [Spectrometer or GC-IR Module Hardware Problems](#)

Spectrometer or GC-IR Module Hardware Problems

These problems fall into two categories:

- Low infrared throughput
- Chromatographic problems within the GC-IR module.

Low infrared throughput can be recognized if you keep a record of the peak-to-peak interferogram voltage, a single-beam spectrum, the results of the Performance Test (from the Bench Diagnostic program), and a 100% line. You should record the flow cell temperature, the number of scans and the resolution. By keeping this information, you can monitor the system integrity over time.

Causes of low infrared throughput may involve:

- Alignment of the beamsplitter or flow cell
- Contamination of the flow cell or flow cell windows
- Failed IR components, including the source and detector (if not properly cooled). A quick check of the signal from the detector in the main spectrometer will check most of the FT-IR system components.

You can recognize chromatographic problems within the GC-IR module if you first run your GC without the GC-IR module connected (see [“Chromatographic Hardware Problems”](#)). If the GC appears to be operating correctly as a stand-alone instrument, check the transfer lines and flow cell. Sources of leaks or blockages may include:

- **Transfer lines.** Ferrules may be unseated, or graphite or other contaminants may be blocking the lines.

- **Flow cell.** Flow cell windows may be cracked, end cap seals may be unseated, or an end cap may be plugged.

You can isolate leaks in the system by holding an opened but not lit butane lighter in each of the suspected problem areas while flowing carrier gas through the flow cell and into the FID (Flame Ionization Detector). If you used a splitter to connect the GC detector, monitor the FID signal on the GC. The presence of an FID peak for butane means a leak exists.

You can find plugged lines if no infrared signal changes are detected for an injection of a low-boiling solvent (for example, CH₂Cl₂). Monitor this in the GC scan mode.

Related Topics

- [Connecting the GC Detector](#)
- [Chromatographic Hardware Problems](#)

Other Problems

Heater Status not available in software

If you don't see the Heater Status item in the Series menu of OMNIC software, make sure the GC beam path is selected. To do this, either press the Touch Point button on the top of the GC-IR module or choose **Collect > Experiment Setup**, select the **Bench** tab and set **Sample Compartment** to GC.

