thermo scientific

TGA Interface

with OMNIC Paradigm software

User Guide

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ThermoFisher SCIENTIFIC

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

For Research Use Only. This instrument or accessory is not a medical device and is not intended to be used for the prevention, diagnosis, treatment, or cure of disease.

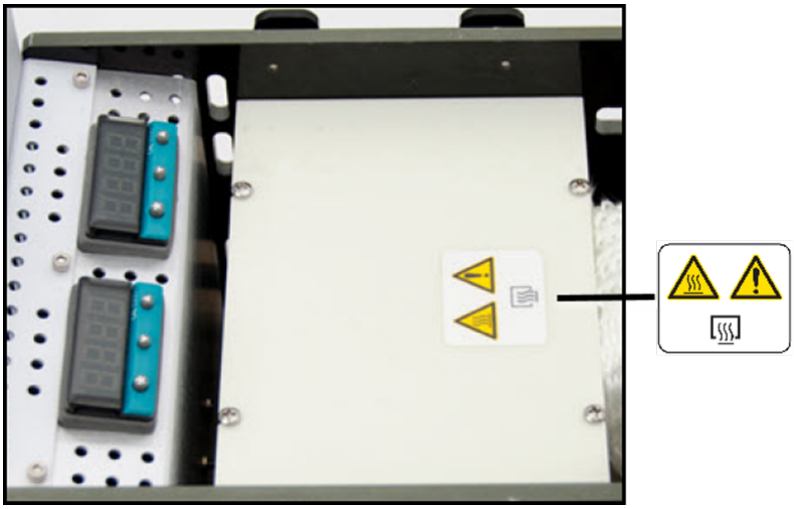
WARNING

Avoid an explosion or fire hazard.

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

Safety label locations

The TGA-IR Module comes with numerous safety labels. Heed the label content to avoid injury or hazard.



Chapter 1

TGA-IR Module

The TGA-IR Module allows you to couple your Thermo Scientific Nicolet™ iS™50 and iS20 spectrometers with a Thermogravimetric Analysis (TGA) furnace to run TGA-IR deformulation experiments when used with our OMNIC Paradigm software. The infrared spectrometer and the accompanying software provide state-of-the art tools to identify evolved gases and gas mixtures.

Figure 1-1: Nicolet iS50 spectrometer with TGA-IR Module installed

A	Nicolet iS50 FTIR spectrometer
B	TGA-IR Module

Before operating the TGA-IR Module, you should have a working knowledge of your FTIR spectrometer and the OMNIC™ Paradigm software. You will also need to know the fundamentals of thermogravimetric analysis and the operating characteristics of your TGA furnace.

WARNING

Avoid personal injury or equipment damage. Read and understand the accompanying documentation and all safety labels before attempting to install or use this equipment.

Using this document

This document describes how to install, operate, and maintain the TGA-IR Module and conduct TGA-IR experiments. It includes important safety precautions; always read and follow these precautions. For more information about TGA-IR Module safety, refer to the TGA-IR Module Site and Safety Supplement, which is included in your Thermo Scientific electronic documentation set.

Conventions used

This document uses these conventions for providing safety as well as other important information:

CONVENTIONS USED IN THIS DOCUMENT

DANGER

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

WARNING

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

CAUTION

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

NOTICE

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

Note Contains helpful supplementary information.

Tip Provides helpful information that can make a task easier.

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Chapter 2

About the TGA-IR Module

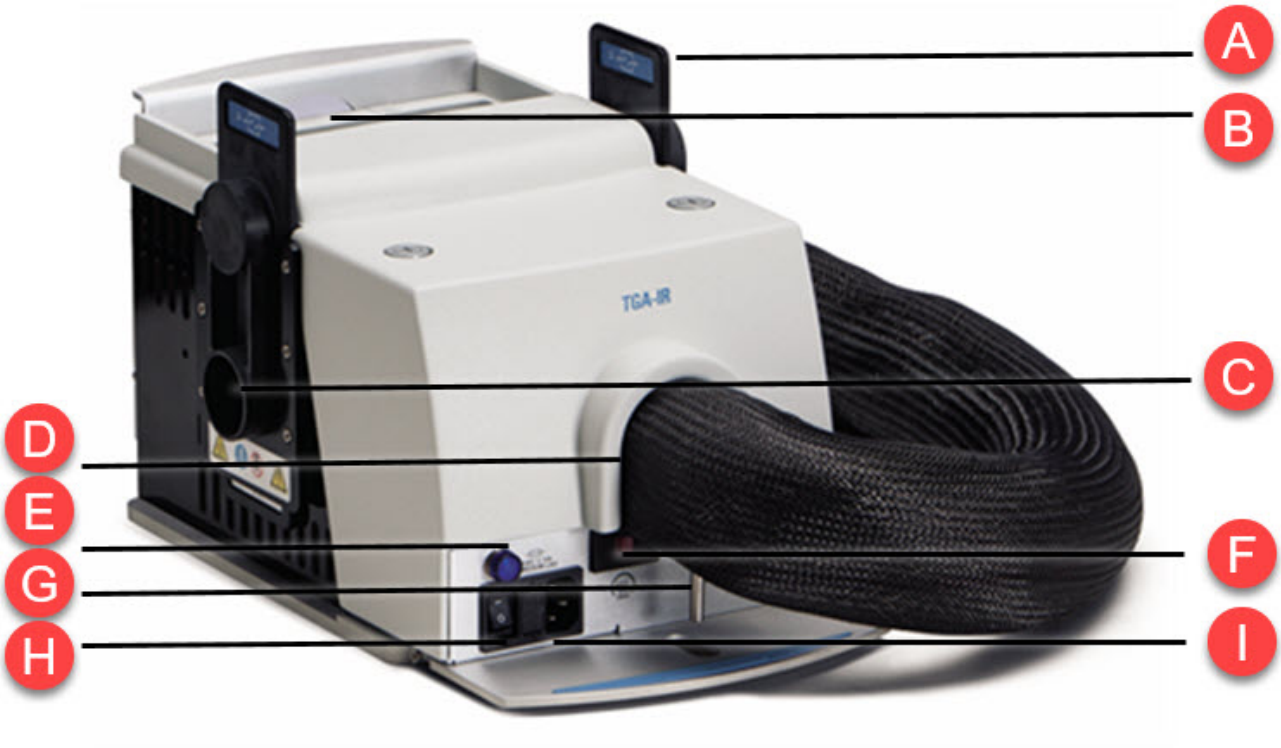
The TGA-IR Module fits in the sample compartment of a Nicolet Series FTIR spectrometer or AEM, and connects to the TGA furnace through a heated transfer line.

Figure 2-1: Nicolet iS50 spectrometer with TGA-IR Module installed

A	Nicolet iS50 FTIR Spectrometer
B	Temperature controllers (2)
C	TGA-IR Module
D	Heated transfer line

Important features

Figure 2-2: TGA-IR Module front view



A	**Beam port shutters (2)**
B	**Air vents**
C	**Beam ports (2)**
D	**Transfer line**
E	**Power button**
F	**Voltage select switches (2)**
G	**Exhaust tube**
H	**Power switch**
I	**AC power receptacle**

TGA-IR Module features include:

- Temperature controllers. Two temperature controllers allow you to specify the temperature of the flow cell and transfer line. See "Setting the flow cell and transfer line temperature" for more information.

  NOTICE

  The temperature controllers are configured at the factory and should be adjusted only by increasing or decreasing the temperature setting. Do not attempt to reprogram the controllers.

  Transfer line. Evolved sample gases from the TGA furnace travel through this precisely heated line to the heated flow cell inside the TGA-IR Module and then out the exhaust tube, which must be connected to a fume hood (via an optional peristaltic pump if used). The flow cell is where the sample interacts with the infrared beam to produce the detector signal in the FTIR spectrometer.

- Beam ports. Infrared light from the spectrometer passes through these ports to the flow cell in the TGA-IR Module and then continues back to the detector in the spectrometer.

- Beam port shutters. Depending on how these shutters are installed, they allow or prevent the infrared beam to pass through the TGA-IR Module and back to the spectrometer. The shutters slide into the holders on the right and left sides of the TGA-IR Module. They must have the end labeled “Open” positioned on top before the system can collect data.

Open

Allows the infrared beam to enter and exit the TGA-IR Module during data collection and seals out the laboratory environment.

Closed

Blocks beam ports in spectrometer while the TGA-IR Module heats up, allowing any excessively humid air to escape vertically.

NOTICE

Closing the beam port shutters prevents any excessively humid air released from the TGA-IR Module while it heats up from damaging sample compartment windows or other moisture-sensitive components in the spectrometer.

- AC power receptacle. Connector for the AC power cord input.
- Voltage select switches. Must be set to match the voltage of the local electricity supply before the power cord for the TGA-IR Module is plugged into a power outlet.

NOTICE

Always verify that the voltage select switches are set correctly for local electricity requirements before you plug in the power cord for the TGA-IR Module. If the switches are set incorrectly, the module may overheat and cause a failure. See "Setting up the TGA-IR system" for details.

- Power switch. Powers the TGA-IR Module on and off. A built-in fuse automatically shuts off power to the instrument if an over-current condition occurs. For information on recovering from an over current condition, see "Replacing fuses".
- Power indicator. Illuminates when the power is on.
- Air vents. Release excess heat generated by the TGA-IR Module.

CAUTION

Avoid fire hazard. To prevent the TGA-IR Module from overheating, never block the air vents on the module housing.

Specifications

Table 2-1:
TGA-IR Module specifications

Feature	Specification
Carrier gas (from TGA furnace)	
Recommended materials	Typically nitrogen (inert) or air (oxidative)
Recommended pressure	5 psi or less
Recommended flow rate[1]	30 mL/min to 100 mL/min[2]
Transfer line	Connects to 1/8-inch Swagelok™ fitting on most commercially available TGA furnaces
TGA furnace connector	
Vertical movement	20 cm (8 inches)
Horizontal movement	20 cm (8 inches)

[1]Attach a flow meter to exhaust tube on TGA-IR Module to measure flow rate.

[2]Higher flow rates will greatly reduce TGA-IR sensitivity and TGA balance stability. Check the documentation that came with your TGA furnace for recommended flow rates.

Feature	Specification
Flow cell (inside TGA-IR Module)	
Window material	Potassium bromide (KBr) or zinc selenide (ZnSe)
Volume (total gas volume inside cell)	1.332 cubic inches (21832.65 cubic mm)
Pathlength (inside dimension window-to-window)	3.945 inches (100.2 mm)
Recommended pressure/vacuum[1]	1 psi (7 kPa) to -3 psi (21 Kpa) vacuum
Variable-flow peristaltic pump (optional)[2]	
Pump tubing	Silicon (0.25 in internal diameter)
Recommended flow rate	0.4 to 85 mL/min
Operating temperatures[3]	
Flow cell	Ambient to 300 °C (572 °F)
Transfer line	Ambient to 300 °C (572 °F)
FTIR detector (inside FTIR spectrometer)	Typically DTGS or MCT-B (for faster data collection; for example, to capture dynamic events)

WARNING

Avoid fire and explosion hazard.

Do not connect a flammable carrier gas to the TGA-IR system.

Keep the workspace clear of flammable materials when the TGA-IR Module heaters and TGA furnace are powered on and for up to 2 hours after the TGA-IR Module and furnace are turned off.

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[1]Higher pressures could cause leaks or damage flow cell windows in TGA-IR Module.

[2]A pump or similar source of suction can be used to draw TGA gases through flow cell at the optimal rate. The pump must be suitable for liquids and gases. Attach pump to TGA-IR Module exhaust tube. Direction of flow should pull air through TGA-IR system.

[3]Both temperature controllers require 30 min to heat from 20 °C to 200 °C (60 min from 20 °C to 300 °C).

Compatible software

Table 2-2:
Software compatible with the TGA-IR Module

Software	Use for...
OMNIC Paradigm™	Collecting and analyzing your data. For more information, see "Performing a TGA-IR measurement".
TQ Analyst	Creating quantitative and classification methods that can be used with OMNIC Paradigm software. TQ Analyst provides an extensive suite of chemometrics features you can use to identify raw materials, perform quantitative analysis and take spectral measurements. For more information, refer to the help system in TQ Analyst software.
Security Suite	Security Suite provides software for security administration, audit management, control access, and event logging.

Safety and operating precautions

Follow these precautions to avoid injury to yourself or others and avoid damaging the TGA-IR Module during setup or operation.

- The transfer line is configured at the factory to accommodate a TGA furnace that is located on the right side of the spectrometer. If your TGA furnace is on the left side of the spectrometer, gently bend the transfer line so it exits the flow cell to the left. Do not twist the transfer line or exceed its minimum bend radius of 7.6 cm (3.0 in).
- Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in). Support the transfer line while you transport the TGA-IR Module to avoid damaging or breaking it.
- Do not touch the exposed flow cell windows inside the beam ports on both sides of the TGA-IR Module. Fingerprints will permanently damage the windows and degrade system performance.
- Make sure the TGA-IR Module voltage select switches are set correctly before you plug in the power cord. If the switches are set incorrectly for the AC line voltage of your power source, the module may heat more slowly than expected or overheat and cause a failure.
- Before you disconnect the transfer line or its electrical connectors, turn off the TGA-IR Module and unplug the power cord. The transfer line and its electrical connectors serve as the electrical ground for the flow cell. Do not operate the TGA-IR Module when the transfer line is disconnected.
- After you install the TGA-IR Module, insert the beam port shutters in the “closed” position before you start the heaters. Leave the shutters in the closed position for the recommended time (4 hours) before you continue, or you may permanently damage the sample compartment windows or other moisture-sensitive components in the spectrometer.
- The temperature controllers are configured at the factory and should be adjusted only by increasing or decreasing the temperature setting. Do not attempt to reprogram the controllers.
- To prevent the flow cell windows from becoming contaminated, do not turn on the flow cell or transfer line heaters unless the carrier gas from the TGA furnace is flowing through the flow cell.
- If your TGA furnace has a separate heater for the evolved gas outlet, we recommend that you turn on that heater before you start the analysis.
- Operating temperatures below 200 °C (392 °F) can cause the transfer line to become clogged.

- Do not use harsh detergents, solvents, propellant gas dusters (“canned air”), chemicals, or abrasives to clean the TGA-IR Module housing; these can damage the finish and show up as contaminates during the FTIR analysis.
- Do not use excessive amounts of liquid to clean the module housing or allow the liquid cleaner to get inside the module.

WARNING

Avoid health hazard. Do not inhale the TGA-IR exhaust. Keep the work area properly ventilated and connect the exhaust tube to a source of slight vacuum if used and then to a fume hood.

WARNING

Avoid fire and explosion hazards.

Do not connect a flammable carrier gas to the TGA-IR system.

Keep the workspace clear of flammable materials when the TGA-IR Module heaters and TGA furnace are powered on and for up to 2 hours after they are powered off.

Never block the vents on the TGA-IR Module housing or the module may overheat.

CAUTION

Avoid burn hazards.

Do not touch the flow cell windows, the transfer line fittings, or the exhaust tube with your body 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.

If the temperature indicator is red and an “over temperature failure” message appears on a temperature controller display, turn off the TGA-IR Module power switch and do not touch the flow cell windows, the transfer line fittings, or the exhaust tube. Contact us for assistance.

For more information, refer to the *TGA-IR Module Site and Safety Supplement*, the site and safety guide that came with your FTIR spectrometer, and the documentation that came with your TGA furnace.

Chapter 3

TGA-IR sampling

Thermogravimetric analysis (TGA) measures a sample's weight change in a controlled atmosphere as a function of temperature or time. The change in weight occurs as the sample decomposes or evaporates and is usually accompanied by evolved gases.

Combining the TGA system with an FTIR spectrometer provides a simple and accurate technique for identifying the evolved gases from a TGA experiment. This provides information on sample characteristics such as thermal stability or decomposition pathways.

The sample is loaded onto the balance of the thermogravimetric analyzer. A furnace heats the sample at a programmed rate from room temperature to high temperatures. The analyzer plots a weight loss curve of the data file as a function of temperature or time.

A carrier gas continuously sweeps evolved gases from the furnace tube chamber. The carrier gas may be inert, such as nitrogen, or oxidative, such as air. As the compounds in the sample vaporize, the carrier gas sends them through a heated transfer line to a heated flow cell in the TGA-IR Module. There the gases are scanned by the infrared beam and the spectral data recorded. The gases exit the flow cell through the exhaust tube, which should be vented to a fume hood via tubing or an optional aspirator or peristaltic pump, if used.

The TGA-IR Module is a single removable assembly that mounts in the sample compartment of the spectrometer or AEM. The flow cell and transfer line can be removed for cleaning or replacement. See "Maintenance" for more information.

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Chapter 4

Setting up the TGA-IR system

This section explains how to set up a TGA-IR system, which includes the following components:

- Thermo Scientific Nicolet iS50 (or other compatible spectrometer) or the AEM (Auxiliary Experiment Module)
- Thermo Scientific TGA-IR Module
- Thermogravimetric analyzer (TGA furnace)

Follow the steps in these sections to set up the TGA-IR system:

1. "Preparing the TGA furnace"
2. "Preparing the FTIR spectrometer"
3. "Installing the TGA-IR Module"
4. "Setting the flow cell and transfer line temperature"
5. "Checking the detector signal"

After you have set up the system, see "Performing a TGA-IR measurement" for information on using the TGA-IR Module to collect sample data.

Preparing the TGA furnace

Follow the instructions in the documentation to set up the furnace. For best results, place the furnace on a strong, flat, vibration-free table. It is advisable to use a source of clean dry air or nitrogen as the carrier gas, and to set the carrier gas pressure and flow rate as follows:

Table 4-1:
Carrier gas recommended pressure and flow rate

Pressure	Flow rate
5 psi or less	30 mL/min to 100 mL/min[1]

WARNING

Avoid fire and explosion hazard. Do not connect a flammable carrier gas to the TGA-IR system.

NOTICE

The TGA-IR Module is configured to connect to a TGA furnace that is positioned on the right side of the spectrometer. If your TGA furnace is on the left side, gently bend the transfer line so it exits the flow cell to the left. Do not twist the transfer line or exceed its minimum bend radius of 7.6 cm (3.0 in).

Preparing the FTIR spectrometer

This section describes how to configure a Thermo Scientific FTIR spectrometer or AEM for use with the TGA-IR Module.

Note Purge is recommended for the spectrometer connected to a TGA-IR Module. Purging the spectrometer ensures spectral features in TGA-IR data relate to the gas phase sample and not to changes in the room environment.

Note The Nicolet iS10 and iZ10 spectrometer require a sample compartment extension to accommodate the TGA-IR Module. For more information, call your local Thermo Scientific representative.

[1]Higher flow rates will greatly reduce TGA-IR sensitivity and TGA balance stability. Check the documentation that came with your TGA furnace for recommended flow rates.

❖ To configure the FTIR spectrometer or AEM

1. Remove any sample or accessory from the sample compartment of the spectrometer or AEM where you will be installing the TGA-IR Module.
2. Remove the sample compartment cover if necessary.

   Refer to the documentation that came with your spectrometer or AEM for instructions.
3. Remove the standard sample compartment transmission baseplate if it is installed.
4. If your spectrometer is sealed and desiccated, we recommend that you install a purge kit.

   To order a purge kit, use the information at the beginning of this document to contact us. After the purge kit is installed and connected to a source of dry air or nitrogen at the proper pressure and flow rate, you can remove the two sample compartment windows from the spectrometer (not necessary for the iS10 and iZ10 spectrometers).

   NOTICE

   - If you are converting a sealed and desiccated spectrometer to a purged spectrometer, install the purge kit before you remove the sample compartment windows. For information on how to install purge equipment, set the controls for the first time, and inspect and clean the purge filter, find “Installing Purge Controls” in Spectrometer Help Topics, available from the Help menu in OMNIC Paradigm software. For instructions to remove sample compartment windows, search for that topic in the spectrometer Help in OMNIC Paradigm software.
   - If you choose not to purge the spectrometer, make sure you install the beam port shutters in the “closed” position while the TGA-IR Module heats up for the first time and wait the recommended time (4 hours) at the recommended temperature (300 °C) before you begin analyzing samples. Closing the beam ports prevents any excessively humid air released from the TGA-IR Module from damaging the sample compartment windows or other moisture-sensitive components in the spectrometer. Before you begin analyzing samples, switch the beam port shutters to the “open” position to allow the IR beam to pass through the module.
5. Turn on the spectrometer and verify that it is operating properly before you install the TGA-IR Module.

   Refer to any topics on verifying you spectrometer's performance in the spectrometer user guide.

Installing the TGA-IR Module

This section explains how to install the TGA-IR Module in a compatible Thermo Scientific spectrometer or AEM and attach the module to your TGA furnace. Before you begin, make sure you have "Preparing the TGA furnace" and the "Preparing the FTIR spectrometer". The TGA-IR Module power switch should be off and the power cord unplugged at both ends.

Tools needed:

- 7/16 inch open ended wrench

❖ **To install the TGA-IR Module**

1. Remove the protective caps from the beam ports on both sides of the TGA-IR Module.

   Figure 4-1: Remove both beam port caps

2. Install the TGA-IR Module in the sample compartment of the spectrometer or AEM.

   Lift the TGA-IR Module using the designated hand holds and gently place it in the sample compartment. The transfer line should be loosely coiled and supported as shown below.

Figure 4-2: Lift points to properly carry TGA-IR Module

A	**Beam port cap (2)**
B	**Lift point (2)**

NOTICE

Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in). Support the transfer line while you transport the TGA-IR Module to avoid damaging or breaking it.

Do not touch the exposed flow cell windows inside the beam ports on both sides of the TGA-IR Module. Fingerprints will permanently damage the windows and degrade system performance.

Line up the alignment pins on the sample compartment baseplate with the alignment holes in the bottom of the TGA-IR Module. The base of the TGA-IR Module should sit flat against the bottom of the sample compartment.

Figure 4-3: TGA-IR Module properly installed in spectrometer sample compartment

3. Engage the anti-rock clip at the back of the sample compartment.

   The anti-rock clip for the Nicolet iS50 spectrometer is shown below (your spectrometer or AEM may have a different style clip).

Figure 4-4: Nicolet iS50 spectrometer anti-rock clip fully engaged

4. Install both beam port shutters in the closed position on each side of the TGA-IR Module.

   Install the beam port shutters with the white “closed” label on top while the TGA-IR Module heats up for the first time. Leave them installed until the module has been heated at 300 °C for at least 4 hours to ensure that no moisture remains inside the module.

Figure 4-5: Install beam port shutters in “closed” position (white label at top)

NOTICE

Make sure you install the beam port shutters in the “closed” position while the TGA-IR Module heats up for the first time (and after being stored). Wait the recommended time (4 hours) at the recommended temperature (300 °C) before you continue or you may permanently damage the sample compartment windows or other moisture-sensitive components in the spectrometer.

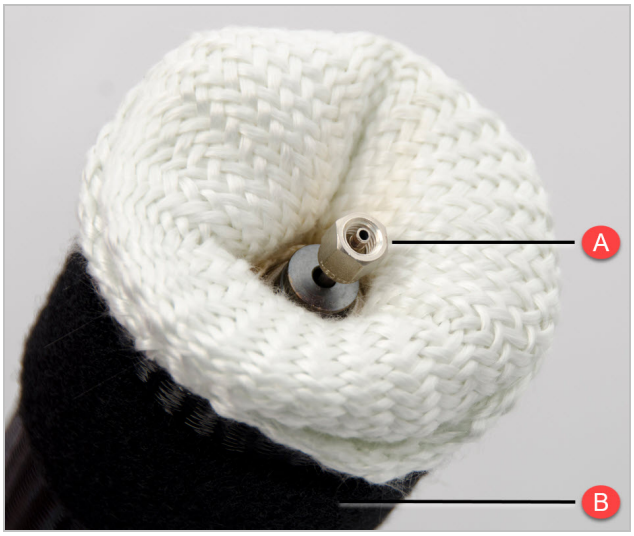
5. Connect the opposite end of the transfer line to the TGA furnace.

NOTICE

- Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in).
- The outside surface of the transfer line is abrasive; if it consistently rubs against another surface such as the outside of your TGA furnace, it may cause scratches.
- Make sure the transfer line is supported while you are connecting it to the TGA furnace and afterwards. Do not allow the transfer line to hang unsupported between the TGA-IR Module and the TGA furnace. The extra weight my interfere with the transfer line's lifetime performance.
- The transfer line is configured at the factory to accommodate a TGA furnace that is located on the right side of the spectrometer. If your TGA furnace is on the left side, gently bend the transfer line so it exits the flow cell to the left. Do not twist the transfer line or exceed its minimum bend radius of 7.6 cm (3.0 in).

Remove the Velcro™ strap from the end of the transfer line and gently spread apart the insulation to expose the Swagelok nut.

Figure 4-6: Free end of transfer line ready to connect to TGA furnace



A	**Swagelok nut**
B	**Velcro strap**

Lower the TGA furnace using the application software for the furnace. This will make the plumbing connection more accessible.

Slide the transfer line onto the Swagelok fitting attached to the evolved gas outlet connector on the TGA furnace and tighten the nut finger-tight. See the documentation that came with the furnace for the location of its evolved gas outlet.

Use two 7/16-inch open ended wrenches to slowly tighten the Swagelok nut until you feel resistance, then continue tightening the nut 1/8 of a turn more. To ensure the best seal, do not overtighten the Swagelok nut.

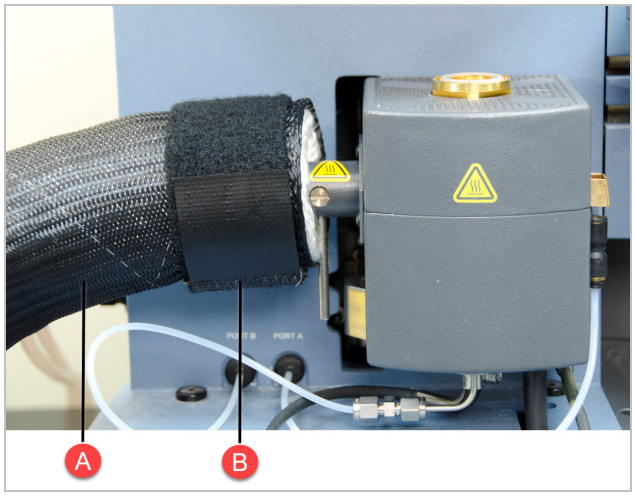
Note If your TGA furnace does not have a 1/8-inch fitting, contact the TGA furnace manufacturer to order a 1/8-inch adapter.

Note If the TGA furnace is a horizontal model with about 7 inches of vertical travel, you may need to gently bend the transfer line into a soft “S curve” to compensate for the furnace’s open/close mechanism.

Use both hands to stretch the protective tubing over the exposed insulation so the tubing extends almost to the end of the transfer line.

Position and secure the strap at the end of the transfer line to hold the tubing and the insulation securely around the connector. Make sure the buckle is not at the top of the transfer line or it may interfere with the oven when the furnace is up. Be careful not to compress the insulation too much by overtightening the strap.

Figure 4-7: Example of TGA-IR attachment at TGA furnace



A	**Stretch the tubing to cover the insulation.**
B	**Position and tighten the strap. Do not over-tighten the strap!**

Make sure the table supports the transfer line to reduce wear at the connections. Here are examples of the TGA-IR Module installed in right and left side spectrometer configurations with the transfer line properly connected and supported.

Figure 4-8: Transfer line properly connected and supported, right side configuration

Figure 4-9: Transfer line properly connected and supported, left side configuration

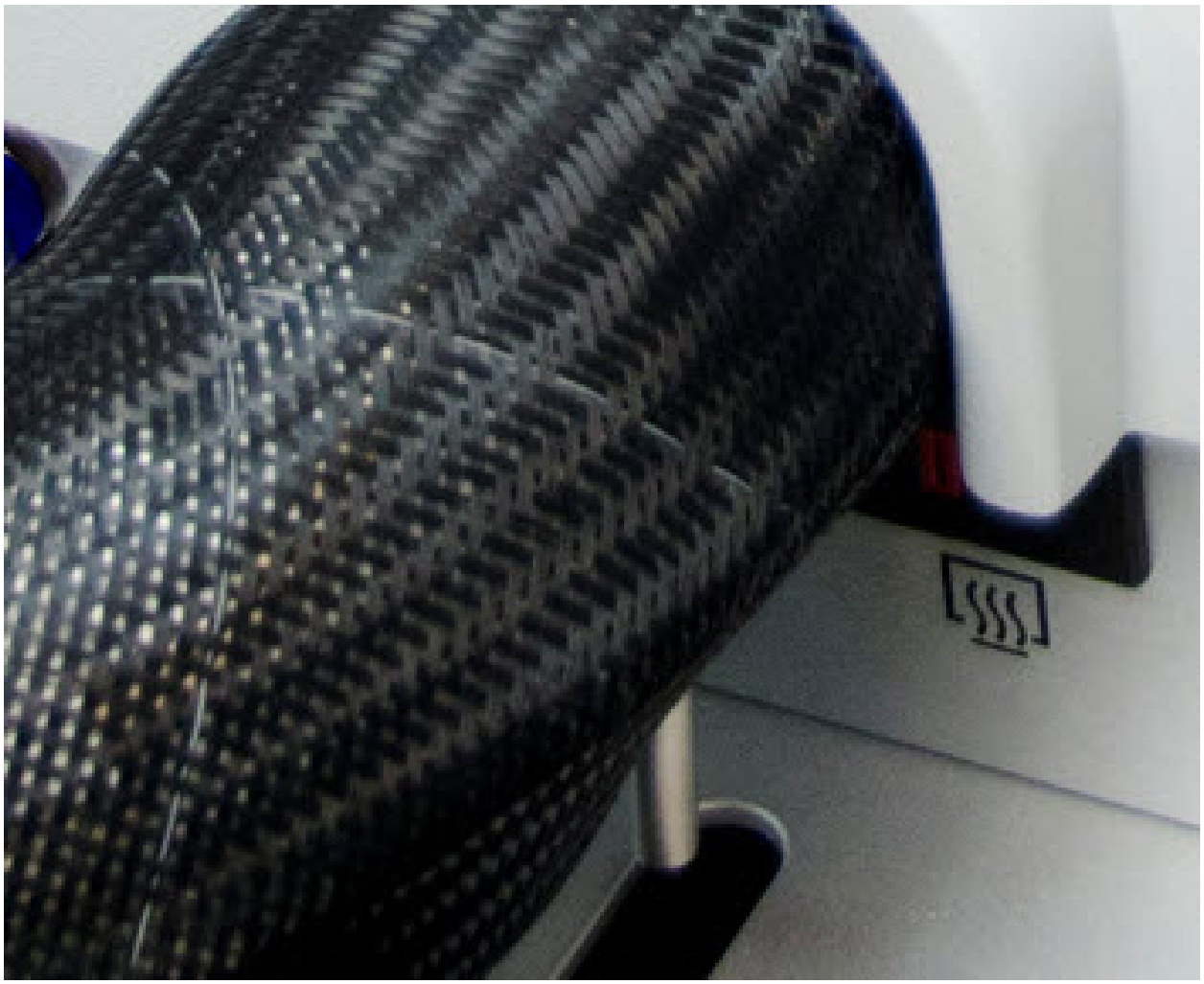
6. Route the exhaust tube outlet to a pump system (or similar source of suction), if used, and then to a fume hood.

   You can order an optional tubing kit for this purpose.

   To set up the pump, refer to the instructions that came with it. Set the pump speed between 60 mL/min and 80 mL/min (flow rate should be 0.4 to 85 mL/min).

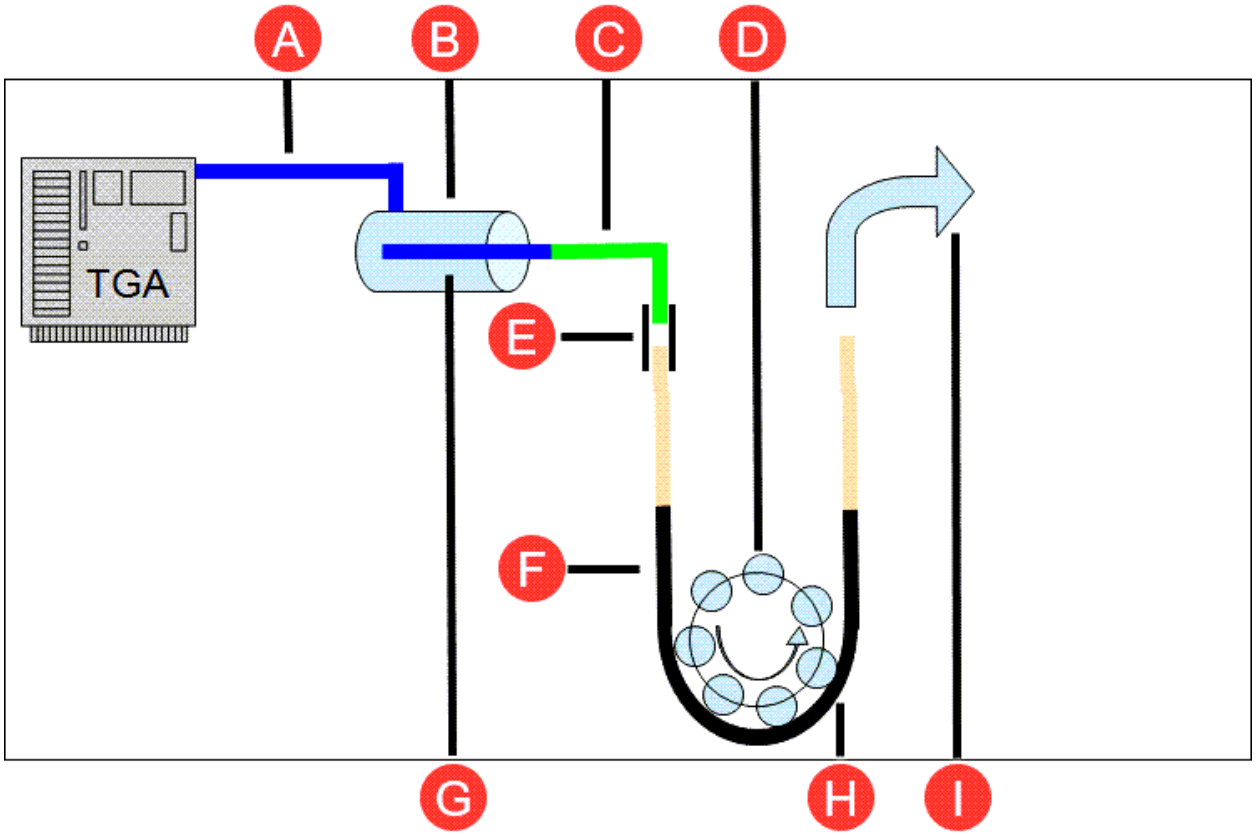
   Note We recommend connecting an aspirator or peristaltic pump to the exhaust tube to help draw carrier gas through the TGA-IR system, especially when using a high resolution TGA furnace (which typically have a low flow rate).

Figure 4-10: TGA-IR Module exhaust tube



If you plan to use a pump, use clear, flexible Tygon™ or other non-reactive tubing to connect the exhaust tube on the TGA-IR Module to the pump inlet. Make sure you connect the pump so it draws the sample/carrier gas mixture out of the TGA-IR Module. Use another piece of tubing to direct the pump outlet to a fume hood or other venting system. The image below shows the proper connections.

Figure 4-11: Connecting the exhaust tube to a peristaltic pump

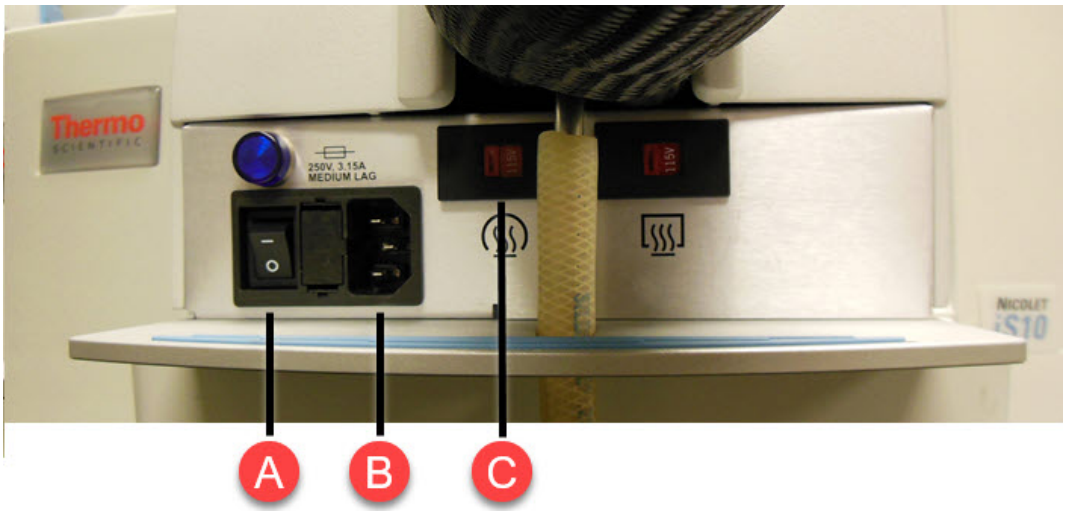


A	Transfer line
B	Cell
C	Tubing
D	Purge tubing
E	Soft rubing to make connection
F	Soft tubing through rollers
G	Exhaust tube
H	Peristaltic pump
I	To exhaust (hood)

7. If using a remote trigger for the TGA-IR Module, connect the cable.
8. Verify that the voltage select switches on the TGA-IR Module are set correctly for the local electricity requirements and then connect the power cord.

   Slide both voltage select switches to 115 V or 230 V to match the AC line voltage of your power source.

   Figure 4-12: Voltage select switches



A	**Power switch**
B	**Power receptacle**
C	**Voltage select switch (2)**

NOTICE

Make sure the TGA-IR Module voltage select switches are set correctly before you plug in the power cord. If the switches are set incorrectly for the AC line voltage of your power source, the module may heat more slowly than expected or overheat and cause a failure.

Ensure the power switch for the TGA-IR Module is in the off position, then plug one end of the AC power cord into the power receptacle on the front of the module. Plug the other end of the power cord into a properly grounded AC power source.

Setting the flow cell and transfer line temperature

Two embedded temperature controllers heat the TGA-IR Module transfer line and flow cell. The flow cell and transfer line temperature should be stable before you start collecting data.

Table 4-2:
Flow cell and transfer line operating temperature specifications

Component	Operating Range[1]
Flow cell[2]	200 °C to 300 °C (392 °F to 572 °F)
Transfer line[3]	200 °C to 300 °C (392 °F to 572 °F)

The TGA-IR Module must be plugged in and powered up before you set the flow cell and transfer line temperature. The carrier gas from the TGA furnace should flow through the flow cell at the recommended rate (TGA furnace door must be closed). Install the beam port shutters in the “closed” position.

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[1]Infrared throughput, and therefore sensitivity, decreases with increased temperature; do not set controllers unnecessarily high.

[2]Residue on flow cell windows can affect system performance. To prevent residue from accumulating, set flow cell temperature 10 degrees higher than condensation temperature of sample material.

[3]Operating at temperatures less than 200 °C (392 °F) can cause transfer line to become clogged. High boiling materials require a higher operating temperature.

NOTICE

- Install the beam port shutters in the “closed” position while the TGA-IR Module heats up for the first time (and after it has been stored). Wait the recommended time (4 hours) at the recommended temperature (300 °C) before you attempt to collect data. Closing the beam ports allows any excessively humid air released from the TGA-IR Module to escape vertically and prevents the sample compartment windows and other moisture-sensitive components in the spectrometer from becoming damaged. Before you begin analyzing samples, switch the beam port shutters to the “open” position to allow the IR beam to pass through the beam ports to and from the TGA-IR Module.
- To prevent the flow cell windows from becoming contaminated, do not turn on the flow cell or transfer line heaters unless the carrier gas from the TGA furnace is flowing through the flow cell.

❖ **To set the flow cell and transfer line temperature**

NOTICE

The temperature controllers are configured at the factory and should be adjusted only by increasing or decreasing the temperature setting. Do not attempt to reprogram the controllers.

- To increase a temperature set point, hold down the ∗ button and press s.
- To decrease a temperature set point, hold down the ∗ button and press t.

A	**Transfer line controller**

B	**Flow cell controller**
C	**Temperature indicators**

After the heaters have reached their set points, they will remain at that temperature until you turn off the TGA-IR Module power switch. The time required to reach the target temperature depends on the starting temperature and the target temperature (see "Specifications" for details).

The tables below describe the temperature controller status and failure indicators.

Table 4-3:
Figure 4-13: Temperature controller LED status indicators

LED status	Meaning
Off	Powered off or not heating
On steady or blinking rapidly	Actively heating
Blinking slowly	At target temperature

Table 4-4:
Figure 4-14: Temperature controller error conditions and alarms

Error	Meaning
inPt and FaiL	Description: Input or sensor fault Action: Call Technical Support
burn	Description: Heater fault Action: Call Technical Support
dAtA and FaiL	Description: Non-volatile memory fault (failed temperature controller) Action: Cycle the power to the TGA-IR Module. If error persists, call Technical Support

Error	Meaning
 **(alternating with the process temperature; LED in lower right stays lit)**	Description: Over temperature error Action: Reset alarm and recycle power to TGA-IR Module[1]
Heats very rapidly or very slowly	Description: Voltage selector switches may be set incorrectly. For example, if voltage switches are set to 115 V and input voltage is 230 V, the heaters will heat too rapidly and eventually blow the fuses. If voltage switches are set to 230 V and input voltage is 115 V, the heaters will heat very slowly. Action: Turn off the TGA-IR Module power switch and make sure voltage select switches are set to match the input voltage of the local electrical supply.

CAUTION

Avoid burn hazard. If the “over temperature” error message appears on a temperature controller display, turn off the TGA-IR Module power switch and do not touch the flow cell windows, the transfer line fittings, or the exhaust tube on the TGA-IR Module. Contact us for assistance.

Checking the detector signal

Once you have installed and connected the TGA accessory, view the detector signal in OMNIC Paradigm software to ensure that everything is working properly before you start to measure any samples. The software shows a live display of the detector signal.

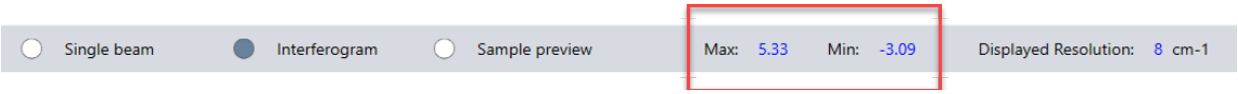
[1]If an error condition occurs, press the s and t keys once simultaneously to reset the temperature controller and clear the error, then turn off the TGA-IR Module power switch, wait 1 minute, and turn the power switch back on. If that doesn’t correct the problem, call our local service representative.

❖ To check the detector signal

1. Open OMNIC Paradigm software. The software automatically loads the default, commonly used settings as *Smart TGA - Factory Preset,* but you can create your own settings based on your analysis needs.

2. Select More to see the live interferogram. The voltage of at least one of the interferogram peaks (positive or negative) should be greater than 5 volts for KBr windows and 4 volts for ZnSe windows. If both are less than the specifified minimum, contact technical support for assistance.

   Figure 4-15: Live interferogram voltage



Chapter 5

Performing a TGA-IR measurement

This section covers how to collect backgrounds and measuring data using your Module.

CAUTION

Avoid fire hazard. To prevent the TGA-IR Module from overheating, never block the vents on the module housing.

Preparing the TGA-IR system for sampling

Follow these steps to prepare the TGA-IR system for sampling after it has been turned off. The TGA-IR system must have been properly installed and the installation verified. See "Setting up the TGA-IR system" for details.

WARNING

Avoid fire and explosion hazard.

Do not connect a flammable carrier gas to the TGA-IR system.

Keep the workspace clear of flammable materials when the TGA-IR Module heaters and TGA furnace are powered on, and for up to 2 hours after you turn them off.

❖ To prepare the TGA-IR system for sampling

1. Turn on the power switch for the TGA furnace and start the carrier gas flow. Make sure the sample door is closed.

   Make sure the carrier gas for the TGA furnace is set to 5.0 psi or less and the flow rate is between 30 mL/min and 100 mL/min. Refer to the operating manual that came with your TGA furnace for more information.

   NOTICE

   To prevent the flow cell windows in the TGA-IR Module from becoming contaminated, make sure carrier gas from the TGA furnace is flowing through the flow cell before you turn on the flow cell and transfer line heaters.

2. Turn on the peristaltic pump (if one is used), then set the pump speed to 60 to 80 mL/min. The flow rate should be 0.4 to 85 mL/min.

3. Turn on the power switch for the spectrometer.

4. Install the beam port shutters with the “closed” label on top on both sides of the TGA-IR Module.

   NOTICE

   Closing the beam ports prevents any excessively humid air released from the TGA-IR Module while it heats up from damaging the sample compartment windows and other moisture-sensitive components in the spectrometer.

5. Turn on the power switch on the front of the TGA-IR Module.

6. Set the flow cell and transfer line heaters to 300 °C.

7. Wait at least 4 hours for the heaters to stabilize and burn off any moisture in the system.

8. Install the beam port shutters in the “open” position before you begin analyzing samples.

   Remove both shutters and flip them over so the blue “open” label is on top, then reinstall them to allow the IR beam to pass through the TGA-IR Module.

   Figure 5-1: Install beam port shutters in open position (blue label at top)

A **Beam port shutters in the "open" position (blue label at top)**

Samples compatible with TGA-IR

Any sample that can be measured by TGA will work with the TGA-IR application. TGA and TGA-IR are useful for two types of heat activated analyses: deformation (where intact molecules of the material vaporize) and breakdown (where materials such as carbon dioxide, water, and ammonia form under high heat). Typical sample types include rubbers, plastics, and resins.

Analyzing a test sample

This section takes you through the steps of a typical TGA-IR measurement using a CaOx (calcium oxalate monohydrate) test sample and a DTGS detector in the FTIR spectrometer.

Before you begin, follow the instructions on "Preparing the TGA-IR system for sampling" and wait at least 4 hours for the transfer line and flow cell temperatures to stabilize and burn off any moisture in the system.

❖ To analyze a test sample

1. Take a high-temperature platinum or ceramic TGA pan.
2. Close the TGA furnace to permit the carrier gas to flow from the furnace to the TGA-IR Module.

   This step is necessary for correct background collection.
3. Specify the ramp rate and target temperature for the TGA furnace.

   For our CaOx test sample, we used a 20 °C per minute ramp from ambient to 900 °C, with a 5 minute hold at 900 °C. This should result in a run time of about 48 minutes ([900-30]/20+5=48.5 minutes).

   Note If your TGA furnace has a separate heater for the evolved gas outlet, make sure that heater is turned on before you continue.
4. Adjust the carrier gas flow rate for your sample material.

   For our CaOx test sample, we used a carrier gas flow rate of 35 mL/min.

   Note The slower the carrier gas flow rate, the higher the sensitivity you can expect. The trade-off at low flow rates is loss of temporal relationship between the weight loss curve and the IR data. The optimum carrier gas flow rate depends on the particular sample and the heating rate of the furnace. A flow rate of 50 mL/min to 60 mL/min is a good starting point for heating rates of 15 C°/min to 30 C°/min. For slow heating rates, for example 5 C°/min or less, use 40 mL/min to 50 mL/min.
5. Use the temperature controllers on the TGA-IR Module to manually set the flow cell and transfer line to the proper operating temperature for your sample material (must be between 200 °C and 300 °C).

   To prevent residue from accumulating in the transfer line and flow cell, set their temperatures 10 degrees higher than the condensation temperature of your sample material. For CaOx, we left the transfer line and flow cell temperatures at 300 °C.

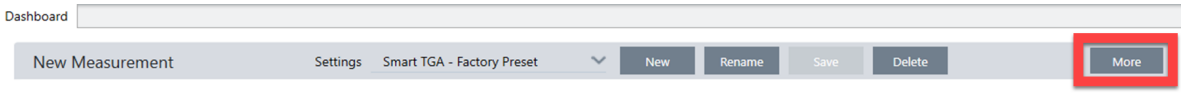
   NOTICE

   Operating temperatures below 200 °C (392 °F) can cause the transfer line to become clogged. High boiling materials will require a higher operating temperature.
6. Open OMNIC Paradigm software and set up a TGA-IR measurement

a. The software automatically loads the default settings as Smart TGA - Factory Preset, but you can create your own settings based on your analysis needs.

b. In the Dashboard, select **More** to view additional settings.

Figure 5-2: TGA - More settings



Note Make sure the **Analysis type** is set to *Time Series*. This should already be the case with the preset settings.

Note Make sure that **Sampling accessory** shows as *Smart TGA*. If it does not, the intrument is not installed or recognized correctly.

c. Enter a **Measurement name** (for example, "TGA measurement"). If you do not, the default title will be the current time and date.

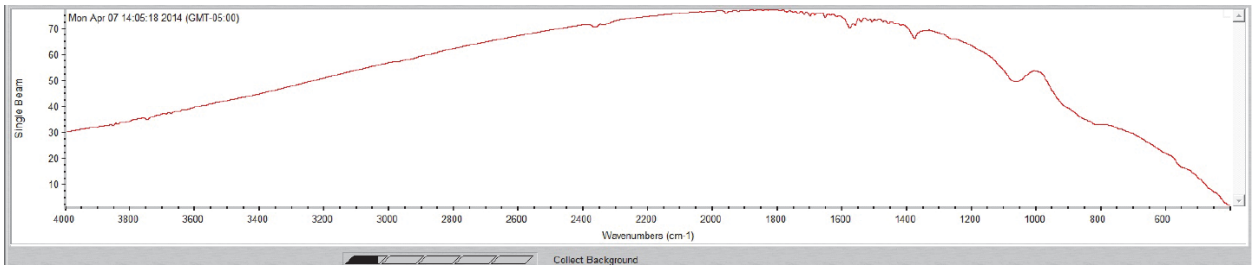
Note For an introduction to data collection and measurement, see the Measure samples section of the OMNIC Paradigm User Guide online at https://knowledge1.thermofisher.

7. Select **Measure Time Series** and wait for the process to complete. This will create a new Spectra tab and open a background preview.

8. Select **Start Background Measurement** and wait for the process to complete.

9. Place a measured quantity of the sample onto the pan in the TGA furnace and start the TGA run. For our test, we are using 50 mg.

Figure 5-3: Collecting a background spectrum



NOTICE

For most samples (CaOx is an exception), a weight loss of about 10 mg usually gives an adequate infrared signal. For example, is a sample loses 50% of its weight during the TGA run, a 20 mg sample size will probably be accurate. Do not use excessively large sample sizes. Large weight losses, in terms of mass, can be accompanied by aerosols that cause IR baseline shifts.

10. Click **Start Time Series Measurement** to measure the sample data and wait for the process to complete.

    Note If you are using an external trigger, TGA-IR data collection will start automatically when you start the TGA run. For more information, see "Configuring series data collection to start automatically".

    The Gram-Schmidt profile will appear in a time response display. The peaks in the Gram-Schmidt profile indicate when different sample compounds vaporized, passed through the flow cell, and absorbed infrared energy.

11. To view the spectrum at a specific point in time, select the Gram-Schmidt profile. The crosshair indicates the point you have currently selected.

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

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

    b. Enter the desired name of the new search and select **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.

Import TGA data

You can import TGA data to your series collection in order to add weight, temperature, and derivative weight to the results.

❖ **To import TGA data:**

1. Export the data from the TGA instrument.
2. While viewing the series data, go to **File > Time Series > Import TGA file**.
3. Select the TGA file and select **Open**.

   The temperature, weight, and derivative weight profiles are now added to the **Profiles** tab.

   For additional information, consult the OMNIC Paradigm User Guide.

Configuring series data collection to start automatically

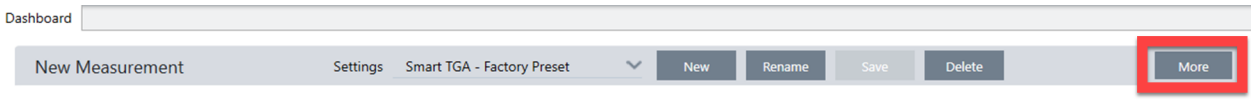
If you have the optional Thermo Scientific remote start cable for your spectrometer, you can set up OMNIC Paradigm software to start automatically at the start of each TGA sample analysis. To order a cable, call your local Thermo Scientific sales or service representative or use the information at the beginning of this document to contact us.

❖ **To configure OMNIC Paradigm software for remote start**

1. Connect the remote start cable.

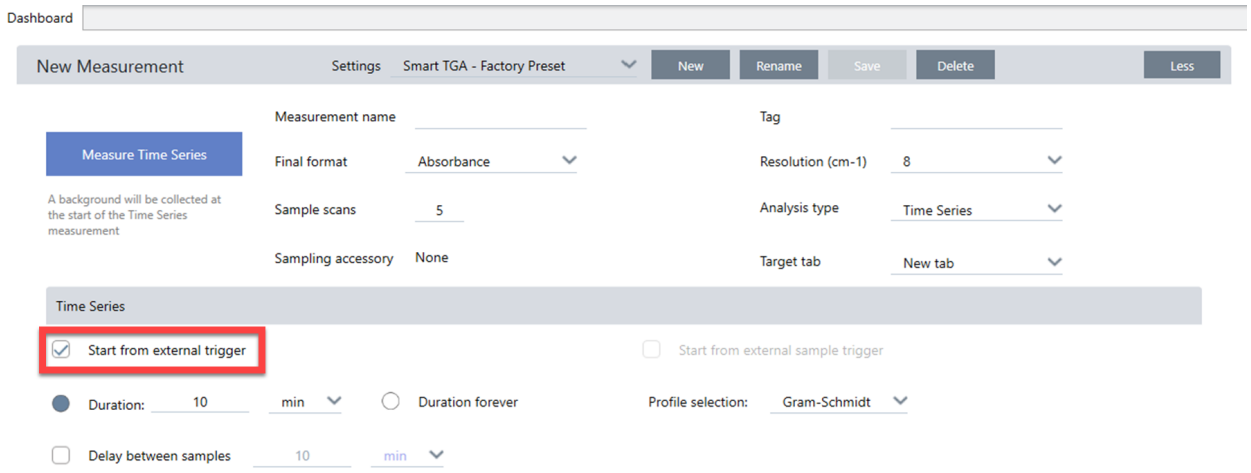
   Plug one end of the remote start cable into the Auxiliary Signals port on the back of the spectrometer. Connect the other end into the data collection trigger signal port for your TGA furnace. For more information, see the documentation that came with your TGA furnace and with the remote start kit.
2. Start OMNIC Paradigm software.
3. In the Dashboard, choose **Time Series** under **Analysis type**.
4. Select **More** in the upper right corner to see additional settings.

   Figure 5-4: TGA - More settings



5. Check **Start with external trigger**.

Figure 5-5: TGA - Start from external trigger checkbox



Note To configure your TGA furnace for remote start, see the user guide that came with the furnace.

Chapter 6

Turning off the TGA-IR system

Follow these steps to turn off the TGA-IR system.

❖ To turn off the TGA-IR system overnight

1. Set the flow cell and transfer line heaters to 125 °C (257 °F).

> **NOTICE**
>
> Leave the beam port shutters in the “open” position and the carrier gas flowing through the TGA-IR Module.

❖ To turn the TGA-IR system completely off

1. Turn off the power switch on the front of the TGA-IR Module.

> **NOTICE**
>
> If the spectrometer is purged with dry air or nitrogen, leave the beam port shutters in the “open” position and the carrier gas flowing through the TGA-IR Module. If the spectrometer is sealed and desiccated (with KBr sample compartment windows), install the beam port shutters in the closed position.

2. Wait 2 hours for the unit to cool.

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Chapter 7

Removing the TGA-IR Module

Tools needed:

- Two 7/16 in open ended wrenches
- Non-powdered latex-free gloves (to handle fiberglass insulation around transfer line)

❖ **To remove the TGA-IR Module**

1. Turn off the power switch on the front of the TGA-IR Module and unplug the power cord.

CAUTION

Do not touch the flow cell windows, the transfer line fittings or the exhaust tube with your body when the heaters are on, and for up to 2 hours after you turn them off. Temperatures can exceed 300 °C (572 °F).

2. Wait 2 hours for the TGA-IR Module and transfer line to cool.
3. Turn off the carrier gas at the TGA furnace.
4. Disconnect the transfer line from the evolved gas outlet on the TGA furnace.

   Loosen the strap and let it slide down the transfer line.

   Gently pull the insulation away from the connector.

   Use the two 7/16-inch open ended wrenches to loosen the nut until the transfer line is released.

   Gently smooth the fiberglass insulation back over the connector (to keep insulation from creasing).

Use both hands to stretch the protective tubing back over the exposed insulation so the tubing extends to the end of the transfer line.

Position and secure the strap at the end of the transfer line to secure the tubing and the insulation around the connector during storage. Be careful not to compress the insulation by overtightening the strap.

5. Remove the beam port shutters from the TGA-IR Module.
6. Release the anti-rock clip at the back of the sample compartment.

   Figure 7-1: Release anti-rock clip

7. Lift the TGA-IR Module straight up using the designated hand holds.

   The transfer line should be loosely coiled and supported as shown below.

Figure 7-2: Lift points to properly carry TGA-IR Module

NOTICE

- Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in). Support the transfer line while you transport the TGA-IR Module to avoid damaging or breaking it.
- Do not touch the exposed flow cell windows inside the beam ports on both sides of the TGA-IR Module. Fingerprints will permanently damage the windows and degrade system performance.

8. Replace the protective caps in the beam ports on the right and left side of the TGA-IR Module and store the module properly in a clean, dry environment. See "Storing the TGA-IR Module".

Figure 7-3: Install beam port caps before storing TGA-IR Module

A	**Beam port cap (2)**
B	**Lift point (2)**

NOTICE

See "Storing the TGA-IR Module" for important information about how to properly store the module to prevent damage to internal components due to excessive moisture, dust and dirt.

9. Install the sample compartment cover on the spectrometer, if applicable.

Chapter 8

Maintenance

This section covers maintenance of your TGA Module.

CAUTION

Avoid burn hazard. Do not touch the flow cell windows, the transfer line fittings, or the exhaust tube with your body 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.

Storing the TGA-IR Module

When the TGA-IR Module is not in use for a short period (a day or two), leave it in the spectrometer or AEM with the power on and the carrier gas flowing through it. Set the flow cell and transfer line heaters between 120 °C and 130 °C (248 °F and 266 °F). This keeps the module and transfer line clean and dry, and minimizes the time needed to restart.

If you need to remove the module from the spectrometer, see "Removing the TGA-IR Module".

Figure 8-1: Install beam port caps before storing TGA-IR Module

If possible, store the module in its original plastic shipping bag and container, or its equivalent. Add a desiccant pack to the plastic bag before you seal it to minimize moisture absorption into the thermal insulation as well as protect KBr windows in the flow cell.

NOTICE

- Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in). Support the transfer line while you transport the TGA-IR Module to avoid damaging or breaking it.
- Do not touch the exposed flow cell windows inside the beam ports on both sides of the TGA-IR Module. Fingerprints will permanently damage the windows and degrade system performance.
- After you remove the TGA-IR Module from storage and install it in the spectrometer, make sure you install the beam port shutters in the “closed” position while the TGA-IR Module heats up for the first time. Wait the recommended time (4 hours) at the recommended temperature (300 °C) before you continue or you may permanently damage the sample compartment windows or other moisture-sensitive components in the spectrometer.

Cleaning the TGA-IR Module housing

If the outside of the TGA-IR Module needs cleaning, turn it off, disconnect the power cord and wait 2 hours for the TGA-IR Module and transfer line to cool. Then use a clean, soft cloth, slightly dampened with mild soap solution, to clean the outside of the module and wipe it dry.

NOTICE

- Do not use harsh detergents, solvents, propellant gas dusters (canned air), chemicals, or abrasives to clean the TGA-IR Module housing; these can damage the finish and show up as contaminates during the FTIR analysis.
- Do not use excessive amounts of liquid to clean the module housing or allow the liquid cleaner to get inside the module.

Flow cell maintenance

If you are analyzing appropriate samples (see "Samples compatible with TGA-IR") at the recommended operating temperature (see "Setting the flow cell and transfer line temperature" section) and following the installation and operating instructions in this guide, the flow cell should not require frequent cleaning or replacement. However, the flow cell may benefit from a maintenance cleaning after 3 to 4 years of regular use. If system performance degrades significantly, the flow cell windows may be contaminated or damaged. Contact your local sales or service representative to order new ones.

Follow these steps to clean the flow cell and to clean or replace the flow cell windows.

Tools needed:

- One large, slotted screwdriver
- #1 Phillips head screwdriver
- #2 Phillips head screwdriver
- Two 7/16-inch open ended wrenches
- 9/16-inch open ended wrench
- 1/2-inch open ended wrench
- 3/16-inch hex key
- 7/64-inch hex key
- Nitrile gloves for handling flow cell windows
- One of the following replacement kits:

- TGA-IR flow cell window replacement kit (if you are replacing the KBr or ZnSe windows in the flow cell). This includes all necessary gaskets and washers.
- TGA-IR flow cell gasket replacement kit (if you plan to clean and reinstall the KBr or ZnSe windows). This includes all necessary gaskets and washers, but no windows.

NOTICE

- Replace KBr or ZnSe windows only with replacement parts supplied by us. Leave the new windows in their sealed and desiccated packaging until you are ready to install them in the flow cell.
- Do not leave KBr windows exposed to room air any longer than necessary; prolonged exposure to humidity will permanently damage the windows.
- Wear nitrile gloves or finger cots to handle replacement windows and hold the windows only by their edges. Fingerprints (even while wearing gloves) will permanently damage the windows and degrade system performance.
- Do not allow liquids to come into contact with flow cell windows.

❖ To remove the flow cell assembly

1. Set the transfer line and flow cell heaters for the TGA-IR Module to 20 °C (68 °F).
2. Turn off the power switch for the TGA-IR Module and unplug the power cord.

CAUTION

Do not touch the flow cell windows, the transfer line fittings or the exhaust tube with your body when the heaters are on and for up to 2 hours after you turn them off. Temperatures can exceed 300 °C (572 °F).

3. Wait 2 hours for the TGA-IR Module and transfer line to cool.
4. Remove the beam port shutters from their slots on both sides of the TGA-IR Module.
5. Remove the main cover from the TGA-IR Module.

   Use the large slotted screwdriver to rotate the two cover screws counter clockwise to release the cover, then lift the cover straight up and off the TGA-IR Module.

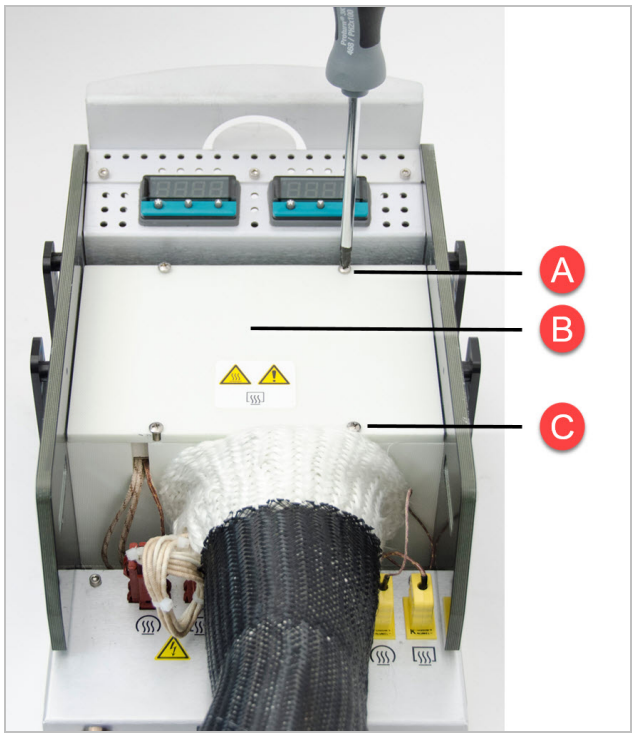
Figure 8-2: Release TGA-IR Module main cover

6. Remove the flow cell compartment cover.

   Use a #2 Phillips head screwdriver to loosen the two back screws that secure the flow cell compartment cover. This will prevent them from falling inside the TGA-IR Module.

   Remove the two front screws that secure the cover and then slide the cover forward and off the TGA-IR Module.

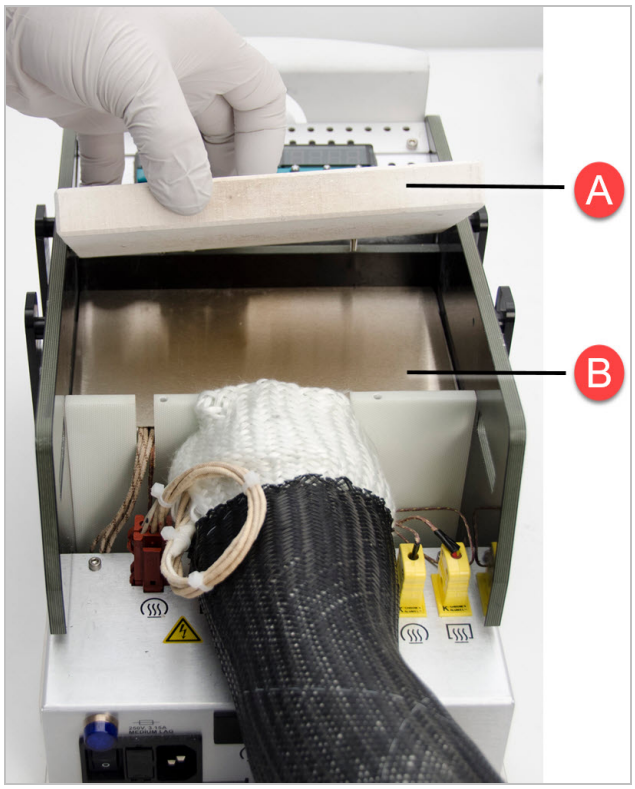
Figure 8-3: Remove flow cell compartment cover



A	**Loosen these two screws so they don't fall inside the TGA-IR Module.**
B	**This is the flow cell compartment cover.**
C	**Remove these two screws and slide the cover forward to remove it from the TGA-IR Module.**

7. Remove the insulating block and metal heat shield from the flow cell compartment and set them aside.

Figure 8-4: Remove insulating block and metal heat shield



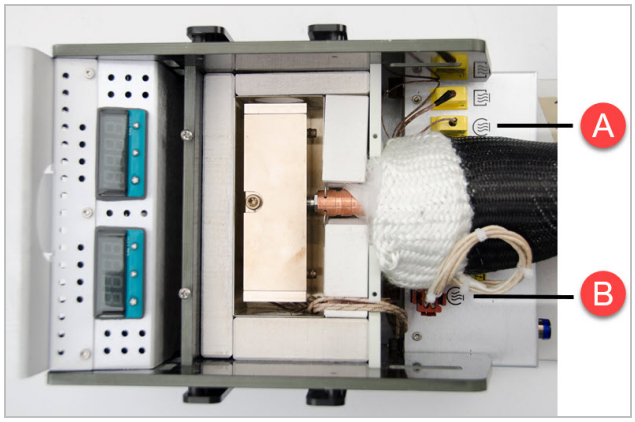
A	**Remove the insulating block.**
B	**Remove the metal heat shield underneath the insulating block.**

NOTICE

Save the insulating block and heat shield. You will need to replace them after you reinstall the flow cell.

8. Unplug the AC power and temperature controller connectors for the transfer line.

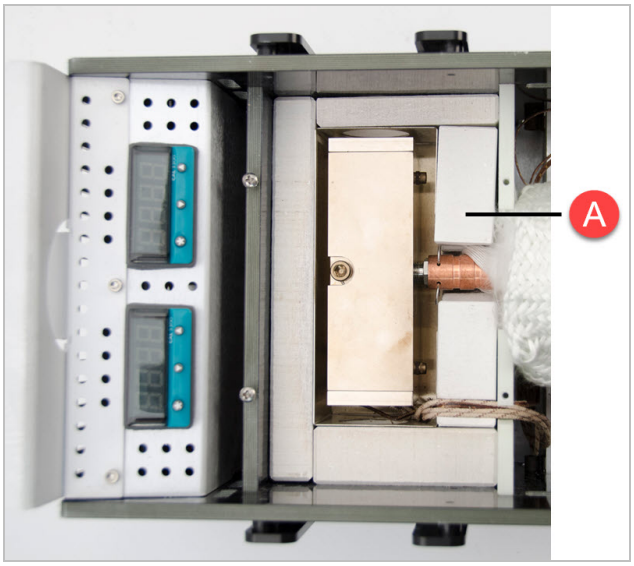
Figure 8-5: Unplug AC power and temperature controller connectors for transfer line



A	**Disconnect temperature controller connector for the transfer line.**
B	**Disconnect AC power connector for transfer line.**

9. Remove the two insulating blocks to expose the flow cell connectors.

Figure 8-6: Remove insulating blocks to expose flow cell connectors



A	**Remove insulating blocks (2)**

NOTICE

Save the insulating blocks. You will need to replace them after you reinstall the flow cell.

10. Disconnect the transfer line from the flow cell inlet.

NOTICE

Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in).

Remove the two cotter spring pins from the copper sleeve.

Slide the sleeve back to expose the Swagelok connector and nut. Use a flat-blade screwdriver or similar tool, if necessary, to push the copper sleeve out of the way.

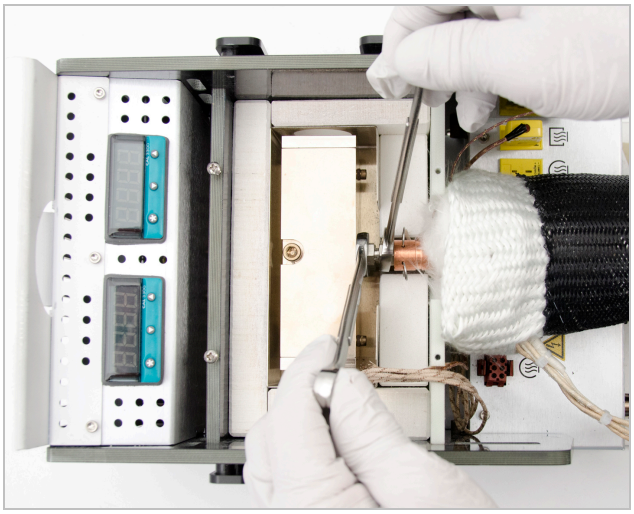
Insert the two cotter pins into their original holes in the copper sleeve to hold the sleeve out of the way, behind the nut.

Figure 8-7: Remove cotter pins and slide copper sleeve toward transfer line to expose connecting nut

A	**Slide the copper sleeve towards the transfer line.**
B	**Remove cotter pins (2).**

Use two 7/16-inch open ended wrenches to loosen the Swagelok nut until the transfer line is released.

Figure 8-8: Loosen Swagelok nut to release transfer line



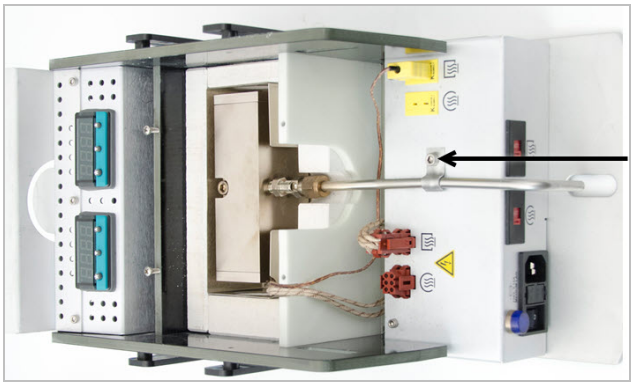
Carefully set the transfer line aside.

Note If the flow cell windows are contaminated, the transfer line may also be clogged. Follow these instructions to clean it.

11. Use a #2 Phillips head screwdriver to loosen the clip that secures the exhaust tube to the TGA-IR Module.

    Slide the clip off the exhaust tube.

    Figure 8-9: Loosen the clip securing the exhaust tube to the TGA-IR Module



12. Use a 3/16-inch hex key to remove the center screw from the flow cell assembly.

Figure 8-10: Remove the center screw from the flow cell assembly

13. Lift the flow cell assembly (with the exhaust tube connected) out of its housing and set it on a table.

Figure 8-11: Remove flow cell from TGA-IR Module (with exhaust tube connected)

A	**Remove the flow cell.**
B	**Leave the exhaust tube connected.**

14. Disconnect the exhaust tube from the flow cell assembly.

Use a 9/16-inch and 1/2-inch open ended wrench to loosen the Swagelok nut until the exhaust tube is released. Set the exhaust tube aside.

Figure 8-12: Disconnect the exhaust tube from the flow cell assembly

❖ **To disassemble the flow cell for cleaning**

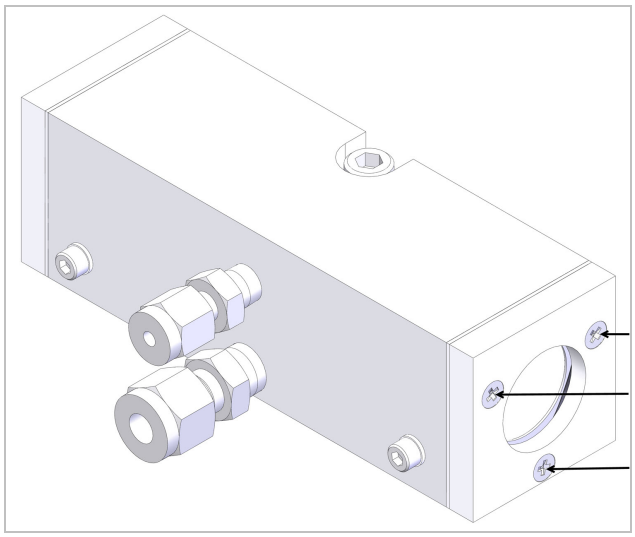
NOTICE

- Wear nitrile gloves to handle KBr or ZnSe windows and hold them only by the edges. Fingerprints, even while wearing gloves, will permanently damage the windows and degrade system performance.
- Do not allow liquids to come into contact with the flow cell windows.

1. Remove the KBr or ZnSe window from each end of the flow cell.

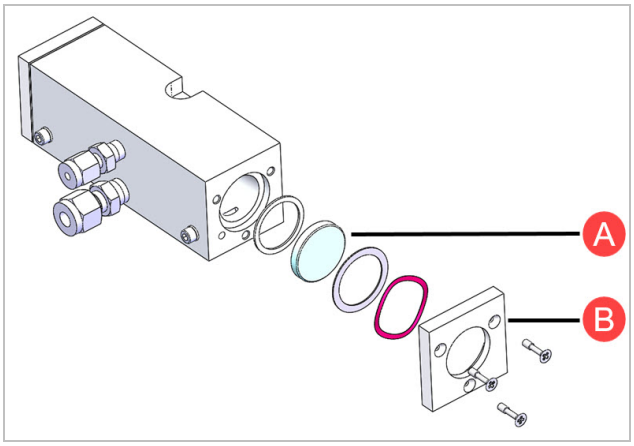
   Use a #1 Phillips head screwdriver to remove the three screws that secure the end cap on one end of the cell body.

Figure 8-13: Remove end cap screws on flow cell assembly



Gently remove the end cap, KBr or ZnSe window, and all gaskets and washers. Discard the gaskets and washers (keep the end cap and the window if you plan to clean it).

Figure 8-14: Remove end cap, gaskets, washers, and KBr or ZnSe window



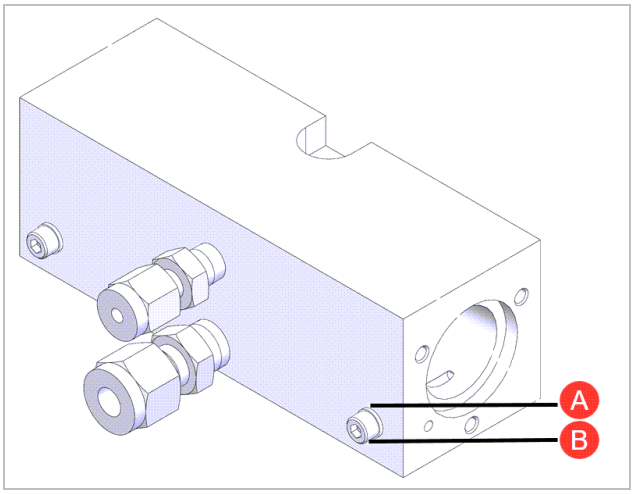
A	**KBr o ZnSe window**
B	**End cap**

Repeat the steps above to remove the end cap and window from the other end of the flow cell.

2. Remove the manifold plugs from the flow cell.

Use a 7/64-inch hex key to remove the manifold plug and gasket at both ends of the flow cell.

Figure 8-15: Remove manifold plugs and gaskets on both ends



A	**Gasket (2)**
B	**Manifold plug (2)**

Remove the gaskets from the plugs. Set the plugs aside. Discard the gaskets (both kits include a new set of them).

❖ To clean the metal flow cell components

Clean the flow cell body, end caps (2), manifold plugs (2, with gaskets removed), and exhaust tube in an ultrasonic bath. Cleaning time will vary depending on the bath type, solution, temperature, and amount of soil.

❖ To clean the KBr or ZnSe flow cell windows

NOTICE

- Wear nitrile gloves to handle the flow cell windows and hold them only by the edges. Fingerprints, even while wearing gloves,will permanently damage the windows and degrade system performance.
- Do not allow liquids to come into contact with the windows.

To remove dust from a flow cell window, blow it off with a gentle stream of pure nitrogen gas. Do not use compressed air (from a can or compressor). Contaminants will damage the windows.

You can remove most deposits from a KBr window by carefully polishing it with a laboratory wipe and ethanol or another suitable dry solvent, or by using a polishing kit for salt windows (available from a laboratory equipment supply company). ZnSe windows are easily scratched and cannot be polished to remove residue.

Note If the flow cell windows are extremely dirty or damaged, contact us to order new ones. Replacement windows include replacement gaskets, washers, and screws.

❖ To assemble the flow cell

1. Install the manifold plugs and gaskets in the flow cell body.

   Install two new gaskets from the replacement kit on the two manifold plugs and insert them into the flow cell body. Use a 7/16-inch hex key to tighten the plugs.

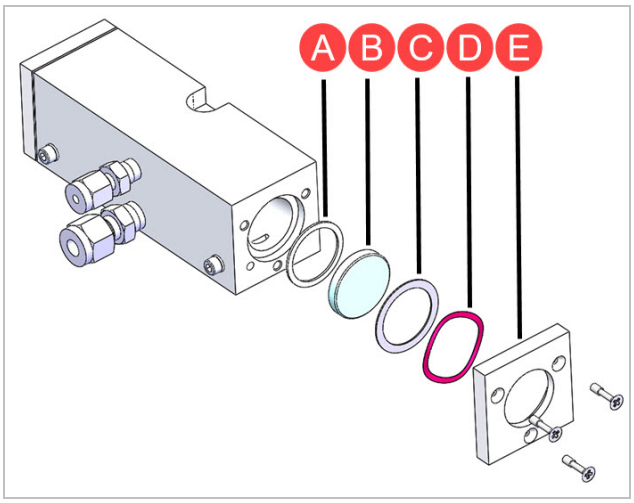
2. Install the flow cell windows and their associated gaskets and washers in the flow cell body.

   NOTICE

   - Wear nitrile gloves to handle flow cell windows and hold them only by the edges. Fingerprints (even while wearing gloves) will permanently damage the windows and degrade the system performance.
   - Be careful not to scratch the windows with the washers, screws, or screwdriver blade.
   - Do not push on the window, gasket, or end cap in any way that bends or distorts the window; the window can crack or break. Damage or breakage during installation is not covered by the warranty.

   On one end of the flow cell body, carefully insert the following parts from the replacement kit in the order shown below and then start the three screws that secure the end cap. Before you tighten the screws, verify that the window opening is not blocked by a portion of a gasket.

Figure 8-16: Install windows, gasket, washers, end caps, and screws on both ends of flow cell



A	Small round gasket (place in flow cell window cavity)
B	KBr or ZnSe window (for ZnSe, make sure coated side faces out of the flow cell to avoid contact with sample material)
C	Large round washer (align with gasket and window)
D	Wave washer (align with washer, gasket, and window; should fit into end cap recess)
E	End cap (align with everything else)

To tighten the screws, hold the cell body with your thumb and finger to keep everything aligned, then use a #1 Phillips head screwdriver to partially tighten each screw, rotating from one to the next until all three screws are secure.

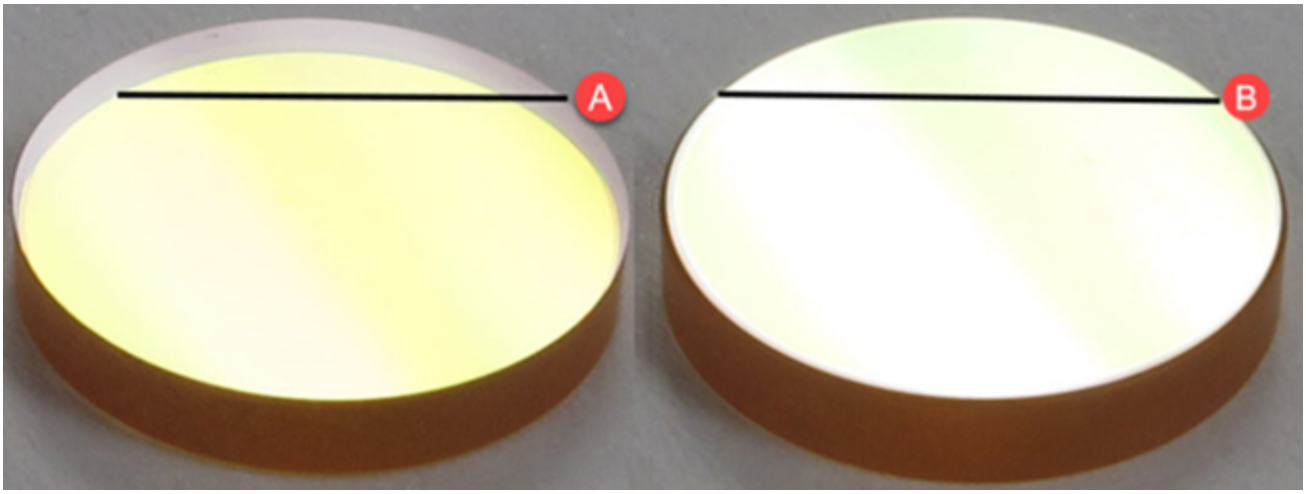
❖ To determine the coated side of a ZnSe window

If you are installing ZnSe windows in the flow cell, make sure the coated side faces out of the flow cell to avoid contact with sample material. To determine the coated side, inspect both sides of the window at an angle to the ceiling light.

When the uncoated side faces up, the bottom edge of the window is visible through the window material.

When the coated side faces up, the light is fully reflected off the coating and you cannot see the bottom edge.

Figure 8-17: Determining the coated side of a ZnSe window



A	**Uncoated side facing up; bottom edge is visible.**
B	**Coated side facing up; bottom edge is invisible (coated surface is opaque).**

❖ **To install the flow cell assembly**

1. Connect the exhaust tube to the flow cell assembly.

   Slide the exhaust tube onto the bottom (flow cell outlet) connector on the flow cell assembly, then tighten the Swagelok nut with a 9/16-inch and a 1/2-inch open ended wrench.

Figure 8-18: Connect exhaust tube to bottom (flow cell outlet) connector

2. Install the flow cell assembly in the flow cell compartment.

   Line up the pin holes on the bottom of the flow cell assembly with the alignment pins on the bottom of the flow cell compartment.

   Figure 8-19: Line up pin holes on flow cell with alignment pins in flow cell compartment

Position the flow cell assembly with the large screw hole facing up; the connectors should face the front of the TGA-IR Module.

Figure 8-20: Install flow cell assembly in flow cell compartment

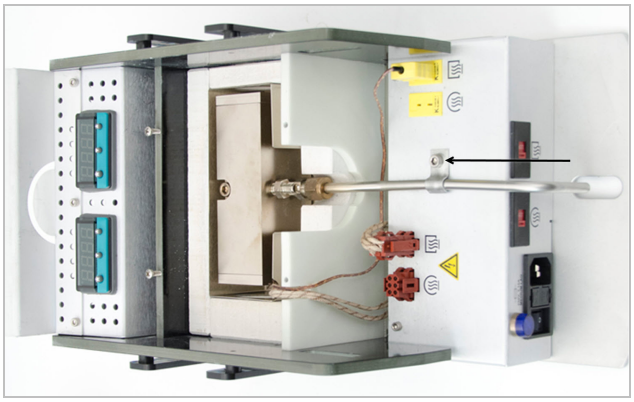
The flow cell assembly should sit flat against the heater plate at the bottom of the compartment.

Insert the large center screw through the flow cell assembly and tighten it with a 3/16-inch hex key.

Figure 8-21: Insert and tighten center screw to secure flow cell assembly

3. Slide the clip over the exhaust tube and tighten it with a #2 Phillips head screwdriver.

   Figure 8-22: Tighten clip that secures exhaust tube to TGA-IR Module



4. Connect the transfer line to the flow cell assembly

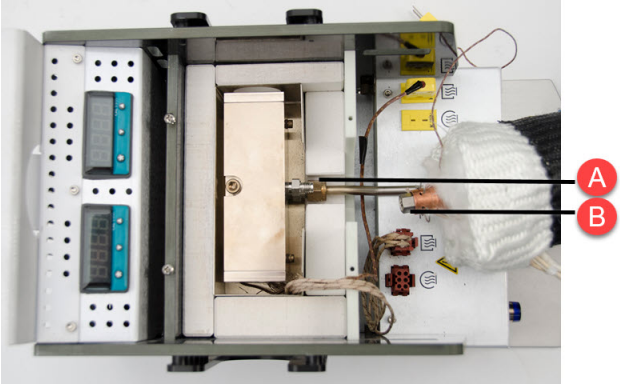
   Orient the transfer line so it will extend in the direction of the TGA furnace when the flow cell is installed; there should not be excessive bending at either end of the transfer line.

   NOTICE

   Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in).

   Slide the transfer line onto the top flow cell connector and tighten the Swagelok nut finger tight.Slide the transfer line onto the top flow cell connector and tighten the Swagelok nut finger-tight.

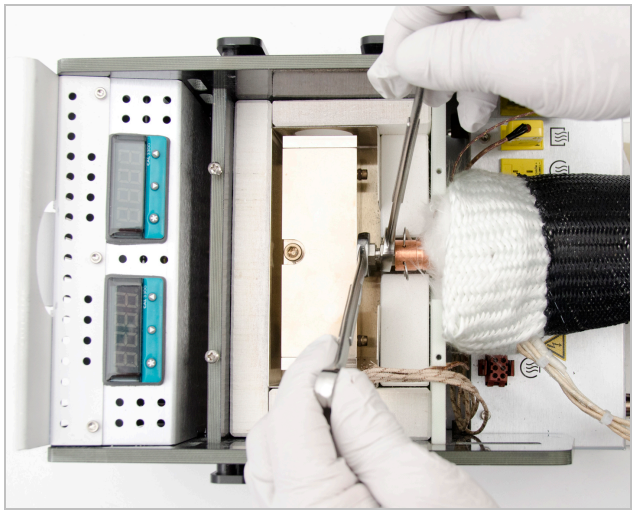
   Figure 8-23: Connect transfer line to top (flow cell inlet) connector and tighten finger-tight



A	Flow cell inlet connector
B	Transfer line connector

Use two 7/16-inch open ended wrenches to slowly tighten the Swagelok nut until you feel resistance, then continue tightening the nut 1/8 of a turn more. To ensure the best seal, do not overtighten the Swagelok nut.

Figure 8-24: Use two 7/16-inch open ended wrenches to tighten the nut

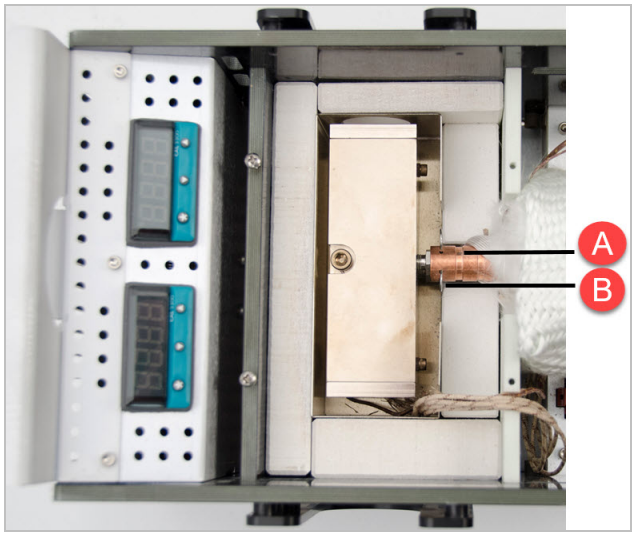


Remove the two cotter pins from the copper sleeve.

Slide the copper sleeve toward the flow cell so it covers the Swagelok nut.

Insert the two cotter pins to secure the sleeve. If you position the sleeve correctly, the pins will easily slide into place.

Figure 8-25: Slide copper sleeve toward TGA-IR Module and insert the cotter pins

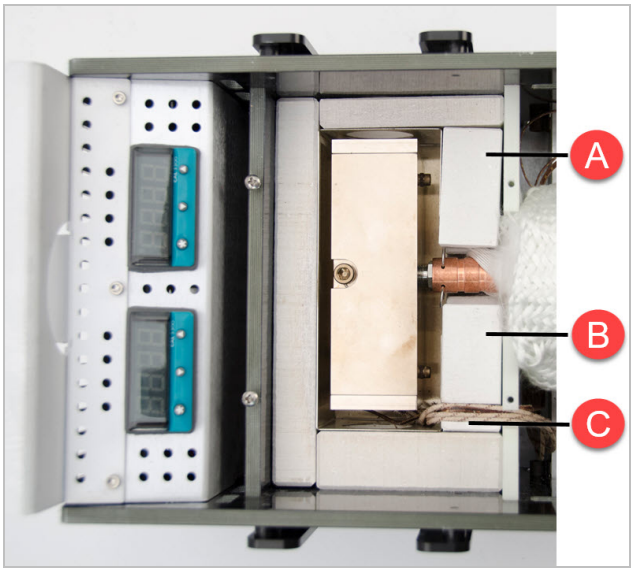


A	**Slide copper sleeve toward flow cell.**
B	**Insert cotter pin.**

5. Install the two insulating blocks.

   Insert the block with the groove at the left.

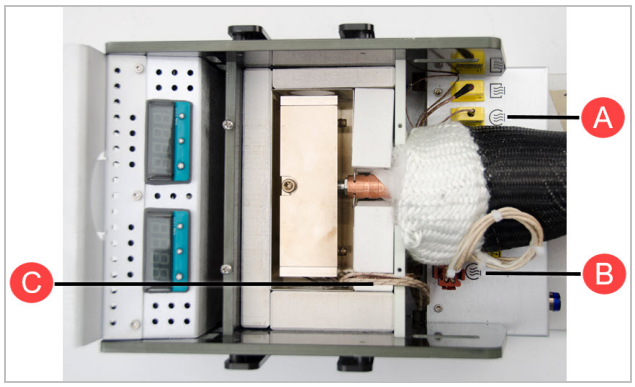
   Figure 8-26: Install insulating blocks (grooved block goes on left side)



A	**Insert plain insulating block here.**
B	**Insert grooved insulating block here (groove faces up).**
C	**Groove.**

6. Plug the AC power and temperature controller connectors for the transfer line into their corresponding receptacles.

Figure 8-27: Attach transfer line power and temperature controller connectors



A	**Plug in the temperature controller connector for the transfer line.**
B	**Plug in the AC power connector for the transfer line.**
C	**Press the wires into the slot.**

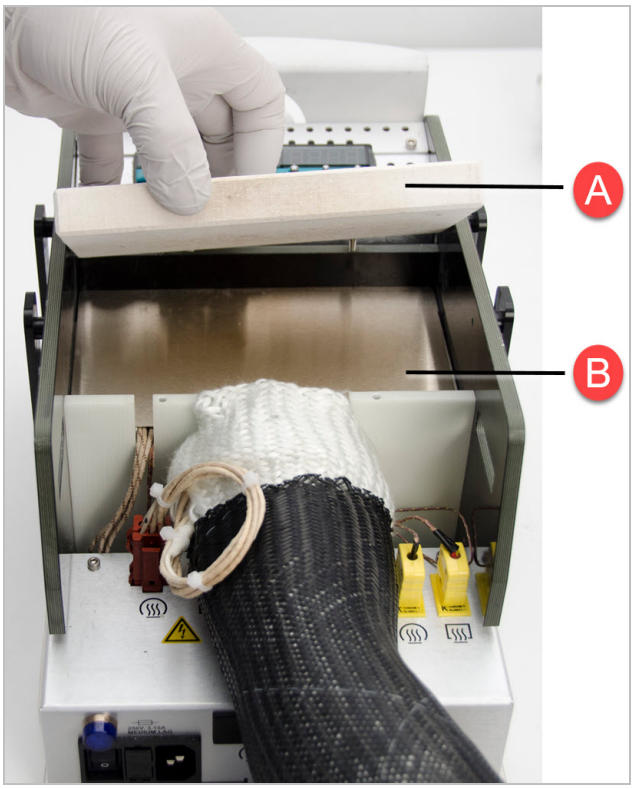
Gather the wires and press them into the slot at the edge of the flow cell compartment.

7. Install the heat shield and insulating block.

   Place the metal heat shield over the flow cell (make sure the wires are neatly gathered into the slot so the heat shield lies flat).

   Place the large insulating block over the heat shield.

Figure 8-28: Install metal heat shield and large insulating block

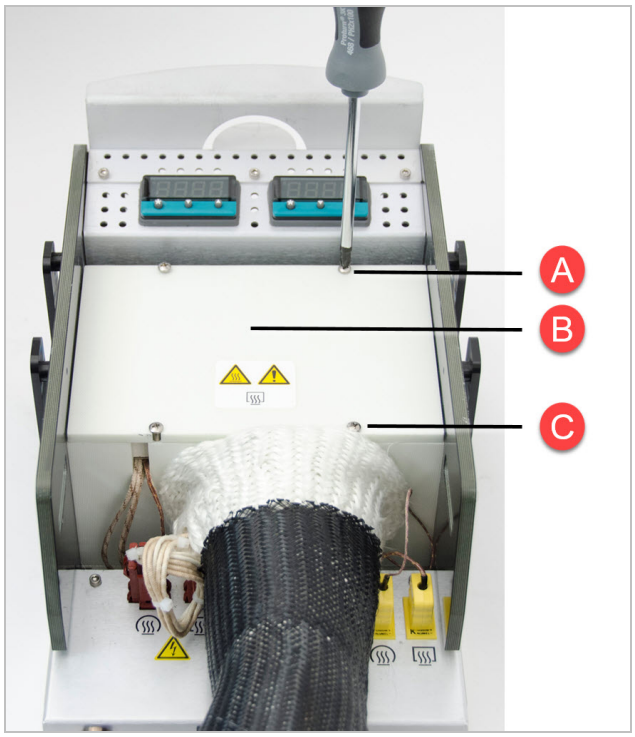


A	**Install the insulating block on top of the heat shield.**
B	**Install the metal heat shield first.**

8. Replace the flow cell compartment cover and insert or tighten the screws.

   Slide the cover back onto the TGA-IR Module and use a #2 Phillips head screwdriver to tighten the 4 screws that secure the cover.

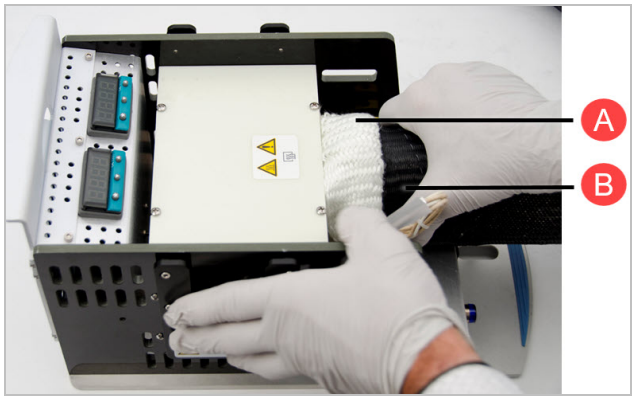
Figure 8-29: Install flow cell compartment cover and insert or tighten screws



A	Tighten back screws (2).
B	Slide cover over flow cell compartment.
C	Replace and tighten front screws (2).

9. Use your hand to stretch the protective tubing over the exposed insulation so the tubing and the insulation press against the flow cell compartment with no air gaps.

Figure 8-30: Smooth out transfer line and stretch tubing to cover exposed insulation



A	**Press insulation against the flow cell compartment with no gaps.**
B	**Stretch protective tubing to cover the exposed insulation.**

Note The transfer line should press against the flow cell compartment with no air gaps so that the specified operating temperature is maintained as the sample material enters the flow cell.

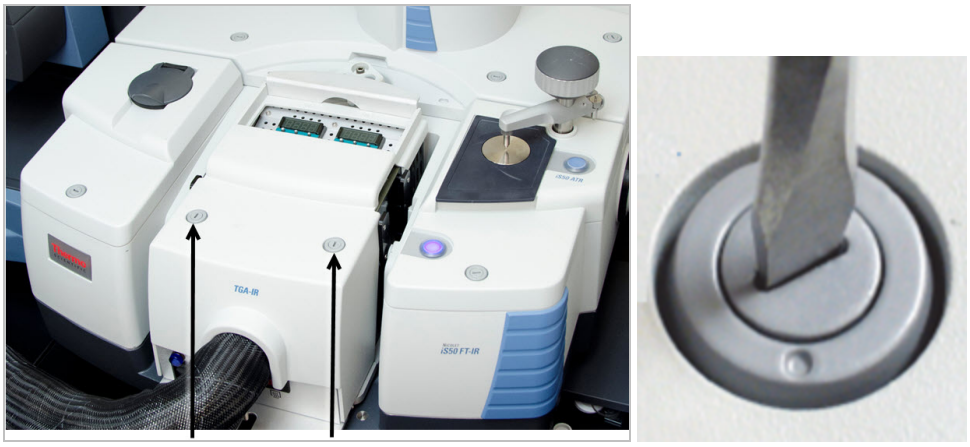
10. Replace the TGA-IR Module main cover.

    Press the electrical wires toward the center of the module, away from the edges (to avoid catching them in the cover latch).

    Figure 8-31: Press electrical wires away from edges of TGA-IR Module

Install the cover and then use a large flat-blade screwdriver to rotate the two screws clockwise to the locked position.

Figure 8-32: Install and secure TGA-IR Module main cover



11. Install the beam port shutters in the closed position.
12. Make sure the TGA-IR Module power switch is off.
13. Plug in the power cord for the TGA-IR Module and turn on the power switch.

NOTICE

To prevent the flow cell windows from becoming contaminated, make sure that carrier gas is flowing through the flow cell before you turn on the flow cell and transfer line heaters. See "Performing a TGA-IR measurement" for more information.

14. Check the signal, verify performance, and follow the instructions to prepare the TGA-IR system for sampling. See "Checking the detector signal" and "Preparing the TGA-IR system for sampling" for more information.

Transfer line maintenance

If you are analyzing appropriate samples (see "Samples compatible with TGA-IR") at the recommended operating temperatures and following the installation and operating instructions in this guide, the transfer line should not require frequent cleaning or replacement. The transfer line may benefit from a maintenance cleaning after 3 to 4 years of regular use. Use the following procedure to clean the transfer line.

CAUTION

Avoid electrical shock. Before you disconnect the transfer line or its electrical connectors, turn off the TGA-IR Module and unplug the power cord. The transfer line and its electrical connectors serve as the electrical ground for the flow cell. Do not operate the TGA-IR Module when the transfer line is disconnected.

If your TGA-IR system clogs up frequently, contact your local sales or service representative.

NOTICE

Handle the transfer line gently and do not exceed its minimum bend radius of 7.6 cm (3.0 in).

❖ To disconnect the transfer line

1. Set the heaters for the TGA-IR Module to 20 °C (68 °F).
2. Turn off the power switch for the TGA-IR Module and unplug the power cord

CAUTION

Do not touch the flow cell windows, the transfer line fittings, or the exhaust tube with your body when the heaters are on and for up to 2 hours after you turn them off. Temperatures can exceed 300 °C (572 °F).

3. Wait 2 hours for the TGA-IR Module and transfer line to cool.
4. Follow steps 4 through 7 of the instructions to remove the flow cell to disconnect the transfer line.
5. Follow these steps to disconnect the other end of the transfer line from the TGA furnace.

❖ To clean the transfer line

Tools needed:

- 0.042 in to 0.06 in diameter stainless-steel spring wire

Gently straighten out any kinks so the transfer line is fairly straight. Then use compressed dry air or nitrogen to blow any loose particles out of the transfer line.

If the transfer line requires more thorough cleaning, gently feed a 0.042-inch to 0.060-inch diameter stainless steel spring wire through the inside of the transfer line. Then use compressed dry air or nitrogen again to blow any loose particles out of the transfer line.

Note If your transfer line is extremely dirty, damaged, or if you can't insert the wire, contact us to order a new one.

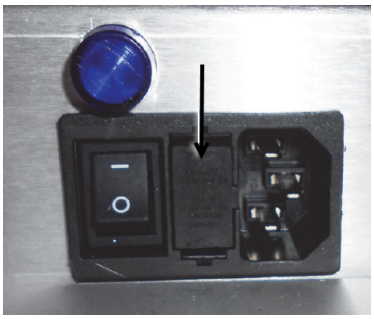
❖ **To connect the transfer line to the TGA-IR Module**

1. Follow steps 4 through 8 of the instructions to install the flow cell to connect the transfer line to the TGA-IR Module.
2. Follow these steps to connect the other end of the transfer line to the TGA furnace.
3. Follow the instructions to "Preparing the TGA-IR system for sampling"

Replacing fuses

Two fuses protect the TGA-IR Module from excessive current. Use replacement fuses of the correct size and specifications (see below). The fuse box is at the front of the TGA-IR Module, next to the power switch.

Figure 8-33: Location of fuse box



If a fuse blows, the TGA-IR Module will automatically power off. Try turning the power back on. If the fuse is bad, the TGA-IR Module will immediately power off again. Replace the fuse.

Tools needed:

- Two small straight blade screwdrivers
- Two 3.15 A medium time-delay, low-breaking capacity, GMC series glass tube fuses, size 5x20 mm

❖ To replace the fuses

1. Turn off the power switch for the TGA-IR Module and unplug the power cord.
2. Use two small straight blade screwdrivers to gently press in the tabs on both ends of the fuse box and then slide the box out of the TGA-IR Module.

   Figure 8-34: Remove fuse box

3. Replace the two fuses with two new ones of the correct size and with the same specifications.

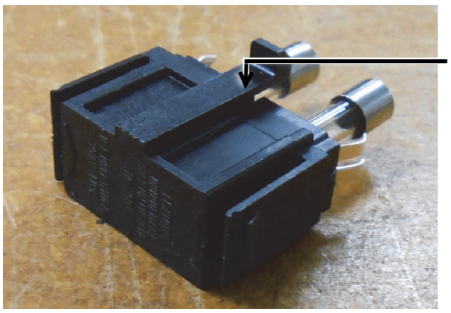
   Figure 8-35: Replace both fuses

4. Install the fuse box.

   Insert the fuse box with the prong facing the right side of the TGA-IR Module and slide the box into the module.

Figure 8-36: Install fuse box



5. Plug in the power cord for the TGA-IR Module and turn on the power switch.

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Chapter 9

Troubleshooting

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

For more troubleshooting tips, find “Troubleshooting” in Spectrometer Help Topics, available from the Help menu in OMNIC Paradigm software.

Power indicator does not light

If the power indicator on the front of the TGA-IR Module does not light after you turn on the power switch, check the following:

- Make sure the power switch is on and the voltage select switches are set correctly for the local electricity supply.

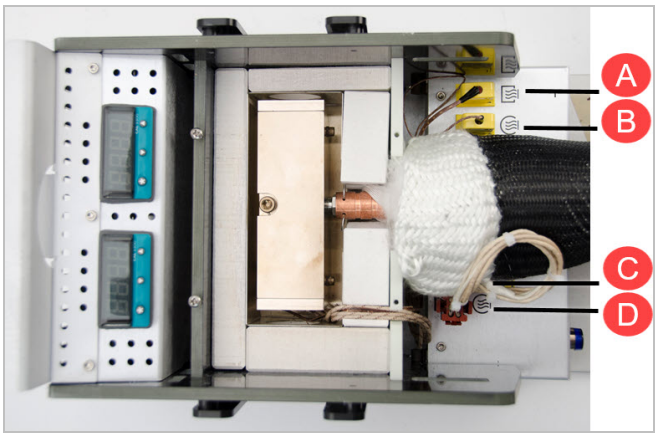
- Inspect and replace the fuses if necessary.

If the problem persists, use the information at the beginning of this document to contact Technical Support.

No heat or heats slowly

If the transfer line or flow cell does not heat up or reach the set point after you set the temperature controllers, turn off the power switch and unplug the power cord. Then check to make sure that the transfer line and flow cell electrical connectors are plugged into the correct receptacles (not reversed) and ensure that both temperature controllers are set properly.

Figure 9-1: Electrical connectors for flow cell and transfer line



A	**Temperature controller connector for flow cell.**
B	**Temperature controller connector for transfer line.**
C	**Power connector for flow cell.**
D	**Power connector for transfer line.**

If the problem persists, the heater element may need replacement. Use the information at the beginning of this document to contact Technical Support.

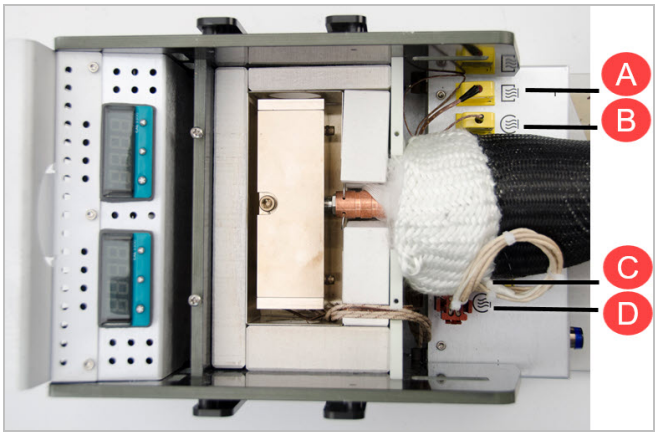
Transfer line and flow cell heat too rapidly (or very slowly)

If the transfer line and flow cell heat up very rapidly or very slowly, turn off the TGA-IR Module power switch and disconnect the AC power cord. Then check that the voltage select switches are set correctly for the local electricity supply. For example, if the voltage switches are set to 115 V and the input voltage is 230 V, the heaters will heat too rapidly and eventually blow the fuses. If the voltage switches are set to 230 V and the input voltage is 115 V, the heaters will heat very slowly. If the problem persists, contact Technical Support.

Input/fail error

If a temperature controller displays a high temperature value and then displays an “input/fail” message, check that the transfer line and flow cell electrical connectors are plugged into the correct receptacles. The connectors are located under the TGA-IR Module main cover. There are two connectors for the transfer line and two for the flow cell.

Figure 9-2: Electrical connectors for flow cell and transfer line



A	**Temperature controller connector for flow cell.**

B	**Temperature controller connector for transfer line.**
C	**Power connector for flow cell.**
D	**Power connector for transfer line.**

Temperature overshoot or undershoot

If the flow cell or transfer line temperature overshoots (or undershoots) the specified target temperature, check that the flow cell and transfer line electrical connectors are plugged into the correct receptacles (and not reversed). See "Input/fail error" for more information.

No spectroscopic IR signal

If no IR interferogram signal appears on the Bench tab in Experiment Setup, check the following:

- Optical path is blocked. Check that beam port shutters are in installed with the “Open” label on top.
- MCT detector not cooled. Fill the detector dewar with liquid nitrogen. See Spectrometer Help Topics in the OMNIC Paradigm software Help menu for more information.
- Wrong detector selected in Experiment Setup > Bench tab.
- Detector oversaturated or ADC overflow. Use a lower Aperture setting to reduce the size of the aperture or reduce the Gain setting on the Bench tab in Experiment Setup.
- Select the Diagnostic tab in Experiment Setup and click Align to align the spectrometer.

If the problem persists, use the information at the beginning of this document to contact Technical Support.

No detectable sample gas

If the Gram-Schmidt profile has no sample peaks, check the following:

- Too little sample. Increase the sample amount and restart the analysis.
- Dirty or clogged transfer line. Clean or replace the transfer line.

If the problem persists, contact Technical Support.

Spectroscopic IR signal is reduced

Low infrared throughput is a problem you may encounter with your TGA-IR Module. To help recognize when this is happening, keep a record of peak-to-peak interferogram voltages, single-beam spectra, and performance. Record the flow cell temperature, number of scans collected, and resolution setting. By keeping track of this information, you can monitor the system integrity over time. If the spectroscopic signal in the TGA-IR Module shows a sudden loss in intensity, check the following:

- For spectrometers that are sealed and desiccated (rather than purged with dry air or nitrogen), the sample compartment windows may be damaged or fogged.

  Remove the TGA-IR Module and inspect the condition of the sample compartment windows in the spectrometer (a small flashlight is recommended). The windows should be clear (like glass), clean, and free of fingerprints. If they are scratched, broken, or fogged, replace them. For more information, open Spectrometer Help Topics from the Help menu in OMNIC Paradigm software and find the “Replacing Sample Compartment Windows” topic.

  > **Note** If you can’t tell the condition of the windows by inspecting them, remove the windows. If the signal intensity improves significantly, replace the windows.

  If the signal is still not satisfactory after you have replaced the windows, contact Technical Support.

- Spectrometer is misaligned. Use the Align feature in Experiment Setup of OMNIC Paradigm software to align the interferometer. See Spectrometer Help Topics in OMNIC Paradigm software for more information.
- Flow cell or flow cell windows are contaminated. Clean the flow cell and clean or replace the flow cell windows. See "Flow cell maintenance" for more information.
- IR beam is blocked, or partially blocked, by the beam port shutters. Make sure both shutters are installed in the open position (blue label on top).
- Spectrometer component has failed. See the Troubleshooting section of Spectrometer Help Topics.

If the problem persists, contact Technical Support.

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