

Thermo Fisher SCIENTIFIC GC-IR Module OMNIC Paradigm Software User Guide

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GC-IR Module OMNIC Paradigm Software

**GC-IR Module
with OMNIC Paradigm software**



User Guide

269-362201 Revision A November 2023 **ThermoFisher**
SCIENTIFIC

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WARNING

Avoid an explosion or fire hazard.

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

Chapter 1

GC-IR Module

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



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 to provide safety and other special information:



DANGER

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



WARNING

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



CAUTION

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

NOTICE

Follow instructions with this label to avoid damaging the system hardware or losing data. Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

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About the GC-IR Module

The 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 FTIR spectrometer as well as the following gas chromatographs and autosamplers:

Thermo Scientific TRACE™ 1300 series gas chromatographs with autosamplers

Agilent™ 7890 gas chromatograph

Figure 1-1: GC-IR system with spectrometer and TRACE 1310 GC



A Nicolet iS50 FTIR spectrometer

B GC-IR Module

C Thermo Scientific TRACE 1310 GC

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

From the front

Figure 1-1: GC-IR Module front view



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

A Detector fill port

B External beam port

C GC-IR Touch Point

D Transfer line

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](#).

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

GC-IR 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.

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.

GC-IR status indicator has the following states:

LED status Meaning

On Ready for use

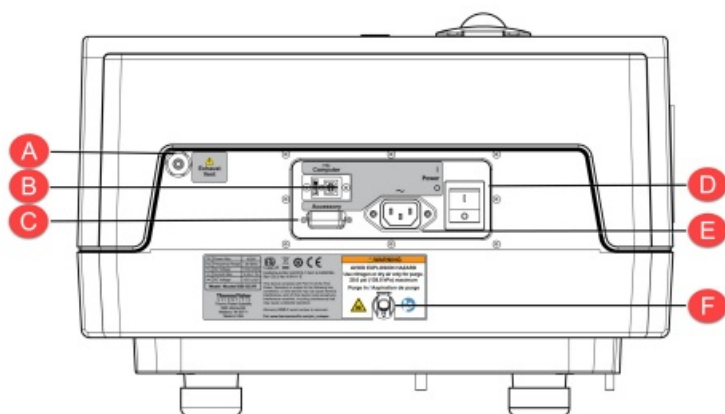
Off System is not configured for GC-IR sampling

Blinking Optics are reconfiguring

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Back panel features

Figure 1-2: GC-IR Module back panel



A 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.

B Computer: Connector for the USB communications cable.

C Accessory: Connector for the detector cable. See Cabling the GC-IR Module for details.

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D 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.

E AC power: Connector for the power cord.

F Purge: Connects to the purge gas supply. For more information, see Connecting a purge source.



WARNING

Avoid hazard. 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.

Specifications

The following items are the specifications for the GC-IR Module.

Feature Specification

Compatible gas

chromatographs

Maximum temperature of transfer/exhaust lines and flow cell

Thermo Scientific TRACE 1300 series Agilent 7890

300 °C

Temperature status output Continuous

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Feature Specification

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.

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

Software compatible with the GC-IR Module

Software Use for...

Collecting, processing, analyzing, and searching time

OMNIC Paradigm

series data.

For more information, see the Your first experiment section.

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Software Use for...

Creating quantitative and classification methods that can be used with OMNIC Paradigm software. This software provides an extensive suite of chemometrics

TQ Analyst

Security Suite 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.

Security Suite provides software for security administration, audit management, control access, and event logging.

GC-IR sampling

In a GC-IR experiment, the sample is injected into the column of a gas chromatograph that separates the sample components. The GC column is routed through a heated, glass-lined, stainless steel transfer line onto 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*. Thermo Scientific GC-IR Module User Guide 11

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 describes how to prepare the system for running GC-IR experiments.



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 GC-IR Module.

Note Provide necessary space such that the unit can be easily plugged or unplugged.



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.

Figure 1-1: 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.

Plug one end of the detector cable into the Accessory port

on the back of the GC-IR Module. The other end goes to

Detector

Exhaust

Remote trigger (optional)

Communications^a

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.

Connect a length of 1/8-inch 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*.

Note: You can connect a flow meter to the exhaust vent to measure the carrier gas flow rate through the flow cell.

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 *Configuring series data collection to start automatically*.

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.

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

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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 document 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).

Adjust the GC-IR Module purge gas supply pressure and flow rate.

Figure 1-2: Recommended purge pressure and flow for the 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

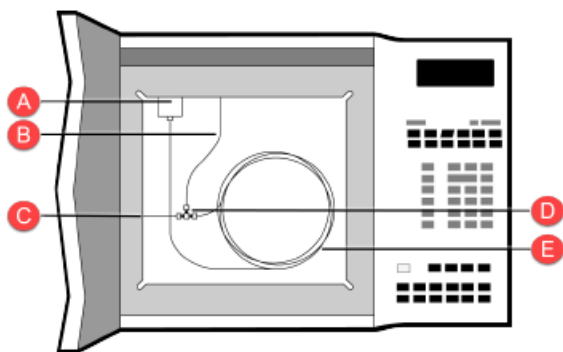
To protect flow cell windows from contamination, make sure that a GC column is installed and helium make-up gas is flowing through the flow cell before you turn on the flow cell or transfer line heaters.

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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, provided the GC column is routed to both the IR detector and the GC detector.

Figure 1-3: GC-IR Module column connections (without the splitter)



A GC injector

B Make-up gas line

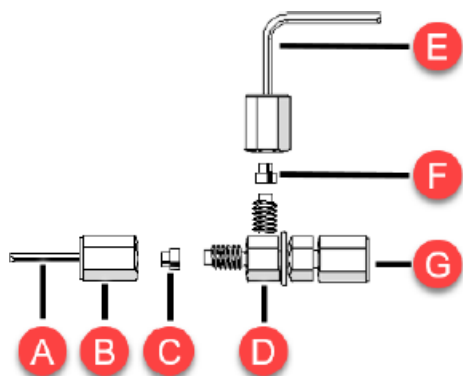
C Transfer line to GC-IR Module

D Tee union

E GC column

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 1-4: Transfer line and make-up gas line connections



A 1/16 in (0.2 cm) OD stainless steel, glass-lined transfer line

B Larger ID cap nut

C Graphite ferrule (size GFF/16)

D Tee union

E Make-up gas line

F Graphite ferrule (size GFF/16)

G Smaller ID cap nut

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❖ 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. Take care not to plug the transfer line with graphite from the ferrule after you slide it over the end.

A Transfer line

B Larger ID cap nut

C Tee union

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D Graphite ferrule (size GFF/005 for 0.25 mm ID columns)

E Smaller ID cap nut

F Standard-bore 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.

Note If a splitter is used to connect the GC detector, stop here and follow the instructions in Connecting the detector to connect the other end of the GC column to the splitter.

2. Start OMNIC Paradigm software. The software should load the preset settings for GC-IR. If not, go to Configure > Sample Location > GC-IR to load the default settings.

3. 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. To display the interferogram, select More in the upper right hand side of the Dashboard to see additional settings, then select

Interferogram.

4. 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.

5. Thread the free end of your GC column through the cap nut and a ferrule that fits onto the column.

6. 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.

7. While watching the live display, carefully feed the column through the Tee union and transfer line and into the flow cell.

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The live display shows the detector signal. The signal is represented as an interferogram as well as numerically as minimum and maximum voltage values.

8. 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.

NOTICE

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.

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

9. Slowly withdraw just enough column so that the interferogram regains its original height.

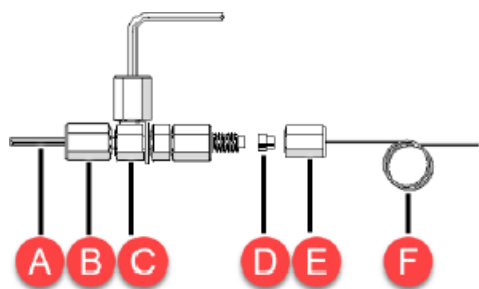
10. Use a 5/16-in wrench and a 1/4-in wrench to tighten the column nut on the Tee union until you feel the nut start to offer resistance. Then tighten the nut another one-quarter turn.

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.

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Figure 1-6: Connections for a wide-bore capillary GC column



A Transfer line

B Larger ID cap nut

C Tee union

D Graphite ferrule (size GFF/10 for 0.53 mm ID column)

E Smaller ID cap nut

F 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 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 detector to connect the other end of the GC column to the splitter.

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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 a 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.

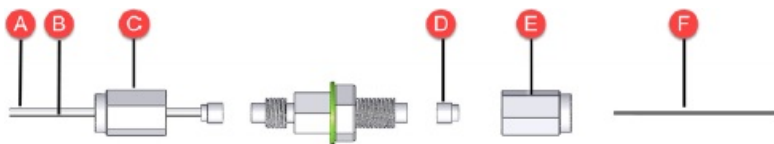
Connecting the detector

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. If desired, you may omit this step 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 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 1-7: GC detector connection using a 10:1 splitter



A Smaller diameter (lighter) capillary tubing connects to GC detector using new GFF/005 ferrule

B Larger diameter (darker) capillary tubing connects to GC detector using new new GFF/005 ferrule

C Cap nut

D Graphite ferrule (use appropriate size for your column)

E Cap nut

F GC column

❖ 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. Thermo Scientific GC-IR Module User Guide 23

2. Connect the the GC column to the splitter.

a. Remove the cap nut from the long 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

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.

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.

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.

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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.

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.

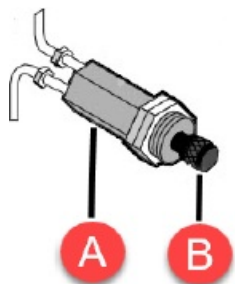
❖ To measure and adjust the make-up gas flow rate

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 1-8: Make-up gas supply valve



A Make-up gas supply valve

B Knurled knob

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.

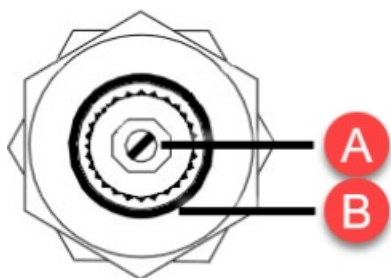
7. Measure the flow rate again.

This is the make-up gas flow rate.

8. 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 1-9: Make-up gas flow rate adjustment screw



A Adjustment screw

B Knurled knob (top view)

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.

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.

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Figure 1-10: GC-IR Module detector dewar cover



Equipment needed:

- one small plastic laboratory funnel
- one laboratory vacuum bottle (one-quart, glass- or stainless steel-lined)
- 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, 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.

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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 1-1:

Flow cell and transfer line recommended temperature

Normal Operating Range^a Acceptable Operating Range

200 to 280 °C 30 to 300 °C

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.

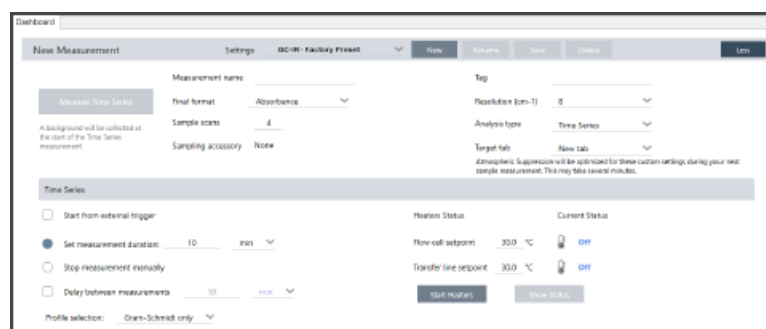
aWe generally recommend a flow cell and transfer line temperature 10 C° hotter than the maximum GC oven temperature program setting being used for the run. Infrared throughput, and therefore sensitivity, decreases with temperature, so do not set the controllers unnecessarily high.

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❖ To set the flow cell and transfer line temperature

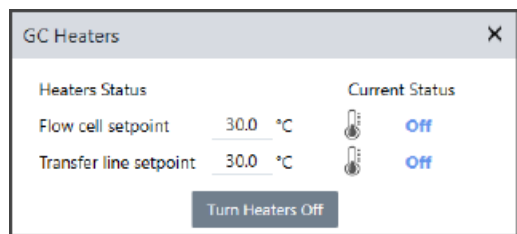
1. Make sure a GC column is connected to the GC-IR Module and the helium make-up gas is flowing through the flow cell. For more information, see Measuring and adjusting the make-up gas flow rate.
2. Turn on the power switch for the GC-IR Module.
3. Press the Touch Point on the top of the GC-IR Module.
4. Open OMNIC Paradigm software. Select More to see additional options.

Figure 1-11: GC-IR factory settings in OMNIC Paradigm software



5. Choose Start Heaters. A dialog box is displayed.
6. Select Turn Heaters On, then enter a value between 30 and 300 °C for each heater and then choose Update Heaters.

Figure 1-12: GC Heaters dialog box in OMNIC Paradigm software



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The table below describes the functions of the status indicators:

Figure 1-13: Heater Status LED and failure modes

Indicator Function

LED states are:

- Gray – GC-IR Module is powered off or the USB communications cable is not connected (GC-IR Module to Heater status Possible failure errors computer)
- Yellow – Heating
- Green – At setpoint temperature
- Red – Failed (a message describes the error)
- 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

If an error condition occurs, turn off the GC-IR Module power switch, wait 1 minute, and turn the power switch back on. If that doesn't correct the problem, call our local service representative.

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CAUTION

Avoid burn hazard. If the temperature indicator is red and an "over temperature failure" message appears, turn off the GC-IR Module or TGA interface power switch and do not touch the transfer line, flow cell, or exhaust 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.

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.

Figure 1-14: 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
--	--------

Configuring series data collection to start automatically

If you have the optional Thermo Scientific remote start cable for the Nicolet spectrometer, you can set up OMNIC Paradigm software to start automatically after each sample is injected onto the GC.

To order a cable, call your local Thermo Scientific sales or service representative or use the information at the beginning of this document or help system to contact us.

To install the cable, follow the instructions in the spectrometer documentation set.

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❖ To configure OMNIC Paradigm software for remote sampling

1. Start OMNIC Paradigm software.
2. In the Dashboard, choose Time Series under Analysis type.
3. Select More in the upper right corner to see additional settings.
4. Check the Start with external trigger box.

Data collection begins with the start of the next full scan following the trigger pulse relay contact close. No data is collected from partial scans.

For high resolution scans or slow scan speeds, there is a potential for time lag.

❖ To configure a Trace 1300 series GC for remote sampling

1. Select GC Configuration > Handshaking.

2. See the TRACE 1300 user guide in the Thermo Fisher Knowledge base for more information. 3. Set Remote Start to use a Low to High transition.

Figure 1-15: Trace 1300 series GC Configuration screen



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Extended heated transfer line

Your GC-IR Module can be configured with an extended heated transfer line and can then be connected to your instrument of choice by a Thermo Fisher representative.

Figure 1-1: Extended heated transfer line with an uncovered GC-IR unit



NOTICE

- Handle the transfer line gently and do not exceed its bend radius of 7.6 cm (3.0 in). Support the transfer line while you transport the instrument to avoid damaging or breaking it.
- The outside surface of the transfer line is abrasive; if it consistently rubs against another surface, it may cause scratches.
- Make sure the transfer line is supported while you are connecting it as well as afterward. Do not allow the transfer line to hang unsupported, as the extra weight may interfere with the transfer line's lifetime performance.

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).

CAUTION

Avoid shock hazard. Do not let the hot part of the heated transfer line touch the power cord, as the cord is not rated for such temperature. Failure to keep the transfer line away from the power cord could result in electrical shock or damage to the cord.

Specifications

- Tip: 1/16-inch outside diameter tubing (can connect to equivalent Swagelok fittings)
- Length: 2.5 meters
- Operating temperatures: ambient to 300 °C (572 °F)

Usage

A Thermo Fisher representative will connect the transfer line to your instrument of choice.

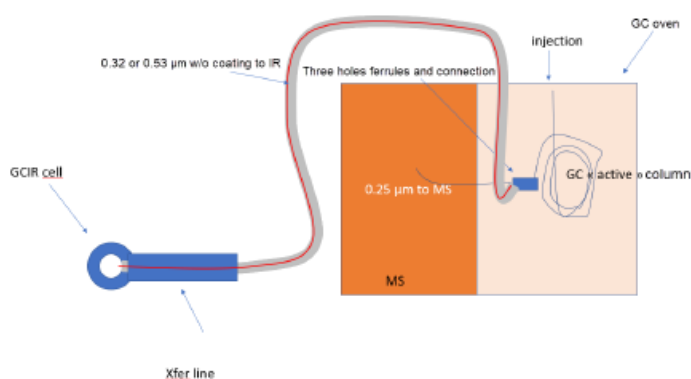
❖ To connect the heated transfer line

1. Insert the 1/16-inch tube into the GC oven using an accessory module appropriate for the type of GC that you are using. Use the large hex nut on the transfer line to anchor the heated transfer line to the accessory module.
2. Cut the 1/16-inch tube to the desired length. Be careful to ensure that the end is square and that no metal chips remain inside the tube.

Note Make sure that your table or work surface properly supports the transfer line to reduce wear at the connections.

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Figure 1-2: Diagram



Columns and tubing configurations

Different tubing diameter configurations are available depending on what type of instrument the heated transfer line must be connected to. Columns come in different diameters, so it is essential to select the right tubing

configuration for your specific needs.

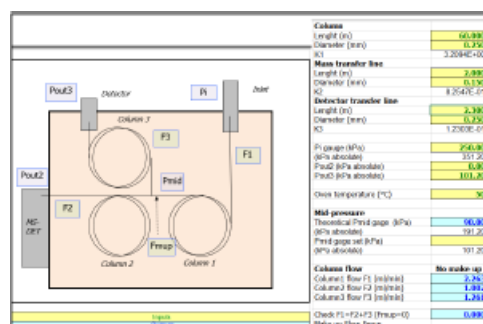
The appropriate configuration is decided at the time of purchase, but you may contact us to be referred to a Thermo Fisher Application Specialist to change configurations, or for assistance with connecting the transfer line to your instrument.

Note We recommend that you do not perform any calculations or configuration steps without the aid of an Application Specialist.

If your configuration requires a splitter, refer to the latest version of the TRACE 1300 and TRACE 1310 Dual Detector Microfluids Installation Guide and follow the steps for your GC instrument. A typical installation would not normally require a splitter.

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Figure 1-3: Calculations to define GC and mass spec needs



Checking performance

Ensure that the instrument is working well by running performance verification (PV) workflows and checking the system status.

Performance verification and qualification workflows

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

Note If the GC-IR Module fails the performance test, consult the Troubleshooting information or contact our local service representative for assistance.

Test Description

Nicolet FTIR – Factory Qual ification

Runs factory-recommended tests and all qual ification tests.

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Test Description

Nicolet FTIR – PV Test Measures basic performance based on factory recommended tests.

Nicolet FTIR – CP Qualification Executes the qualification tests as defined in the Chinese Pharmacopeia.

Nicolet FTIR – JP Qualification Executes the qualification tests as defined in the Japanese Pharmacopeia.

Nicolet FTIR – PHEUR Qualification

Executes the qualification tests as defined in the European Pharmacopeia.

Nicolet FTIR – USP Qualification Executes the qualification tests as defined in the U.S. Pharmacopeia.

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 demonstrates how to use the GC-IR system to analyze a sample.

Preparing the GC-IR system for sampling

Follow the steps in this section to prepare the GC-IR system and measure a sample. 38 GC-IR Module User Guide Thermo Scientific

❖ 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 Paradigm software. The preset factory settings will populate automatically, but you may create your own settings based on your analysis needs.
4. Press the GC-IR Touch Point to configure the instrument optics for GC-IR sampling. Alternatively, you can go to Configure > Sample location > GC-IR in OMNIC Paradigm software.
5. Power on the GC and set the oven temperature. Refer to the operating manual for your gas chromatograph more information.
6. 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 that make-up gas is flowing through the flow cell before you turn on the flow cell and transfer line heaters.

7. In OMNIC Paradigm software, enter a setpoint temperature for the GC-IR transfer line and flow cell (it 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 or TGA interface power switch and do not touch the transfer line, flow cell or exhaust line. Contact us for assistance.

8. Wait for the spectrometer and heaters to stabilize.

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.

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Figure 1-1: 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. In the OMNIC Paradigm software Dashboard, select Measure Time Series. This opens a new spectra tab with a background preview.
3. Select Start Background Measurement to collect a background. Wait for the process to complete.
4. Inject a sample into the GC and start the GC run.
5. Select Start Series Measurement and wait for the process to complete.

A Gram-Schmidt profile will appear, which is a plot showing how overall spectral intensity changed during the experiment.

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 ration 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.

6. To view a spectrum collected at a particular time, select the Gram-Schmidt profile. The crosshair indicates the point you currently have selected.

7. To identify the components that were measured, complete a Profile Search.

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a. In the series tab, select Search on the left-hand side. Then select New.

b. Enter the desired name and click OK. Then select Save.

c. Select Apply Search to display the results.

For more information on library searches, see the Analyze data topics in the OMNIC Paradigm User Guide at our online knowledge base.

8. 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.

Maintenance

This section covers basic maintenance of the GC-IR, including how to reset the circuit breaker and how to maintain the carbon filter.

CAUTION

Avoid burn and fire hazards. Do not touch the flow cell housing, transfer line or

exhaust line with your body or allow contact with a flammable substance when the

heaters are on and for up to 2 hours after you turn them off. Temperatures can

exceed 300 °C (572 °F). Never remove the insulating material from the transfer line.

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, then turn the power switch back on.

If the power indicator still does not light, contact our service representative in your area. Thermo Scientific GC-IR Module User Guide 41

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, the GC detector is already connected (see Connecting the detector). 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.

Spectrometer or GC-IR Module hardware problems

These problems fall into two categories:

- Low infrared throughput
- Chromatographic problems within the GC-IR Module

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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 FTIR 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.

Other problems


HEATER STATUS NOT AVAILABLE IN SOFTWARE

If you don't see the Heater Status item in the 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 go to Configure > Sample Location > GC-IR in OMNIC Paradigm software.

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Documents / Resources

	<p>Thermo Fisher SCIENTIFIC GC-IR Module OMNIC Paradigm Software [pdf] User Guide 269-362201, GC-IR Module OMNIC Paradigm Software, GC-IR Module, OMNIC Paradigm Software, Paradigm Software, Software</p>
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References

- [User Manual](#)

[Manuals+](#), [Privacy Policy](#)

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