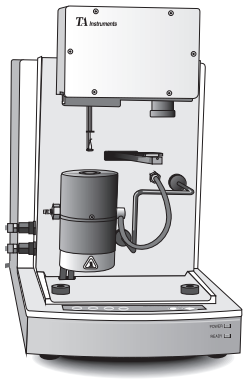
TA Instruments

Thermal Analysis & Rheology



TGA 2050

Thermogravimetric Analyzer

Operator's Manual

PN 925603.001 Rev. D (Text and Binder)
PN 925603.002 Rev. D (Text Only)
Issued July 2000

Notice

The material contained in this manual is believed adequate for the intended use of this instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. This publication is not a license to operate under or a recommendation to infringe upon any process patents.

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

NOTE: A NOTE highlights important information about equipment or procedures.

♦ ***CAUTION:*** **A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.**

!WARNING **A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.**

Hotlines

To TA Instruments Thermal Analysis

For Technical Assistance ..... (302) 427-4070

To Order Instruments and
Supplies ..... (302) 427-4040

For Service Inquiries ..... (302) 427-4050

General Information ..... (302) 427-4000

Safety

Electrical Safety

You must unplug the instrument *before* doing any maintenance or repair work; voltages exceeding 110 volts AC are present in this system.

WARNING

High voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

WARNING

After transport or storage in humid conditions, this equipment could fail to meet all the safety requirements. Refer to the NOTE on page 2-10 for the method used to dry out the equipment before use.

Chemical Safety

Use only the purge gases listed in Table 1.2 in Chapter 1. Use of other gases could cause damage to the instrument or injury to the operator.

WARNING

Do not use hydrogen or any other explosive gas in the TGA 2050 furnace.

WARNING

If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

Safety
(continued)

WARNING

If you are routinely evaluating materials in the TGA that lose a large amount of volatile hydrocarbons (*e.g.*, lubricating oils), you need to clean the furnace more frequently to prevent dangerous buildup of debris in the furnace. This is particularly important if oxygen is used as a purge gas in the TGA 2050. See Chapter 5 for instructions.

WARNING

The TGA 2050 furnace assembly also contains refractory ceramic fiber (RCF) insulation. This insulation is enclosed within the furnace housing. The furnace housing should only be disassembled for replacement of EGA furnace sample tube or furnace assemblies. Refer to instructions provided with the sample tube or furnace replacement kits for procedures for handling RCF insulation.

Thermal Safety

After running an experiment, allow the open furnace and thermocouple to cool down before you touch them.

WARNING

During a sample run, the furnace base can be hot enough to burn skin. Avoid contact with the furnace base during experiments.

Mechanical Safety

WARNING

Keep your fingers and all other objects out of the path of the furnace when it is moving. The furnace seal is very tight.

Using This Manual

Chapter 1	Describes your TGA 2050 instrument and its specifications.
Chapter 2	Describes how to connect the TGA to the rest of your system and how to install the system.
Chapter 3	Describes how to run TGA experiments.
Chapter 4	Provides technical information on the procedures described in Chapter 5 and explains principles of TGA operation.
Chapter 5	Describes how to perform routine maintenance, replace the thermocouple, remove and reinstall the furnace, and diagnose power problems; also provides an explanation of the confidence test.
Appendix A	Lists TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.
Index	Lists the page numbers of important topics for your reference.

CHAPTER 1: Introducing the TGA 2050

Introduction

Your TA Instruments Thermogravimetric Analyzer (TGA) 2050 is a thermal weight-change analysis instrument that can be used with any of the TA Instruments PC-based controllers.

The TGA 2050 measures the amount and rate of weight change in a material, either as a function of increasing temperature, or isothermally as a function of time, in a controlled atmosphere. It can be used to characterize any material that exhibits a weight change and to detect phase changes due to decomposition, oxidation,or dehydration. This information helps the scientist or engineer identify the percent weight change and correlate chemical structure, processing, and end-use performance.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instruments
- Enables you to set up experiments and enter constants
- Stores experimental data
- Runs data analysis programs.

The TGA 2050 executes thermal experiments that have been set up on the controller. See Figure 1.1 on the next page for an illustration of a TGA system.

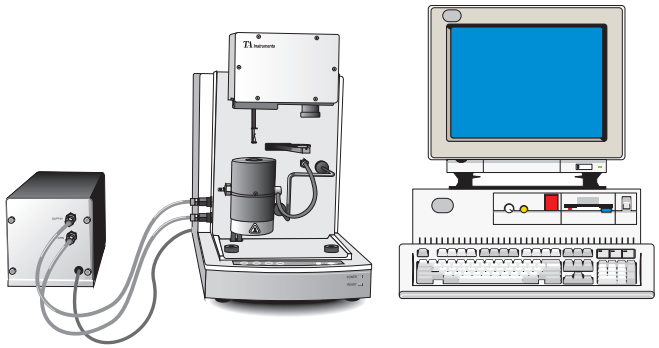

Figure 1.1 Thermogravimetric Analyzer (TGA) 2050 with PC-Based TA Instruments Thermal Analyzer

Components

The TGA 2050 has five major components, illustrated in Figure 1.2:

- The balance, which provides precise measurement of sample weight. The balance is the key to the TGA system.
- The sample platform, which loads and unloads the sample to and from the balance.
- The furnace, which controls the sample atmosphere and temperature.
- The cabinet, where the system electronics and mechanics are housed.
- The heat exchanger, which dissipates heat from the furnace.

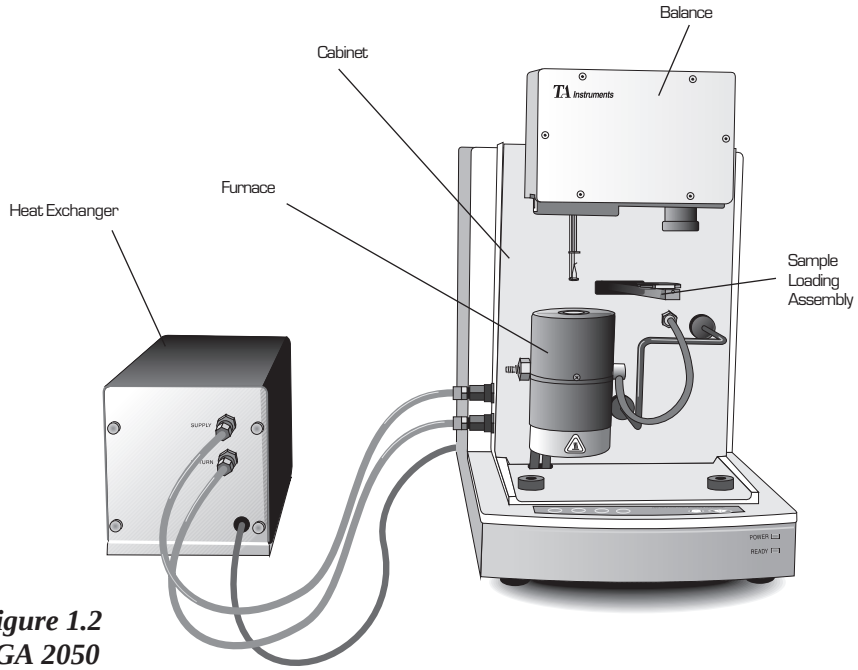

Figure 1.2
TGA 2050
Components

The 2050 Instrument

The instrument keypad (Figure 1.3) contains keys that control local operations at the instrument (automatic balance tare, furnace elevator, sample loading platform, and experiment start and stop functions).

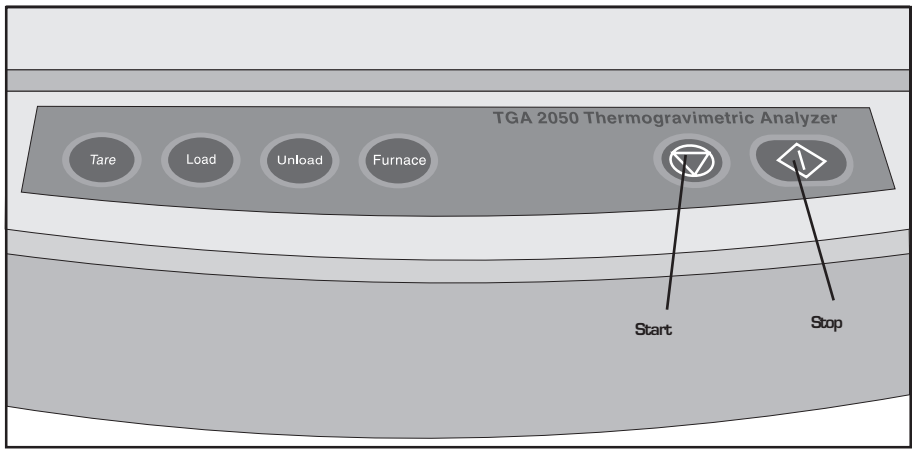

Figure 1.3
TGA 2050 Keypad

Table 1.1 explains the functions of the instrument keys.

NOTE: Experiment information and instrument constants are entered from the controller keyboard, not the instrument keypad.

Table 1.1
Instrument Keypad
Function Keys

Key/Function	Explanation
TARE	Zeros the weight of an empty sample pan: automatically loads the pan from the sample platform, raises the furnace to protect the pan from air currents, weighs the pan, stores the weight as an offset, and then unloads the pan.
LOAD	Loads a sample pan from the sample platform onto the balance.
UNLOAD	Unloads the sample pan from the balance onto the sample platform.
FURNACE	Toggles between the furnace closed (up) and furnace open (down) functions, depending on where the furnace is when you press the key. This key can be pressed while the furnace is moving to reverse the direction of movement.
START	Begins the experiment. This is the same function as **Start** on the controller.

(table continued)

Table 1.1
Instrument Keypad
Function Keys
(continued)

Key/Function	Explanation
STOP	If an experiment is running, this key ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions go into effect, and the data that has been generated is saved. This is the same function as **Stop** on the controller. If an experiment is not running (the instrument is in a stand-by or method-end state), the STOP key will halt any activity (air cool, all mechanical motion, *etc*.).

Automatic Keypad Functions

Some of the TGA instrument keys automatically perform additional functions under certain conditions:

- START automatically loads the sample pan and closes the furnace, if necessary, before beginning the experiment.
- TARE, LOAD, and UNLOAD automatically open the furnace if necessary.
- START can be pressed while a sample load is in progress.

Front Panel Lights

The front panel green power light shows that the instrument is on; the yellow light shows that the instrument is ready.

Accessories

Gas Switching Accessory

The TA Instruments Gas Switching Accessory can be used to turn the purge gas on and off or to switch between two different purge gases during TGA experiments.

Other Accessories

The TGA can be used with many standard analytical accessories offered by various manufacturers, including vacuum, FTIR, gas chromatographs, mass spectrometers, and evolved gas analyzers. Consult the appropriate local instrument manufacturer for further information.

Specifications

Table 1.2
TGA 2050
Specifications

Temperature range	25°C to 1000°C
Thermocouple	Platinel II*
Heating rate	0.1 to 50°C/min
Operating line voltage	115 volts, 50/60 Hz
Energy consumption	1.5 kVA

*Platinel II is a registered trademark of Engelhard Industries.

Table 1.3
Sampling System

Sample pans	
Types	Platinum, Alumina ($Al_2 0_3$), Aluminum
Volume capacity	Platinum: 50 μL, 100 μL Alumina: 100 μL, 250 μL, 500 μL Aluminum: 100 μL
Weighing capacity[1]	1.0 gm
Balance measurement[2]	
Resolution	0.2 μg
Accuracy	$\leq \pm$ 0.1%
Ranges	100 mg range: -10 mg to 100 mg 1000 mg range: -100 mg to 1000 mg

♦ ***CAUTION:*** **[1] The total mechanical capacity of the balance is 5 gm. In order to avoid damaging the balance assembly, never allow the total weight of the sample, tare weight, hang-down wires, and pans to exceed 5 gm.**

[2] The TGA balance mechanism is sensitive to changes in the surrounding room temperature. For optimum accuracy, you must regulate the ambient temperature.

(*table continued*)

Table 1.3
Sampling System
(continued)

Furnace Atmosphere Purge gases	Helium, nitrogen, oxygen, air, argon[3]
Purge rate	Up to 100 cc/min

WARNING

[3] Do not use hydrogen or any other explosive gas in the TGA 2050. Oxygen may be used. However, the furnace must be kept clean of hydrocarbons to prevent combustion.

CHAPTER 2: Installing the TGA 2050

Unpacking/Repacking the 2050

NOTE: Due to the delicate mechanisms contained in the TGA 2050 balance, the balance will be unpacked later, see page 2-21.

Refer to Figures 2.1 to 2.3 while unpacking your instrument.

Unpacking the 2050

WARNING **Have an assistant help you unpack this unit. Do not attempt to do this alone.**

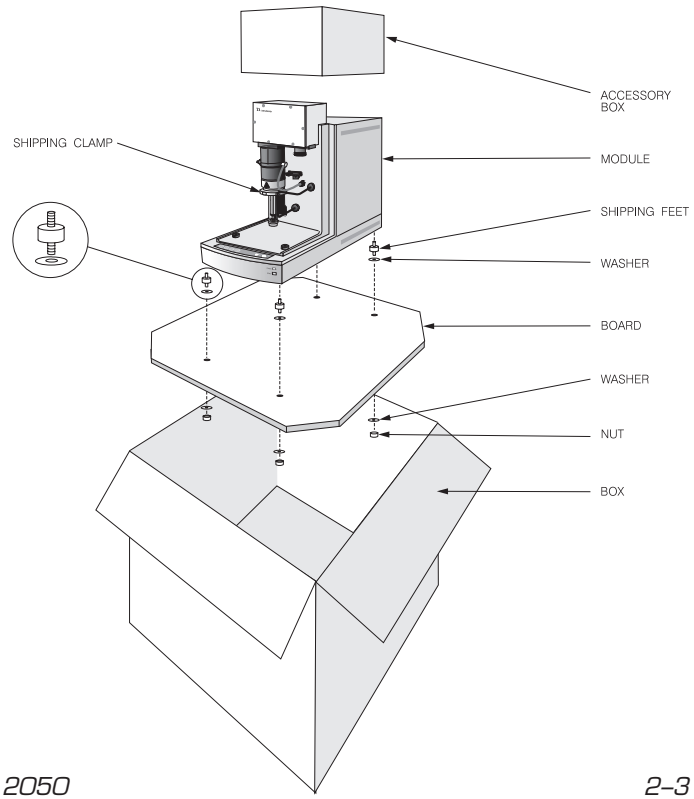

Figure 2.1
Shipping Boxes

1. Open the shipping carton and remove the accessory box.

2. Remove the cardboard packing insert.

3. Stand at one end of the box with your assistant facing you at the other end. Lift your end of the unit out of the box as your assistant lifts his/her end.

4. Place the unit on a lab bench with one side hanging over the edge of the bench (see Figure 2.2). **Someone must be holding onto the unit at all times while it is in this position.**

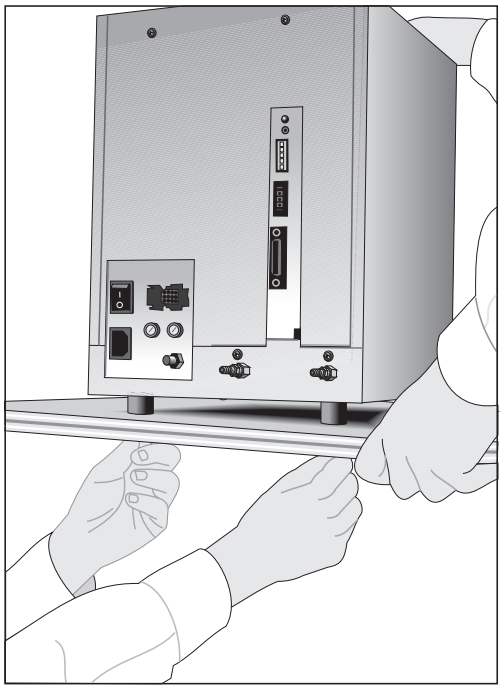

Figure 2.2
Installing Mounting Feet

5. While your assistant holds the unit, use a wrench to remove the two nuts and washers from the bottom. Then lift and rotate the unit so that the other end hangs over the edge of the bench. **Someone must hold onto the unit at all times while it is in this position.** While your assistant holds the unit, remove the two nuts and washers from the other side.

6. Slide the unit completely onto the lab bench. Have your assistant hold one side up while you unscrew and remove the black rubber shipping feet from the bottom. Then rotate the unit and remove the shipping feet from the other side in the same manner.

7. Have your assistant lift one side of the unit while you install two mounting feet on one side (see Figure 2.3). Screw in about 1/4-inch of the threaded mounting post into the unit. Rotate the unit and install the two remaining mounting feet on the unit in the same manner.

8. Have your assistant lift the entire unit while you slide the plywood board out from under the unit.

9. Turn the instrument to face front and use a wrench to remove the furnace clamp from the unit. Retain this clamp in case the instrument needs to be shipped in the future.

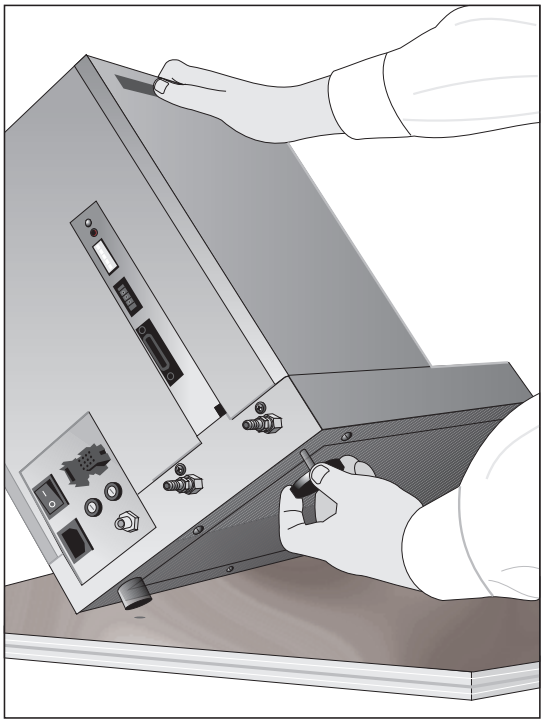

Figure 2.3 Installing the Mounting Feet

10. Follow the directions beginning on page 2-7 to install the instrument before you unpack the balance.

Repacking the 2050

To pack and ship your instrument, use the hardware retained during unpacking and reverse the instructions found on pages 2-3 to 2-5.

Installing the Instrument

Before shipment, the TGA 2050 instrument is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Installation involves the following procedures, described in this chapter:

- Unpacking the instrument and its components and accessory kit
- Inspecting the system for shipping damage and missing parts
- Filling the heat exchanger
- Connecting the TGA to the TA Instruments controller
- Connecting the heat exchanger cable and water lines, purge gas lines, accessories, and power cable
- Unpacking the balance
- Installing the hang-down wires
- Leveling the instrument and aligning the hang-down wires
- Adjusting the sample platform.

If you wish to have your TGA installed by a TA Instruments Service Representative, call for an installation appointment when you receive your instrument.

♦ ***CAUTION:*** **To avoid mistakes, read this entire chapter before you begin installation.**

Inspecting the System

When you receive your TGA, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

If the instrument is damaged, notify the carrier and TA Instruments immediately.

If the instrument is intact but parts are missing, contact TA Instruments.

A list of TA Instruments phone numbers can be found in Appendix A of this manual.

Choosing a Location

Because of the sensitivity of TGA experiments, it is important to choose a location for the instrument using the following guidelines. The TGA should be:

In
- ... a temperature controlled area.
- ... a clean, vibration-free environment.
- ... an area with ample working and ventilation space. (Refer to the specifications in Chapter 1 for the instrument dimensions).

On
- ... a stable work surface.

Near
- ... a power outlet (115 volts AC, 50 or 60 Hz, 15 amps). A step up/down line transformer may be required if the unit is operated at a higher or lower line voltage.
- ... your TA Instruments thermal analysis controller.
- ... compressed lab air and purge gas supplies with suitable regulators and flow meters.

Away from
- ... dusty environments.
- ... exposure to direct sunlight.
- ... direct air drafts (fans, room air ducts).
- ... poorly ventilated areas.
- ... noisy or mechanical vibrations.

NOTE:

Drying out the instrument may be needed, if it has been exposed to humid conditions. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following method to dry out the instrument (refer to Chapter 4 for further information):

1 Ramp at 10°C/min to 400°C
2 Isothermal for 30 min.

Filling the Heat Exchanger

The heat exchanger contains a liquid reservoir that supplies the instrument with coolant to dissipate heat from the furnace. The coolant exits the heat exchanger through the supply line, circulates to the furnace, and comes back to the reservoir via the return line as seen in Figure 2.4 (for instructions on how to connect the water lines, turn to page 2-13). To fill the heat exchanger, follow the directions given below.

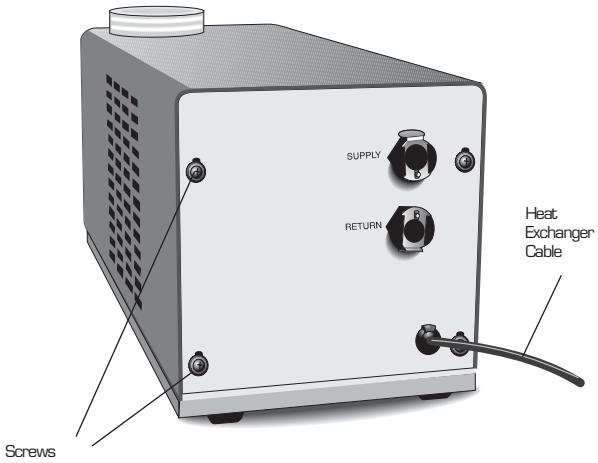

Figure 2.4 Rear Panel of Heat Exchanger

1. Unscrew the water reservoir cap on the heat exchanger bottle (see Figure 2.5).

Figure 2.5
Heat Exchanger Bottle

2. Pour one half bottle of TA Instruments TGA Conditioner (PN 952377.001) into the water reservoir bottle. Then fill the bottle to the inner rim (see Figure 2.5) with distilled water.

NOTE: After the system has been started, recheck the level of water in the reservoir bottle and refill to the inner rim with distilled water, if necessary.

♦ ***CAUTION:*** **Do not put any water other than distilled water in the heat exchanger reservoir.**

3. Replace and tighten the water reservoir cap.

Connecting Cables and Lines

To connect the cables and water and gas lines, you will need access to the TGA instrument's rear panel. All directional descriptions are written on the assumption that you are facing the back of the instrument.

NOTE: Connect all cables before connecting the power cords to outlets. Tighten the thumbscrews on all computer cables.

♦ ***CAUTION:*** **Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.**

!WARNING **Protect power and communications cable paths. Do not create tripping hazards by laying them across accessways.**

Heat Exchanger Cable and Water Lines

1. Locate the cooling accessory connector on the left rear of the instrument cabinet (Figure 2.6).

2. Connect the heat exchanger cable to the connector. The heat exchanger cable is the only cable that fits into this connector.

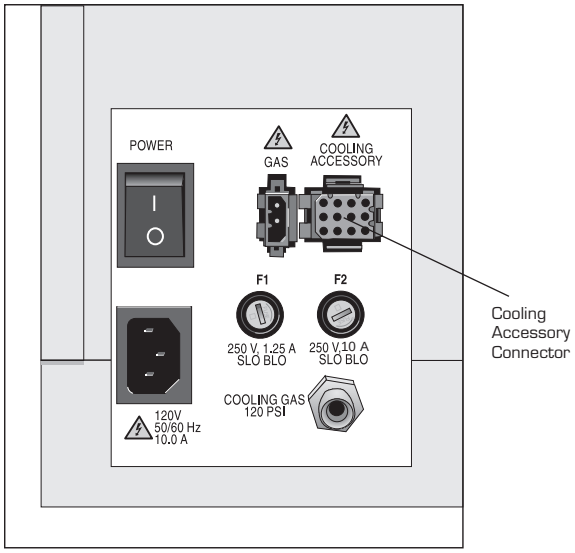

Figure 2.6
TGA Connector Panel

3. Remove the water lines from the packing.

4. Connect one end of the water line marked “SUPPLY” to the connector labeled “SUPPLY” on the left front of the instrument cabinet.

5. Connect the other end of the water line marked “SUPPLY” to the connector labeled “SUPPLY” on the heat exchanger.

6. Connect one end of the unmarked water line to the connector labeled “RETURN” on the left front of the instrument cabinet.

7. Connect the other end of the unmarked water line to the connector labeled “RETURN” on the heat exchanger.

   Figure 2.7 on the next page illustrates the correct water line connections for the TGA and heat exchanger.

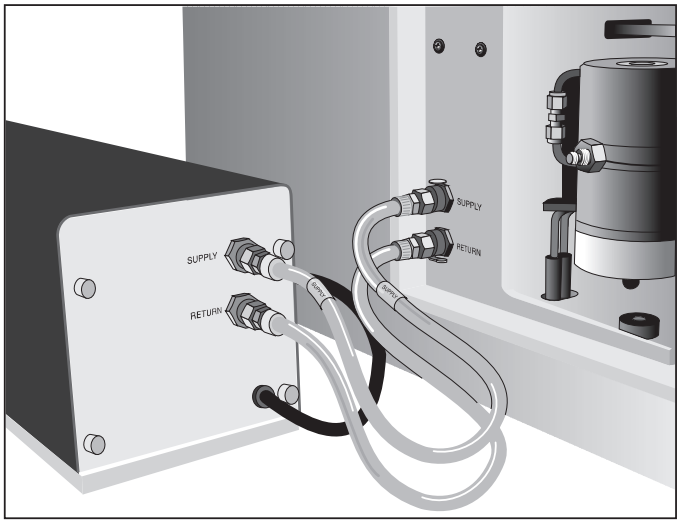

Figure 2.7 Heat Exchanger Water Line Connections

NOTE:

Air trapped in the heat exchanger system must be purged before starting the first run. After installation of the TGA is complete, turn on the instrument by placing the HEATER and POWER switches in the ON position. Then start the heat exchanger pump by turning on Air Cool from the controller. Refill the coolant reservoir as needed. Repeat this process untill all of the air has been purged from the system and the instrument stops reporting an "Err 119."

GPIB Cable

1. Locate the GPIB connector on the right rear of the TGA instrument (See Figure 2.9).

2. Connect the GPIB cable to the connector. The GPIB cable is the only cable that fits into the connector.

3. Tighten the hold-down screws on the connector.

4. Connect the other end of the GPIB cable to the controller or to the GPIB cable of another TA Instruments instrument connected to the controller.

5. Select an address from 1 to 9. Then use the binary address switches on the TGA connector panel to set the desired address (Table 2.1). Figure 2.8 shows a instrument address of 7.

NOTE: If you have a multi-instrument system, each instrument must have a different address.

If you change the address after the TGA is powered on, you must press the TGA's Reset button to enter the new address. Wait 30 seconds after releasing the Reset button; the yellow ready light should begin to glow steadily. Reconfigure the instrument.

Table 2.1
Binary Address Settings*

Address	Switch Pattern 1 2 3 4 5
1	0 0 0 0 1
2	0 0 0 1 0
3	0 0 0 1 1
4	0 0 1 0 0
5	0 0 1 0 1
6	0 0 1 1 0
7	0 0 1 1 1
8	0 1 0 0 0
9	0 1 0 0 1
*0 = OFF; 1 = ON	

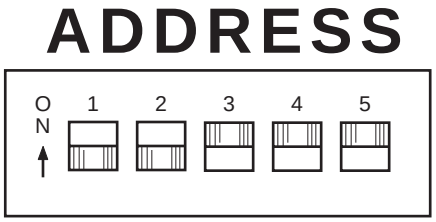

Figure 2.8
Binary Address Switches

Purge Lines

WARNING

Do not use any liquid in the purge lines.

1. Locate the PURGE and BALANCE PURGE fittings on the back of the TGA instrument (Figure 2.9).

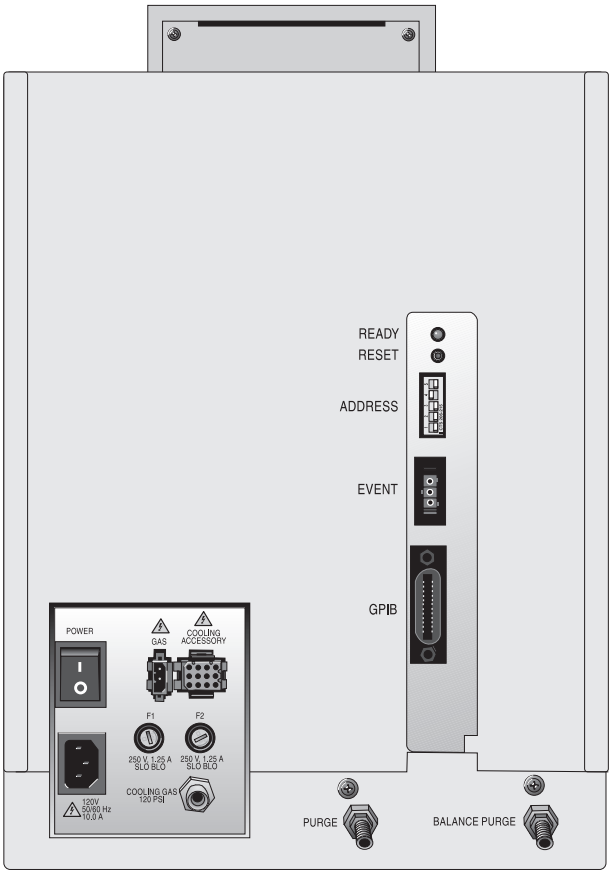

Figure 2.9
TGA PURGE Fittings

2. Make sure that the pressure of your purge gas source does not exceed the manufacturers' recommended pressures for flowmeters and other regulated devices you are using.

NOTE: If you are using laboratory purge, rather than bottled purge, you will need to install an external drier.

♦ ***CAUTION:*** **The use of corrosive gases in the TGA 2050 are not recommended.**

!WARNING **Use of an explosive gas as a purge gas is dangerous and is not recommended for this instrument. For a list of the purge gases that can be used with the TGA instrument, see Chapter 1. Oxygen may be used as a purge gas, but the furnace must be kept clean of volatile hydrocarbons to prevent combustion.**

3. Connect a length of 1/4-inch I.D. flexible tubing from each of the PURGE fittings to a flowmeter (consult your compressed gas vendor for specific requirements). Then connect each flowmeter to the purge gas source.

4. The recommended setting for the purge rate is 100 cc per minute or less, with a flow distribution of 10 percent to the balance chamber and 90 percent to the furnace.

Cooling Gas Line

1. Locate the COOLING GAS fitting, a 1/4-inch compression fitting on the left side of the TGA cabinet back, marked with a 120 psig maximum warning label (Figure 2.10).

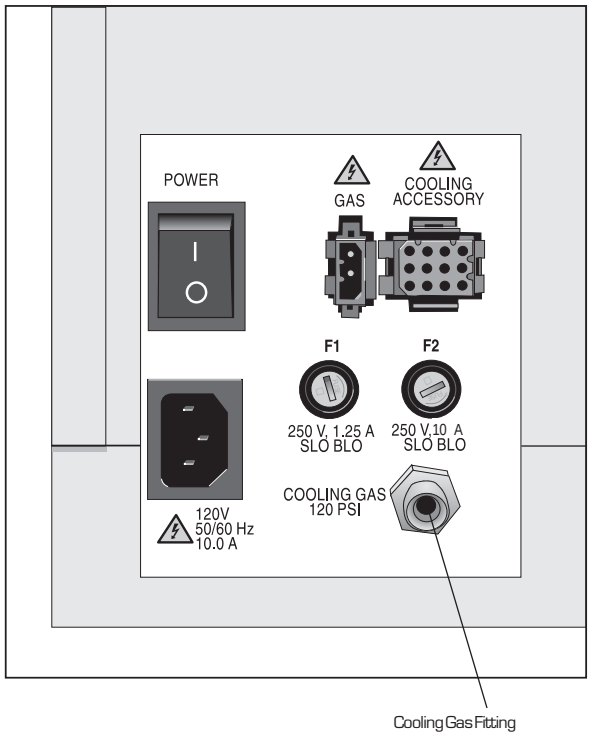

Figure 2.10 TGA COOLING GAS Fitting

2. Make sure your compressed lab air source is regulated to between 25 and 120 psig and is free of oil and water vapors.

3. Connect a compressed lab air line to the COOLING GAS fitting.

NOTE: Nitrogen may also be used as a cooling gas.

Power Cable

1. Make sure the TGA POWER switch, located on the back of the instrument, (Figure 2.11) is in the OFF position.

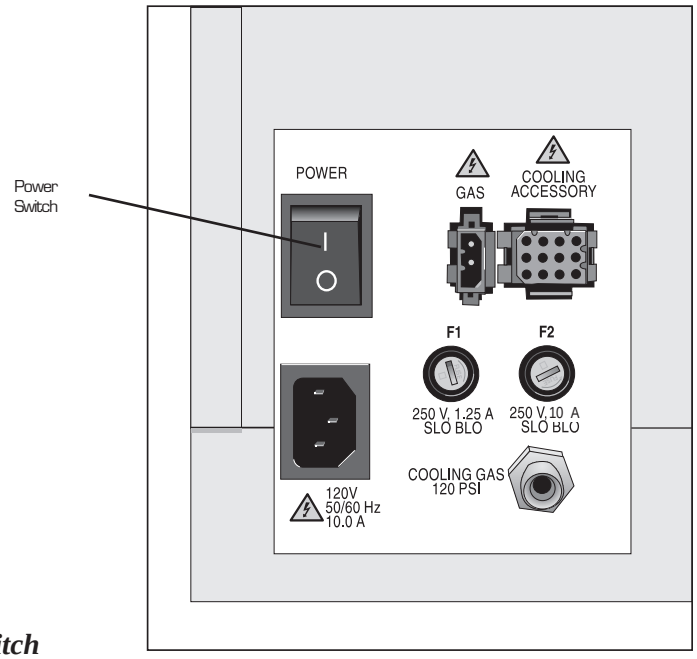

Figure 2.11
TGA POWER Switch

2. Plug the power cable into the TGA.

♦ ***CAUTION:*** **Before plugging the TGA power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.**

3. Plug the power cable into the wall outlet.

Unpacking the Balance

♦ ***CAUTION:*** **When unpacking the balance, be careful not to damage the balance arm or hang-down loops.**

1. Using the 7/64-inch ball driver supplied in your TGA accessory kit, loosen and remove the six screws securing the balance chamber faceplate to the instrument.

2. Take off the faceplate.

3. Loosen and remove the thumbscrew holding the balance cover on the sample (left) side of the balance mechanism (Figure 2.12), and take off the cover. Repeat this step for the tare (right) side of the balance mechanism.

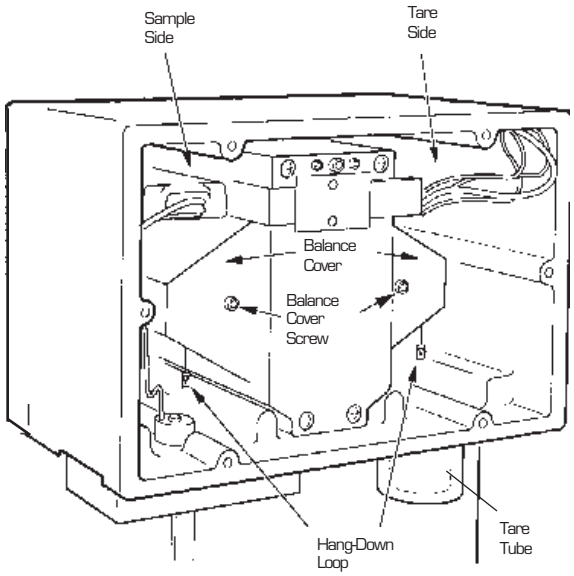

Figure 2.12 Interior of Balance Chamber Before Unpacking

4. Using tweezers, remove the foam insert from around the screw hole (Figure 2.13):

   a. Gently compress the foam with the tweezers, being careful not to touch the balance.

   b. Remove the foam insert from the balance chamber.

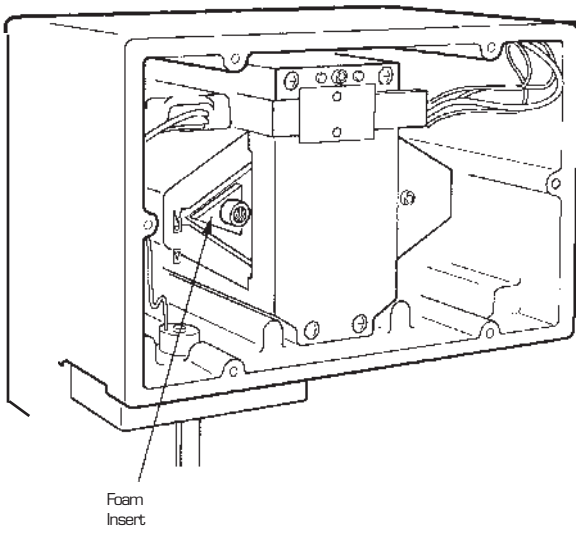

Figure 2.13 Removal of Foam Insert from Balance Chamber

5. Replace the sample side cover and screw.

6. Repeat the procedure to remove the foam insert in the tare (right) side of the balance.

Installing the Hang-Down Wires

♦ ***CAUTION:*** **During installation, take care not to bend the hang-down wires or damage the hang-down loops.**

1. Turn on the TGA instrument (see Chapter 3 for proper start-up order).
2. Press the FURNACE key to lower the furnace.
3. Locate the sample hang-down wire in your TGA Accessory Kit.
4. Hold the wire in your hand so that the doubly bent top hook is pointing to the left and the bottom hook is pointing to the right.
5. Carefully insert the bottom of the hang-down wire into the top of the furnace far enough so that you can insert the top of the wire into the hang-down tube without bending the wire (Figure 2.14 on the next page).

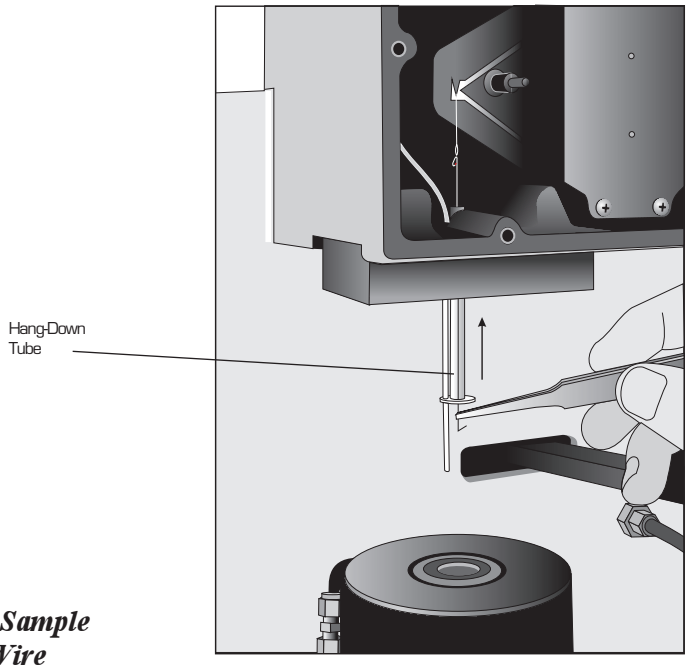

Figure 2.14
Installing the Sample Hang-Down Wire

6. Thread the hang-down wire up through the hang-down tube into the balance chamber, and hook the top of the wire over the top of the tube (see Figure 2.15 on the next page).

NOTE:

To make the hang-down loops easier to see, we suggest sliding a piece of white paper into the balance chamber behind each loop before you hook the hang-down wire into it. (Do not forget to remove the paper when finished.)

7. Grasp the top hook of the hang-down wire with brass tweezers. Being careful to keep the top hook pointing to the left, pass the double bend through the hang-down loop so the wire is hanging from the loop.

8. Unscrew and remove the tare tube.

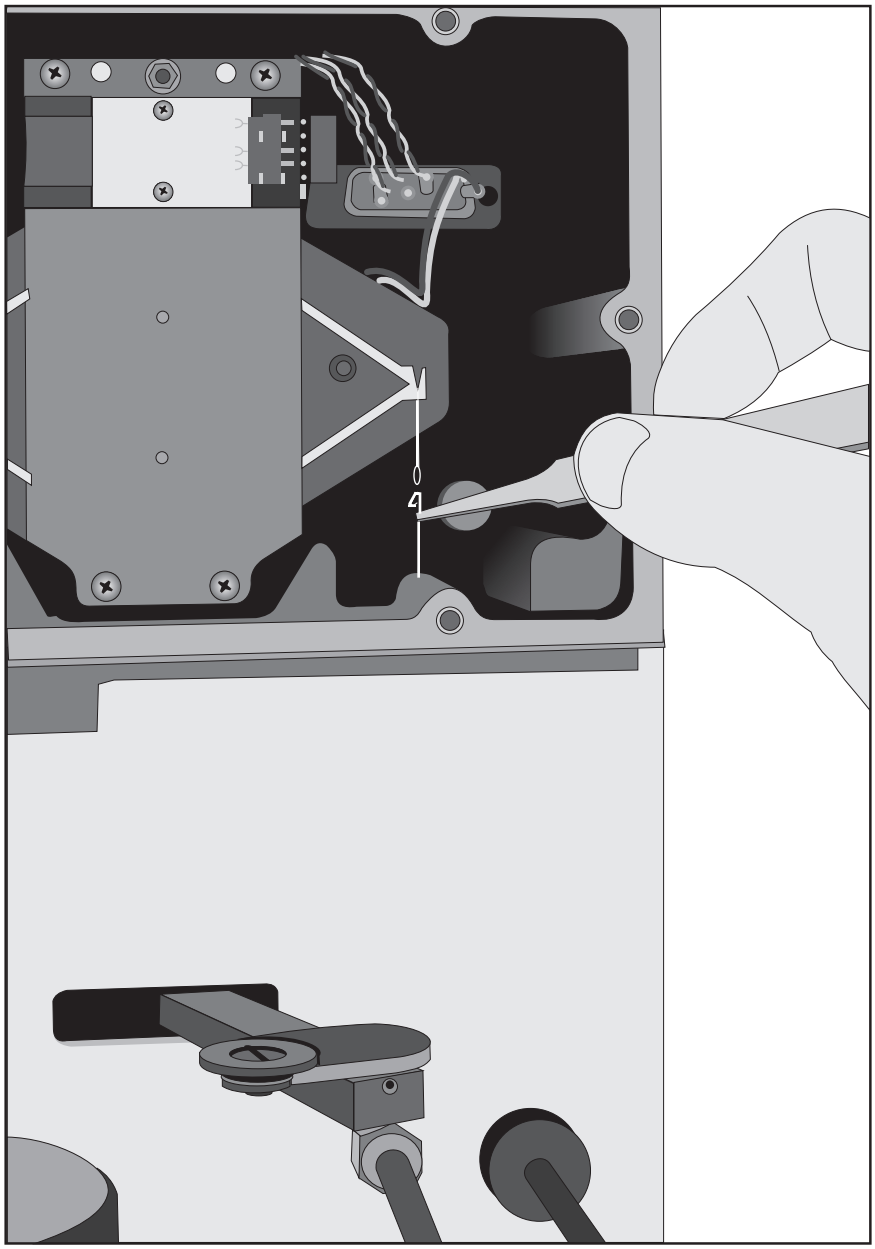

Figure 2.15
Installing the Tare
Hang-Down Wire

9. Locate the tare hang-down wire in your accessory kit.

10. Hold the wire in your hand so that the doubly bent top hook is pointing to the left and the bottom hook is pointing to the right.

11. Using brass tweezers, insert the tare hang-down wire into the balance chamber on the tare side and down through the hole above the tare tube connection, taking care not to bend the wire.

12. Being careful to keep the top hook pointing to the left, pass the double bend through the hang-down loop so the wire is hanging from the loop.

13. Select the sample pan you will use in your experiments and load one of the same size and type onto the tare hang-down wire.

14. Replace the tare tube and finger-tighten it to compress the O-ring seal.

    You are now ready to align the hang-down wires.

Aligning the Sample Hang-Down Wire

To avoid weight signal noise, the TGA instrument must be level so that the sample pan and hang-down wire hang inside the furnace and hang-down tube without touching them. The angle at which the pan hangs is very sensitive to slight irregularities in benchtop surfaces, so it is important that you select a sturdy table or bench for your TGA.

Once you have your TGA in a satisfactory location, adjust the top and bottom of the sample hang-down wire and level the instrument using the following procedures.

To align the top of the sample hang-down wire:

1. Place an empty sample pan on the sample platform.

2. Press the LOAD key on the instrument keypad. The TGA will automatically lower the furnace (if necessary), move the sample platform over to the furnace, and load the pan onto the balance.

   If the pan will not automatically load, place the pan manually (using brass tweezers) on the sample hang-down wire and continue with the procedure. Use the Sample Platform Adjust procedure (page 2-31) to correct loading after completing sample hang-down wire alignment.

3. Check to see whether the top end of the sample hang-down wire is hanging freely and roughly centered within the top of the hang-down tube inside the balance chamber.

4. If the wire is not roughly centered inside the hang-down tube, turn the balance adjustment screw (Figure 2.16) with the 7/64-inch ball driver until the wire is centered.

   Turning the balance adjustment screw clockwise will move the wire backwards; turning the screw counterclockwise will move the wire frontwards.

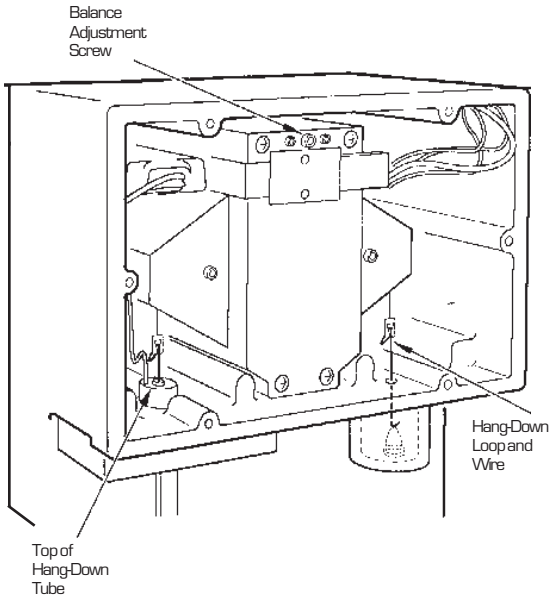

Figure 2.16
Location of Balance Adjustment Screw

To align the bottom of the hang-down wire:

1. Press the FURNACE key to raise the furnace just to the bottom of the sample pan, and press STOP.

2. Check the alignment of the sample pan within the furnace. It should hang freely, roughly centered, and should not be touching the sides of the furnace or the hang-down tube (Figure 2.17).

3. If the sample pan is not centered and hanging freely within the furnace, level the TGA instrument by adjusting the feet on the bottom. Turn the feet clockwise to lengthen or counterclockwise to shorten the legs. Continue adjusting until the pan hangs correctly.

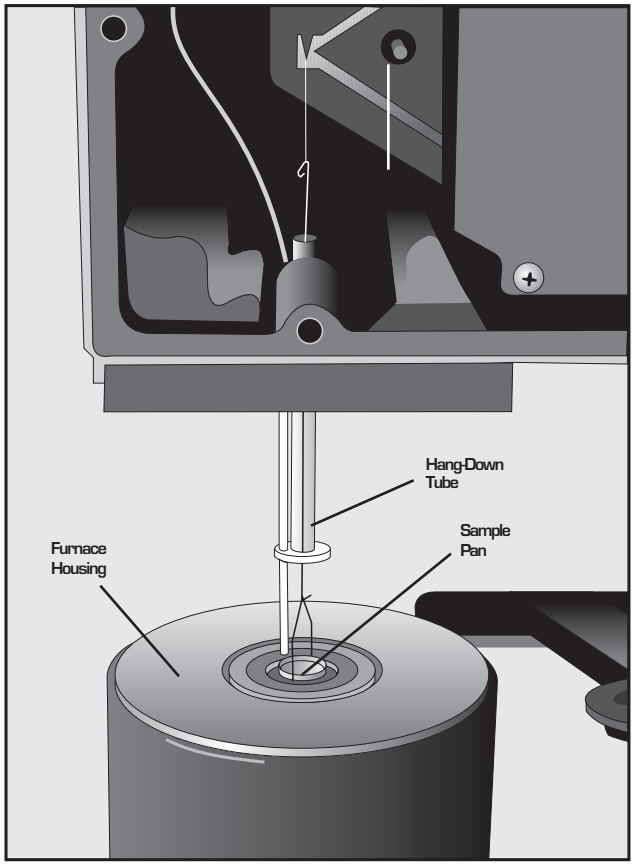

Figure 2.17 Aligning the Sample Pan in the Furnace

4. Press the FURNACE key to lower the furnace.

5. Press the UNLOAD key to remove the sample pan from the furnace.

6. Replace the balance chamber faceplate and its 6 screws.

   If you had to load the sample pan manually in order to align it in the furnace, you should now adjust the sample platform using the procedure described on the next page.

Adjusting the Sample Platform

If the sample hang-down wire fails to pick up a sample pan during an automatic loading procedure, you will need to adjust the position of the sample platform, using the Sample Platform Adjust procedure. This procedure is part of TGA instrument control, see the online help and documentation for further information.

Starting the TGA 2050

NOTE: Allow the TGA to warm up for at least 30 minutes before performing an experiment.

1. Check all connections between the 2050 and the controller. Make sure each component is plugged into the correct connector. (For installation information, see Chapter 2.)

2. Press the instrument power switch, located on the rear of the instrument, to the ON position. The green power light on the front of the instrument should turn on and the yellow Ready light should flash.

3. If the green power light fails to come on, recheck the power connections to the instrument and the power source. Also, check instrument power fuse F1 on the rear of the instrument to see if the fuse is blown.

Shutting Down the Instrument

Before you decide to power down your TGA instrument, consider the following:

- All of the components of your thermal analysis system are designed to be powered on for long periods.
- The electronics of the TGA and the controller perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, turning the system and its components on and off frequently is discouraged.

When you finish running an experiment on your TGA and wish to use the thermal analysis system for some other task, leave the instrument on; it will not interfere with whatever else you wish to do.

If you do need to power down your instrument for any reason, simply press the POWER switch to the OFF (0) position.

CHAPTER 3: Running Experiments

Overview

All of your TGA experiments will have the following general outline. In some cases, not all of these steps will be performed.

- Entering experiment information through the TA controller (sample and instrument information)
- Creating and loading the thermal method on the controller
- Attaching and setting up external accessories as required (*e.g.,* purge gas, air cool, gas switching accessory)
- Selecting and taring the sample pan
- Loading the sample
- Starting the experiment
- Unloading the sample at the end of the experiment.

To obtain accurate results, follow procedures carefully and check calibration periodically (once a month). Consult the online help and documentation for details on calibrating the TGA and conducting experiments.

Before You Begin

Before you set up an experiment, ensure that the TGA and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections between the TGA and the controller
- Connected heat exchanger cable and water lines
- Connected all gas lines
- Powered on each unit
- Installed all appropriate options
- Configured the instrument online with the controller
- Become familiar with controller operations
- Calibrated the instrument, if necessary.

Calibrating the TGA

The TGA 2050 requires two kinds of calibration: temperature calibration and weight calibration. Both of these calibrations should be performed when you first install the instrument and at least once a month thereafter. Consult the see the online help and documentation for further information.

Temperature Calibration

Temperature calibration involves analyzing a material for its Curie Temperature, and comparing the observed value with the standard literature value. The standard most often used is nickel with a curie temperature of 354.4°C (NIST Certificate for GM761). The Curie Temperature corresponds to the extrapolated endpoint on the "s-shaped" thermal curve generated utilizing a magnet.

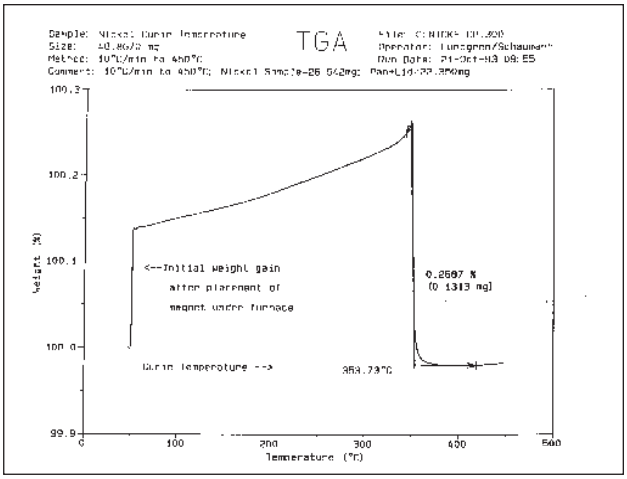

Figure 3.1
Curie Temperature Determination

Weight Calibration

TGA weight calibration uses standard reference weights to calibrate the accuracy of the TGA balance system. This calibration is conducted through the controller.

Running a TGA Experiment

Experimental Procedure

All of your TGA experiments will have the following general outline.

- Selecting and preparing a sample. This involves preparing a sample of the appropriate size, selecting the sample cup, and placing the sample in the cup.
- Taring and loading the sample cup on the balance.
- Entering experimental information through the TA controller.
- Creating and selecting the thermal method on the controller.
- Setting up any external accessories.
- Starting the experiment.

Preparing the Sample

Selecting Sample and Tare Pans

Three kinds of sample pans are available for the TGA 2050: platinum, alumina ceramic, and aluminum. The platinum pans come in 50 and 100 μL sizes, the ceramic pans come in 100, 250, and 500 μL sizes, and the aluminum pans are 100 μL in size. The criteria for choosing a sample pan are as follows:

- For most experiments, platinum is the desirable choice. It is easy to clean and does not react with most organics and polymers. Ceramic pans are more porous and are therefore more easily contaminated. There are some conditions, however, in which other types of pans are desirable, as explained below.

- Use ceramic pans for samples that might amalgamate or react with platinum (*e.g.*, metals, corrosives, inorganics).

- Use aluminum pans when disposability is desired. Aluminum pans are meant for one time use in experiments that do not go above 600°C and for samples that do not react with aluminum (*e.g.*, corrosives, acidic materials, or anything that would alloy with aluminum).

- If your sample will melt during the experiment, use a pan that is deep enough to prevent spilling (the deepest pan is the 500 μL ceramic pan).

The platinum and ceramic pan types are reusable. To clean between experiments, use a Bunsen burner or a propane torch, or run the pan through a hot thermal program in the TGA to burn out any residue. Aluminum pans are disposable, do not attempt to clean and reuse them.

Once you have selected the proper sample pan, remove the tare tube, and using brass tweezers, put the same type and size pan on the tare hook.

NOTE: Whenever a different type of pan is selected, the balance must be mechanically tared using step 1 of the weight calibration.

Taring the Sample Pan

Taring the sample pan ensures that the weight measured by the balance reflects the weight of the sample only. You should tare the sample pan before each experiment, even if you use the same pan in consecutive experiments.

When you tare a pan, the TGA reads the weight of the empty pan and then stores the weight as an offset, which is subtracted from subsequent weight measurements. For optimum accuracy, the weight reading must be stable before it is accepted as an offset. If you use the automatic tare procedure, the TGA will determine when the weight reading is sufficiently stable; or you can determine the acceptability of the weight reading by taring the system manually. Both tare procedures are explained here.

Automatic Tare

Because the TGA 2050 has two weight ranges, taring is done for both ranges. The tare weight is stored by the instrument for both weight ranges.

1. Place the empty sample pan on the sample platform.

2. Press the TARE key on the instrument keypad or from the controller. The TGA will automatically load the pan, raise the furnace (to protect the pan from air currents), weigh the pan, store the weight as the offset for each weight range, and unload the pan.

Manual Tare

Manual tare operates in the weight range indicated, by storing the current reading as an offset, and estimates the tare weight for the other weight range (typically the 1-gm range). The estimate is accurate if the TGA 2050 has been tared or weight-calibrated recently.

1. Place the empty sample pan on the sample platform.

2. Press the LOAD key to load the pan onto the balance.

3. Press the FURNACE key to close the furnace, to protect the pan from air currents.

4. Observe the weight reading on the controller's Signal Display window screen (Signal A Weight).

5. Wait for the Signal A Weight to stabilize, and then choose **Auto Zero** to store the displayed weight as the offset.

6. Press the UNLOAD key to open the furnace and unload the pan.

Loading the Sample

After taring the sample pan, load the sample into the TGA furnace as follows:

1. Place the sample in the sample pan, and position the pan on the sample platform (Figure 3.2).

   The wire on the bottom of the sample pan should align with the groove in the panhole, so that the sample pan can be picked up by the sample hang-down wire.

NOTE: Always use brass tweezers to handle the sample pans.

♦ ***CAUTION:*** **Manually loading the sample pan onto the hang-down wire may damage the balance mechanism.**

Figure 3.2 Sample Pan Ready to Load

2. Press the LOAD key. The TGA will automatically load the sample pan onto the balance.

3. Position the thermocouple at the edge of the sample pan, rather than in the middle, for best results (Figure 3.3).

NOTE: The position of the thermocouple should be the same as it was during temperature calibration.

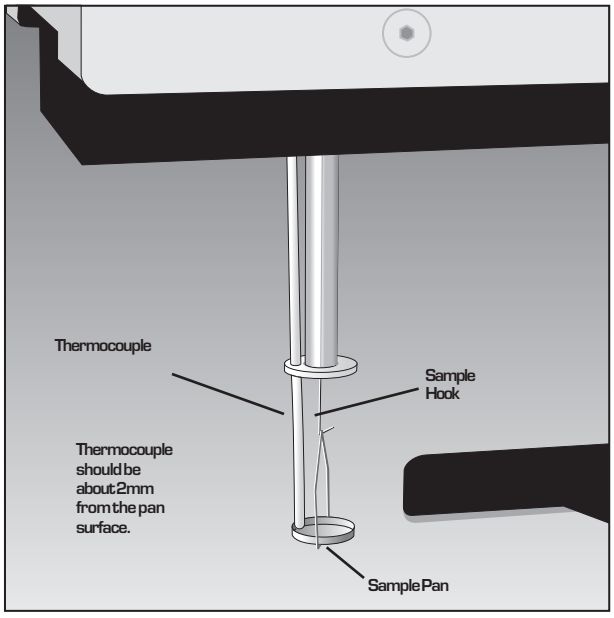

Figure 3.3 Adjusting the Thermocouple

4. Press the FURNACE key to close the furnace by moving it up around the sample, until it is fully closed as shown in Figure 3.4.

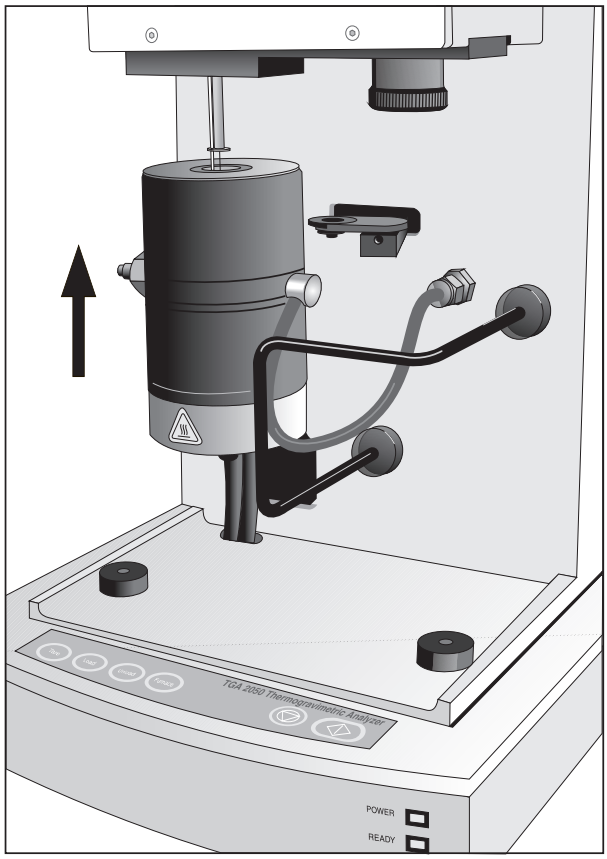

Figure 3.4
Furnace Closing

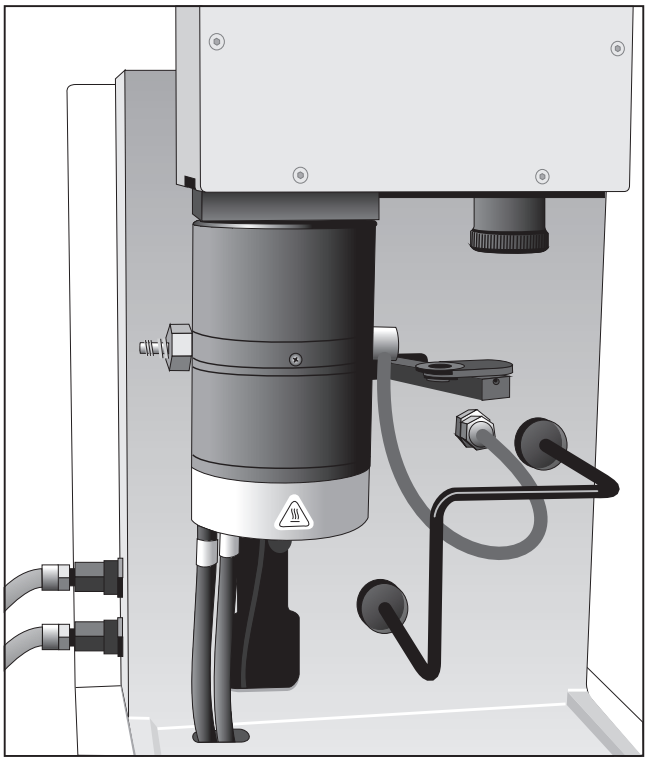

Figure 3.5
Furnace in
Fully Closed Position

Setting Up an Experiment

Once you have prepared the sample, the next step in your experiment is to enter the needed information in the TA controller. All of the controller functions described in this section are accessed through the Instrument Control screen. Refer to the online help and documentation to learn how to perform the following steps.

1. **Select the Instrument.**

2. **Select the Instrument Mode.**

3. **Enter Sample Information.**

4. **Enter Instrument Information.**

5. **Create and Select Thermal Methods.**

The first time you use your TGA you will need to create at least one thermal *method* to control experiments. Each method is made of several *segments*, or individual instructions (*e.g.*, Equilibrate, Ramp), that control the state of the instrument.

Setting Up Accessories

If your experiment requires external accessories, ensure that they are turned on, and make any necessary adjustments before you start your experiment. Make sure that the system can achieve the conditions of all segments in the method.

This section describes how to use the following accessories with the TGA 2050:

- Air cool option
- Purge gas
- TA Instruments Gas Switching Accessory.

The TGA can also be used with other accessories, such as vacuum, FTIR, gas chromatographs, mass spectrometers, and evolved gas analyzers. Consult the appropriate local instrument manufacturer for further information.

Using a Purge Gas

You can control the sample atmosphere during TGA experiments by connecting a purge gas to the system. Purge gas is distributed separately to two parts of the TGA: the furnace and the balance chamber.

The balance purge maintains a positive pressure in the balance chamber to prevent decomposition products from contaminating the sensitive balance mechanism. The balance purge flows from the balance chamber via two routes: down

the hang-down tube and through an outlet in the balance chamber to the right of the hang-down tube. It then exits across the sample pan along with the furnace purge.

The purge flow through the furnace is horizontal to the sample (Figure 3.6), permitting rapid removal of decomposition products from the sample environment.

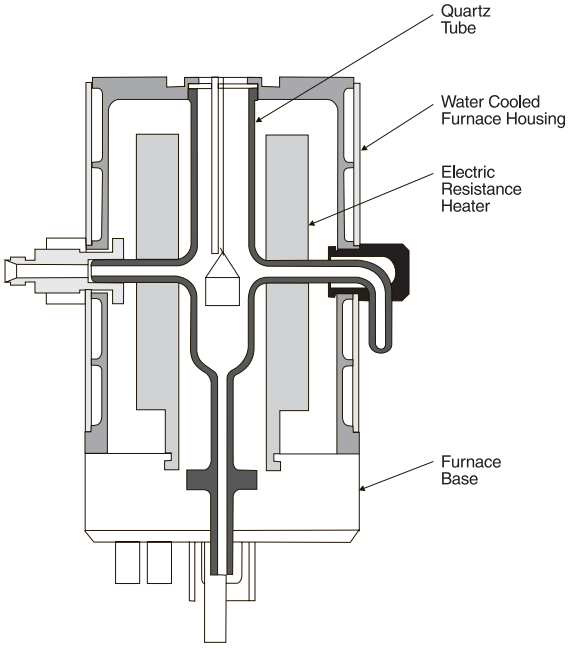

Figure 3.6
Furnace Purge

You can choose nitrogen, oxygen, helium, air, or argon for your purge environment. Do not use any other gases in the TGA 2050.

WARNING

Do not use hydrogen or any other explosive gas in the TGA 2050 furnace. Oxygen may be used as a purge gas, but the furnace must be kept clean of volatile hyrdrocarbons to prevent combustion.

WARNING

Do not use any liquid in the purge lines.

Purge gas can be obtained from a pressurized cylinder or an in-house supply source. Gas supplied from an in-house source should be passed through a sieve dryer to remove any trace of moisture before in enters the TGA.

It is important to maintain the proper ratio of flow rates between the balance chamber and the furnace housing. Having the separate balance chamber purge prevents decomposition gases from entering the balance chamber. The recommended setting for the purge rate is 100 cc per minute or less. The flow distribution should be 10 percent to the balance chamber and 90 percent to the furnace.

To maintain this flow distribution, you will need to connect a flowmeter to each of the purge fittings on the back of the TGA instrument. Set the purge gas flow rates by adjusting these meters. The PURGE port goes to the TGA furnace, and the BALANCE PURGE goes to the balance chamber.

Before you start the purge gas, make sure that the desired gas is connected to the purge ports, that all lines are clear, and that your supply of purge gas is sufficient for the experiment. Always maintain constant purge flow rates and distribution throughout your experiment; changing the purge during an experiment can affect the data.

Using the Air Cool Option

You can program the system to air cool the furnace automatically at the end of the experiment by selecting this option from the TA controller. After the air cool is activated, it will continue to run for the desired time.

Before you start an experiment that uses the air cool option, ensure that the supply valve from the air source is open and that the pressure is regulated to between 25 and 120 psig. Nitrogen can also be used as a cooling gas.

NOTE: Air Cool can be used with the furnace closed. However, if the temperature is above 500°C, the furnace will cool naturally until it is 500°C or less, then Air Cool will begin.

Using the Gas Switching Accessory

You can use the Gas Switching Accessory to turn the purge gas on and off or to switch between two different purge gases during a TGA experiment. Before starting an experiment that uses the Gas Switching Accessory, make sure its power switch is on, and make sure the necessary gas sources are properly connected.

The Gas Switching Accessory can be controlled by the Gas segment in the method (see the online help and documentation) or by the Gas control option.

Connect the Gas Switching Accessory to the purge port only, when switching between gases during an experiment. Attach the inert gas to GAS 1 and the other gas to GAS 2.

Consult your Gas Switching Accessory operator's manual for further instructions.

Starting an Experiment

You can begin the experiment using either the START key on the TGA instrument keypad or select **Start** on the controller. When you start, the system automatically begins the set-up procedures. It loads the sample pan and closes the furnace, if necessary, and then begins the selected method and data collection starts.

If you wish to start collecting data during instrument setup, you can use the *forced start* feature. This is most useful for samples that loose a significant amount of weight during the set-up period (*i.e.*, samples with volatile solvents). When a forced start is initiated, the current sample weight is stored as the initial weight, data collection is started immediately, and the instrument status changes from "Set Up" to "Started." The method begins when the normal set-up procedures are completed.

NOTE:

Once the experiment is started, operations are best performed at the controller keyboard. The TGA 2050 is very sensitive to motion and might pick up the vibration caused by pressing a key on the instrument keypad.

Forced Start

Press the START key on the instrument keypad *while* the controller status line displays "Set Up."

Stopping an Experiment

If for some reason you need to discontinue the experiment, you can stop it at any point by using either the STOP key on the TGA instrument keypad or **Stop** on the controller screen. Another function that stops the experiment is **Reject** on the controller. However, the Reject function discards all of the data from the experiment; the Stop function saves any data collected up to the point at which the experiment was stopped.

NOTE: The Heat Exchanger will continue to run as long as the Air Cool option is activated or until the indicated temperature is below 50°C.

♦ ***CAUTION:*** **The REJECT function discards all experiment data.**

Unloading the Sample

If you select the "Furnace Open and Unload" method-end option on the controller, the TGA will automatically unload the sample at the end of the run. If you need to unload the sample manually, wait until the run and all method-end operations are complete, and then press the UNLOAD key. The sample pan may not line up with the sample platform groove at method end.

Use in an Oxygen-Free Atmosphere

A few extra precautions are necessary to ensure an oxygen-free environment.

Purge Gas System

- Use a high purity, inert gas of grade 5 or better. It may be necessary to use an oxygen trap in-line, depending on the purity grade.
- Choose a 2-stage regulator of diaphragm construction for high purity applications.
- Use copper or stainless steel tubing from the gas regulator, to the flowmeters, and to the TGA purge inlet ports.
- Allow the TGA to prepurge (under closed conditions) for at least 30 minutes after first turning on your purge gas. Increase the standard purge rate during this time.

  100 cc/minute flow into the balance chamber and 100 cc/minute into the furnace.

Instrument Setup

- Readjust your flowmeter(s) for standard operating flow rates.

  10 cc/minute flow into balance chamber and 90 cc/minute into the furnace.

- Tare sample pan (as needed).

- Load sample and close system

- Purge 20 minutes, if possible, before starting a run.

- When run is complete, allow the furnace to cool in the closed position. This can be done by changing the method-end conditions to leave the furnace closed at method end.

- When using air or oxygen during an experiment, introduce new gas through furnace purge port only and switch back to the inert gas before cooling down.

NOTE: When the TGA is idle, leave the system closed and continue purging with the inert gas.

CHAPTER 4: Technical Reference

Description of the TGA 2050

The TGA 2050 operates on a null balance principle. Physically attached to a taut-band meter movement, the balance arm is maintained in a horizontal reference position by an optically actuated servo loop. When the balance is in a null position, a flag located on top of the balance arm blocks an equal amount of light to each of the photodiodes (the light is supplied by a constant current infrared LED). As sample weight is lost or gained, the beam becomes unbalanced, causing an unequal amount of light to strike the photodiodes. The unbalanced signal, called the error signal, is acted upon by the control circuitry and reduced to zero, or nulled. This is accomplished by an increase or decrease in the current to the meter movement, causing it to rotate back to its original position (null position). The change in current necessary to accomplish this task is directly proportional to the change in mass of the sample. This current is converted to the weight signal.

The TGA 2050 has two weight ranges: 1 gm and 100 mg. Both ranges are continuous over their weight loss operating range, which means that the entire weight loss range can be viewed without any loss of information. The weight loss operating ranges are:

- 1 gm to 0 μg for the 1 gm range
- 100 mg to 0 μg for the 100 mg range.

Negative weight (tare imbalance) is limited to 150 mg in the 1 gm range, and to 15 mg in the 100 mg range. Range control is automatic.

During normal operation of the TGA, the sample may evolve gases. To prevent back diffusion of these liberated gases to the balance chamber, the balance chamber is purged with an inert gas (10 cc/min). An inert gas must be used to prevent contamination or corrosion of the balance.

Heating rate and sample temperature are measured by the thermocouple located above the sample. This enables the controller to program and maintain the sample environment at your selected rate.

Components

The TGA 2050 has five major components, illustrated in Figure 4.1: the balance, the sample loading assembly, the furnace, the cabinet, and the heat exchanger.

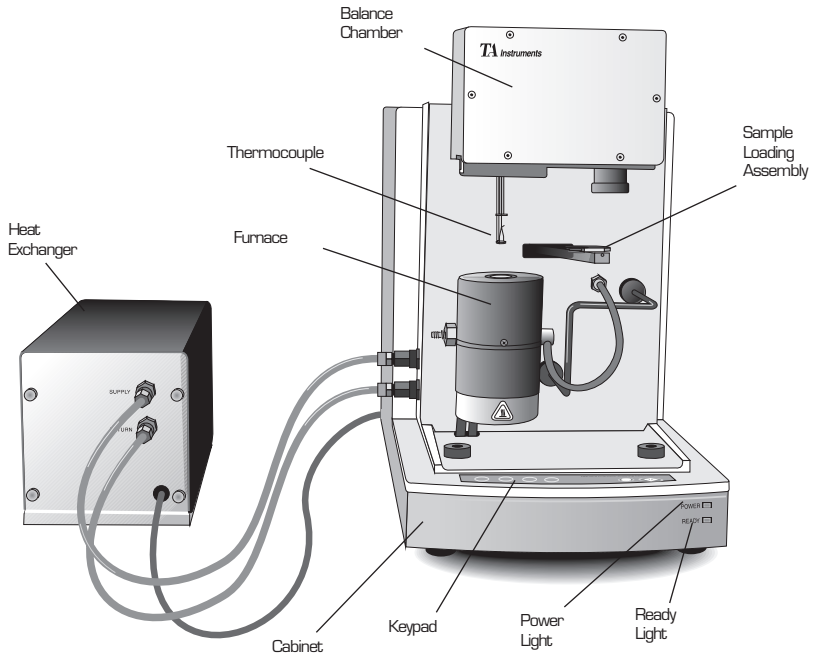

Figure 4.1
TGA 2050 Components

The **balance**, the most important part of the TGA system, provides precise measurement of the sample weight. The **sample loading assembly** automatically loads and unloads samples from the TGA balance. The **furnace** controls the sample atmosphere and temperature. The **cabinet** contains the system electronics and mechanics. The **heat exchanger** dissipates heat from the furnace.

Balance

The TGA balance assembly (shown in Figure 4.2 below) consists of the balance meter movement, the balance arm, the balance arm sensor, the hang-down wire assemblies, the sample pan, and the tare pan.

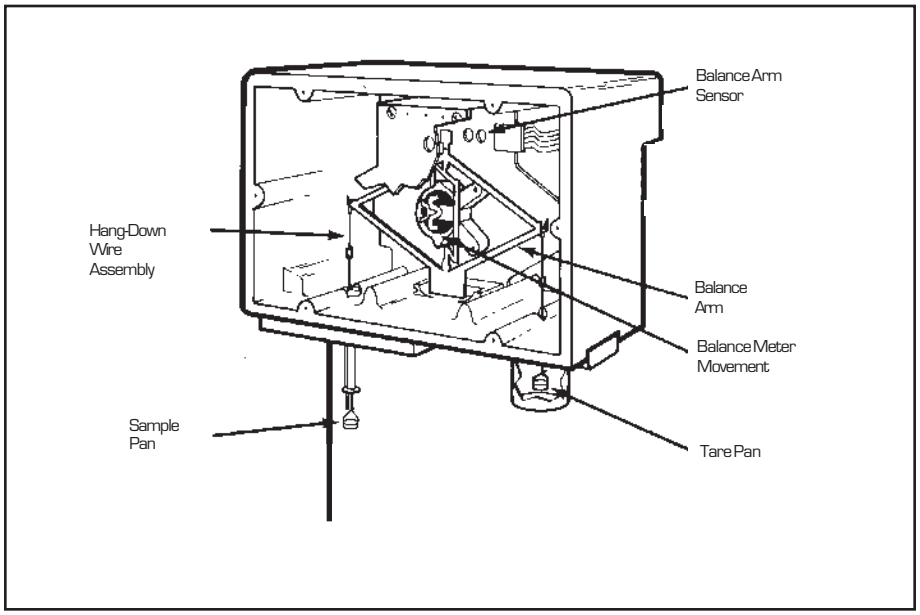

Figure 4.2
TGA Balance Assembly

The **balance meter movement** is a taut-band meter movement to which the balance arm is attached.

The **balance arm** is a rhombic piece of aluminum attached to the meter movement. It is in a null balance system. A hang-down loop is attached to each end to hold the hang-down wires.

The **balance arm sensor** is a printed circuit board assembly that detects the null position of the meter movement. The balance beam sensor is mounted above the balance arm. It is used in conjunction with the analog circuitry to maintain a null position.

The TGA has two **hang-down wire assemblies:** one for the tare pan and one for the sample pan. Each assembly consists of a hang-down wire and loop. The hang-down wire has hooks at each end and connects the pan to the loop. The loop has eyelets at each end; it is used to connect the hang-down wire to the balance arm. The longer hang-down wire (4 inches) is for the sample pan.

Sample pans are available in platinum in 50 and 100 μL sizes, alumina ceramic in 100, 250, and 500 μL sizes, and aluminum in 100 μL size. All pans are 0.4 inch in diameter.

The **tare pan** holds the counterbalance weight that mechanically subtracts out the weight of the sample pan.

Sample Loading Assembly

The sample loading assembly (Figure 4.3) is a platform that pivots the sample pan to the furnace area, where the pan engages the hang-down wire from the balance assembly. It also pivots the platform away from the furnace area for easy sample loading and unloading.

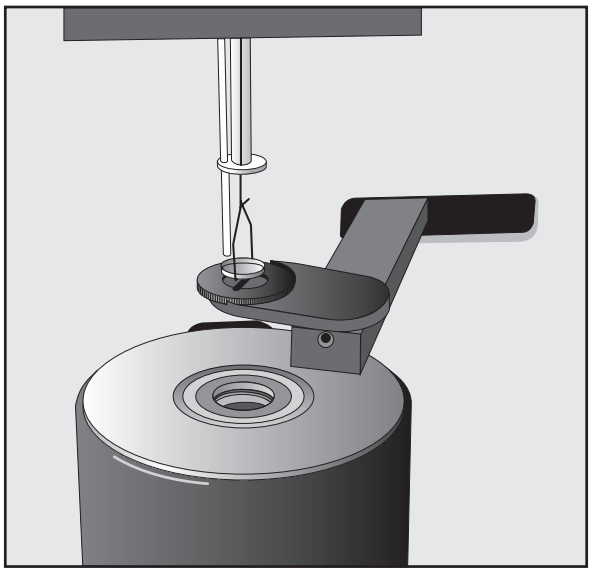

Figure 4.3 Sample Loading Assembly

Furnace

The TGA furnace consists of a quartz glass sample tube surrounded by an electric resistance heater, both of which are contained within a water-cooled furnace housing. The housing is mounted to a furnace base that raises and lowers the furnace for sample loading and unloading. See Figure 4.4.

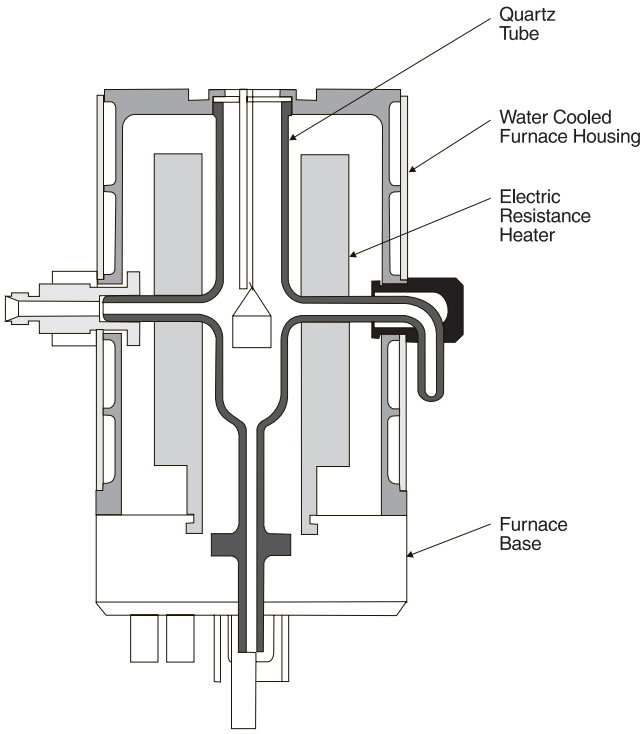

Figure 4.4 Furnace

The **sample tube** has a purge gas inlet that passes through the right side of the furnace housing. A fitting on the left side of the housing allows connection of a transfer line to carry exhaust gas to a spectrometer such as a FTIR. Because the heater is external to the sample tube, evolved gases from sample decomposition within the sample tube do not come in contact with the resistance elements or the furnace ceramic refractory.

Cooling air enters through the furnace base and passes upward between the outside of the sample tube and the inside of the furnace, completely separating the cooling air from the sample and the sample zone.

The **furnace** is a resistance heater wound on alumina ceramic, which allows sample zone temperatures as high as 1000°C with heating rates up to 50°C/min.

A Platinel II* **thermocouple** is positioned in the furnace, just above the sample pan, where it monitors the sample environment temperature.

The **furnace base** moves the furnace assembly up around the sample pan to the closed position, or down away from the sample pan to the open position.

* Platinel II is a registered trademark of Engelhard Industries.

Cabinet

The TGA cabinet (Figure 4.5) consists of the cabinet housing, the keypad, the electronics compartment, the purge and cooling gas fittings, and the rear panel.

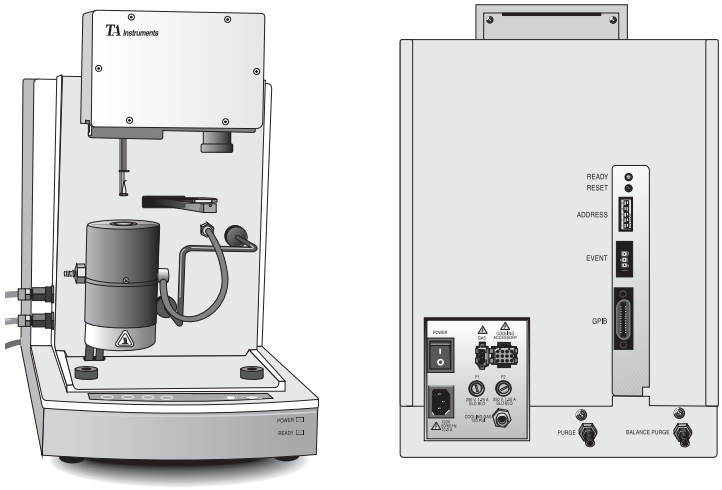

Figure 4.5
TGA Cabinet Components

The TGA **cabinet housing** consists of a base and a rear cover. The base is a single piece of heavy-weight aluminum, designed to provide a stable platform for the TGA instrument parts.

The TGA **instrument keypad** is described in Chapter 1.

The **electronics compartment** contains the electronics that control the instrument functions.

Two **purge fittings** are located on the back of the instrument, one for the balance chamber and one for the furnace housing.

The **cooling gas fitting** is located on the left side of the instrument back and is for furnace cool-down air.

The rear panel (Figure 4.6) has the signal and power connections for the instrument; the fuses; the ready light; the POWER switch; the address switches; the purge, gas, cooling accessory, and GPIB connections; and the Reset button.

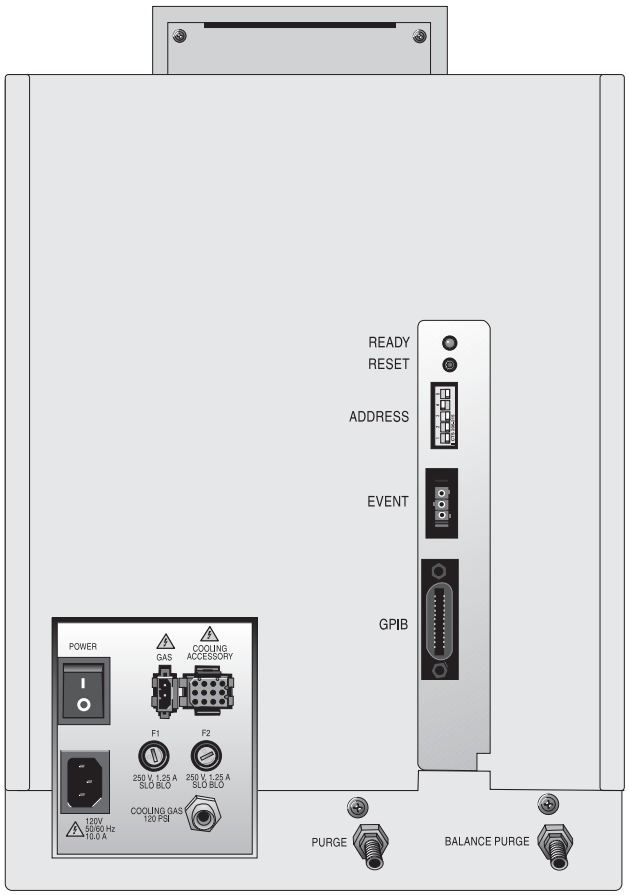

Figure 4.6
Rear Panel

Heat Exchanger

The heat exchanger (Figure 4.7) consists of a fan, a radiator, a water reservoir, a pump, a temperature switch, and a flow switch.

The **fan** blows cool air through the radiator.

The **radiator** exchanges heat between the water and air.

The **water reservoir** holds additional water that may be required by the system.

The **pump** circulates the water through the system.

The **temperature switch** detects over-temperature conditions, which could be caused by failure of the fan.

The **flow switch** detects lack of flow, which could be caused by failure of the pump or a leak or blockage in the system.

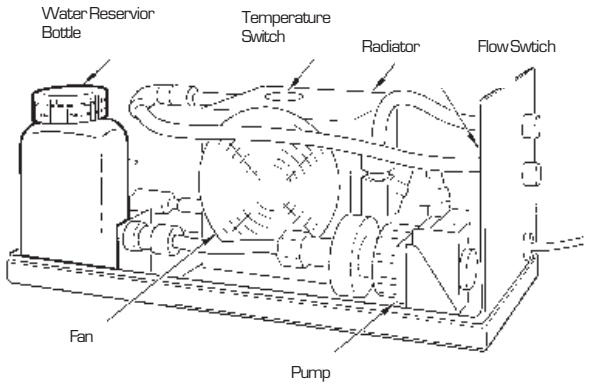

Figure 4.7
Heat Exchanger

Theory of Operation

Thermogravimetric analysis (TGA) is a thermal analysis technique for measuring the amount and rate of change in sample mass as a function of temperature and time. It is used to characterize any material that exhibits weight loss or phase changes as a result of decomposition, dehydration, and oxidation. Two modes are commonly used for investigating thermal stability behavior in controlled atmospheres: (1) dynamic, in which the temperature is increased at a linear rate, and (2) isothermal, in which the temperature is kept constant.

Status Codes

Status codes are continuously displayed at the top of the controller screen.

Table 4.1
Method Status Codes

Code	Meaning
Air Cool	The furnace air cool line has been opened to cool the furnace.
Calib	The TGA Calibration program is running.
Closing	The furnace assembly is closing.
Cold	The instrument heater cannot supply heat fast enough to keep up with the thermal program. This may be caused by a large ballistic jump in the program, a faulty heater, or a faulty control thermocouple signal.
Complete	The thermal method has finished.

(table continued)

Table 4.1
Method Status Codes
(continued)

Code	Meaning
Cooling	The heater is cooling, as specified by a Ramp segment.
Ending	The method is complete and the furnace is cooling until it can Air Cool or open and unload.
Equilb	The temperature is being equilibrated to the desired set point.
Heating	The heater temperature is increasing, as specified by a Ramp segment.
Holding	Thermal experiment conditions are holding; the program is suspended. Select **Resume** to continue the run.
Hot	The temperature is beyond the set point, and the instrument cannot remove heat fast enough to follow the thermal program. This is usually caused by a large ballistic jump to a lower temperature.

(table continued)

Table 4.1 (continued)

Code	Meaning
Initial	The temperature is being equilibrated to the desired set point. When the temperature has reached equilibrium, the status will change to "Ready."
Iso	The thermal program is holding the current temperature isothermally.
Jumping	The heater is jumping ballistically to the set point temperature.
Load	The TGA is loading a sample pan onto the balance.
No Power	No power is being applied to the heater. Check the heater fuse.
Opening	The furnace assembly is opening.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Use the **Start** function to continue the method.

(table continued)

Table 4.1
Method Status Codes
(continued)

Code	Meaning
Reject	The experiment has been terminated and the data erased.
Repeat	The method is executing a repeat loop that does not involve temperature control segments.
Set Up	The system is loading the sample, closing the furnace, and letting the weight stabilize before beginning the first segment.
Stand by	The method and method-end operations are complete.
Started	The TGA is still setting up to start the experiment (see **Set Up** above), but the initial weight has been measured and data collection has begun. The thermal method will start when the normal setup process has been completed.

(table continued)

Table 4.1 (continued)

Code	Meaning
Tare	The TGA is measuring the weight difference between an empty sample pan and the tare pan. The measured weight is used as an offset so that the displayed weight value indicates the weight of the sample only.
Temp	The heater is in stand-by mode, and the experiment has been terminated.
Unload	The TGA is unloading a sample from the balance.

CHAPTER 5: Maintenance and Diagnostics

Overview

The procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel.

WARNING

Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

Routine Maintenance

Inspection

Examine the instrument periodically to keep it free of dust, debris, and moisture. Keep the furnace area clean. Any sample spillage of residue should be removed before the next experiment.

Cleaning the Instrument Keypad

You can clean the TGA instrument keypad as often as you like. The keypad is covered with a silk-screen Mylar* overlay that is reasonably water resistant but not waterproof or resistant to strong solvents or abrasives.

A household liquid glass cleaner and paper towel are best for cleaning the instrument keypad. Wet the towel, not the keypad, with the glass cleaner, and then wipe off the keypad.

Cleaning the Quartz Furnace Tube

!WARNING

If the TGA is used to evaluate materials using an oxygen purge, the furnace should be cleaned routinely to prevent build-up of volatile hydrocarbon residues that could combust.

!WARNING

Do not touch the furnace sample tube with your bare fingers. Skin oils may cause devitrification of the quartz glass, resulting in severely reduced sample tube life.

WARNING

Do not insert metallic instruments inside the sample tube to scrape or chip contaminants from the sample tube as breakage may result.

*Mylar is a registered trademark of the DuPont Company.

To clean the furnace quartz sample tube, use the following procedure:

WARNING

Do not disturb the hangdown wire and furnace thermocouple located directly above the furnace when cleaning the furnace, as damage may result.

1. Press the FURNACE key to open the furnace completely.

2. Remove any sample pans.

3. Remove the rubber cap located on the underside of the furnace base.

4. Place a small cup under the furnace tube. Rinse the furnace tube using a solvent (such as alcohol) to remove debris. The solvent will drain out of the bottom of the tube into the cup.

5. Using a soft bristle brush (we recommend a flexible bottle brush), gently slide the brush up and down to clean out the inside of the furnace tube, allowing the handle to bend freely (see Figure 5.1 on the next page).

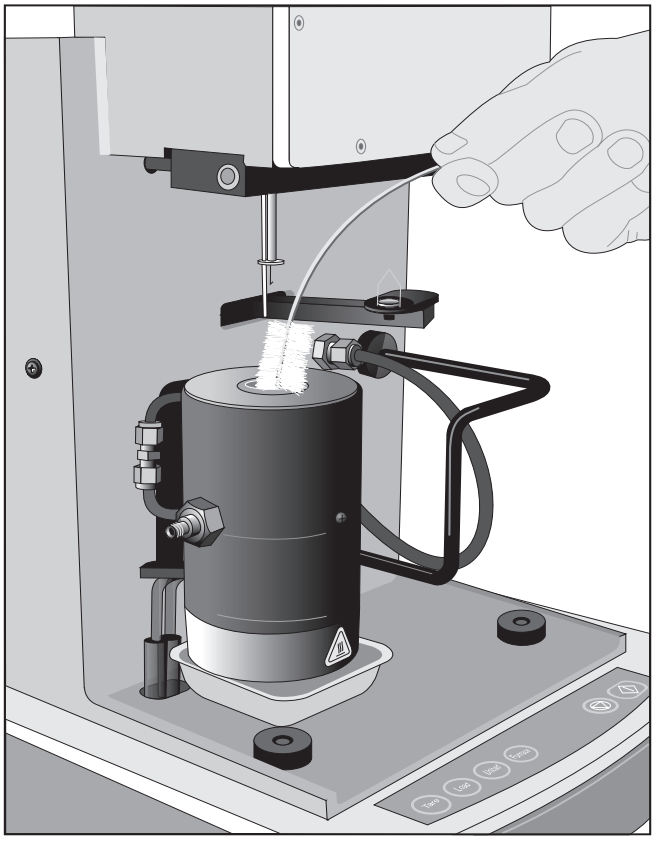

Figure 5.1 Cleaning the Inside of the Funace Tube

6. Rinse the furnace tube with the solvent again.

7. Replace the rubber cap on the quartz tube stem when you have completed the cleaning procedure.

8. Purge the system with nitrogen for one hour.

9. Heat the furnace to 900°C to remove any remaining solvent.

Heat Exchanger

The heat exchanger does not require any maintenance other than to maintain the level and quality of the liquid coolant. If the level drops too low, or the coolant becomes contaminated, this could result in problems with your instrument.

WARNING **Do not put any liquid other than distilled water in the heat exchanger reservoir.**

Maintaining Heat Exchanger Coolant

You should check the level and condition of the heat exchanger coolant periodically. We recommend routine checks every three to six months, depending on use of the instrument.

Add distilled water to the reservoir, if necessary, to keep the reservoir at least 2/3 full. If algae growth is visible, drain the reservoir bottle, refill it with distilled water, and add TA Instruments TGA Conditioner, as described in the next section.

Draining and Refilling the Water Reservoir

Drain and refill the heat exchanger water reservoir as follows:

1. Turn off the POWER switch and disconnect the heat exchanger cable and water lines from the instrument cabinet (see Chapter 2 for instructions).

2. Unscrew and remove the water reservoir cap.

3. Drain the coolant and flush out the system as follows:

   a. Lift the heat exchanger and dump out the contents of the water reservoir bottle.

   b. Fill the bottle to 2/3 full with distilled water only and replace the cap.

   c. Reconnect the heat exchanger cable and water lines to the instrument cabinet.

   d. Turn on the POWER switch.

   e. Turn on the pump by activating Air Cool (on the controller), and allow the water to circulate for several minutes.

   f. Turn off the pump by deactivating Air Cool, and check the clarity of the water in the reservoir bottle.

   g. If the water clarity is still unacceptable, disconnect the heat exchanger cable and water lines from the instrument cabinet, and repeat steps a through f.

   h. Continue repeating this procedure until you are satisfied with the clarity of the water in the bottle after it has circulated.

4. Dispose of the water and fill the bottle with TGA Conditioner (PN 952377.001) and fresh distilled water as directed in Chapter 2 on pages 2-10 and 2-11.

5. Turn on the pump again by activating Air Cool, and circulate the water until the air bubbles disappear from the water lines. (You may see “Err 119” on the instrument display until all the air has been removed.)

6. Replace and tighten the water reservoir cap.

Replacing the Thermocouple

1. Unload the sample pan and open the instrument's furnace.

2. Using the ball driver supplied in your TGA accessory kit, loosen and remove the six screws securing the balance chamber faceplate to the instrument.

3. Take off the faceplate.

4. Push the thermocouple up from the bottom, to feed it back into the balance chamber (Figure 5.2).

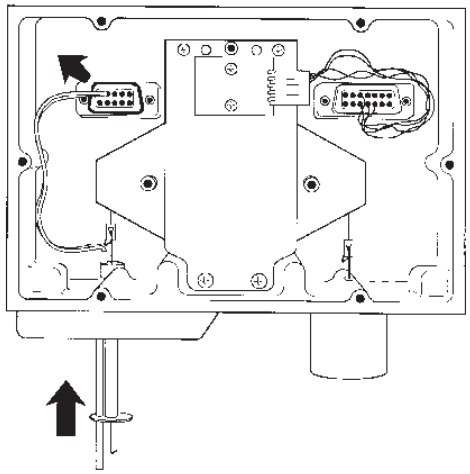

Figure 5.2 Removing the Thermocouple

5. Unplug the thermocouple from its connector and remove the thermocouple from the balance chamber.

6. Plug the new thermocouple carefully into the connector.

7. Thread the new thermocouple down through the hole next to the hang-down tube.

8. Thread the end of the thermocouple just through the ceramic disk at the end of the hang-down tube.

9. Load a sample pan to make sure that the end of the thermocouple does not touch it (Figure 5.3).

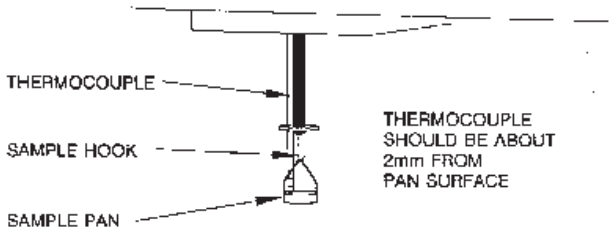

Figure 5.3 Checking Position of Thermocouple

10. Make sure that the hang-down wire does not touch the top of the thermocouple inside the balance chamber.

11. Replace the balance chamber faceplate and screws.

Diagnosing Power Problems

Fuses

The TGA contains internal fuses that are not user serviceable. If any of the internal fuses blow, a hazard may exist. Call your TA Instruments service representative.

The only fuses that you should service yourself are the external fuses, located on the TGA's rear panel. Both are housed in safety-approved fuse carriers, labeled F1 and F2 (Figure 5.4).

WARNING

Always unplug the instrument before you examine or replace the fuses.

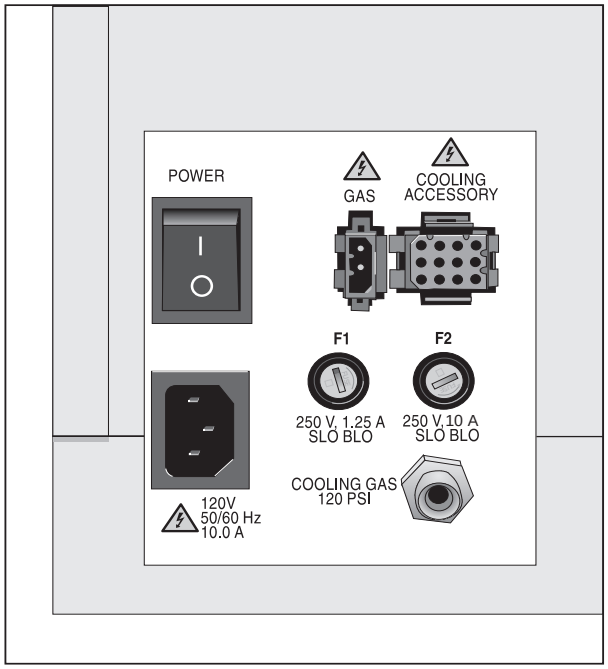

Figure 5.4 Fuse Locations

Fuse 1 is in the circuit between the main electrical input and the POWER switch. All power for internal operations and instrument functions, except heater power, passes through this fuse. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Fuse 2 protects the heater coils in the furnace. Because fuse 2 does not power the internal logic, you may not know that this fuse is blown until you try to heat a sample; the TGA passes the confidence test with fuse 2 open.

Fuse 2 is always checked at the beginning of a method. Power supplied by this circuit is switched by a computer-controlled relay. If fuse F2 is open at the start of a run, error 73 "No heater power at startup" is displayed, then the run is terminated.

Power Failures

A power failure caused by a temporary drop in line voltage results in one of two responses by the TGA instrument:

- If the drop is fairly large and of long duration (20 milliseconds or more), the system will reset and go into its power-up sequence when power resumes.

- If the drop is small or of short duration, the system may halt. The instrument will not restart until it is reset. To reset, press the Reset button on the TGA's back panel.

The TGA is designed for a nominal line voltage of 115 volts AC (+ 10%), 50 or 60 Hz. It should not be operated outside this range. Low line voltage may result in poor instrument operation; high line voltage may damage the instrument.

TGA 2050
Test Functions

The TGA 2050 has three levels of test and diagnostic functions:

- The confidence test that is run every time the instrument is started.
- Cycling test functions that continuously test specific.
- A manufacturing verifier test mode that coordinates and logs the results of a sequence of confidence test and drift runs.

These test functions are always present in the instrument. They are designed to aid manufacturing and service in checking and repairing the instrument.

The Confidence Test

The TGA 2050 confidence test is run each time the instrument is turned on or reset. The confidence test checks most of the computer and interface components in the system.

A standard 2050 instrument takes about 16 seconds to run the confidence test. During the test the yellow ready light will flash several times, indicating that the test is proceeding normally. At the end of the test the ready light will turn on and the instrument can be configured online.

If the confidence test is completed without any fatal errors detected, the ready light will turn on and remain on steady and the instrument will sound a beep. The instrument is now ready to configure online to the instrument controller.

If a fatal problem is detected during the confidence test, the ready light will remain off or continue to flash periodically. The instrument cannot be configured online when fatal errors are detected. Contact your TA Instruments service representative for assistance.

If the confidence test detects a non-fatal problem during startup, a confidence test error message will be displayed on the controller when the instrument is configured online. Non-fatal error 15, “CMOS RAM checksum error,” is displayed immediately after new instrument software is loaded into the 2050 instrument. Resetting the instrument should clear this error. If error 15 persists, or if any other confidence test errors are displayed, contact your TA Instruments service representative for assistance.

Replacement Parts

Table 5.1
TGA 2050 Parts List

Part Number	Description
952018.906	100 µL platinum sample pan kit
952018.907	100 µL ceramic sample pan kit
952040.901	Sample hang-down wire
952040.902	Tare hang-down wire
952011.906	Class M calibration weight kit (100 mg and 1 gm)
269931.001	Class M cal. wt. 100 mg
269931.002	Class M cal wt. 1 gm
952018.908	50 µL platinum sample pan kit
952018.909	250 µL ceramic sample pan kit
952018.910	500 µL ceramic sample pan kit
952350.901	Furnace assembly
952014.901	Balance assembly
952017.001	Tare tube
952310.901	Motor drive PCB
952060.901	Analog PCB
952068.901	Sample thermocouple assembly
952080.901	Sample motor assembly
952081.902	Furnace (down) sensor
952081.903	Furnace (up) sensor
952081.904	Sample position sensor
952082.901	Furnace motor assembly
952323.902	Aluminum sample pan kit

(table continued)

Table 5.1 (continued)

Part Number	Description
952384.901	TGA Temperature Calibration Kit
952183.901	Aluminum temperature calibration standard
900905.901	Calcium oxalate sample
990806.901	Air purge valve assembly
952410.001	TGA 2050 keypad assembly
993008.901	Power supply assembly
990850.901	Central processor PCB
993100.901	Communications PCB
990870.901	Triac drive PCB
259508.000	Brass tweezers
259509.000	Spatula, curved, 165 mm long
269845.001	O-ring, furnace housing to balance chamber
269920.002	Balldriver, 0.050-inch
269920.026	Balldriver, 7/64-inch
269930.001	Class C calibration weight kit (1 mg to 500 mg)
952160.901	TGA 2050 Cooling Accessory
952160.903	Heat exchanger fan/ assembly
952162.901	Cooling Accessory tubing
952166.901	Cooling Accessory water reservoir bottle
952172.901	Cooling Accessory pump assembly

(table continued)

Table 5.1 (continued)

Part Number	Description
952161.901	Flow switch assembly
269932.001	Solid state relay
952381.901	Upper furnace core kit, EGA
952382.901	Lower furnace core kit, EGA
952357.901	Furance tube replacement kit, EGA

Appendix A:
Ordering Information

For information or to place an order, contact:

United States:

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: (302) 427-4000
Fax: (302) 427-4001

Overseas:

TA Instruments Ltd.
Europe House
Bilton Centre
Cleeve Road
Leatherhead, Surrey KT22 7UQ
England
Telephone: 44-1-372-360363
Fax: 44-1-372-360135

TA Instruments GmbH
Siemenstrasse 1
8755 Alzenau
Germany
Telephone: 49-6023-30044
Fax: 49-6023-30823

TA Instruments Benelux
Ottergemsesteenweg 461
B-9000 Gent
Belguim
Telephone: 32-9-220-79-89
Fax: 32-9-220-83-21

TA Instruments Japan
Iwata Building #5
28-11 Nishigotanda 2-Chome
Shinagawa-Ku, Tokyo 141
Japan
Telephone: 813-5434-2711
Fax: 813-5434-2770

For technical assistance or service in the United States:

HOTLINE
For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis Helpdesk at (302) 427-4070.

SERVICE
For instrument service and repairs, please call (302) 427-4050.

ORDERING
To oder instruments and supplies, please call (302) 427-4040.

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