

TGA Interface User's Guide

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Introduction

This manual explains how to set up your TGA interface for use with a Thermo Scientific spectrometer. After you have set up the interface, see the OMNIC™ Series software Help system for information on performing TGA/IR experiments.

Before operating the TGA interface, you should have a working knowledge of the OMNIC Series software. You will also need to know the fundamentals of thermogravimetric analysis and the operating characteristics of your thermogravimetric analyzer.

 **Warning**

The *Spectrometer Safety Guide* that came with your system contains important safety information. This guide is available in several languages. Contact our local office for information about the languages that are available. Before you use the TGA interface, read the entire guide. To prevent personal injury and damage to equipment, follow the safety precautions contained in the guide whenever you use the TGA interface or other system components. ▲

Manual conventions

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

Note Notes contain helpful supplementary information. ▲

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

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

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

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

Questions and concerns

In case of emergency, follow the procedures established by your facility. If you have questions or concerns about safety or need assistance with operation, repairs or replacement parts, you can contact our sales or service representative in your area or use the information at the beginning of this document to contact us.

Principles of Operation

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 FT-IR 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 module. A furnace heats the sample at a programmed rate from room temperature to temperatures in excess of 1000 °C. The TGA system plots a weight loss curve of the data file as a function of temperature or time.

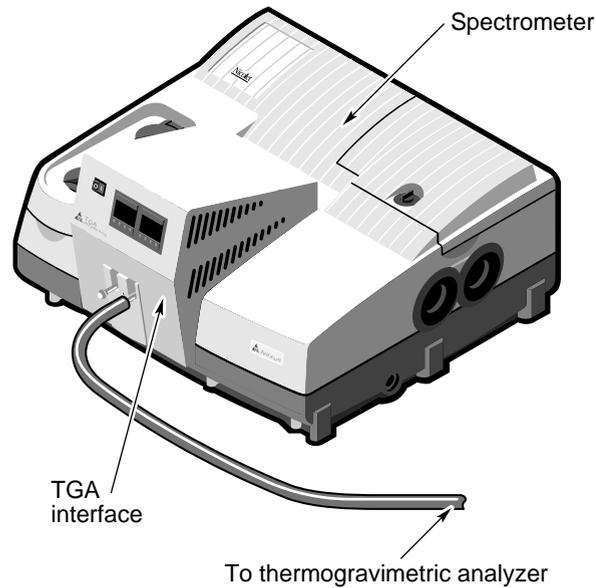
A purge gas continuously sweeps evolved gases from the furnace tube chamber. The purge gas may be inert, such as nitrogen, or oxidative, such as air. As the compounds in the sample vaporize, the purge gas carries them through a heated transfer line and flow cell. Here the gases are scanned by the infrared beam and the spectral data recorded. The gases exit the flow cell through the outlet tube and should be vented to a fume hood or trap.

The TGA interface is a single removable assembly that mounts in the sample compartment of the spectrometer or auxiliary experiment module (AEM). An insulated housing around the flow cell maintains its temperature at a preselected setting, from ambient to 300 °C. The flow cell is a separate assembly that you can quickly insert into or remove from the interface. The cell's simple design makes it easy to disassemble for cleaning.

Configuring the System

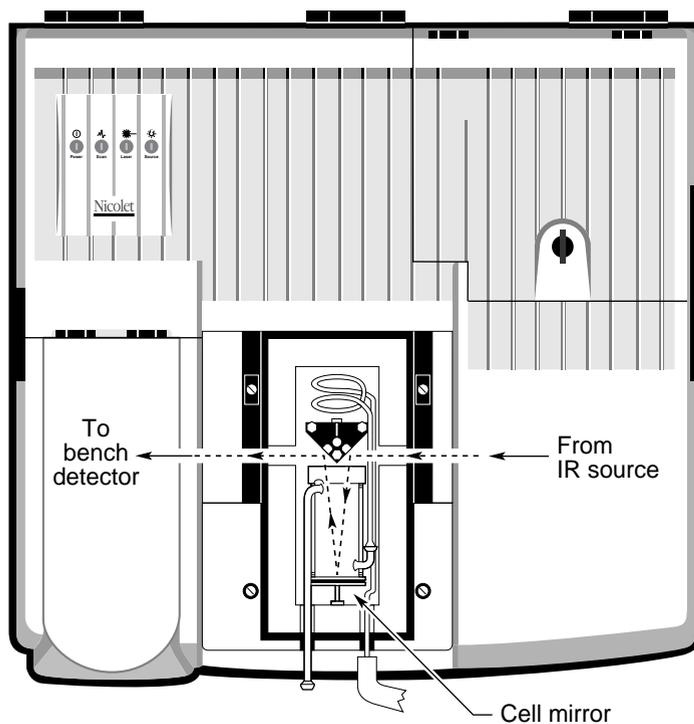
The TGA/IR system includes the following components:

- Thermo Scientific spectrometer
- Thermo Scientific TGA interface
- Thermogravimetric analyzer (TGA)



The TGA interface can also be installed in an auxiliary experiment module (AEM). The installation procedure is essentially the same.

The optical layout of the TGA interface is shown in the following illustration. See the manual that came with your spectrometer for a description of the spectrometer's internal layout.



Installing the TGA Interface

This chapter explains how to install the TGA interface in a Thermo Scientific spectrometer or AEM.

Before you install the TGA interface, measure and record the background signal intensity of your spectrometer (or auxiliary experiment module if you will be installing the interface in an AEM). This value will be used after you install the interface to determine whether the signal intensity through the interface is adequate. The interface signal intensity should be at least 40% of the spectrometer (or AEM) background signal intensity. If it is not, the interface will need to be aligned. The alignment procedure is provided later in this manual. To measure the background signal intensity, follow these steps:

- 1. Remove any sample or accessory from the sample compartment where you will be installing the TGA interface.**
- 2. Select the appropriate experiment or set the experiment parameters individually.**

If you have created and saved an experiment for your TGA work, you can set the parameters in one step by selecting the experiment. You can also choose Experiment Setup from the Collect menu and set the parameters individually. Be sure to set Sample Compartment on the Bench tab of the Experiment Setup dialog box to the location where you will be installing the interface. Make a record of the settings.

3. Record the maximum signal intensity.

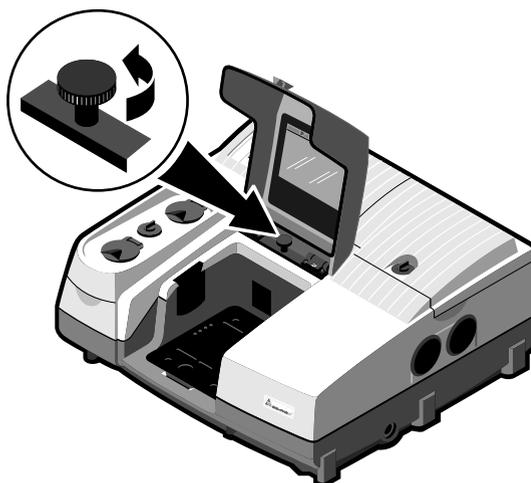
This is the value next to Max above the live display on the Bench tab of the Experiment Setup dialog box. You can leave the dialog box open while you install the interface.

Preparing the spectrometer or AEM

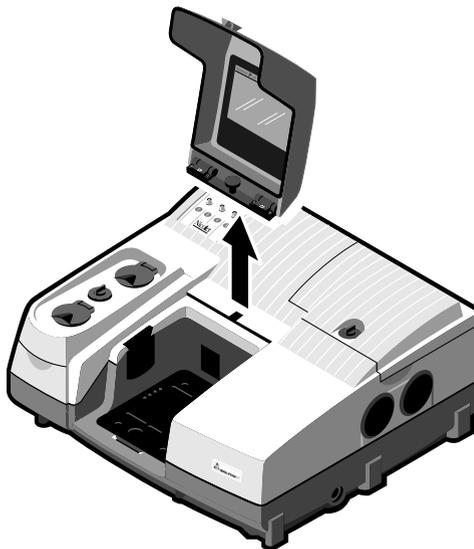
Follow the steps below to prepare the spectrometer or AEM for the TGA interface.

1. Remove the sample compartment cover.

Open the cover and loosen the thumbscrew that fastens the cover to the spectrometer or AEM.



Then lift the cover straight up.



2. Remove the standard sample compartment baseplate.

For instructions on removing the baseplate, see the user's guide that came with your spectrometer or AEM, or choose Replacing Parts from the OMNIC Help menu and then choose the "Removing the snap-in baseplate" topic from the "Replacing parts" book.

3. If your spectrometer is sealed and desiccated, remove the two sample compartment windows and purge the spectrometer.

See the "Replacing sample compartment windows" topic in the "Replacing parts" book of Spectrometer Help Topics. The "Removing the damaged window" procedure available in that topic explains how to remove a window. Also see the Note below.

Note If you have a Magna-IR™ system, contact our representative for information about removing the sample compartment windows. Do not attempt to remove the windows without first contacting us. ▲

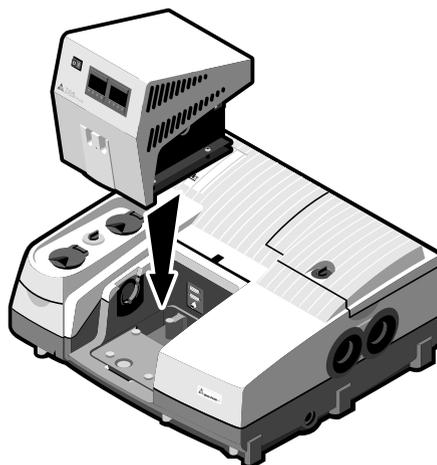
Important If you have a sealed and desiccated spectrometer, you must purge it after removing the sample compartment windows. View “Maintaining your spectrometer” in Spectrometer Help Topics for information on how to install the purge equipment, set the controls for the first time, and inspect and clean the purge filter. (If you have a Magna-IR, the information is in the “Service and maintenance” book of Spectrometer Help Topics.) ▲

Installing the interface

Follow these steps to install the interface:

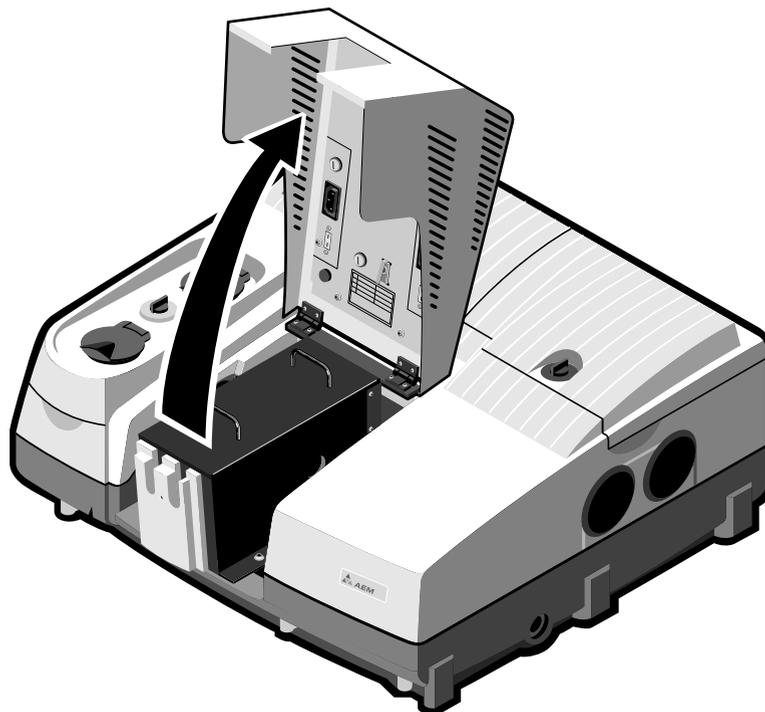
1. Carefully place the interface into the sample compartment.

To do this, hold the TGA interface with one hand under the hinge for the cover and the other hand under the front edge as shown below. Make sure the interface baseplate seats properly on the bottom of the sample compartment. The interface should not rock after it is installed.



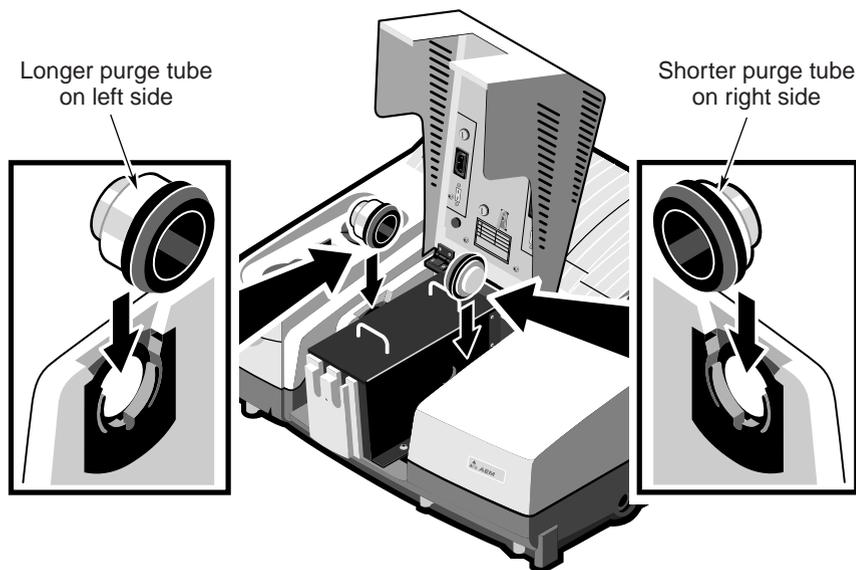
2. Open the interface cover.

It swings upward at the front.

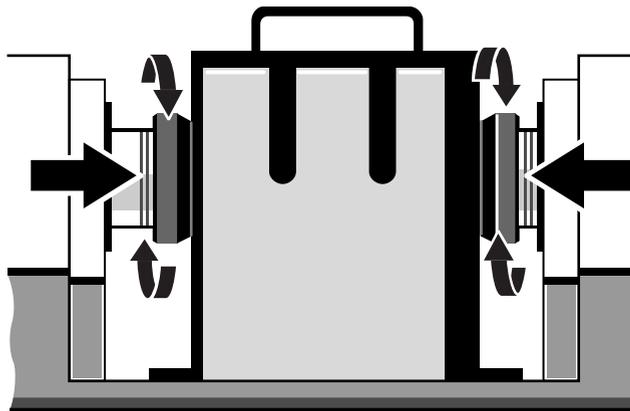


3. Install the purge tubes.

Slide the longer purge tube into the Smart Accessory clip on the left wall of the sample compartment so that the black ring faces the TGA interface, as shown below. Slide the shorter purge tube into the Smart Accessory clip on the right wall of the sample compartment so that the black ring faces the interface.



While holding the white inner ring to prevent it from turning, rotate the black rings of the tubes as shown below until they are snug against the outer walls of the interface. Do not overtighten.



⚠ Warning

AC connectors and hot surfaces may be present in the vicinity of the receptacles. Use care when plugging or unplugging any connector on the underside of the interface cover. ▲

- 3. On the underside of the interface cover, plug the cell heater connector into the receptacle labeled “Heater” on the left, and plug the transfer line heater connector into the receptacle labeled “Heater” on the right.**
- 4. On the underside of the interface cover, plug the cell thermocouple connector into the receptacle labeled “Thermocouple” on the left, and plug the transfer line thermocouple connector into the receptacle labeled “Thermocouple” on the right.**

5. Plug in the interface power cord.

Note The TGA interface uses 110 VAC. Operation using 220 VAC requires a transformer (available from us). Call us for assistance if you plan to use the higher line voltage. ▲

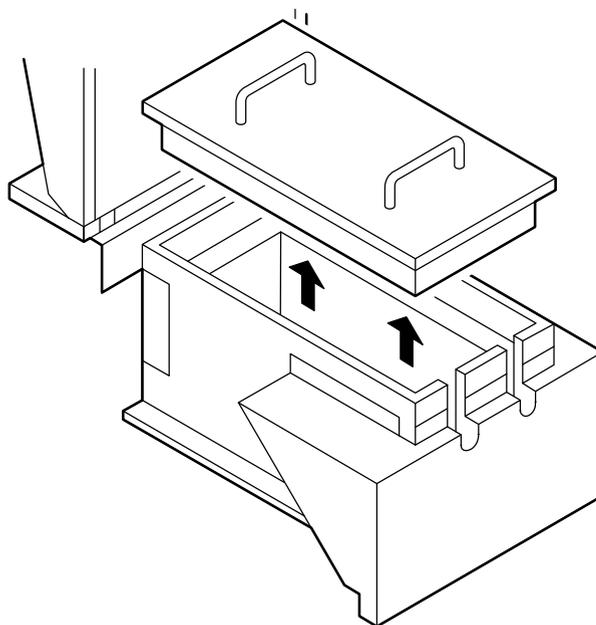
▲ Caution After you plug the interface power cord into the power outlet on the wall, make sure there is at least 10 cm (4 in) of clearance in front of and around the outlet. ▲

Installing the cell

The cell for the interface is shipped in a separate box and must be installed in the furnace. Follow these steps:

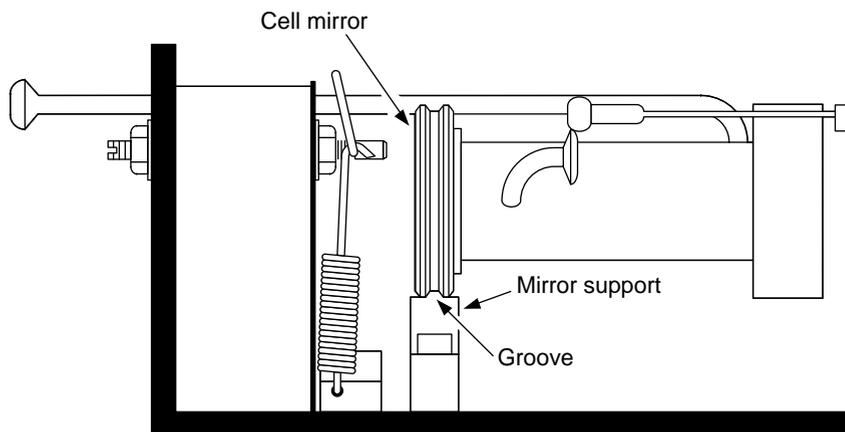
- 1. Open the TGA interface cover.**
- 2. Remove the furnace cover.**

To remove the cover, grasp the insulated furnace cover by the two handles and lift the cover straight up as shown in the following illustration.



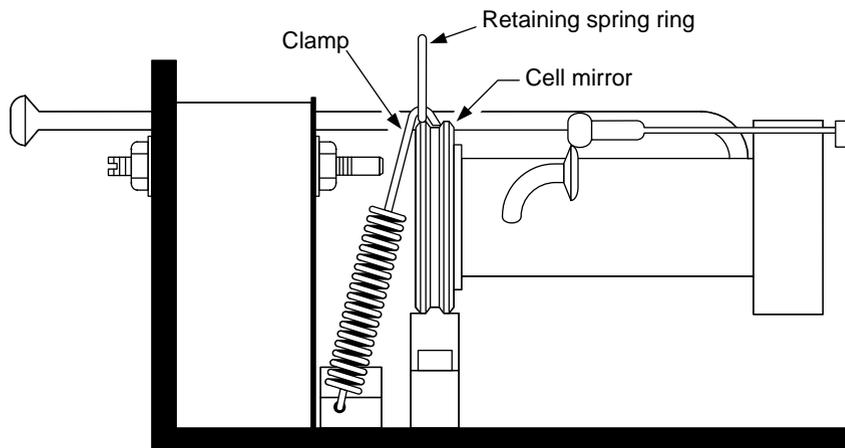
3. Place the cell into the furnace.

To do this, position the cell under the transfer line so that the grooved cell mirror rests on the mirror support as shown below.



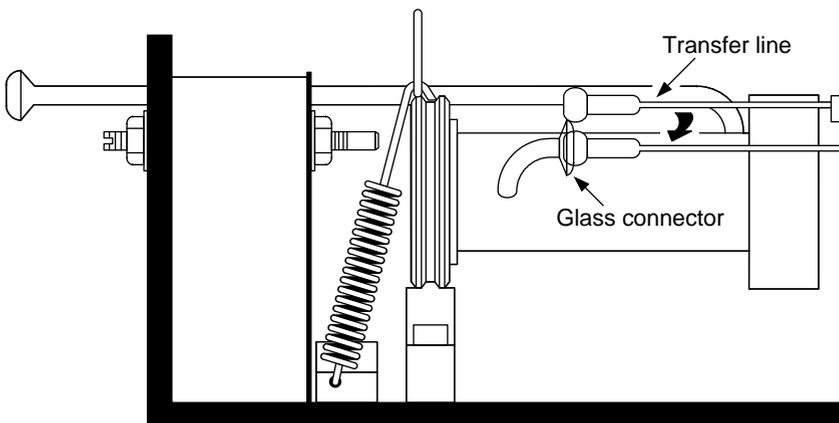
4. Clamp the cell in place.

To do this, pull up the retaining spring ring, slide the clamp toward the cell mirror and hook the clamp over the groove in the edge of the mirror as shown in the following illustration.



5. Connect the transfer line to the glass tube.

To do this, position the end of the line inside the glass connector as shown below.



Note If the ball joint end of the transfer line is not secure in the glass connector, disconnect the line from the connector and then gently expand the transfer line coil at the rear of the interface. This will increase the pressure on the joint when you reconnect the line to the connector. ▲

Aligning the Optics

Your TGA interface was aligned at the factory and normally requires no further alignment. The optics of the interface need to be aligned only if the signal intensity through the interface is less than 40% of the background signal intensity of the spectrometer (or auxiliary experiment module if the interface is installed in an AEM). The signal intensity through the interface is shown above the live display on the Bench tab of the Experiment Setup dialog box. The value shown next to Max should be at least 40% of the value you recorded before installing the interface. If it is not, first contact Customer Support for advice and then, if directed, use the following procedure to align the interface.

After the initial alignment, no further adjustments of the interface optics should be necessary. The pins on the TGA baseplate are designed to align the interface after it is removed and reinstalled.

Warning

If for any reason you need to align the interface optics after the interface has been used, allow the interface furnace to cool for at least 2 hours before using this procedure. ▲

Follow these steps to align the optics:

- 1. Start the OMNIC software.**
- 2. Choose Experiment Setup from the Collect menu and go to the Bench tab.**

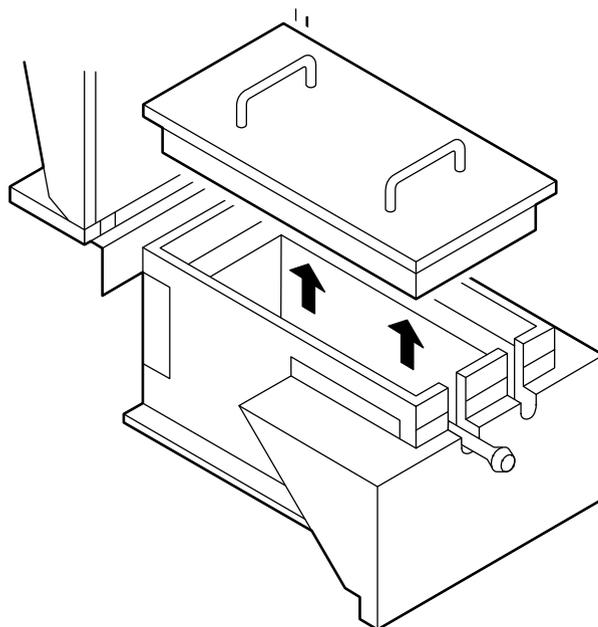
The detector signal is represented graphically in the live display as an interferogram and numerically as maximum (Max) and minimum (Min) voltages. The object of alignment is to maximize the intensity of the detector signal.

(If the current maximum value is at least 40% of the background signal intensity measured before the interface was installed, the interface does not need to be aligned and you can skip the rest of this procedure.)

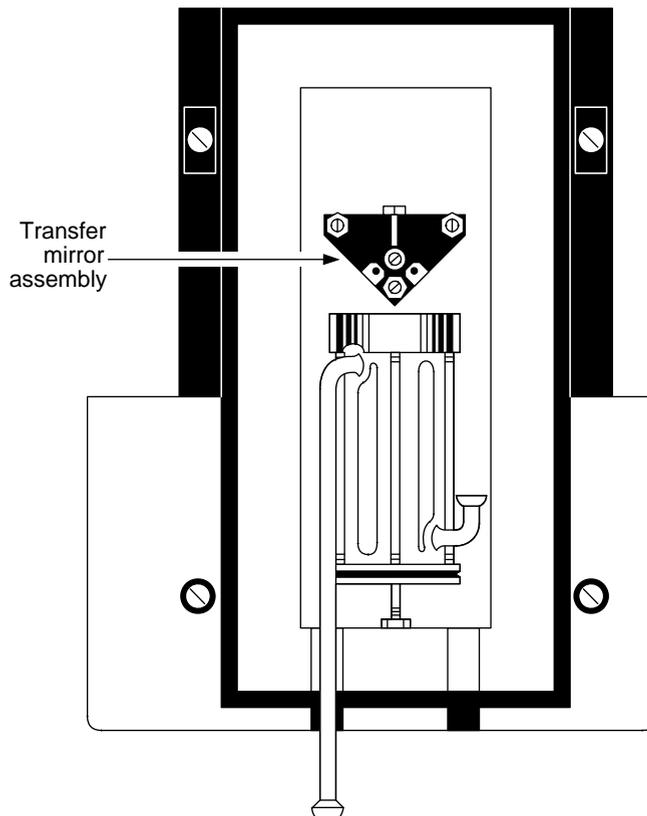
3. Open the TGA interface hinged cover.

4. Remove the furnace cover if it is now in place.

The mirrors are located inside the furnace. Grasp the insulated furnace cover by the two handles and lift the cover straight up as shown in the following illustration.

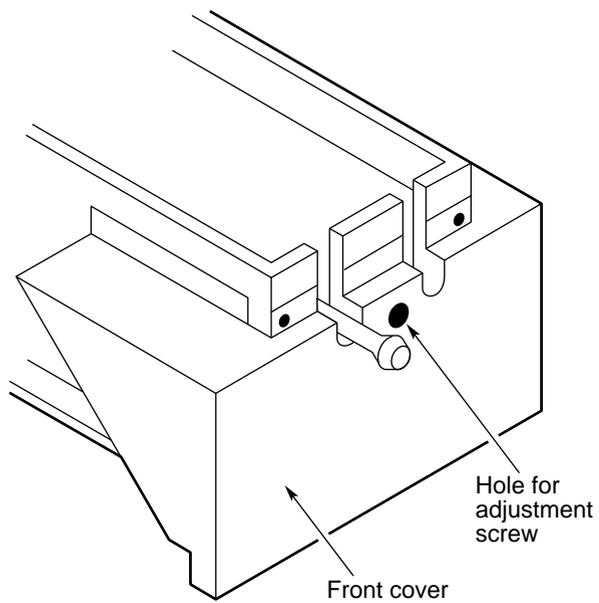


5. **Press down on the transfer mirror assembly and attempt to rotate it back and forth slightly to make sure it is properly seated.**



After seating the transfer mirror assembly, check the maximum value displayed in the Experiment Setup dialog box. If it is now at least 40% of the background signal intensity measured before the interface was installed, there is no need to make any further adjustments. Replace the furnace cover and close the hinged cover. If the maximum value is still less than the required 40% level, go on to the next step.

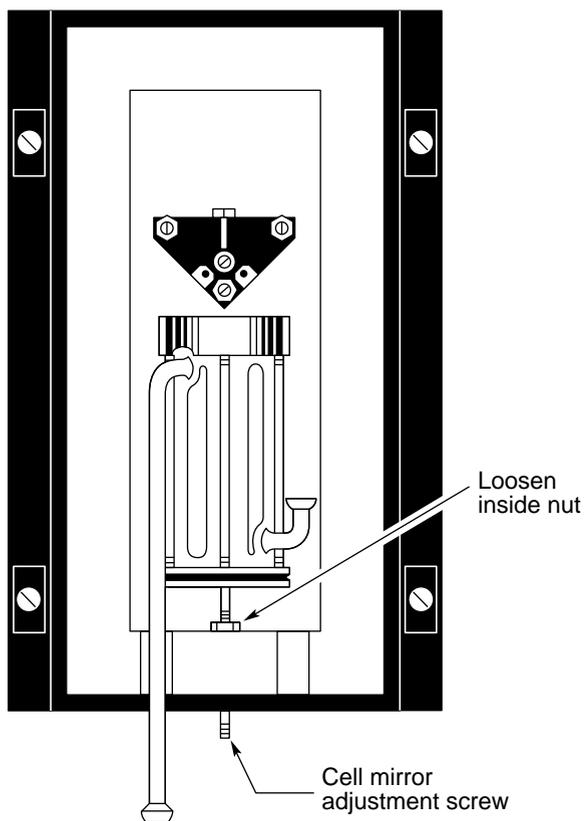
6. Locate the hole in the front cover.



7. Use a flat-blade screwdriver to turn the cell mirror adjustment screw to maximize the detector signal.

A nut inside the interface locks the adjustment screw in place; loosen the nut before turning the screw. Insert the screwdriver through the hole in the cover to turn the adjustment screw. Adjust for maximum signal.

Always retighten the locking nut when you are finished.



Try turning the adjustment screw a small amount in each direction until the maximum signal is obtained.

If you are able to increase the maximum value to at least 40% of the background signal intensity measured before installation, go to the next step.

If you cannot obtain a maximum value of the required 40% level, replace the interface front cover and the furnace cover, close the hinged cover and call us for assistance. Skip the rest of this procedure.

8. Tighten the nut without turning the adjustment screw.

9. Replace the interface front cover and the furnace cover and close the hinged cover.

Note The furnace heater will not turn on unless the hinged cover is closed. ▲

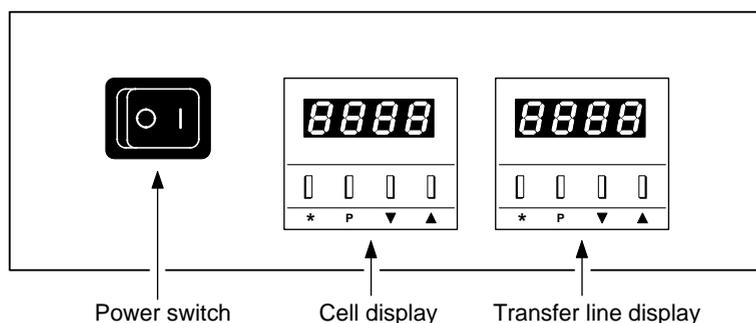
10. Choose OK to close the Experiment Setup dialog box.

Setting the Temperature Controller

The temperature controllers for the TGA interface are part of the cover of the interface. Follow these steps to set the controllers:

- 1. Make sure the power cord for the TGA interface is plugged into an outlet, and then turn on the interface power.**

The power switch is located on the front panel of the TGA interface:

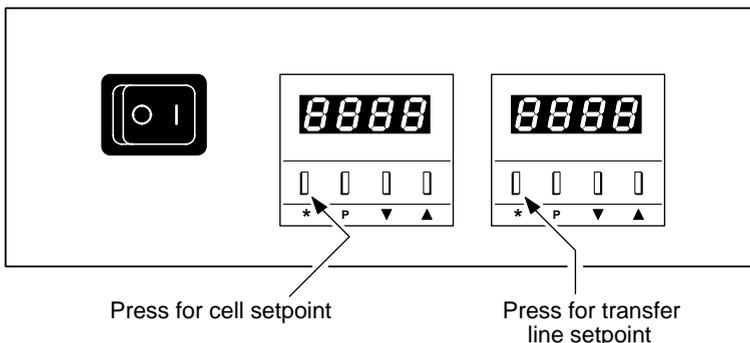


- 2. Make sure the furnace cover is securely seated and close the hinged cover.**

Note The TGA interface has a safety interlock that shuts off the power to the heater when the furnace cover is not securely in place or when the hinged cover is raised. ▲

3. Display the current setpoint temperatures.

To do this, press each button labeled with an asterisk (*) on the front of the temperature controller. The digital display on the left is for the cell and the display on the right is for the transfer line.



4. Adjust the setpoints.

We recommend a cell temperature of 250 °C and a transfer line temperature of 225 °C for most TGA/IR applications. To adjust a setpoint, hold down the setpoint display button (labeled *) and press the down arrow button (labeled ▼) or up arrow button (labeled ▲). Do this for both digital displays. See the preceding illustration for the location of the buttons.

Important Do not set the cell setpoint above 300 °C or the transfer line setpoint above 225 °C! Higher temperatures may damage the interface. ▲

If a ▲ symbol appears in the upper-left corner of the controller display, the temperature of the heated zone currently exceeds the setpoint. If a ▼ symbol appears in the lower-left corner of the display, the temperature of the heated zone is currently lower than the setpoint. If a ■ symbol appears at the left-center of the display, the temperature of the heated zone is at the setpoint. If a ■ symbol appears in the lower-right corner of the display, the heater element power is on.

Preparing the System for Data Collection

This chapter explains how to prepare your TGA/IR system before running an experiment.

Setting up the interface

After your TGA interface is installed, follow these steps to set up the interface:

- 1. Make sure that the TGA is properly connected to the TGA interface.**

The TGA should have been positioned properly during installation. Check to see that no glass components are stressed.

- 2. Prepare a dry nitrogen purge line and connect it to the TGA furnace purge inlet.**

- 3. Adjust the purge flow rate to approximately 50 to 60 mL/min.**

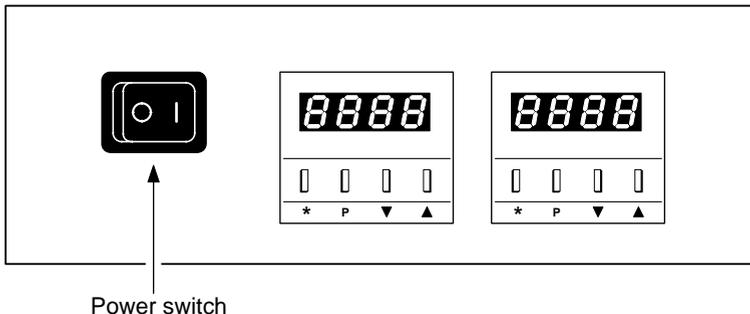
Use a flowmeter to measure the purge flow rate.

Note Higher purge flow rates will greatly reduce the TGA/IR sensitivity.

▲

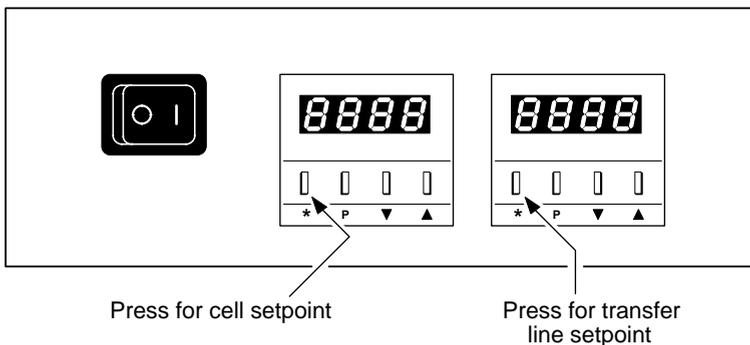
4. Turn on the temperature controller.

Use the power switch on the interface front panel.



5. Display the temperature setpoints.

The current cell temperature is shown by the digital display on the left, and the current transfer line temperature is shown by the display on the right. To display the setpoints for these, press the button labeled with an asterisk (*) under each display (see the following illustration).



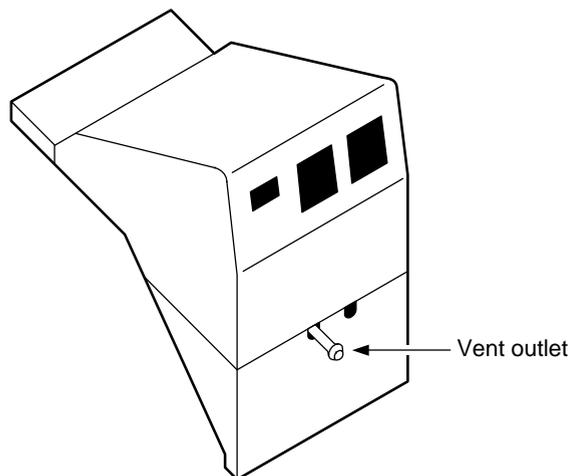
6. Adjust the setpoints.

A temperature of 220 °C is adequate for most TGA/IR applications. To adjust a setpoint, hold down the setpoint display button (labeled *) and press the down arrow button (labeled ▼) or up arrow button (labeled ▲). Do this for both digital displays. See the illustration in the preceding step for the location of the buttons.

Important Do not set the cell setpoint above 300 °C or the transfer line setpoint above 225 °C! Higher temperatures may damage the interface. ▲

Venting the interface

Gases released during a TGA/IR experiment may be hazardous and should be vented in a safe manner. The gases are released through the vent outlet on the TGA interface (see the following illustration).



The venting apparatus you use should be connected to the vent outlet with a 12/5 ground glass socket fitting using the provided clamp. We recommend using an aspirator to draw out the exhaust gases.

⚠ Warning

Allow the vent outlet to cool before connecting a venting apparatus to it. ▲

Important

The TGA cell is not designed for vacuum operation. ▲

Your TGA interface is now ready for operation. See the OMNIC Series Help system for instructions on collecting spectra.

Operating Tips

Here are a few tips that will help you obtain the best results with your TGA/IR system:

Allow plenty of time (2 hours) for the spectrometer to thermally stabilize after you turn on the interface.

The slower the purge flow you select, the higher the sensitivity you can expect. The tradeoff at low flow rates is loss of temporal relationship between the weight loss curve and the IR data. The optimum purge gas flow rate depends on the particular sample and the heating rate of the furnace. A flow rate of 50 to 60 mL/min is a good starting point for heating rates of 15 to 30 C°/min. For slow heating rates, say 5 C°/min or less, use 40 to 50 mL/min.

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

Maintenance

This chapter explains how to replace fuses in the TGA interface and perform cell maintenance.

If you need to replace a transfer line heater, contact our representative for assistance.

▲ Warning

Maintenance procedures for the TGA/IR interface require you to open the interface cover, which exposes the furnace cover. We recommend turning off the interface power and letting the furnace cool before performing a maintenance procedure. If you open the interface cover without letting the furnace cool first, do *not* touch the furnace cover or the cover handles! ▲

Replacing fuses

The TGA interface has three fuses that protect the system against damage from excessive current. The cell and transfer line each have a 2 A, quick-acting, F Type fuse. There is also a 4 A, quick-acting, F Type main fuse.

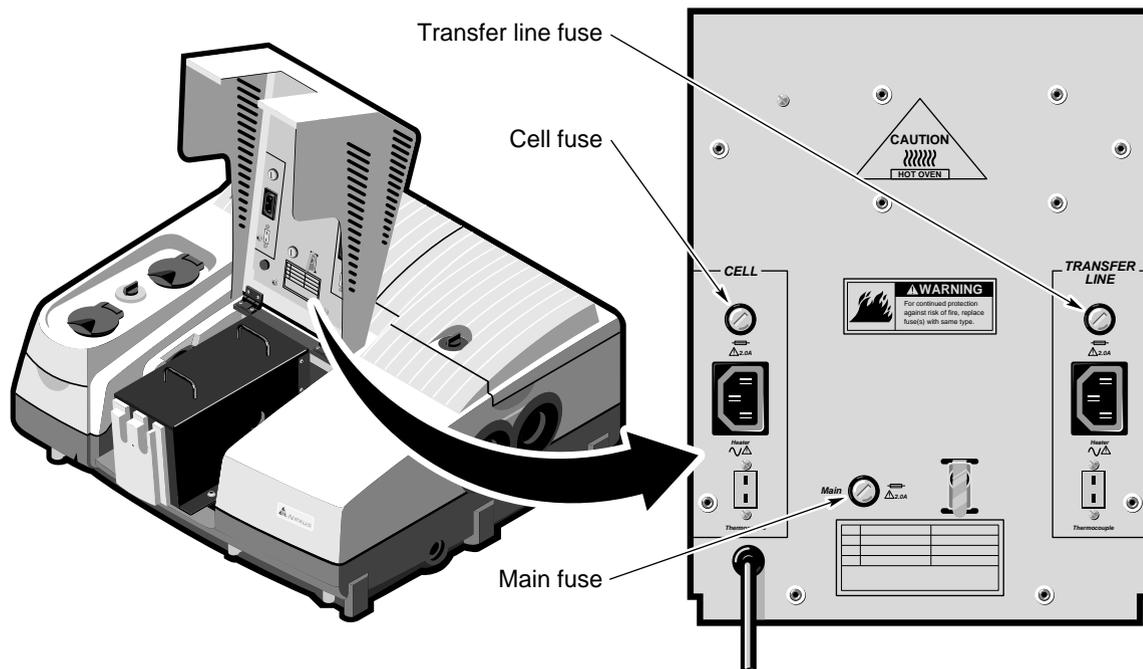
Note The interface temperature controller is protected by an externally mounted thermal fuse. If the cell does not heat after you have used the following procedure to check or replace the cell fuse under the interface cover, the thermal fuse may need replacement. Contact our representative for assistance. ▲

Follow these steps if you need to replace one of these fuses:

- 1. Turn off the TGA interface power.**

2. Open the TGA interface cover.

The fuse holders are on the underside of the cover.



⚠ Warning Alternating current and hot surfaces may be present in the vicinity of the fuse holders. Use care when changing a fuse. ▲

3. Use a flat-blade screwdriver to turn the appropriate fuse holder counterclockwise.

The fuse holder will pop out of the interface cover.

⚠ Warning

To avoid the risk of fire, always replace a fuse with a new fuse of the same type and amperage rating. ▲

4. Replace the fuse with a new fuse of the same type and amperage rating.

To replace the cell fuse or transfer line fuse, use a 2 A, quick-acting, F Type fuse. To replace the main fuse, use a 4 A, quick-acting, F Type fuse.

5. Insert the fuse holder into the hole in the interface cover and turn it clockwise to lock it in place.

6. Close the TGA interface cover.

7. Turn on the TGA interface power.

Cell maintenance

Use the following procedure to clean the cell mirror, body or IR window or to check or replace gaskets.

- 1. Turn off the TGA temperature controller and unplug the power cord.**

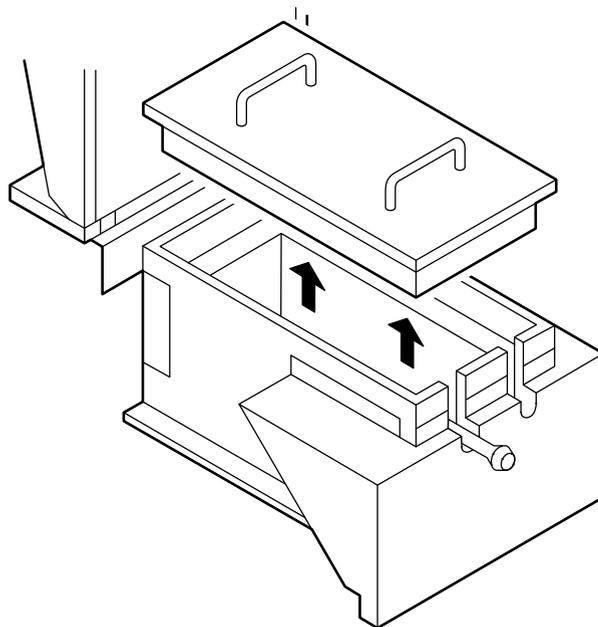
The ON/OFF switch is on the underside of the hinged cover. Allow the TGA interface to cool for at least two hours before continuing.

▲ Warning The furnace cover may be very hot. Do *not* touch it. ▲

- 2. Open the TGA/IR interface.**

- 3. When the interface is cooled sufficiently, remove the furnace cover.**

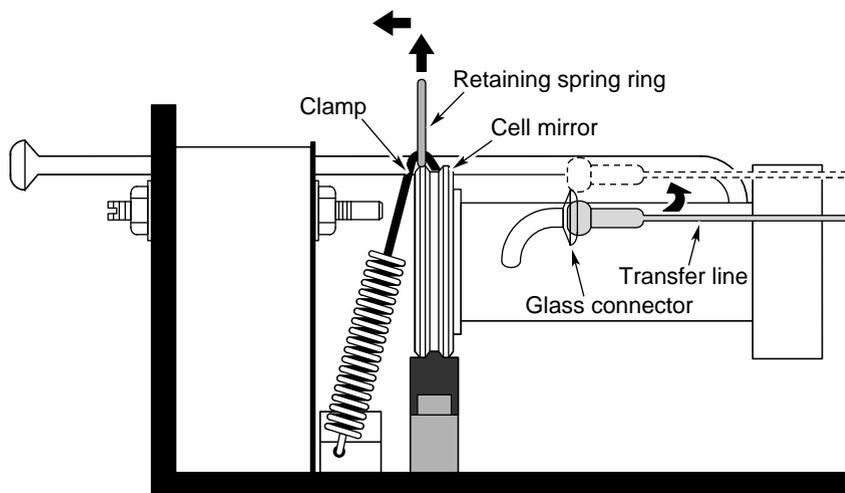
To do this, grasp the insulated furnace cover by the two handles and lift the cover straight up.



⚠ Warning The transfer line may be very hot. Do *not* touch it until cooled sufficiently. ▲

4. Remove the cell assembly.

First carefully disconnect the transfer line from the end of the glass tube. The cell is held in place by a spring-loaded clamp. Lift up the ring attached to the clamp and pull the clamp away from the cell mirror to release the cell assembly (see the following illustration).



5. Replace the cell assembly with a clean cell assembly or disassemble it for cleaning.

Follow these steps to disassemble the cell assembly:

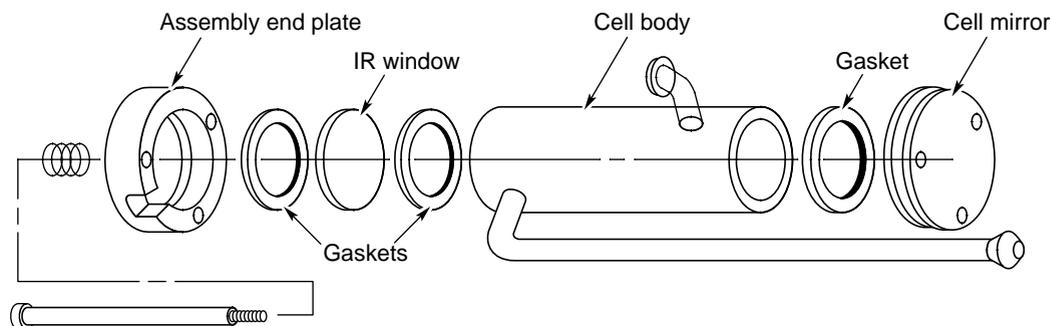
Remove the three retaining rods from the cell.

Slide the mirror plate and gasket off the assembly.

Pull the cell body from the assembly.

Remove the IR window and the gaskets on either side of the window.

The following illustration shows the parts of the cell assembly:



7. Replace the IR window or clean it as you would any IR salt plate.

If you are using a KBr window, you can remove most deposits by polishing the window with a laboratory wipe and ethanol (or other suitable solvent) or by using a polishing kit for salt windows (available from your laboratory equipment supply company).

8. Clean the mirror.

To clean the mirror, gently swab it with a solvent-soaked Chem Wipe.

9. Clean the glass cell body.

To clean the cell body, soak it in a cleaning bath suitable for cleaning laboratory glassware.

Remember that the IR beam does not contact the walls of the cell body, so it is not necessary for it to be immaculately clean. All that is required is to remove the materials which may volatilize at the cell operating temperature or be reactive to evolved gases.

10. Replace the gaskets if they are cracked or deformed.

11. Reassemble the cell assembly.

Replace the IR window and gaskets, cell body, gasket and mirror as shown in the preceding illustration.