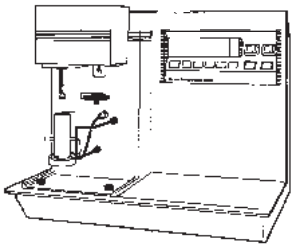
TA Instruments 109 Lukens Drive, New Castle, DE 19270

Thermal Analysis & Rheology

A Subsidiary of Waters Corporation



TGA 2950 CE

Thermogravimetric Analyzer

Operator's Manual

PN 825602.001 Rev. D (Text and Binder)
PN 825602.002 Rev. D (Text Only)
Issued July 2000

109 Lukens Drive
New Castle, DE 19720

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.**

Helplines

To TA Instruments

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

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

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

Sales ..... (302) 427-4000

Safety

This equipment has been designed to comply with the following standards for safety:

- IEC 1010-1/1990 and A1/1992
- IEC 1010-2-010/1992
- EN 61010-1/1993
- EN 61010-2-010/1994
- UL 3101-1, First Edition.

CE Compliance

In order to comply with the Electromagnetic Compatibility standards of the European Council Directive 89/336/EEC (EMC Directive) and Directive 73/23/EEC on safety as amended by 93/68/EEC, the following specifications apply to the TGA 2950 Autosampler CE:

- *Safety:*
  EN 61010-1/1993
  EN 61010-2-010/1994

- *Emissions:*
  EN 55011: 1991, CISPR 11:1990 Group 1 Class B (30–1000 MHz) Radiated
  EN 55011: 1991, CISPR 11:1990 Group 1 Class B (0.15–30 MHz) Conducted

- *Immunity:*
  EN 50082-1: 1992 Electromagnetic Compatibility—Generic immunity standard Part 1.Residential, commercial, and light industry.
  — IEC 801-2: 1991, 8 kV air discharge, direct. No change of state.

Safety

(continued)

— IEC 801-3: 1984, 27 - 500 MHz, 3V/m. No response above 10.00 mg sample weight and 2.00°C sample temperature.
— IEC 801-4: 1988, Fast transients common mode 1 kV AC power. No change of state.

Instrument Symbols

The following label is displayed on the TGA 2950 CE instrument for your protection:

Symbol	Explanation
	This symbol, on the front of the TGA 2950 CE furnace, indicates that a hot surface may be present. Do not touch this area or allow any material that may melt or burn to come in contact with this surface.

Please heed the warning label and take the necessary precautions when dealing with the furnace. The *TGA 2950 CE Operator's Manual* contains cautions and warnings that must be followed for your own safety.

Safety
(continued)

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. 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 of the safety standards indicated. 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 1.4 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 2950 CE furnace or the TGA 2950 CE EGA furnace.

WARNING

Oxygen can be used as a purge gas in the TGA 2950 CE. However, the furance must be kept clean so that volatile hyrdocarbons (which might combust) are removed.

Safety

(continued)

WARNING

The TGA 2950 CE furnace assembly contains a layer of refractory ceramic fiber (RCF) insulation. This insulation is completely encapsulated within the ceramic subassembly, which is not meant to be disassembled. If the subassembly should break in such a way as to expose the RCF insulation, we recommend that you dispose of it as you would any refractory material.

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.

WARNING

The TGA 2950 CE EGA furnace assembly also contains refractory ceramic fiber (RCF) insulation. This insulation is enclosed within the furnace housing. The furnace housing should be disassembled only 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.

WARNING

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

Safety

(continued)

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 (see Figure 1.1) 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.

Lifting the Instrument

The TGA 2950 CE is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:

WARNING

Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

Using This Manual

Chapter 1	Describes your TGA 2950 CE instrument and its accessories and specifications.
Chapter 2	Describes how to unpack and install the TGA 2950 CE and how to connect it to the rest of your system.
Chapter 3	Describes how to run TGA experiments and set up the accessories.
Chapter 4	Provides technical information and explains principles of TGA operation.
Chapter 5	Describes how to perform routine maintenance, replace the thermocouple 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.
Appendix B	Describes the High Resolution option, including installation, usage, and applications.

Using This Manual

(continued)

Appendix C Provides instructions needed to operate the TGA Autosampler option to automatically load and run samples.

Appendix D Provides instructions needed to install and use the EGA furnace with the TGA 2950 CE.

CHAPTER 1: Introducing the TGA 2950 CE

Introduction

Your TA Instruments Thermogravimetric Analyzer (TGA) 2950 CE is a thermal analysis instrument used for evaluating changes in sample weight. It is used in conjunction with a TA Instruments thermal analysis controller and associated software, to make up a thermal analysis system.

The Thermogravimetric Analyzer 2950 CE 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.

Components

The TGA 2950 CE has five major components, illustrated in Figure 1.1:

- 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.

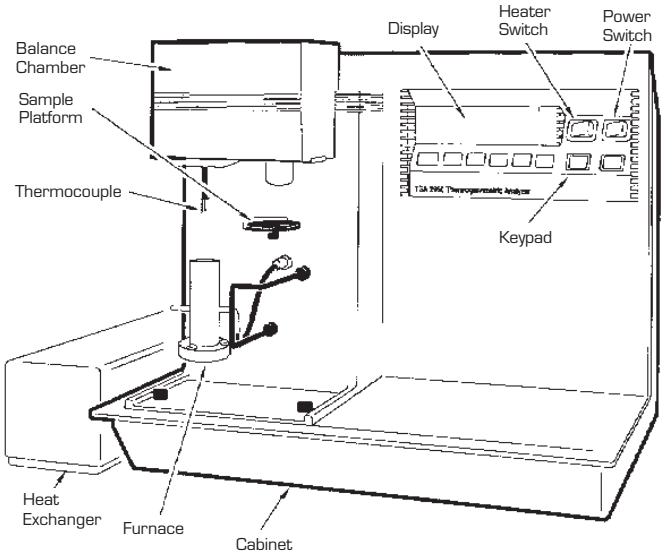


Figure 1.1 TGA 2950 CE Components

The 2950 CE Instrument

The parts of the TGA 2950 CE instrument that provide for operator control are:

- The instrument display
- The instrument keypad.

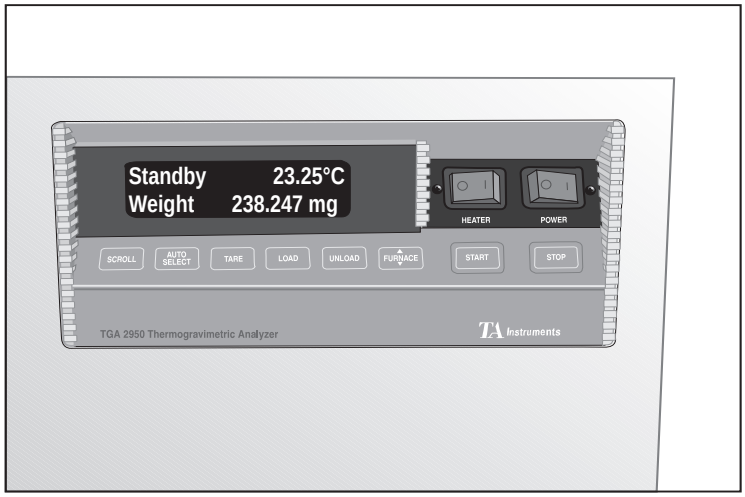


Figure 1.2
TGA 2950 CE
Display and Keypad

2950 CE Display

The TGA instrument display is the lighted area of the keypad (Figure 1.2). It contains two rows of 20 characters each.

During normal operation, the display is segmented into three areas. The left eight characters on the upper line show the instrument status; the right nine characters show the sample temperature; and the bottom line is a realtime signal display (*e.g.,* weight).

A pound sign (#) after the weight signal indicates that the balance reading has not yet stabilized. When the weight stabilizes, the pound sign will disappear.

2950 CE Keypad

The instrument keypad (see Figure 1.2) contains the keys found in Table 1.1 and the HEATER and POWER switches.

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

Table 1.1
TGA 2950 CE Keypad Function Keys

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display. For more details on the experiment, refer to status and signal displays on the controller.
TARE	Zeros the displayed 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.

(table continued)

Table 1.1
(continued)

Key/Function	Explanation
LOAD	Loads a sample pan from the sample platform onto the balance.
UNLOAD	Unloads the sample pan from the balance onto the sample platform.
Δ 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. *Forced Start* can be done by pressing the START key while the status line displays "Set Up." Forced start begins collecting data during instrument setup.

(table continued)

Table 1.1
TGA 2950 CE 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 standby or method-end state), the STOP key will halt any activity (air cool, all mechanical motion, *etc.*).
REJECT (Hold down SCROLL and press STOP)	If an experiment is running, SCROLL-STOP 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 *discarded*. This is the same function as **Reject** on the controller.

(table continued)

Table 1.1
(continued)

Key/Function	Explanation
NOTE:	The SCROLL key operates normally (scrolls the realtime signals) until the STOP key is pressed. If an experiment is not running, SCROLL-STOP works like the STOP key.
AUTOSELECT	This key appears only on instruments with an autosampler installed. See Appendix C for details.

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.

HEATER Switch

The HEATER on/off switch (see Figure 1.2) turns the power to the instrument heater on and off. This switch should be in the ON (1) position before you start an experiment.

NOTE: The light in the HEATER switch will glow only after an experiment is initiated. If the heater loses power during an experiment, the HEATER switch will continue to glow, even if it is switched to the OFF (O) position, until the "STAND BY" status code is displayed.

POWER Switch

The POWER switch (see Figure 1.2) turns the power to the instrument on and off.

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.

Evolved Gas Analysis Furnace

The TGA 2950 CE EGA furnace is an accessory to the instrument that allows you to perform combined TGA and evolved gas analysis experiments.

NOTE: The EGA furnace can be installed only by a service representative. To arrange for installation of this furnace, contact TA Instruments.

Other Accessories

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

Specifications

Tables 1.2 through 1.4 contain the technical specifications for the TGA 2950 CE. Only values with tolerances or limits are guaranteed data. Values without tolerances are for information only. See page *xv* for EMC Conformity Specifications.

Table 1.2
TGA 2950 CE
Operating Parameters

Temperature range	25°C to 1000°C
Thermocouple	Platinel II*
Heating rate with standard furnace with EGA furnace	 0.1 to 100°C/min 0.1 to 50°C/min
*Platinel II is a registered trademark of Engelhard Industries.	

Table 1.3
TGA 2950 CE
Instrument
Characteristics

Operating line voltage	120 volts ± 10%, 50/60 Hz
Energy consumption	1.5 kVA

Table 1.4
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	0.1 μg
Accuracy	$\leq \pm$ 0.1%
Ranges	100 mg range: 0.1 μg to 100 mg 1000 mg range: 1 μg 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 1.4
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 2950 CE furnace or the TGA 2950 CE EGA furnace. Oxygen may be used. However, the furnace must be kept clean of hydrocarbons.

CHAPTER 2: Installing the TGA 2950 CE

Unpacking/Repacking the 2950 CE

NOTE: These instructions are also found as separate unpacking instructions in the shipping box.

You may wish to retain all of the shipping hardware, the plywood, and boxes from the instrument in the event you wish to repack and ship your instrument.

Unpacking the 2950 CE

Refer to Figures 2.1 to 2.3 while unpacking your instrument.

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

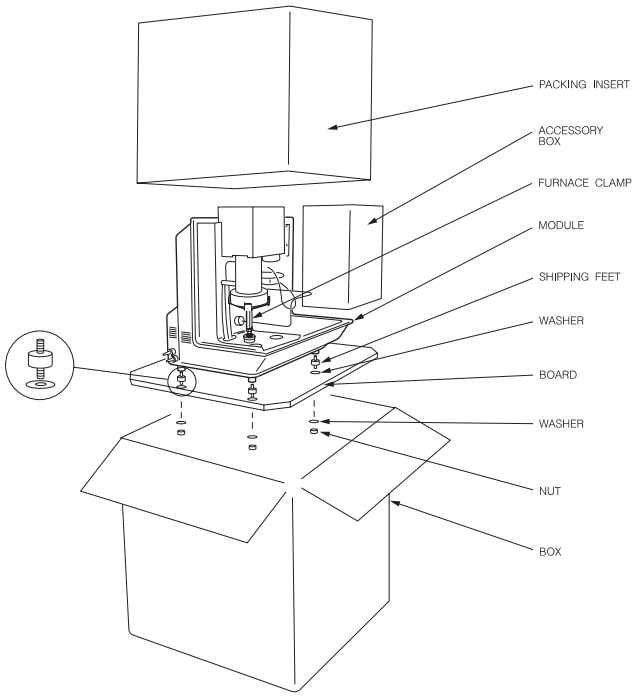


Figure 2.1
Shipping Boxes

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.**

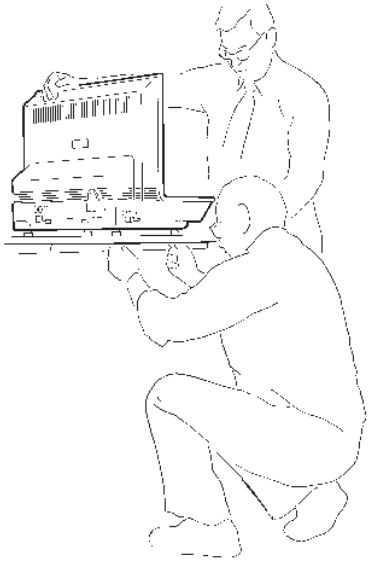


Figure 2.2
Removing the Plywood Board

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 be holding 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 the entire unit while you slide the plywood board out from under the unit.

8. Have your assistant lift one side of the unit while you use a wrench to install two mounting feet on the other side (see Figure 2.3). Rotate the unit and install the two remaining mounting feet in the same manner.

9. Remove the furnace clamp before turning on the power to the unit.

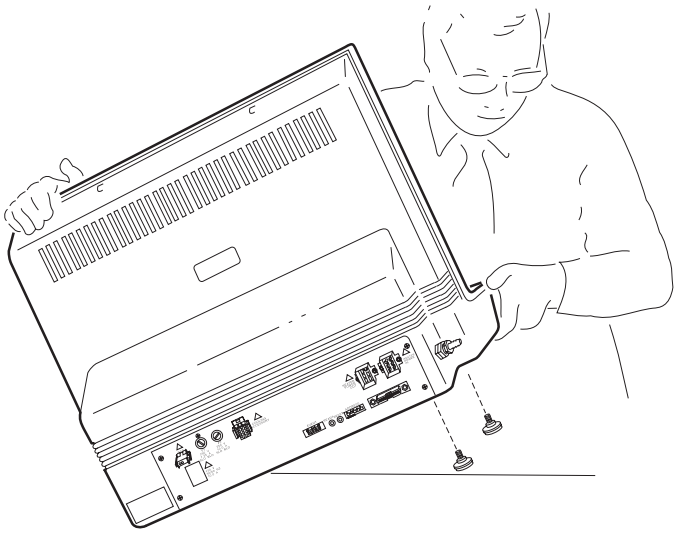


Figure 2.3 Installing the Mounting Feet

Repacking the 2950 CE

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 2950 CE 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:

- 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 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 2950 CE, 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.

On
- ... a stable work surface.

Near
- ... a power outlet (120 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 flowmeters.

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

After you have decided on the location for your instrument, refer to the next several sections to unpack and install the TGA 2950 CE.

WARNING

Drying out the instrument may be needed, if it has been exposed to humid conditions. Certain ceramic materials used in this equipment may absorb moisture, causing leakage currents to exceed those specified in the applicable standards until moisture is eliminated. 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 3 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 2-12). To fill the heat exchanger, follow the directions given here.

Figure 2.4
Rear Panel of
Heat Exchanger

1. Unscrew the water reservoir cap on the heat exchanger (see Figure 2.4).

Figure 2.5
Heat Exchanger Bottle

2. Add TA Instruments TGA Conditioner (PN 952377.901) into the water reservoir bottle. Refer to the instructions on the bottle for the amount of conditioner to add to the reservoir. 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 it to the inner rim if necessary.

!WARNING **Do not put any liquid 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 with 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 the cables across accessways.**

Heat Exchanger Water Lines

1. Remove the water lines from the packing.
2. Connect one end of the water line marked "SUPPLY" to the connector labeled "SUPPLY" on the right side of the instrument cabinet. The connector will snap in. (To disconnect the line, press down on the release tab.)
3. Connect the other end of the water line marked "SUPPLY" to the connector labeled "SUPPLY" on the heat exchanger.

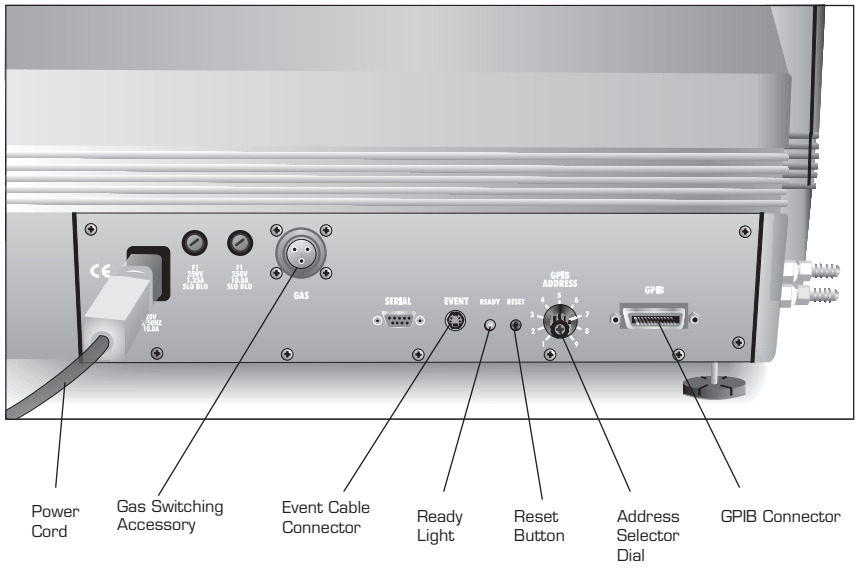


Figure 2.6
TGA 2950 CE
Connector Panel

4. Connect one end of the unmarked water line to the connector labeled “RETURN” on the right side of the instrument cabinet.

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

NOTE:

The TGA heat exchanger has been electronically de-coupled from the instrument in order to meet EMC requirements and therefore must be turned on and off manually by the operator. Remember to turn the heat exchanger on prior to running an experiment, and off after completion of an experiment. Allowing the heat exchanger to run continuously will not harm the instrument, but may reduce the operating life of your heat exchanger.

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

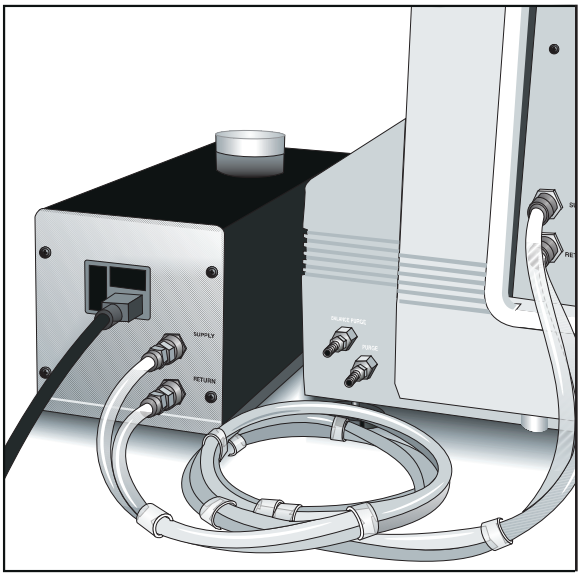


Figure 2.7
CE Heat Exchanger Water Line Connections

NOTE:

Air trapped in the heat exchanger system must be purged before starting the first run. Turn on the heat exchanger POWER switch and allow the water to circulate until all 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 2.6).

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. (For more information, see your controller manual.)

5. Select an address from 1 to 9. Then use the address selector dial on the TGA connector panel to set the desired address. Figure 2.8 on the next page shows an 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 until the instrument completes its start-up displays, then reconfigure the instrument with the controller to bring the instrument back online.

NOTE: The instrument's GPIB address is displayed during startup and can also be viewed on the instrument's status display.

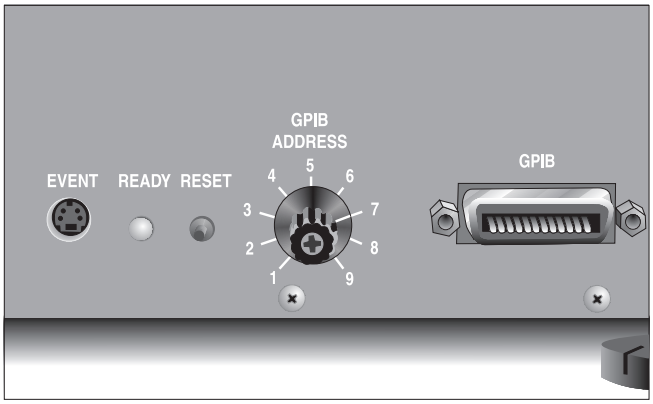


Figure 2.8
Address Selector Dial
(Showing an Address of 7)

Purge Lines

WARNING

Do not use any liquid in the purge lines.

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

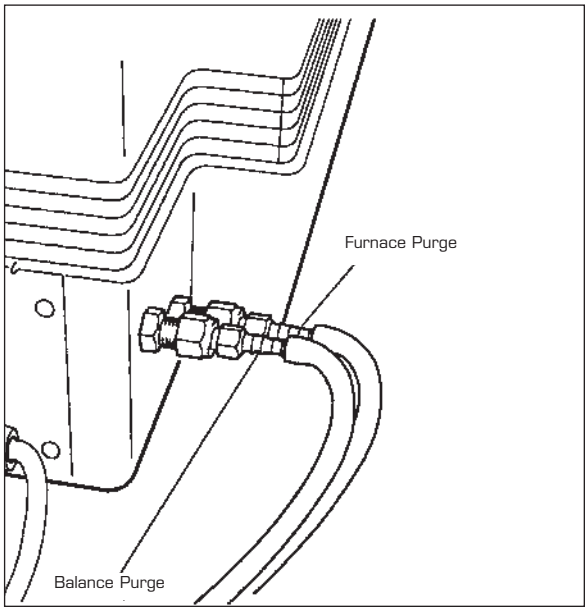


Figure 2.9
TGA PURGE Fittings

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 is 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; however, 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 mL per minute or less. The flow distribution should be as follows: (a) for the standard furnace, 40 percent to the balance chamber and 60 percent to the furnace, and (b) for the EGA furnace, 10 percent to the balance and 90 percent to the furnace.

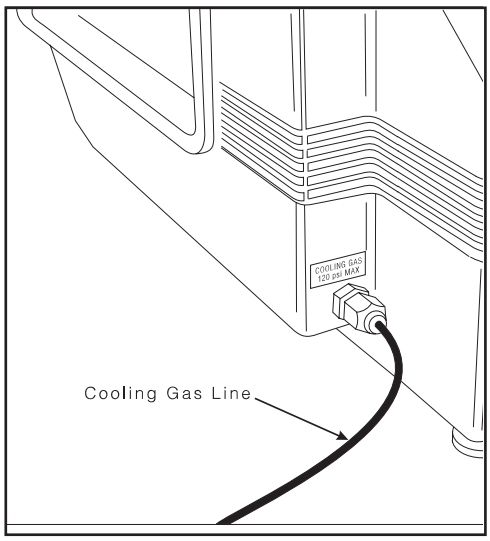


Figure 2.10
TGA COOLING GAS Fitting

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).

2. Make sure your compressed air source is regulated to between 25 and 120 psi 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

NOTE: The accessory kit contains two power cords. The ferrite loaded power cord provides the TGA protection against electromagnetic interference (EMI). To ensure best test results, this power cord must be used for the TGA instrument, while the standard power cord is used with the heat exchanger.

1. Make sure the TGA POWER switch is in the OFF (0) position.

2. Plug the ferrite-loaded power cord into the TGA.

♦ ***CAUTION:*** Before plugging the TGA power cord 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 cord 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 2.11), and take off the cover.

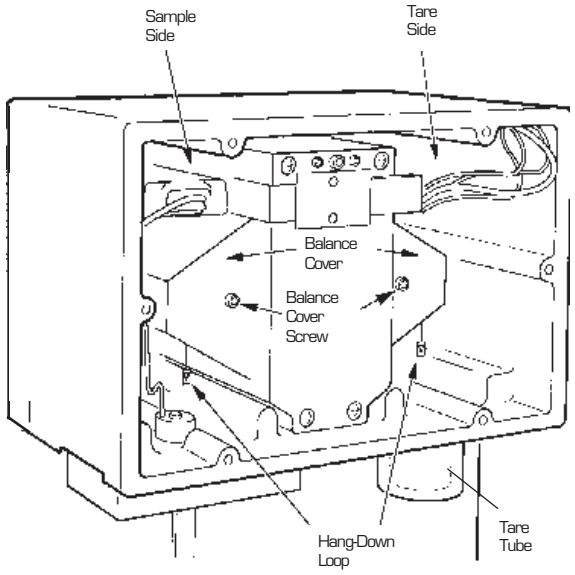


Figure 2.11
Interior of Balance Chamber Before Unpacking

4. Using tweezers, remove the foam insert from around the screw hole (Figure 2.12):

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

   b. Remove the foam insert from the balance chamber.

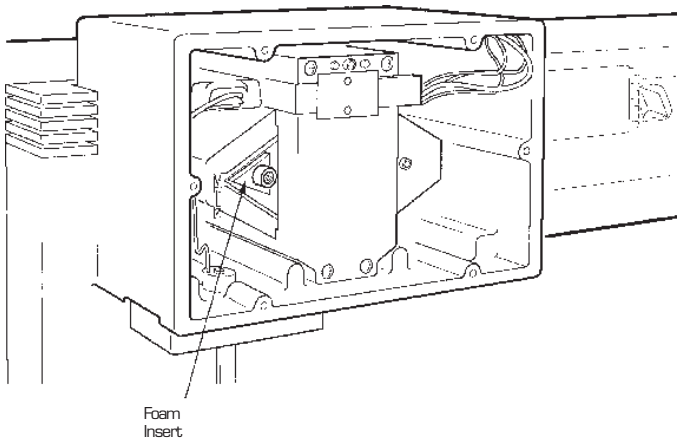


Figure 2.12 Removal of Foam Insert from Balance Chamber

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.

Starting the 2950 CE

1. Check all connections between the TGA 2950 CE and the controller. Make sure each component is plugged into the correct connector.

2. Press the instrument POWER and HEATER switches to the ON (1) position. The instrument will run an internal confidence test, which is run each time you power on the unit.

NOTE: The HEATER and POWER indicator lamps may flicker under low AC voltage conditions.

3. Watch the instrument display during the confidence test for any error messages that may be indicated. If an error occurs, make a note of the test number in which the error occurred, and call TA Instruments for service.

After the confidence test, the screen will briefly display the system status, indicating the amount of data storage memory available and the GPIB address. Next follows the copyright display, and then the standby display, shown on the next page.

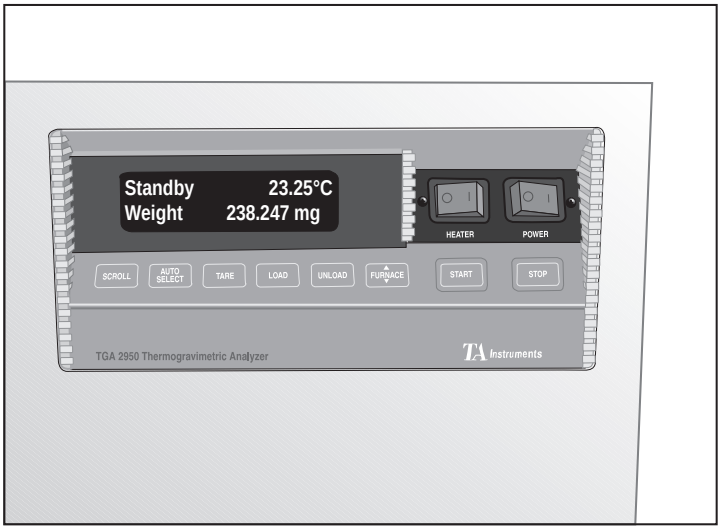


Figure 2.13
TGA 2950 CE
Standby Display

4. Bring the instrument online with the TA controller.

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

5. Make sure the heat exchanger POWER switch is OFF.

NOTE: The accessory kit contains two power cords. The ferrite loaded power cord provides the TGA protection against electromagnetic interference (EMI). To ensure best test results, this power cord must be used for the TGA instrument, and the standard power cord is used with the heat exchanger.

6. Plug the standard power cord into the heat exchanger.

7. Plug the power cord into the wall receptacle.

8. Turn the heat exchanger POWER switch ON.

NOTE:

The TGA heat exchanger has been electronically de-coupled from the instrument in order to meet EMC requirements and therefore must be turned on and off manually by the operator. Remember to turn the heat exchanger on prior to running an experiment, and off after completion of an experiment. Allowing the heat exchanger to run continuously will not harm the instrument, but may reduce the operating life of your heat exchanger.

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 instrument.
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 thermocouple tube without bending the wire (Figure 2.14).

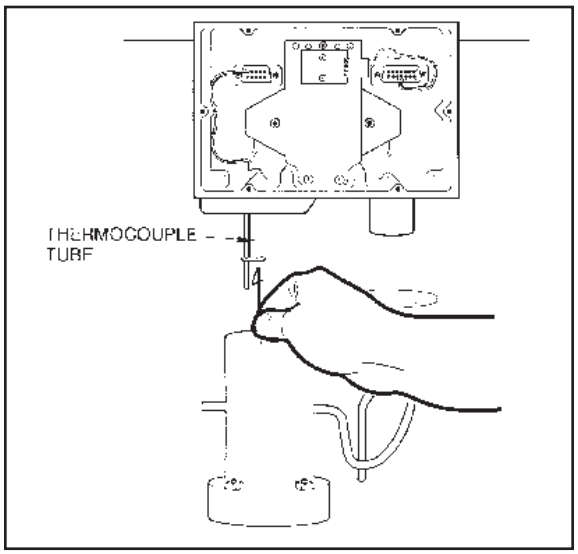


Figure 2.14
Installing the Sample Hang-Down Wire

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

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.

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 (Figure 2.15 on next page).

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.

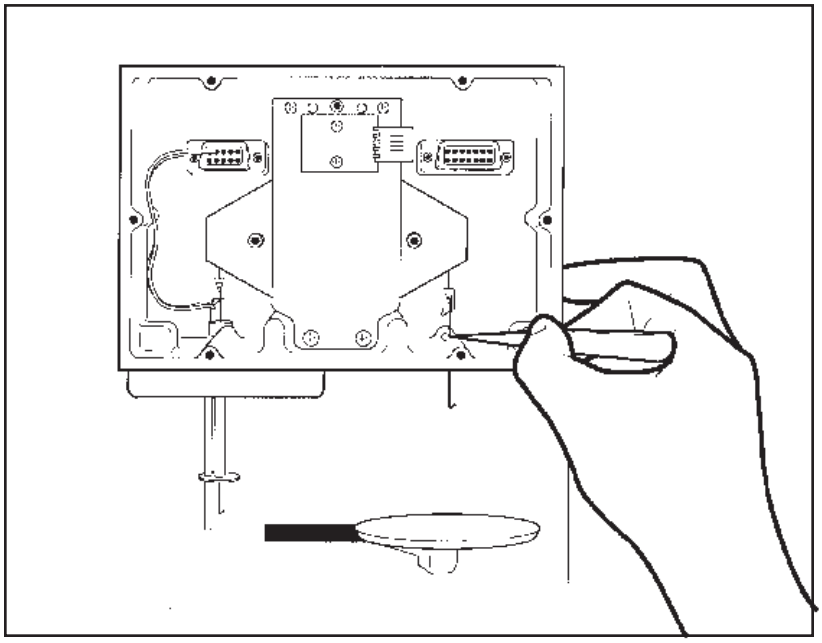


Figure 2.15
Installing the Tare Hang-Down Wire

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 thermocouple 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, you will need to 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 it manually (using brass tweezers) on the sample hang-down wire and continue with the procedure. Use the Instrument Control Sample Platform Adjust procedure 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 is roughly centered within the top of the thermocouple tube inside the balance chamber.

4. If the wire is not roughly centered inside the thermocouple 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.

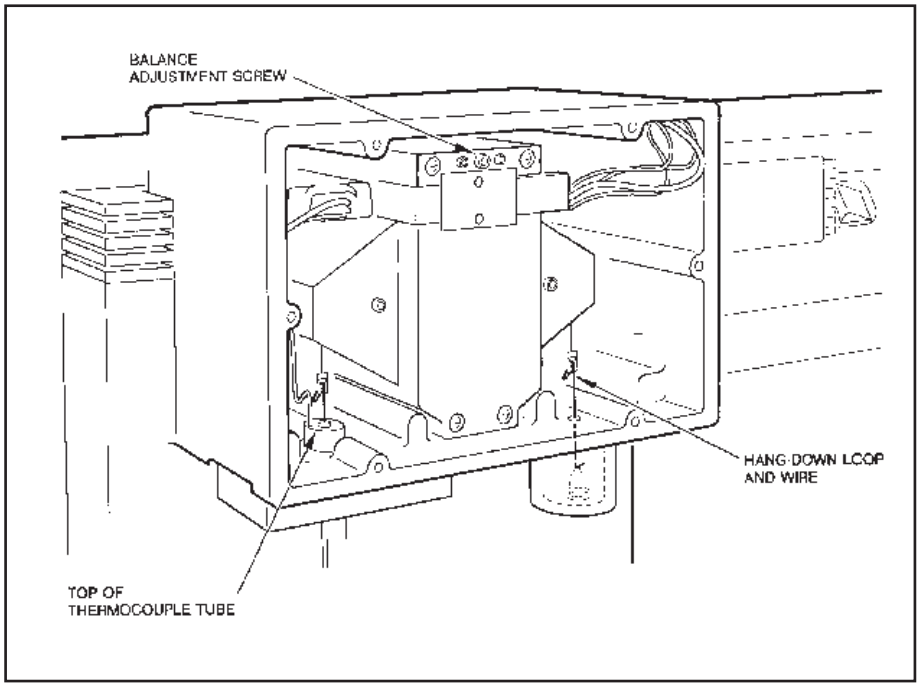


Figure 2.16
Location of Balance Adjustment Screw

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 thermocouple 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.

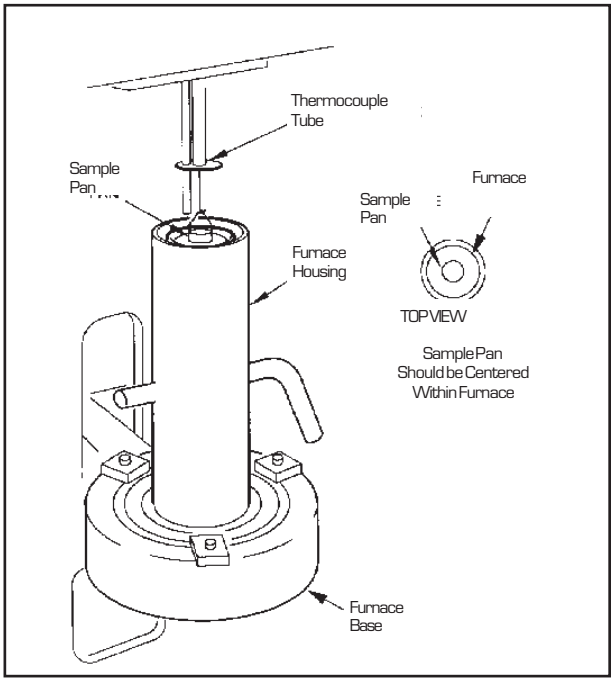


Figure 2.17 Aligning the Sample Pan in the Furnace

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. The method used to adjust the platform position is called the Sample Platform Adjust procedure. This procedure is part of the Instrument Control software program. Refer to the online help and documentation for further information on adjusting the sample platform shown in the figure below.

NOTE: This procedure assumes that the instrument has been properly leveled and that the sample hang-down wire is straight.

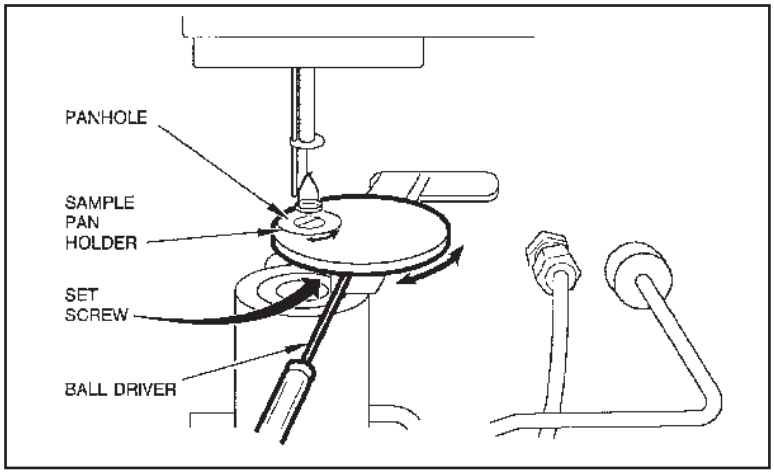


Figure 2.18 Sample Platform Assembly

Shutting Down the 2950 CE

Turning the system and its components on and off frequently is discouraged. When you finish running an experiment on your instrument 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 your system will not be used for longer than five days, we suggest that you turn it off. To power down your instrument for any reason, simply press the POWER and HEATER switches to the OFF (0) position.

CHAPTER 3: Running Experiments

Overview

This chapter provides step-by-step instructions on how to run TGA experiments. Explanations of terminology and how the instrument operates are given in Chapter 4.

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 throughthe TA controller (sample and instrument information)
- Creating and selecting the thermal method
- Selecting and taring the sample pan
- Loading the sample
- Setting the purge gas flow rate
- 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).

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 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 TGA, if necessary.

Calibrating the TGA

To obtain accurate experimental results, you should calibrate the instrument when you first install it. For the best results, however, you should recalibrate periodically.

Three types of calibration are needed for the TGA 2950 CE: temperature, weight, and sample platform calibration. All three calibration procedures are performed through the Instrument Control software. This section provides a brief description of these types of calibration; for details, refer to the online help and documentation for further information.

Temperature Calibration

Temperature calibration is useful for TGA experiments in which precise transition temperatures are essential. To temperature-calibrate the TGA, you need to analyze a high-purity magnetic standard for its curie temperature, and then enter the observed and correct values in the Instrument Control temperature calibration table; see the online help and documentation for further information. The standard most often used is nickel with a curie temperature of 354.4° C (NIST Certificate for GM761). The observed and correct temperatures correspond to the experimental and theoretical transition temperatures (*e.g.*, curie temperature) of the calibrant. From one to five temperature calibration points (pairs of observed and correct temperature points) can be entered in the calibration table. A multiple-point calibration is more accurate than a one-point calibration.

Weight Calibration

Weight calibration should be performed on the TGA at least once a month. The weight calibration procedure calibrates both the 100-mg and 1-gm weight ranges. The calibration parameters are stored internally in the instrument.

The Instrument Control weight calibration functions guide you through the calibration procedure step-by-step; see the online help and documentation for further information.

Sample Platform Calibration

The sample platform adjustment procedure is used if the sample hang-down wire fails to pick up a sample pan during an automatic loading procedure. The sample platform must be adjusted so that the instrument can properly load and unload the sample pans.

To avoid weight signal noise, the TGA must be level so that the sample pan and hang-down wire hang inside the furnace and thermocouple tube without touching them. The Instrument Control sample platform adjust functions take you step-by-step through the procedure; see theonline help and documentation for further information.

Running a TGA Experiment

Experimental Procedure

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

- Selecting the pan type and material
- Loading the pan
- Taring the empty sample pan
- Loading the sample into the pan
- Entering experiment information through the TA controller (sample and instrument information)
- Creating and selecting the thermal method on the controller
- Attaching and setting up external accessories as required (*e.g.*, purge gas, Gas Switching Accessory)
- Starting the experiment.

Preparing Samples

TGA experiments utilize different types of sample pans, depending upon the type of material that you are analyzing.

Selecting Sample and Tare Pans

Three kinds of sample pans are available for the TGA 2950 CE, 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 pan materials are desirable.

- 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.

- 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 pans 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. Use the first step in the weight calibration procedure to mechanically tare the balance.

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 2950 CE has two weight ranges, taring is done for both ranges. The tare weight is stored by the instrument for the appropriate weight range.

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

2. Press the TARE key on the instrument keypad. 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 2950 CE 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 (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.

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 3.1).

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.

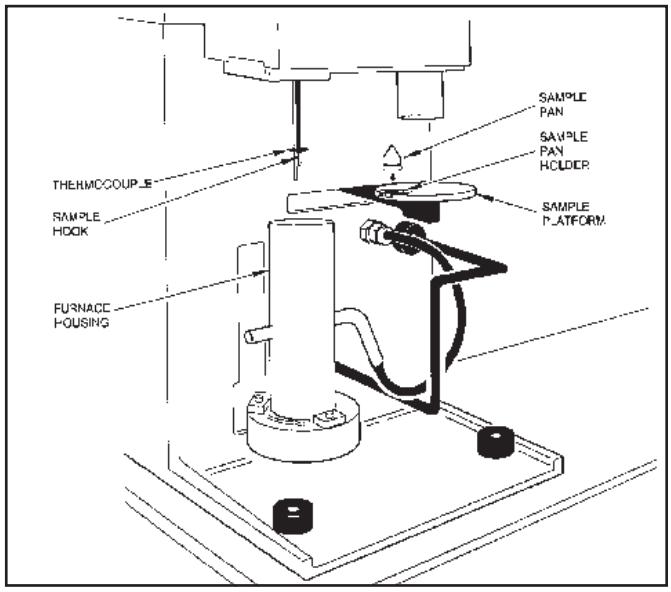


Figure 3.1 Sample Pan Ready to Load

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 3.2).

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

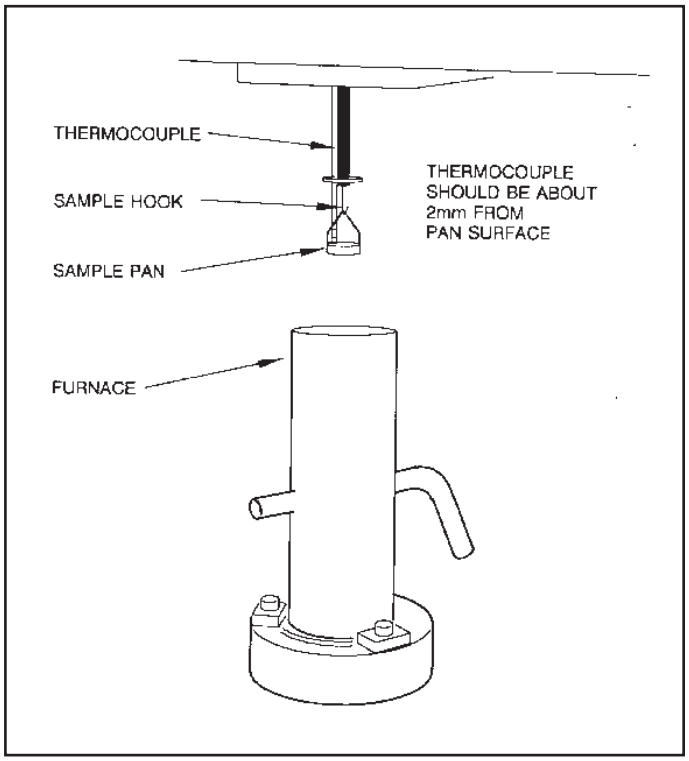


Figure 3.2 Adjusting the Thermocouple

4. Press the FURNACE key to close the furnace by moving it up around the sample (Figure 3.3).

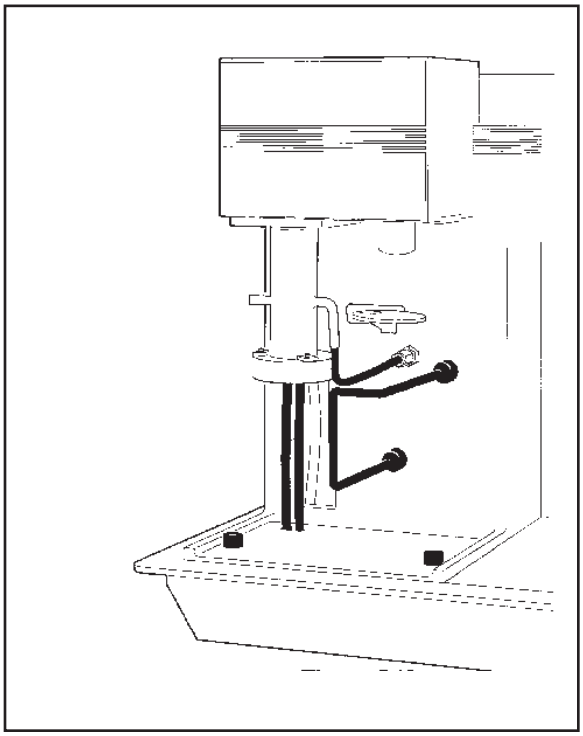


Figure 3.3 Furnace in Closed Position

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 Instrument Control. 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 2950 CE:

- Air cool option
- Purge gas
- TA Instruments Gas Switching Accessory
- Evolved Gas Analysis (EGA) Furnace (see also Appendix D).

For the TGA Hi-Res™ Option, see Appendix B, and for the TGA Autosampler, see Appendix C.

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 the Air Cool Option

You can program the system to air-cool the furnace automatically at the end of the experiment by selecting the air cool method-end condition as one of your instrument parameters (see the online help and documentation for further information). 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 psi. Nitrogen can also be used as a cooling gas.

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

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

The purge flow through the furnace is horizontal to the sample (see Figure 3.4), permitting rapid removal of decomposition products from the sample environment.

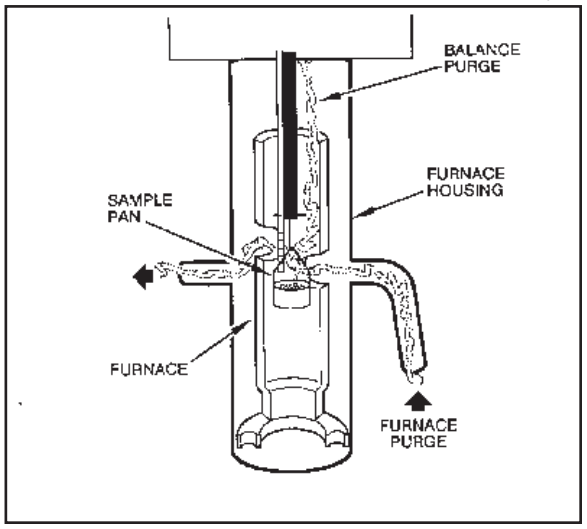


Figure 3.4
Furnace Purge

You can choose nitrogen, oxygen*, helium, air, or argon for your purge environment. Do not use any other gases in the TGA 2950 CE. See the "Chemical Safety" section on page xvii for further details.

! WARNING

Do not use hydrogen or any other explosive gas in the TGA 2950 CE furnace or the TGA 2950 CE EGA furnace. *Oxygen may be used as a purge gas, but the furnace must be kept clean of volatile hydrocarbons 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 it 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 mL per minute or less. When you use the standard TGA 2950 CE furnace, the flow distribution should be 40 percent to the balance chamber and 60 percent to the furnace. When you use the TGA 2950 CE EGA furnace, 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 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 that its power switch is on and that 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 for further information).

When switching between gases during an experiment, connect the Gas Switching Accessory to the purge port only. 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

Before you start the experiment, ensure that the TGA 2950 CE is online with the controller and you have entered all necessary experimental parameters.

NOTE: Once the experiment is started, operations are best performed at the controller keyboard. The TGA 2950 CE is very sensitive to motion and might pick up the vibration caused by pressing a key on the instrument keypad.

Start the experiment by pressing the START key on the instrument keypad, or selecting start on the TGA Instrument Control program (see the online help and documentation for further information). When you press the START key, the system automatically loads the sample pan and closes the furnace if necessary, and then runs the loaded method to completion.

Forced Start

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 methods begin when the normal set-up procedures are completed.

To begin a *forced start*, press START on the instrument keypad while the 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 pressing either the STOP key on the TGA 2950 CE keypad or **Stop** on the TGA Instrument Control program (see theonline help and documentation for further information). Another function that stops the experiment is **Reject.** 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.

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

Unloading the Sample

If you select the method-end option that enables the furnace to open and unload at the end of the experiment, the TGA will automatically unload the sample at the end of the run. (See the online help and documentation for further information.) 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

If you choose to perform TGA experiments in an oxygen-free atmosphere, a few extra precautions regarding the purge gas system and instrument setup are necessary to ensure an oxygen-free environment.

Purge Gas System

When performing TGA experiments in an oxygen-free atmosphere, take the following precautions:

- 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 two-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 to 100 mL/minute flow into the balance chamber and 100 mL/minute into the furnace.

Instrument Setup

When performing TGA experiments in an oxygen-free atmosphere, take the following steps to set up your instrument:

- Readjust your flowmeter(s) for standard operating flow rates: (a) for the standard furnace, 40 mL/minute flow into balance chamber and 60 mL/minute into the furnace or (b) for the EGA furnace, 10 mL/minute into the balance chamber and 90 mL/minute into the furnace.
- Tare sample pan (as needed).
- Load sample and close system.
- Purge for 20 minutes, if possible, before starting a run.
- When run is complete, allow the furnace to cool in the closed position until the temperature is less than 350˚C. This can be automatically programmed into your method; after the last segment, add an "Equilibrate at 350˚C" step. Another option is to change method-end conditions to leave the furnace closed at method end.
- When using air or oxygen during an experiment, introduce new gas through the furnace purge port only, and switch back to nitrogen before cooling down.

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

CHAPTER 4: Technical Reference

Description of the TGA 2950 CE

The TGA 2950 CE 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 the same 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 unequal amounts 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 2950 CE has two weight ranges: 1-gm and 100-mg. Both ranges are continuous over their weight loss operating range, meaning 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 0.0 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 (standard furnace = 40 mL/min, EGA furnace = 10 mL/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.

Components

The TGA 2950 CE has five major components, illustrated in Figure 4.1: the balance, the sample loading assembly, the furnace, the cabinet, and the heat exchanger.

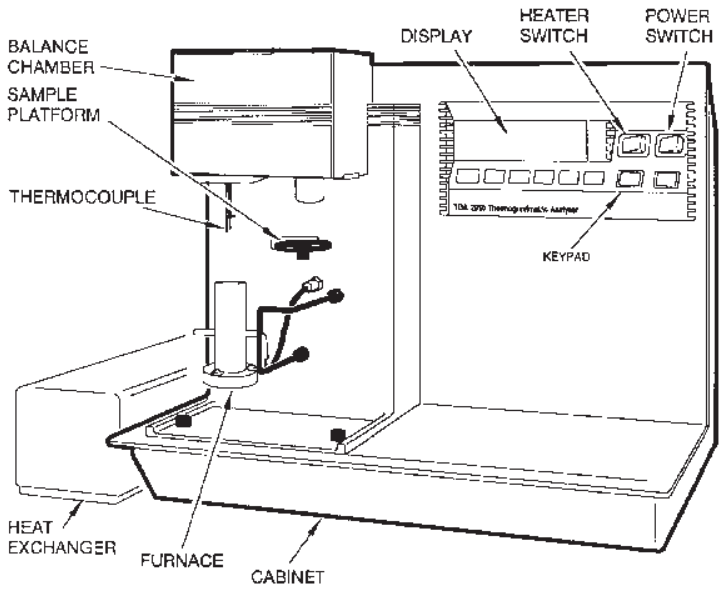


Figure 4.1
TGA 2950 CE Components

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 (Figure 4.2) 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.

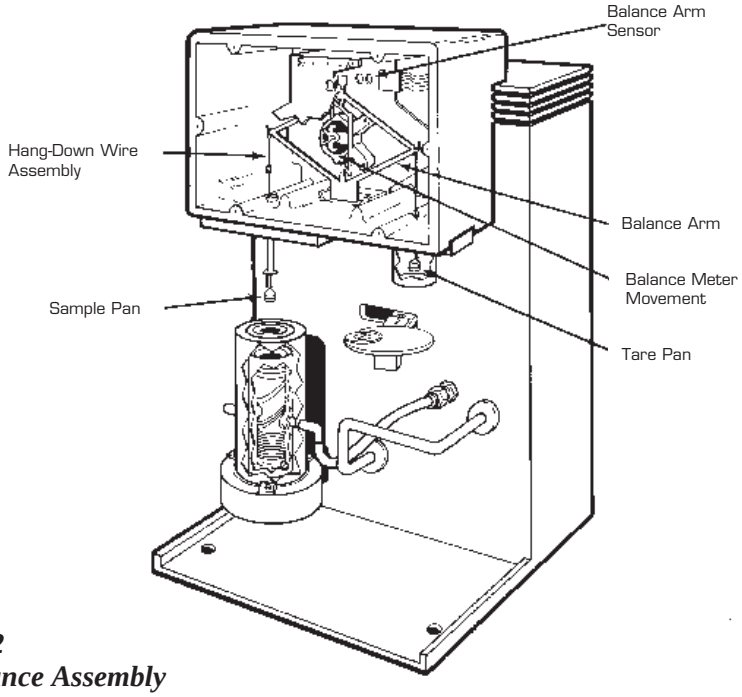


Figure 4.2
TGA Balance Assembly

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. All pans are 0.4 inches in diameter.

The *tare pan* holds the counterbalance weight that mechanically subtracts out the weight of the sample pan and hang-down wire.

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.

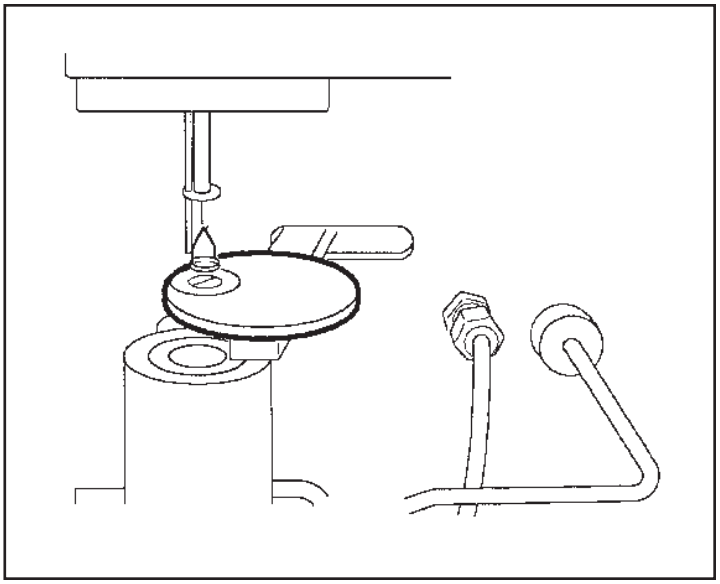


Figure 4.3
Sample Loading Assembly

Furnace

Two types of furnaces may be used with the TGA 2950 CE instrument, the standard TGA furnace and the Evolved Gas Analysis (EGA) furnace. The EGA Furnace (see Appendix D) is an optional accessory that allows you to connect a spectrometer to the instrument so that the gases evolved by sample decomposition can be analyzed.

TGA Standard Furnace

The TGA standard furnace (Figure 4.4) consists of a furnace housing, a heater, and a furnace base that moves these parts up (to closed position) and down (to open position).

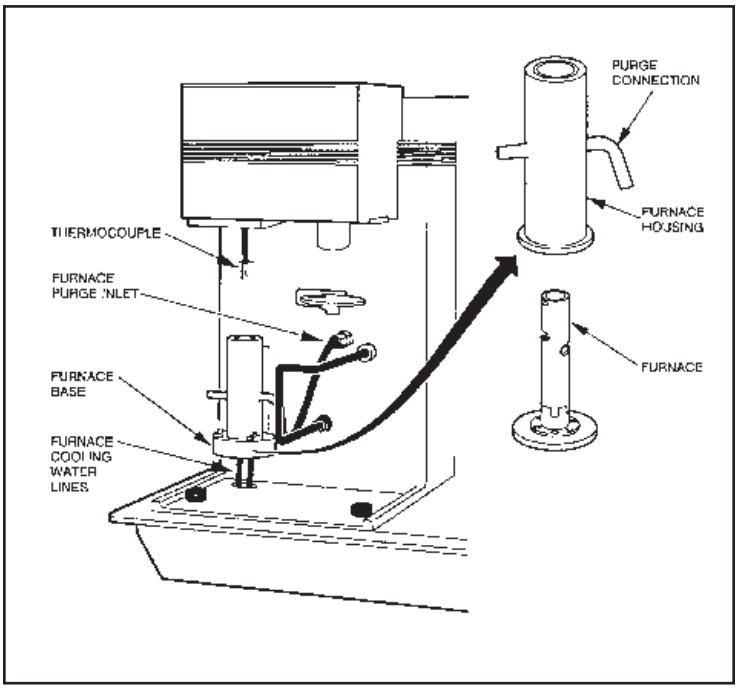


Figure 4.4
TGA Furnace

The stainless steel *furnace housing* has a purge opening on either side to allow gas flow or evacuation. Purge gas enters through a purge fitting on the right side of the furnace housing. It then passes through an opening in the heater, flows around the sample pan, and exits through openings on the opposite side of the furnace housing and heater. From here, the evolved gas can be sent through appropriate connections to an external analyzer, if desired.

The *heater* is a resistance-wound unit of low thermal mass alumina material that can be heated and cooled rapidly. Controlled heating rates of up to 100°C per minute can be obtained, to an upper limit of 1000°C.

Cooling air enters through the holes in the base of the furnace assembly at the completion of test runs, if desired.

A Platinel II* *thermocouple* extends through the bottom of the balance chamber, down along the hang-down wire, and is positioned 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.

EGA Furnace

The (Evolved Gas Analysis) EGA 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 4.5.

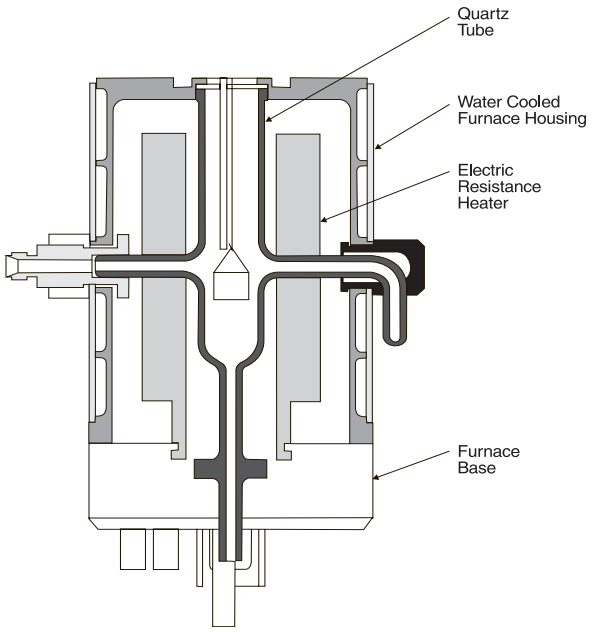


Figure 4.5
EGA Furnace

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 an 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. This design completely separates 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.

The *thermocouple and furnace base* are the same as those described for the standard TGA furnace.

*Platinel II is a registered trademark of Engelhard Industries.

Cabinet

The TGA cabinet (Figure 4.6) consists of the cabinet housing, the instrument display and keypad, the electronics compartment, the purge and cooling gas fittings, and the rear panel.

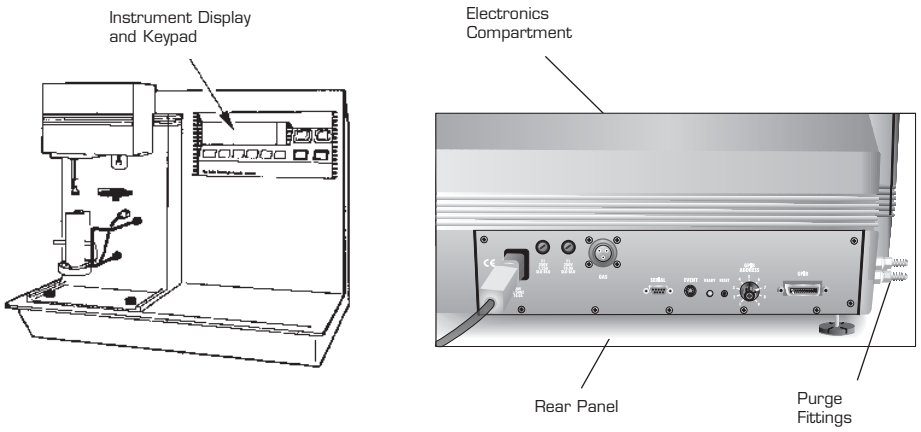


Figure 4.6
TGA Cabinet Components

The TGA *cabinet housing* consists of a base casting, a rear cover, and a sample preparation tray. The base casting is a one-piece casting of heavy-weight aluminum, designed to provide a stable platform for the TGA instrument parts. The rear cover is injection molded using a heavy-gauge thermoplastic material, designed for easy cleaning. The removable sample preparation tray is located on the right side of the instrument. The tray is designed to keep liquids from spilling into the instrument.

The *instrument display and keypad* are described in Chapter 1.

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

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

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

The *rear panel* (Figure 4.7) has the signal and power connections for the instrument, the fuses, the Ready light, the address selector dial, and the Reset button.

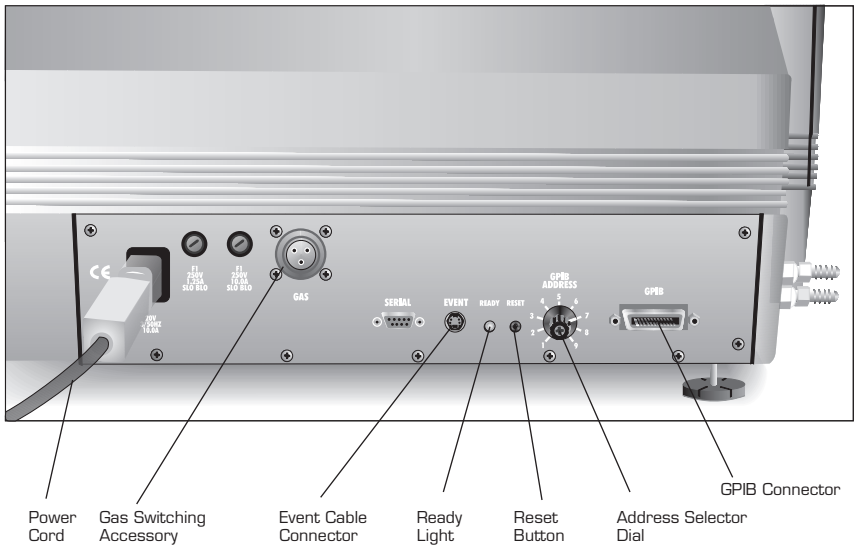


Figure 4.7
Rear Panel

Heat Exchanger

The heat exchanger (Figure 4.8) consists of a fan, a radiator, a water reservoir, and a pump.

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* pushes the water through the system.

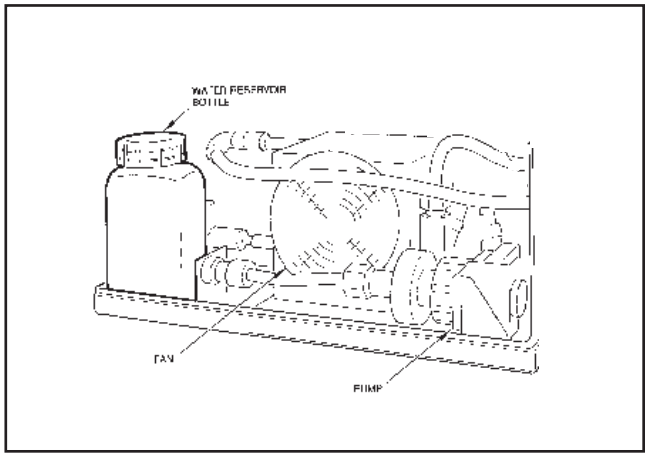


Figure 4.8
Heat Exchanger

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 character strings that are continuously displayed at the top left of the TGA instrument display. These codes tell you what segment in the method is currently being performed by the instrument.

Table 4.1
Status Codes

Code	Meaning
Air Cool	The furnace air cool line has been opened to cool the furnace.
Calib	The TGA is in calibration mode.
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 thermo-couple signal.
Complete	The thermal method has finished.

(table continued)

Table 4.1
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.
Err *n*	An error has occurred. The instrument display will give the error code number (*n*, a two- or three-digit code); the controller screen will also show the complete error message.
Heating	The heater temperature is increasing, as specified by a Ramp segment.
Holding	Thermal experiment conditions are holding; the program is suspended. Choose **Start** to continue the run.

(table continued)

Table 4.1 (continued)

Code	Meaning
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.
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 that the heater switch is in the ON (1) position.

(table continued)

Table 4.1
Status Codes
(continued)

Code	Meaning
Opening	The furnace assembly is opening.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Choose **Start** to continue the method.
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.

(table continued)

Table 4.1 (continued)

Code	Meaning
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.
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 °C	The heater is in stand-by mode, and the experiment has been terminated.
Temp*	Temperature calibration is in effect. The heater is in stand-by mode, and the experiment has been terminated.
Unload	The TGA is unloading a sample from the balance.
Weight #	The weight reading is not stable.

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 or residue should be removed before the next experiment.

Cleaning the Instrument

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 and display.

Cleaning the Furnace Housing

WARNING

Do not touch the EGA furnace sample tube with your bare fingers. Skin oils may cause devitrification of the quartz glass, severely reducing sample tube life. Do not insert instruments inside the sample tube to scrape or chip contaminants from the sample tube, as breakage may result.

TGA Standard Furnace Only

To ensure long furnace life, we recommend that you clean the furnace housing at least once a month to remove condensation materials.

Use the following procedure to remove and clean the furnace housing:

WARNING

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

1. Turn the HEATER power switch to the OFF (0) position.
2. Press the FURNACE key to open the furnace completely.
3. Unload any pans, if necessary.
4. Disconnect the purge tube from the furnace housing purge connection.

5. Loosen and remove the nut and lockwashers holding the ground wire to the rear of the furnace housing, and disconnect the wire from the housing.

♦ ***CAUTION:*** **Take care not to drop the nut or lockwashers into the TGA cabinet.**

6. Loosen the three (3) hold-down screws on the clamps securing the furnace housing flange to the furnace base (see Fig. 5.1), and turn the clamps a quarter-turn clockwise to move them off the flange.

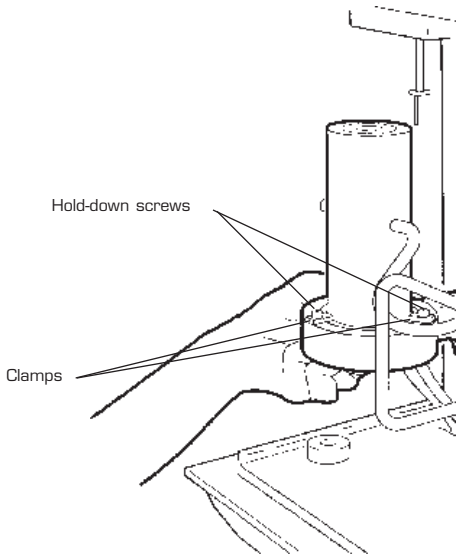


Figure 5.1 Location of Furnace Housing Hold-Down Screws and Clamps

7. Carefully lift the furnace housing up and over the furnace to the left. (You may have to move the thermocouple in order to lift the furnace housing completely off the furnace.)

8. Lay some paper towels down, and invert the furnace housing over them.

9. Clean the inside of the housing with a solvent and cotton swabs.

NOTE: Dry the housing and purge ports with air to remove any traces of solvent before replacing the housing on the furnace.

10. Replace the housing on the furnace by reversing the procedure you used to remove it—lower the housing carefully over the furnace, position the clamps over the housing flange, tighten the hold-down screws, reattach the ground wire with its nut and lockwashers, and reconnect the purge tube.

!WARNING **For continued protection against electric shock, the ground wire *must* be securely connected.**

NOTE: Remember to replace the thermocouple if you had to move it to lift up the furnace housing.

11. Purge the system for 1 hour with nitrogen.

12. After cleaning and replacing the furnace housing, heat the TGA 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, problems with your instrument could result.

WARNING

Do not put any liquid other than distilled water and TA Conditioner 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 heat exchanger POWER switch, and disconnect the power cable and water lines from the heat exchanger (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 power cable and water lines to the heat exchanger.

   d. Turn on the heat exchanger POWER switch.

   e. Allow the water to circulate for several minutes.

   f. Turn off the pump, and check the clarity of the water in the reservoir bottle.

   g. If the water clarity is still unacceptable, repeat steps 1 through 3f.

   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 952377.901) and fresh distilled water as directed in Chapter 2.

5. Turn on the pump again, and circulate the water until the air bubbles disappear from the water lines.

6. Replace and tighten the water reservoir cap.

Replacing the Thermocouple

1. Unload the sample pan and open the 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).

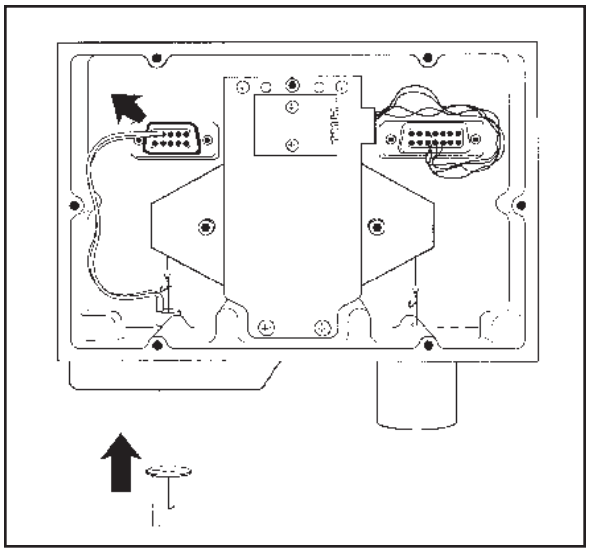


Figure 5.2 Removing the Thermocouple

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

6. Plug the new TGA thermocouple into the connector.

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

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

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

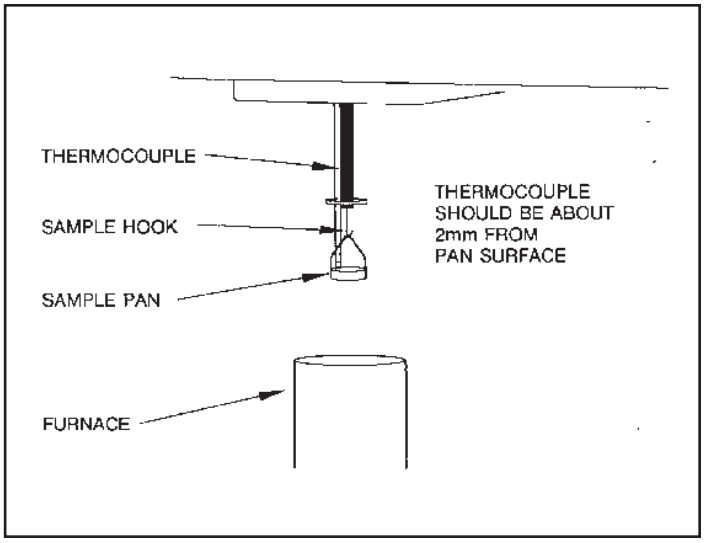


Figure 5.3 Checking Position of Thermocouple

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 blows, 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.**

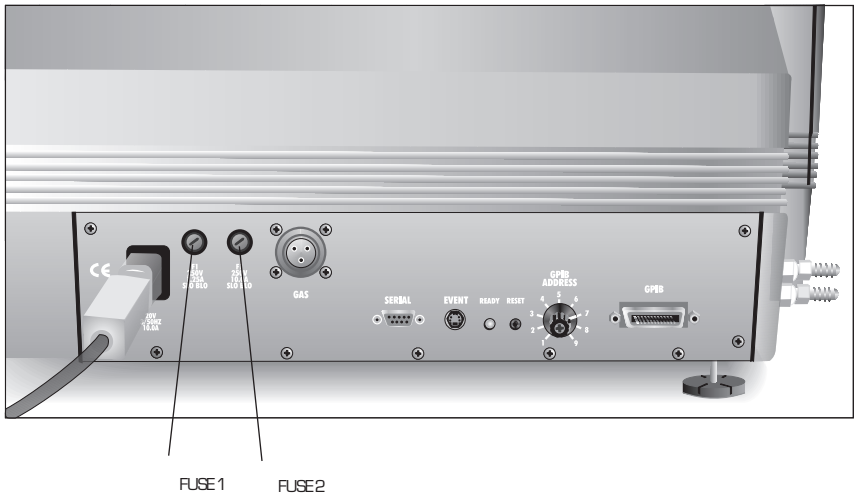


Figure 5.4
Fuse Locations

Fuse 1 is in the circuit between the POWER switch and the instrument control circuits. 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 is in the circuit between the main electrical input and the POWER switch. This fuse protects all internal components, including the furnace and power supplies.If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Furnace Power Check

Furnace power is always checked at the beginning of a method. Power supplied to the furnace is switched by a computer-controlled relay as well as by the HEATER switch located on the instrument's front panel. The HEATER switch must be ON (1) to start a method.

NOTE: The light in the HEATER switch will glow only after an experiment is initiated. If the heater loses power during an experiment, the heater switch will continue to glow, even if it is switched to the OFF (O) position, until the "Stand By" status code is displayed.

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, and you may see “Err F02” on the display. This message means that the system has detected a power failure and has shut down. The instrument will not restart until it is reset. To reset, press the Reset button on the TGA’s back panel.

If “Err F02” appears at startup and remains even after you have tried to restart the instrument, the detection circuitry itself is probably at fault. Do not try to repair it yourself; call your TA Instruments service representative.

The TGA is designed for a nominal line voltage of 120 $\pm$ 10% volts AC, 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 2950 CE Test Functions

The TGA 2950 CE 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 functions.
- 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 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.

When the confidence test is running, the number of the test currently being performed is shown on the display. The test number appears as a two-digit hex number on the lower right of the display. This number is changed as each new test is started. Most of the tests are very brief, so their test numbers may not be apparent.

A standard TGA system takes about 12 seconds to run the confidence test. The longest tests are the RAM tests, which take about 6 seconds.

After the tests are completed, a sign-on message is displayed for 3 seconds. The system then starts running, and the Ready light on the back of the instrument glows.

If an error is detected, an error message is posted on the bottom line of the display. Nonfatal errors are displayed for 3 seconds, and then the confidence test continues. A fatal error occurs when a circuit essential to the operation of the instrument has failed the confidence test; the instrument cannot reliably perform any further functions. The system stops when the fatal error is posted, and the Ready light remains off.

Table 5.1 summarizes the primary confidence tests for the TGA. If any errors occur during the confidence test, call your TA Instruments service representative.

Table 5.1
TGA Confidence Test

Test Number	Area Being Tested
—	CPU logic
30	CMOS RAM
4*n*	Program memory
5*n*	CPU board I/O functions
6*n*	DRAM data storage memory
70	GPIB test
82	Keypad test
A*n*	Analog board tests
B*n*	Drive board tests
D0	Saved memory checksum

Replacement Parts

Replacement parts for the TGA 2950 CE are available from TA Instruments and are listed in Table 5.2.

Table 5.2 List of TGA 2950 CE Parts

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	Calibration weight kit (100 mg and 1 gm)
269931.001	Cal. wt. 100 mg
269931.002	Cal wt. 1 gm
952018.908	50 µL platinum sample pan kit
952323.902	100 µL aluminum sample pan kit
952018.909	250 µL ceramic sample pan kit
952018.910	500 µL ceramic sample pan kit
952384.901	TGA Temperature Calibration kit
952385.901	TGA nickel reference material
952398.901	TGA alumel reference material
852013.901	Furnace assembly

(table continued)

Table 5.2
List of TGA
2950 CE Parts
(continued)

Part Number	Description
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
952082.901	Furnace motor assembly
952121.001	Work surface tray
952183.901	Aluminum temperature calibration standard
900905.901	Calcium oxalate sample
990806.901	Air purge valve assembly
952324.001	TGA 2950 CE keypad assembly
890032.001	Instrument keypad
990828.901	Power supply assembly
990850.901	Central processor PCB
990880.901	Communications PCB
990870.901	Triac drive PCB
990630.901	Overload protection PCB
990810.901	Transformer replacement kit
259508.000	Brass tweezers
259509.000	Spatula, curved, 165 mm long
265303.001	Instrument display
265749.001	O-ring, bottom of furnace housing
200063.029	O-ring, bottom cap plate of furnace

(table continued)

Table 5.2 (continued)

Part Number	Description
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)
852160.901	TGA 2950 CE Cooling Accessory
852160.903	Heat exchanger fan/ assembly
852162.901	Cooling Accessory tubing
952166.001	Cooling Accessory water reservoir bottle
952172.901	Cooling Accessory pump assembly
852161.901	Flow switch assembly
952377.901	Conditioner Kit

Appendix A: Ordering Information

Email address: http://www.tainst.com; click on "Answerman" icon.

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: 1-302-427-4000 or 1-302-427-4040
Fax: 1-302-427-4001

HELPLINE—U.S.A.
For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis Help Desk at 1-302-427-4070.

SERVICE—U.S.A.
For instrument service and repairs, please call 1-302-427-4050.

TA Instruments Ltd.
Europe House, Bilton Centre
Cleeve Road
Leatherhead, Surrey KT22 7UQ
England
Telephone: 44-1372-360363
Fax: 44-1372-360135

TA Instruments GmbH
Max-Planck-Strasse 11
D-63755 Alzenau
Germany
Telephone: 49-6023-9647-0
Fax: 49-6023-9647-77

TA Instruments Belgium
A Division of Waters s.a./n.v.
Raketstraat 60
B-1130 Brussels
Telephone 32-2- 706 00 80
Fax 32-2- 706 00 81

TA Instruments The Netherlands
A Division of Waters Chromatography B.V.
Florijnstraat 19
4879 AH Etten-Leur
Telephone 31-76- 508 72 70
Fax 31-76- 508 72 80

TA Instruments Japan
No. 5 Koike Bldg.
1-3-12 Kitashinagawa
Shinagawa-Ku, Tokyo 140
Japan
Telephone: 813/3450-0981
Fax: 813/3450-1322

TA Instruments France
B.P. 608
78056 Saint-Quentin-Yvelines
Cedex
France
Telephone: 33-1-30-48 94 60
Fax: 33-1-30-48 94 51

TA Instruments Spain
Waters Cromatografia, S.A.
División TA Instruments
Avda. Europa, 21. Pta. Baja
28108 Alcobendas
Madrid, Spain
Telephone: 34-91-203-9100
Fax: 34-91-661-0855

TA Instruments Australia
Unit 3
38-46 South Street
Rydalmere NSW 2116
Autstralia
Telephone: 61-29-9331-705
Fax: 61-29-8981-455

TA Instruments Italy
Division of Waters SpA
via Achille Grandi 27
20090 Vimodrone (MI), Italy
Telephone: 39-02-27421-1
Fax: 39-02-250-1827

Printed in U.S.A.

Appendix B:
High Resolution™ TGA Option

Overview

This appendix describes the use of the TGA 2950 CE High Resolution option.

Some of the benefits provided by the Hi-Res™ option are:

- Improved transition resolution
- Faster survey scans
- Enhanced signature analysis capability
- Transition temperatures closer to isothermal values
- Greater method programming versatility.

Three method programming steps (segments) are used for the high resolution TGA capability:

- High Resolution Ramp
- High Resolution Sensitivity
- Abort Next Segment.

These segments make method programming more versatile and powerful than ever before. The Hi-Res™ ramp can be used alone as a simple single-segment method, or the method segments can be combined with more traditional segments, such as constant heating rate ramps and timed isothermal periods, for maximum programming flexibility.

What these segments do and how to use them to control TGA experiments and improve transition resolution are described in the following sections. For further details about creating methods, refer to the online help and documentation.

Option Installation

The Hi-Res™ TGA option can be installed by qualified service personnel, or you can install it yourself using the separate installation procedure included with the Hi-Res™ option kit. For this option to run on your TGA 2950 CE, the following items are required:

- Either of the following software items:

  — Version 2.0 or higher TGA 2950 CE RMX Instrument Software (included in kit) and version 8.2 or higher RMX controller operating system

  or

  —Any version of *Thermal Solutions/ Advantage* software

- Instrument "Software Option" circuit board (included in kit)

- Hi-Res™ TGA software option key (included in kit)

A TGA instrument with Hi-Res™ capability properly installed can be identified by the "Hi-Res TGA Installed" message on the instrument display screen following the confidence test, and by the letters "HR" in the instrument identification string on the configuration screen of the controller (*e.g.*, "TGA 2950 CE HR V2.0A").

Using Hi-Res™ TGA

Background

Thermogravimetric analysis is particularly useful for observing the thermal decomposition of compounds. When individual thermal decompositions occur at well-separated temperatures, quantitative information about sample composition can be obtained from the percent weight change at each transition. However, many TGA decomposition transitions overlap or appear drawn out in temperature because of the time-dependent nature of the reactions taking place. This overlap substantially reduces the ability to obtain an accurate measurement of weight change and reaction temperature.

It has long been known that using very slow heating rates improves the separation of some overlapping transitions and, thus, increases the resolution of the TGA scan. Another technique for increasing resolution is to raise furnace temperature until the onset of decomposition and then hold temperature isothermally until the decomposition is complete; then to raise the temperature again until the next decomposition begins; and so on, until the final temperature of interest is reached. A third technique is to control the furnace temperature so as to maintain a preselected constant reaction rate (percent/minute). This technique results in slower or even negative heating rates during a transition, giving the reaction more time to reach completion before the next transition is encountered.

The major drawback to these techniques is that they increase substantially the total time required for a measurement, thereby reducing laboratory productivity. Moreover, increasing the measurement time often reduces the accuracy and reliability of the analysis: Exposing the sample to high temperatures for long periods may cause slow time-dependent changes, such as oxidation and absorption, or exposure to changing ambient conditions, such as humidity and pressure.

The TA Instruments Hi-Res™ Technique

The TA Instruments Hi-Res™ technique, dynamic rate TGA (DRTGA), avoids some of the problemes just described. The heating rate of the sample material is dynamically and continuously modified in response to changes in the rate of decomposition of the sample so as to maximize weight change resolution. This technique allows the use of very high maximum heating rates during Hi-Res™ ramp segments while avoiding transition temperature overshoot. The time needed to complete typical Hi-Res™ ramps is often the same as or less than that for a comparable constant heating rate experiment run at a lower heating rate, and the resolution is improved.

The Hi-Res™ Ramp Segment

The Hi-Res™ ramp segment varies the heating/cooling rate of the furnace in response to changes in the rate of decomposition of the sample so as to improve weight change resolution. This segment has the following format:

Ramp <rate>°C/min res <res_setting> to <temp>°C

where:

<rate> is the maximum ramp heating rate (0.01 to 200 °C/minute)

<res_setting> is the resolution setting (–8.0 to +8.0)

<temp> is the ramp final temperature (up to 1000 °C).

Example:

Ramp 50.00°C/min res 4.0 to 800.00 °C

The Hi-Res™ ramp segment operates like the traditional constant heating rate ramp segment, except that the heating rate is varied dynamically during the ramp in response to the derivative of weight change (percent/minute). As percent/minute increases, heating rate is decreased. As percent/minute decreases, heating rate is increased. The heating rate is constrained to the range 0.001 °C/minute (minimum) to the

maximum specified in the ramp segment. The resolution setting is a unitless number used to select the most useful band of percent/minute values for proportional heating rate control.

Higher resolution settings select lower percent/minute values, and generally result in greater resolution and longer experiment times. Lower settings have the opposite effect.

Resolution settings may be selected anywhere in the range of –8.0 to +8.0. Positive settings indicate that dynamic rate mode is to be used during the ramp. Negative settings indicate that constant reaction rate mode is to be used. More details on using each mode are given in the section on advanced Hi-Res™ techniques.

Positive resolution settings are more universally useful and less likely to have undesirable side effects. Although there are no hard and fast rules about which resolution setting to use for a given experiment, the general guidelines offered here may help you select resolution settings for your experiments.

As mentioned, higher resolution settings usually provide better resolution results, and lower settings the reverse. The closer the setting is to zero, the larger the derivative of weight change (percent/minute) must be for a reduction in heating rate to occur. In fact, a resolution setting of exactly zero completely disables the application of the Hi-Res™ technique, resulting in a normal constant heating rate ramp at the maximum rate specified.

A resolution setting of +1.0 produces a TGA scan at 50 °C/minute that roughly approximates the resolution obtained by a constant heating rate scan at 20 °C/minute. In other words, the Hi-Res™ ramp segment enables your instrument

to pass quickly through the baseline sections of your scan at a higher heating rate and to slow down only for transitions, and still obtain the resolution of the slower heating rate scan. For scans that contain a large amount of baseline, this option can result in very significant overall time savings with no loss of resolution. The same speed/resolution relationship applies to other heating rates as well.

Higher resolution settings apply the Hi-Res™ technique more aggressively by reacting to smaller percent/minute values. Some general guidelines for selecting resolution settings are as follows:

- If you are uncertain what the resolution setting and heating rate should be, try setting +3.0 and rate 50 °C/minute. (Negative resolution settings are covered in the section on advanced Hi-Res™ techniques.)

- If you wish to obtain better resolution, try progressively higher resolution settings, 1.0 at a time, while leaving the heating rate the same.

- The most useful resolution settings are 3.0 to 5.0, because they cover the most commonly encountered bands of percent/minute change during typical decompositions. If your decompositions tend to be explosive in nature, with large percent/minute peaks (greater than 50 %/minute), or if you wish to minimize experiment time, use settings less than 3.0. If your decompositions are very gradual (less than 0.5 %/minute peaks), or if it is important to limit the rate of decomposition, try settings higher than 5.0.

Selecting the resolution setting is covered in greater detail in the section on advanced Hi-Res™ techniques.

Guidelines for the choice of heating rates are as follows:

- The most useful maximum heating rates are 10 to 50 °C/minute for positive resolution settings. However, other values are perfectly acceptable when dictated by the needs of your experiment.

- Use lower heating rates when transitions are very closely spaced in temperature, or if the sample material reacts very rapidly. A higher heating rate conventional ramp can be used to skip over baseline sections in order to shorten experiment time. Generally speaking, you do not need to use very slow heating rates, less than 5 °C/minute, because the dynamic rate Hi-Res™ technique automatically reduces heating rate with the accompanying improvement in resolution.

There are no special method programming constraints on Hi-Res™ ramp segments. They may appear anywhere in a method that a normal ramp could appear. You can change the maximum ramp rate, resolution setting, and final temperature of an executing Hi-Res™ ramp using the Modify Segment function on the controller.

Calcium Oxalate Example Scans

In this example, five TGA scans of calcium oxalate monohydrate ($CaC_2O_4 \cdot H_2O$) were run in nitrogen to compare the results using conventional TGA and Hi-Res™ TGA. In all cases, a single ramp or Hi-Res™ ramp segment from ambient to 800 °C was used for the method.

Figures B.1 and B.2 show the results of constant heating rate scans at 20 °C/minute and 1 °C/minute, respectively. Figures B.3, B.4, and B.5 show the results of Hi-Res™ scans at 50 °C/minute with resolution settings 3.0, 4.0, and 5.0, respectively. Figure B.6 shows a composite plot of the derivative of weight loss for each of the five scans. The derivative smoothing window for all plots was set to 5 °C.

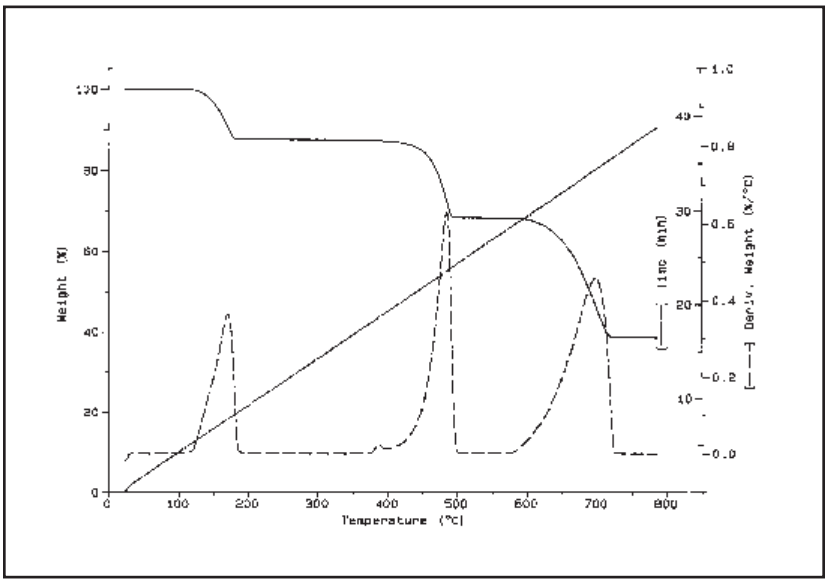


Figure B.1
Constant Heating Rate Scan at 20 °C/minute

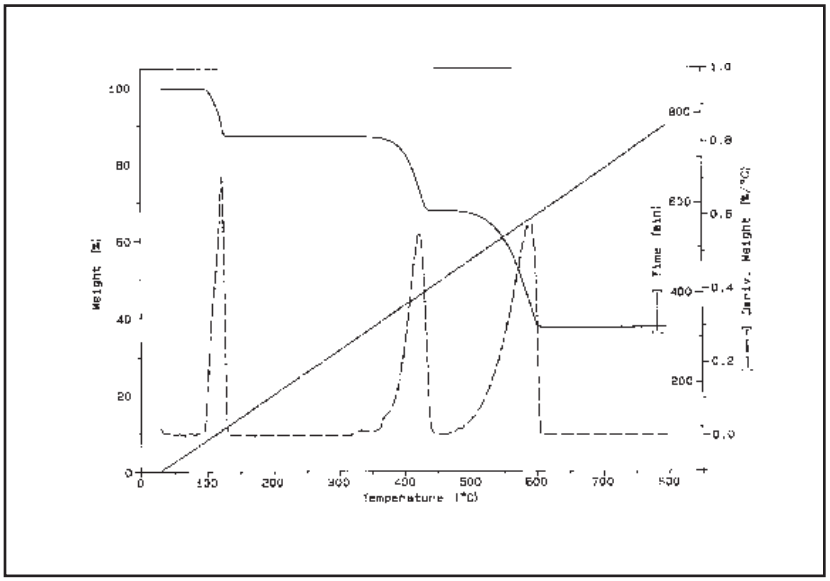


Figure B.2
Constant Heating Rate Scan at 1 °C/minute

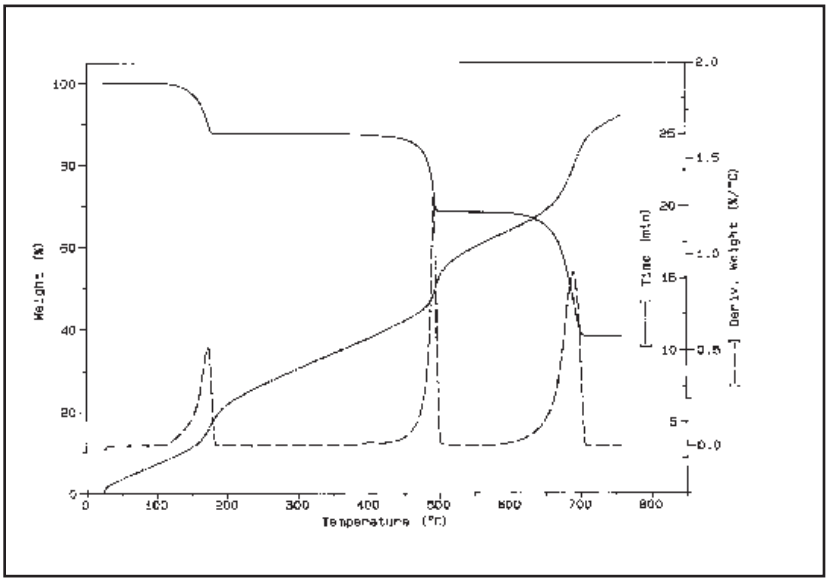


Figure B.3
50 °C/min Hi-Res™ Scan with Resolution Setting 3.0

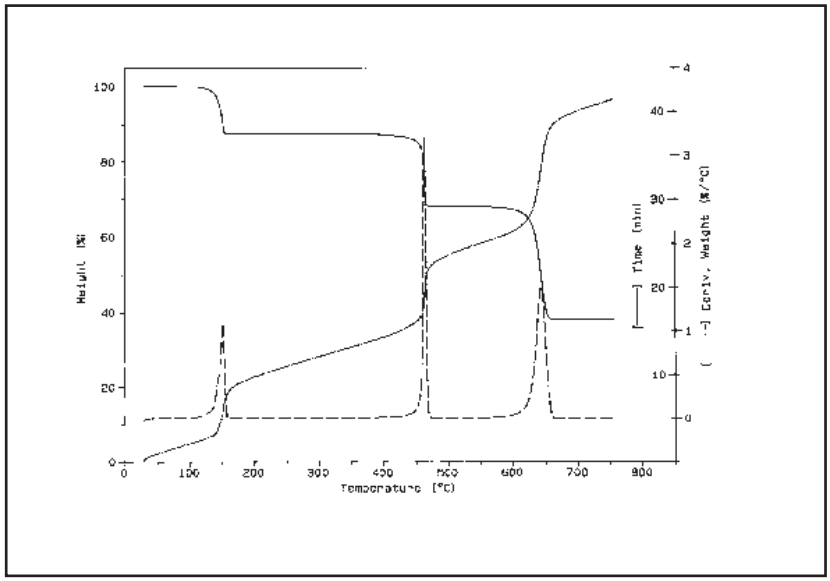


Figure B.4
50 °C/min Hi-Res™ Scan with Resolution Setting 4.0

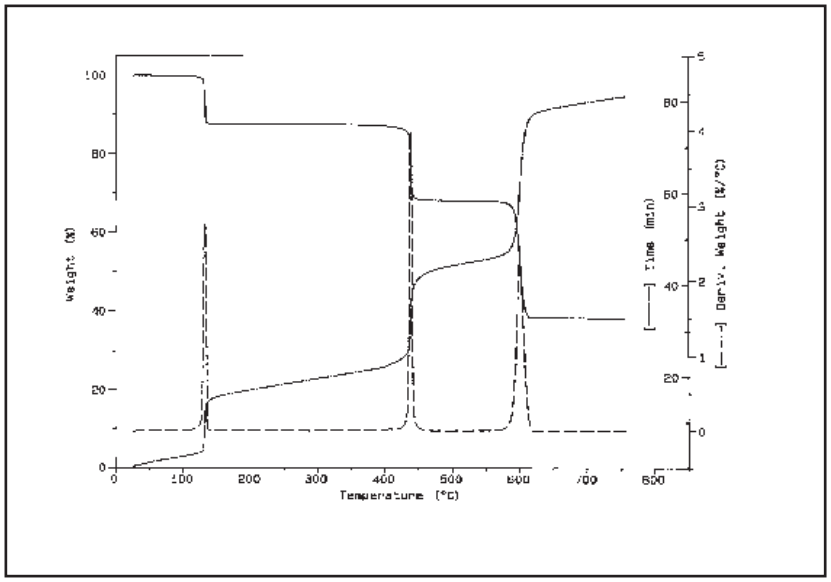


Figure B.5
50 °C/min Hi-Res™ Scan with Resolution Setting 5.0

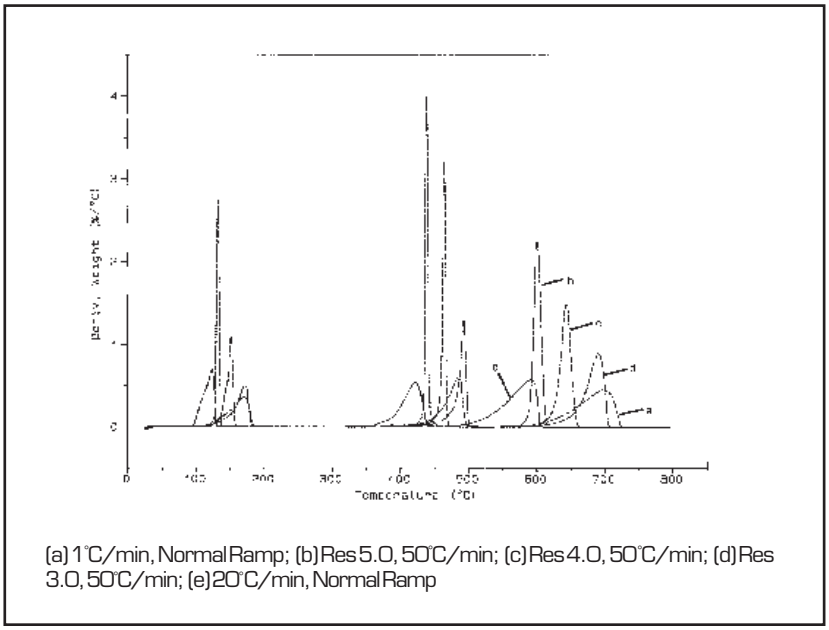


Figure B.6
Composite Weight Loss Derivative Plot from Figures B.1 through B.5

As can be seen in these examples, calcium oxalate has three well-resolved transitions corresponding to the loss of water (first weight loss), carbon monoxide (second weight loss), and carbon dioxide (third weight loss). Comparing the 20 °C/minute and the 1 °C/minute scans (Figures B.1 and B.2) shows some improvement in resolution of the water loss (first transition), but little improvement in the other two transitions.

Note that the transition temperatures in the slower scans are shifted to lower temperatures as expected. The 20 °C/minute scan took 39 minutes, and the 1 °C/minute scan nearly 13 hours, to complete.

Comparing the results of the two constant heating rate scans (Figures B.1 and B.2) with the Hi-Res™ scans (Figures B.3, B.4 and B.5) shows that:

- The resolution 3.0 scan (Figure B.3) gave comparable results in two thirds the time required for the 20 °C/minute scan.
- The resolution 4.0 scan (Figure B.4) gave much better resolution in about the same time as the 20 °C/minute scan.
- The resolution 5.0 scan (Figure B.5) gave a dramatic improvement in resolution in only twice the time required for the 20 °C/minute scan.

Figure B.6 shows a plot of the weight percent derivatives from each of the calcium oxalate scans overlaid on the same scale. Note how much taller and narrower the Hi-Res™ peaks are than those on the conventional scans. Also, transition temperature clearly is reduced by increasing the resolution setting. This effect is normal, because with progressively higher resolution settings, transitions are constrained to lower decomposition rates, which can be maintained only at lower temperatures.

As can be seen in these simple examples, the Hi-Res™ ramp segment is easy to use and provides significant resolution improvement, in the same time frame as conventional constant heating rate TGA. The following section on advanced Hi-Res™ techniques contains valuable information about additional Hi-Res™ methods and adjustments to help you obtain maximum performance from this powerful analysis tool.

Advanced Hi-Res™ Techniques

This section discusses in greater detail how to use the Hi-Res™ features and gives specific advice about setting up your own experiments. It is intended to help you become familiar with the options and adjustments and gain knowledge about what you can and cannot expect from the analysis.

What Can Be Resolved and What Cannot?

"Will Hi-Res™ TGA improve the resolution of my transitions?" is one of the first questions asked by most people when they are introduced to Hi-Res™ TGA. In some applications, resolution enhancement will be minimal. Therefore, some criteria for selecting likely candidates for resolution improvement are needed. Here are some guidelines to use when you are considering a new material, or when you try the Hi-Res™ techique on a sample and no resolution improvement is obtained. (It is worth noting that substantial productivity gains are still possible, even if transition resolution is not improved.)

Unresolved Transitions

The Hi-Res™ techniques provide useful tools to improve transition resolution of many sample materials. Some materials, however, show little or no resolution improvement, because they have transitions that cannot be separated by time

and temperature alone. The transitions are usually overlapped so that the components of interest decompose at or very near the same temperature and at approximately the same rate of reaction. For these materials, you may have to use other techniques separately or in conjunction with TGA, such as vacuum, switching purge gases, semi-pressurized sample containment, or evolved gas analysis.

Considering the following issues can help you decide whether the material you are working with is a good candidate for resolution improvement:

- **Do the components of the sample material decompose at sufficiently different temperatures?** This can often be determined by running a slow heating rate survey scan of the material at 1 °C/minute and comparing the result with that of a 20 °C/minute scan of the same material. Generally, the temperatures of transition are lower in the slower scan. If there is no observable improvement in separation of the components, it is likely that Hi-Res™ TGA will not produce a significant separation of the components either.

- **Choice of purge gas.** Always consider purge gas to be a factor when running survey scans or heating rate trials. Generally, nitrogen and air are the most common choices for purge. In some cases, transitions that appear as one large weight loss in nitrogen are separate in air because of reaction with oxygen in the purge stream. Switching from air to nitrogen may help to eliminate oxidation, which tends to counteract a simultaneous weight loss. A

few materials are reactive with nitrogen and are better run in argon. Also, consider the purity and moisture content of the purge gas. Adding or removing moisture may change the rate or nature of the reactions taking place. When the nature of the sample is relatively unknown, we suggest repeating the scans using both air and nitrogen to see whether any transitions appear or disappear, or shift in temperature or weight loss.

- **Do the components of the sample material have significantly different rates of decomposition?** If so, it may be possible to improve resolution using either stepwise isothermal heating, or the constant reaction rate Hi-Res™ mode (negative resolution settings). To find out, run a conventional constant heating rate ramp up to the transition temperature and then hold isothermally. After the decomposition is complete, plot the derivative of weight percent versus time, and look at the shape of the curve. If the curve shows a continuous exponential decay, the two components of the transition are decomposing at about the same rate. However, if the curve shows a rather rapid exponential decay followed by a very gradual and somewhat constant rate of weight change, the components have different reaction rates, and improvement in separation is probably achievable with the Hi-Res™mode.

- **Controlling the atmospheric pressure with sample containment**. Controlling the atmospheric pressure surrounding the sample while it is in the furnace may improve the resolution. Place the sample in a semi-sealed container such as a hermetic

DSC pan with a very small pin hole (0.1 mm or less in diameter) or in a sample cup with a lid. When the sample starts to react, the evolved gas will build a slight vapor pressure inside the sample container. This pressure may reduce or stop one or more of the overlapped reactions, thereby allowing completion at a higher temperature. To make this test, run the sample using a Hi-Res™ ramp both with and without the sample containment to see whether there is a difference in separation. Generally, the temperature of reaction shifts higher, even if no improvement in separation occurs. The pressure containment technique is particularly helpful when the constant reaction rate Hi-Res™ mode (negative resolution settings) is used.

Selecting a Hi-Res™ Technique

The Hi-Res™ TGA option actually consists of several techniques, all of which control the thermal experiment on the basis of changes in the sample weight. Both absolute weight change and the rate of weight change can be utilized. These capabilities add a whole new dimension to TGA experiments.

We have already given some general guidelines as to how to set some of the parameters involved in using Hi-Res™ TGA. Here, we consider more closely why one technique may give better results than another, and present additional guidelines for selecting techniques and setting parameters.

Dynamic Rate Hi-Res™ Ramp

In dynamic rate Hi-Res™ mode, one or more Hi-Res™ ramp segments are used with positive resolution settings. In this mode, the furnace heating rate is varied between a fixed minimum and the maximum specified in the ramp segment, but is never reduced to zero (isothermal). A mathematical function is used to relate the rate of weight change (percent/minute) to the sample heating rate (°C/minute). Dependent variables in this function are resolution setting, sensitivity setting, and maximum heating rate. Independent variables are both short-term and long-term rates of weight change (percent/minute), time, and temperature. The result is the ability to directly compute the appropriate heating rate for the current weight change conditions.

Because the dynamic rate Hi-Res™ mode reduces heating rate smoothly and only when necessary, it is the fastest and most reliable of the various techniques. This mode gives good results with most temperature-separable transitions. It is preferred for fast survey scans of unknown materials over wide temperature ranges. If no other criteria exists to select a Hi-Res™ technique, then dynamic rate is the preferred choice.

Constant Reaction Rate Hi-Res™ Ramp

In constant reaction rate Hi-Res™ mode, one or more Hi-Res™ ramp segments are used with negative resolution settings. In this mode, the heater control system varies the temperature of

the furnace as required to maintain a constant preselected rate of weight change (percent/minute). Whenever the rate of weight change exceeds the percent/minute threshold, the heating rate of the furnace is reduced, even to the point of cooling if necessary. When the rate of weight change falls below the threshold, the heating rate is increased up to the maximum specified for the ramp segment. Transition resolution is improved because sample heating is reduced or reversed during transitions, allowing one transition to complete at the selected reaction rate before the next transition is begun.

Figure B.10 on page B-49 shows a good example of a material (sodium bicarbonate) that was analyzed using a constant reaction rate ramp at 10 °C/minute with resolution setting –4.0.

Constant reaction rate Hi-Res™ mode is recommended for the following situations:

- **Sample Containment:** When used with semi-pressurized sample containment, constant reaction rate mode becomes even more powerful. The vapor pressure that builds up inside the sample container limits the rate of reaction of the sample. This allows the reaction to complete at a nearly constant rate and temperature. The reaction progresses more uniformly throughout the sample, because vapor pressure gradients in and around the sample material are substantially reduced. The onset of higher temperature reactions is effectively suppressed until lower temperature reactions are complete.

- **Reaction Control:** Constant reaction rate

mode is preferred for any situation in which it is important to limit or control the rate of reaction. Examples are pyrotechnics, self-heating reactions, auto-catalyzing reactions, and gas diffusion reactions. Constant reaction rate mode is also a good choice when you want to accurately determine the transition temperature at a given reaction rate.

- **Small Transition with Large Weight Change:** Another situation in which constant reaction rate heating can be helpful is when the sample material exhibits a relatively large and somewhat constant background weight change, onto which is superimposed a relatively small transition. If you choose a decomposition rate threshold that is close to the background percent/minute at the maximum heating rate, the heating rate will be changed significantly only when the smaller transition occurs.

Derivative of weight change curves that are plotted versus temperature may appear cyclic and have negative peaks as well as positive ones. This effect is caused by the automatic application of cooling whenever the rate of weight change (percent/minute) exceeds the specified set point. In many cases, the appearance of the derivative curve can be improved by increasing the derivative smoothing window in the data analysis program.

Constant reaction rate mode works best at lower heating rates (1 to 10 °C/minute), at which significant reaction rate and transition temperature overshoot can be avoided. Usually, several

scans of the same material are required to determine the best reaction rate threshold to use.

If the sample material is very reactive, or if it is important not to overshoot the selected reaction rate, then even lower maximum heating rates may be required, particularly for very small reaction rates (less than 0.1 %/minute).

Because the heater control system concentrates on a very narrow band of reaction rates, transitions with slightly different reaction rates in the same scan are often given very different treatment. For example, a transition that falls short of the percent/minute threshold may be passed at a fairly high heating rate, with results similar to those of conventional constant heating rate TGA. However, a transition that just crosses the percent/minute threshold may cause a significant reduction in heating rate or even reversal of the heating process. The two transitions, although similar in nature, may appear quite different on a plot of weight change versus sample temperature.

This effect can be observed in the weight loss curve of Figure B.10 (page B-49). Note that the surface water loss at 85 °C and the bicarbonate transition at 100 °C are treated quite differently. This effect is most noticeable at low sensitivity settings. The effect can be reduced or eliminated by using multiple ramp segments in the method, each tailored to the needs of specific transitions. Increasing sensitivity setting may also be helpful. (See the section on adjusting sensitivity setting in constant reaction rate mode).

Weight Gain Experiments

Although most TGA work involves decomposition analysis, some applications involve weight gain, such as in oxidation studies. The Hi-Res™ heating control techniques apply as well to weight gains as to weight losses. In this case, the absolute value of the weight change signal is used for control. Weight gains of up to 200% can be accommodated. The rates of weight gain (percent/minute) and their relationship to heating rate, resolution setting, and sensitivity setting are exactly the same as for weight loss.

No special parameters or controls are needed for Hi-Res™ weight gain analysis. Combinations of weight gain and weight loss in the same TGA scan are handled automatically. It is important to recognize, however, that when weight gain and weight loss transitions overlap, the resultant weight change is additive and may not be separable.

Signature Analysis

For many materials, it is not possible to separate overlapped transitions sufficiently to allow quantitative analysis of weight change. However, this does not mean that no useful information can be gained from the TGA scan of the material. Frequently, particularly in quality control work, an exact determination of sample composition is not needed. Instead, the requirement is only to identify which material of a group of known standards the unknown sample most closely resembles. Another purpose is to identify lot-to-lot variation from an acceptable standard. In both cases the location, size, and shape of the derivative of weight change peaks of the TGA scan, or the weight curve itself, is used to create a unique pattern or "signature" of the sample material. Signature scans of standards and the unknown material are then compared to make the identification or "accept/do not accept" decision.

Because the various components of a sample, when run separately, usually decompose or evolve at unique and reproducible rates and temperatures, people often think that it should be possible to determine exactly what is in an unknown mixture by comparing a scan of it with a library of known TGA scans of the individual components. Unfortunately, this comparison usually does not work, because the various components of a mixture typically interact with one another, so that the resultant scan is unlike either material run separately. The interactions can be chemical or physical. Some examples are evolved gases from one decomposition that slow or accelerate the decomposition or evolution of another component (*e.g.*, CO_2 from oxidizing carbon). Molecular attractions prevent a more

volatile component from evolving when expected and, at the same time, hasten the evolution of another less volatile component (*e.g.*, chain linkage in polymer blends). The physical matrix of the mixture may retard the evolution of a more volatile component, which then evolves at a higher than normal temperature and at a slower than normal rate (*e.g.*, oil evaporating from rubber). Alternatively, the components may react chemically at elevated temperatures and produce new compounds that decompose at different temperatures from those for the individual components.

Sample Quantity and Orientation

As with conventional constant heating rate TGA, sample quantity and orientation in the sample holder can be important during Hi-Res experiments. This is particularly true if the sample is not homogeneous (*e.g.*, a laminated sheet or coated surface). With these types of samples, it is wise to try scans with different surfaces exposed. Be aware that chopping or grinding the material may produce an entirely different result, because physical or chemical properties may be altered.

Exposed Surface Area

Exposed surface area is often important. When samples melt, they usually spread out over the bottom surface of the sample container. This exposes more or less surface area, depending on whether the original configuration was a single block (area typically increases) or a powder (area typically decreases). Usually, with open

sample pans, it is best to try to maximize the exposed surface area at all times, so that evolved gases escape quickly and reactions proceed uniformly. The best way to do so is to use small powdered or thin samples that are uniformly distributed in the sample vessel. When semi-pressurized sample vessels are used, the issue of exposed surface area is far less important.

Generally, sample sizes in the range of 5 to 15 mg are recommended. If the material is self-heating, or autocatalytic, then smaller sample quantities may help with heating control. (This is important for constant reaction rate Hi-Res™ mode.) On the other hand, larger sample quantities (50 to 100 mg) are recommended for reactions in which a very small weight change (less than 1%) is being measured. For maximum weight resolution, it is advisable to keep sample weight below the TGA 2950 CE weight range change at 100 mg.

A problem with very large samples that decompose rapidly and almost completely is that the furnace purge may not be able to remove all of the evolved components, and some contamination of the furnace wall and cooling jacket may result. This contamination may affect the remainder of the experiment or future scans.

Bubble Formation

Whenever medium to large sample quantities are being run, use caution in placing the thermocouple. Some materials, particularly polymers, form a “skin” on the outer surface of the sample as it is heated, inhibiting mass transport of the more volatile components.

Such samples form bubbles, which can rise enough to touch the end of the thermocouple, ruining the experiment and possibly contaminating the thermocouple. Just as the largest chewing gum bubbles are produced by blowing slow prolonged breaths, the largest gas bubbles form in samples that are heated gradually. For this reason, bubble formation is more of a concern for Hi-Res™ TGA than for conventional TGA.

Another effect of bubble formation is a sudden small unexpected change in weight and an accompanying spike in the percent/minute curve as the bubble bursts. An excellent example of bubble noise can be seen in the scan of ethylene-vinyl acetate in Figures B.7 and B.8. In Figure B.7, the effect of bubble formation and bursting can be seen as a sudden drop in weight at about 400 °C during the second weight loss. This appears in the derivative curve as a peak shoulder. Figure B.8 clearly shows the formation and bursting of several large bubbles in the percent/minute curve between 65 and 85 minutes into the run. As mentioned, these effects tend to be more noticeable in Hi-Res™ TGA, in which larger bubbles form because of the slow heating process. The best solution to bubble noise is to decrease resolution setting and/or increase maximum heating rate. Reducing the sample size may also help.

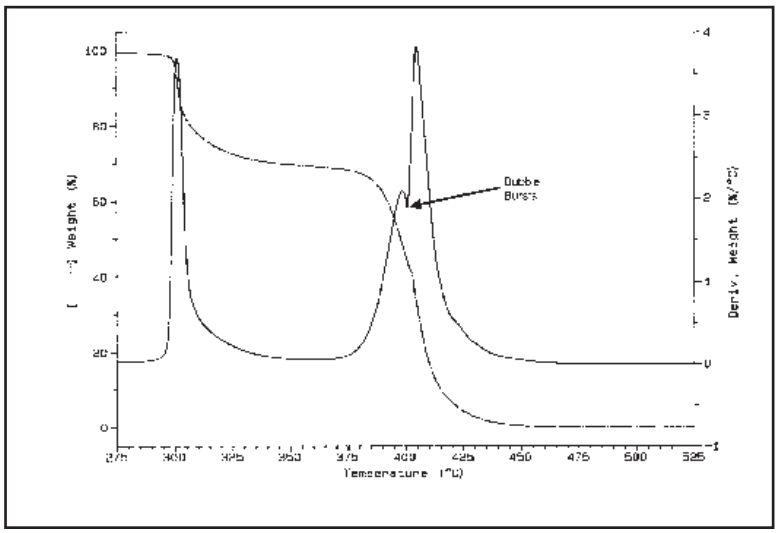


Figure B.7
Ethylene-Vinyl Acetate Scan

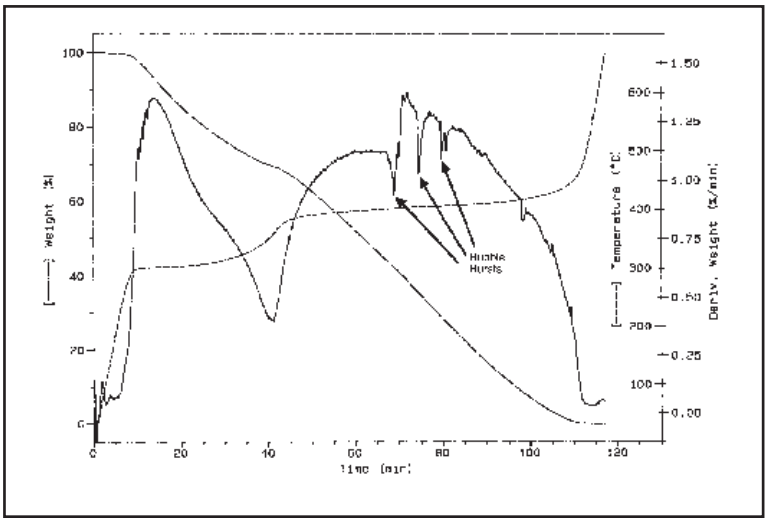


Figure B.8
Ethyl-Vinyl Acetate Scan

Thermocouple Placement

Generally, the thermocouple should be placed 2 to 5 mm above the bottom of the sample container (1 to 4 mm above the edge). Placement 1 mm above the edge is recommended for most applications. Placing it higher does not degrade heater control and is sometimes helpful in avoiding possible adverse effects of sample self-heating or bubble formation.

Data Analysis Effects

Hi-Res™ TGA, because of its technique of changing heating rates dynamically, causes very significant nonlinearity of data points across transition regions when you plot the data points versus temperature. This is not a problem when you are making plots, but if you use automatic limit selection when generating data analysis reports, the analysis program can sometimes become confused about where to place the limits. This situation is signified by curve tick marks and tangent lines that are poorly placed on the weight curve or that may even be plotted off the curve.

The difficulty is caused by the fixed size of the transition onset and endset windows (the first and last 12.5% of the analysis region). The data analysis program assumes that data points are uniformly distributed throughout this region. If the number of data points in the baseline portion of a window is very small compared with the number of points in the sloped portion of the window (the usual case with Hi-Res™ scans),

the tangent lines will be excessively weighted toward this cluster of points and may be nearly vertical or even fall off the curve entirely.

You can obtain better results by moving the transition start and stop limits farther away from the transition, so that the onset and endset windows remain completely on the baseline portion of the curve. If this maneuver fails to help, you can place the transition tick marks manually on the curve.

Derivative Plots

Another data analysis problem caused by unequally spaced temperature data points is unusually shaped or flattened curves when you plot derivative data versus temperature. This problem is caused by the assumption in the data analysis program that data points are equally distributed over a moving window (the smoothing window) used to compute the derivative. If the derivative smoothing window is too wide, derivative peaks will flatten, and apparent resolution will be reduced. If the window is too narrow, derivative peaks will be needle-sharp and noisy.

The default smoothing window is 0.2 minutes for derivatives with respect to time (%/min and %/min/min) and 10 °C for derivatives with respect to temperature (°C/min and °C/min/min). For typical Hi-Res™ TGA scans, you can improve the quality of your derivative plots with respect to temperature by decreasing the temperature window to 5 °C. If extremely sharp results are desired, try a smaller window. Less than 1 °C is not recommended. The smoothing window for time does not normally need adjust-

ment. If the time derivative seems particularly noisy, try increasing the window to 1 minute.

In conventional constant heating rate TGA, the plot of percent/minute and the plot of percent/°C are essentially identical, because there is a direct linear relationship between time and temperature. Percent/minute is conventionally chosen for transition peak analysis and plotting. With Hi-Res™ TGA, there is no linear relationship between time and temperature, because the heating rate is constantly changing. When plotting the derivative of weight change versus temperature for a Hi-Res™ TGA scan, you should use percent/°C. A plot of percent/minute versus temperature can still be useful for locating minor transitions and determining the rate of reactions.

Adjusting the Heating Rate

Heating rate has long been used to control transition resolution. The typical thermal analyst makes most runs at 20 °C/minute and then changes to 5 or 1 °C/minute either for hard-to-resolve transitions, or just to see whether there is anything else interesting going on that might have been missed in the faster scan. Generally, 1 °C/minute scans are not routinely used because of the enormous amount of time required to run them.

With Hi-Res™ TGA, the heating rate is varied automatically to give the benefit of slow heating rates during transitions and fast heating rates during baseline. However, it is still necessary to specify a maximum heating rate. This is because in Hi-Res™ TGA, just as in constant heating

rate TGA, the maximum heating rate affects the results of the experiment and, as such, is an important adjustment to consider.

Heating rate adjustment is particularly critical if a group of overlapped transitions are very closely spaced in temperature. At 50 °C/minute, the normal temperature lags in the TGA furnace can be enough to overshoot the first transition, reducing separation from the other transitions in the group. This effect is most noticeable when the percent/minute baseline preceding a rapid transition is relatively constant and several orders of magnitude below the peak rate of the transition. We recommend running at 20 °C/minute instead of 50 °C/minute for most routine work using positive resolution settings, particularly if you can make only one scan of each unknown material. Running at less than 10 °C/minute is usually not required.

For negative resolution settings, the selection of heating rates is usually quite different, because reaction rate overshoot must be minimized. Generally, 1, 2, 5 and 10 °C/minute are the most useful rates to use with negative resolution settings. Start with 5 °C/minute.

An important difference between the dynamic rate (positive resolution setting) and constant reaction rate (negative resolution setting) Hi-Res™ modes is that the maximum heating rate selected is an upper limit in negative mode but is a gain factor in positive mode. In other words, in negative mode, the maximum heating rate is irrelevant to heating control, except when the reaction rate drops to such a low level that heating rates higher than this maximum limit are required to maintain the selected percent/minute. In negative mode, as long as the maximum

heating rate is high enough to allow completion of the transition at the selected percent/minute set point, but not so high as to cause percent/minute overshoot, transitions will appear largely the same from one heating rate to the next.

A word of caution, however: A*lways keep in mind that the background (baseline) rate of weight change will be accelerated at higher maximum heating rates.* This effect moves the background percent/minute closer to the control point you have selected with the resolution setting. If noise is present in the background percent/minute, it may "fool" the control algorithm into registering the start of a transition and prematurely reducing the heating rate to avoid predicted set point overshoot. In extreme cases, heating rate cycling can occur, particularly at high sensitivity settings. (See Figure B.13 on page B-61) To solve this problem, reduce the maximum heating rate and/or the sensitivity setting.

Adjusting the Resolution Setting

The purpose of the resolution parameter is to select the range of percent/minute values over which the heater control system will vary heating rate in response to changes in the rate of weight change. In dynamic rate mode (positive resolution settings), the range of percent/minute values selected by each resolution setting is fairly wide (about two orders of magnitude). You can adjust this width using the sensitivity parameter.

In constant reaction rate mode (negative resolution settings), the resolution setting specifies the percent/minute value that will be used as the control set point for furnace heating. In this case, the system will adjust heating rate as required to maintain the selected constant rate of weight change. Table B.1 (on the next page) shows the negative resolution settings and their associated percent/minute values.

The process of initially picking and then adjusting resolution setting is not exacting or calculated. It is based largely on experience and some general guidelines, because no single resolution setting will give dramatically different results from all the others. The change from one number to another is rather gradual.

Another reason you might wish to experiment with more than one setting is that some materials react differently from others to increasing or decreasing the resolution setting. This is due to the time-dependent as well as the temperature-dependent nature of transitions, and to the

Table B.1
Percent/Minute Values for
Negative Resolution Settings

Res	%/min	Res	%/min	Res	%min	Res	%/min
-0.1	28.2	-2.1	2.82	-4.1	0.282	-6.1	0.0282
-0.2	25.1	-2.2	2.51	-4.2	0.251	-6.2	0.0251
-0.3	22.4	-2.3	2.24	-4.3	0.224	-6.3	0.0224
-0.4	20.0	-2.4	2.00	-4.4	0.200	-6.4	0.0200
-0.5	17.8	-2.5	1.78	-4.5	0.178	-6.5	0.0178
-0.6	15.8	-2.6	1.58	-4.6	0.158	-6.6	0.0158
-0.7	14.1	-2.7	1.41	-4.7	0.141	-6.7	0.0141
-0.8	12.6	-2.8	1.26	-4.8	0.126	-6.8	0.0126
-0.9	11.2	-2.9	1.12	-4.9	0.112	-6.9	0.0112
-1.0	10.0	-3.0	1.00	-5.0	0.100	-7.0	0.0100
-1.1	8.91	-3.1	0.891	-5.1	0.089	-7.1	0.0089
-1.2	7.94	-3.2	0.794	-5.2	0.079	-7.2	0.0079
-1.3	7.08	-3.3	0.708	-5.3	0.071	-7.3	0.0071
-1.4	6.31	-3.4	0.631	-5.4	0.063	-7.4	0.0063
-1.5	5.62	-3.5	0.562	-5.5	0.056	-7.5	0.0056
-1.6	5.01	-3.6	0.501	-5.6	0.050	-7.6	0.0050
-1.7	4.47	-3.7	0.447	-5.7	0.045	-7.7	0.0045
-1.8	3.98	-3.8	0.398	-5.8	0.040	-7.8	0.0040
-1.9	3.55	-3.9	0.355	-5.9	0.036	-7.9	0.0036
-2.0	3.16	-4.0	0.316	-6.0	0.032	-8.0	0.0032

interaction between the rate of weight change (percent/minute) and heating rate (°C/minute). As percent/minute increases, heating rate is reduced by the control algorithm, but the reduction in heating rate usually causes an accompanying reduction in percent/minute, and vice versa. Therefore, attempting to directly compute optimal resolution settings from percent/minute information gathered from previous runs becomes a very questionable and usually frustrating experience.

Let us then consider what guidelines you can use to help make the selection process easier. As stated earlier, in the section on Hi-Res™ ramps, if you do not know what resolution setting to start with, you should try +3.0 resolution setting and 50 °C/minute heating rate. This gives a rapid scan with moderate application of the Hi-Res™ heating technique. Results should be at least as good as those of a 20 °C/minute conventional scan of the same material, and are usually better. If time permits, it is often helpful to have a constant heating rate 20 °C/minute scan of the material available for comparison.

Useful Resolution Settings

After some experience with Hi-Res™ TGA, you will find that the most useful resolution settings fall within the range +3.0 to +5.0 for the positive numbers and –3.0 to–5.0 for the negative numbers, and that adjustment by ±0.5 is usually adequate. This situation is similar to that for the heating rate: You can adjust the heating rate to any value from 0.01 to 200.0 °C/minute in steps of 0.01 °C, but most people use 1, 5, 10, 20 and 50 °C/minute exclusively, because a finer adjustment does not produce significantly different results.

With these guidelines alone, we have reduced the number of resolution settings that must be dealt with from 80 to only 5 for each Hi-Res™ mode while covering the majority of materials of interest.

The next step is how to proceed after the first Hi-Res™ run is complete. If the first run was made at resolution setting 3.0, try the second one at 4.0. Generally, increasing resolution setting

by a whole number increases the time to complete the TGA scan 2 to 5 times. Therefore, you must consider whether you can afford the added run time as well as the extra setup time. This balance between increased run time and adequate resolution is usually the determining factor in what resolution setting to use.

The benefit of having more range and "resolution" to the resolution setting than seems to be necessary is the rare occasion on which a material requires a very fine adjustment or an extreme treatment. Remember that the maximum heating rate also influences the resolution.

Lower resolution settings allow materials that already have well-separated transitions to be analyzed at super-high heating rates such as 200 °C/minute with excellent resolution in a fraction of the time required at a constant rate of 20 °C/minute. Resolution settings greater than 5.0 are useful in the following situations:

- When you need exact decomposition temperatures
- When the decompositions are explosive in nature
- When overlapped transitions are extremely close but highly temperature selective.

The effect of adjusting the resolution setting in dynamic rate mode while holding other experimental factors constant can be seen in Figure B.13 (page B-61).

Temperature Calibration

TGA temperature calibration is useful if you require accurate transition temperatures. The major cause of temperature inaccuracy in a TGA is thermal gradients between the sample thermocouple and the sample being studied. The magnitude of these gradients is proportional to heating rate. The Hi-Res™ TGA techniques inherently reduce thermal gradients by slowing down the heating rate during transitions.

An active way to reduce the effect of thermal gradients is to temperature-calibrate the TGA. The general procedure for temperature calibration is found in the online help and documentation. Temperature calibration involves analyzing a magnetic standard to determine its curie temperature. The curie temperature corresponds to the extrapolated endpoint on the S-shaped thermal curve.

However, when the calibration is intended for use with Hi-Res™ TGA experiments (*i.e.*, dynamic rate, constant reaction rate, or stepwise isothermal), a slow heating rate conventional ramp of 5 °C/minute or less should be used for calibration. You should use a faster ramp rate only when calibrating for constant heating rate experiments, because the Hi-Res™ heating control system reduces the heating rate during transitions.

Hi-Res™ Transition Temperatures

The TGA provides precise weight measurements coupled with relative temperature information. The resolution setting of a Hi-Res™ ramp controls the reaction rate of sample transitions. It is reaction rate, more than anything else, that determines the apparent transition temperature of a decomposition reaction.

The shift in measured transition temperature caused by changing the resolution setting can easily be an order of magnitude larger than the thermal gradients you are trying to correct with calibration. This effect can be clearly observed in the mixture of bicarbonates example (Figure B.13, page B-61). In light of this fact, it is acceptable in many cases to simply not use temperature calibration when employing the Hi-Res™ TGA techniques for decomposition analysis.

Hi-Res™ Sensitivity Segment

The Hi-Res™ sensitivity segment sets an additional parameter associated with Hi-Res™ ramp segments that you can use to adjust the response of the Hi-Res™ temperature control algorithm. Adjustment is sometimes necessary because of the wide variation in decomposition mechanisms of typical sample materials. This segment has the following format:

Hi-Res™ sensitivity <sens_setting>

where:

<sens_setting> is the Hi-Res™ sensitivity setting (1.0 to 8.0).

Example:

Hi-Res™ sensitivity 2.0.

Hi-Res™ sensitivity segments execute immediately when encountered in a method and simply set the sensitivity setting to the new value provided. The last value set is used for all subsequent Hi-Res™ ramps until a new value is set. If no Hi-Res™ sensitivity segment has been encountered in the method before the execution of a Hi-Res™ ramp segment, the default sensitivity (1.0) is used.

The sensitivity setting is a unitless number, ranging from 1.0 (lowest sensitivity) to 8.0 (highest sensitivity). The setting is used by both the dynamic rate (positive resolution setting)

and the constant reaction rate (negative resolution setting) modes of the Hi-Res™ ramp segment. There is no limit to how many times the setting can be changed during a method. Increasing the sensitivity setting tends to increase experiment time.

Understanding Sensitivity Setting

The TGA 2950 CE Hi-Res™ control algorithms have been designed to respond correctly to most transition situations with the default sensitivity setting of 1.0. In most cases, you do not have to adjust sensitivity setting at all. The key is knowing when and how to make the adjustment.

It is easy to confuse resolution setting and sensitivity setting, because both values can affect the resolution of the TGA scan. However, there is a simple way to think of the difference between the two parameters:

- *Resolution setting* controls the temperature at which the transition will occur (*i.e.*, how far from the theoretical isothermal decomposition temperature) by selecting the reaction rate (%/minute) at which heating rate is reduced. The closer the reaction is to the isothermal decomposition temperature, the lower the reaction rate and the longer the reaction time. You will find that you can use the resolution setting to literally move the measurement of transitions on the temperature axis.(See Figure B.13, page B-61.)

- *Sensitivity setting* controls the response of the Hi-Res™ system to changes in the rate

of reaction (percent/minute). Higher sensitivity settings cause the system to be more reactive or more "sensitive" to small changes in the rate of the reaction. Lower sensitivity settings dampen this response.

Generally, it is best to adjust resolution setting first with sensitivity set to a low value. Then, after you obtain a good result, try increasing the sensitivity to see whether doing so makes any improvement in the resolution.

Use caution when adjusting sensitivity. Over-adjustment can cause oscillation or anomalies in the weight versus temperature curve.

Adjusting Sensitivity in Dynamic Rate Mode

In dynamic rate Hi-Res™ mode (positive resolution settings), the sensitivity setting is used to further increase the resolution of some transitions once an appropriate resolution setting has been determined. This is accomplished by narrowing the range of percent/minute values over which the heating rate is proportionally varied. Higher sensitivity settings result in progressively narrower percent/minute ranges and, usually, in greater resolution.

In this mode, the resolution setting selects the general range of percent/minute values that will drive changes in furnace heating rate. For example, resolution setting 3.0 selects the range of approximately 1.0 to 20.0 %/minute for most of the variation in heating rate. Resolution setting 4.5, however, selects the range of 0.1 to 2.0 %/minute.

Sensitivity setting controls the relative width of the range: Setting 1.0 allows use of the full range. Setting 2.0 reduces the range to about half the full range, setting 3.0 to about a third, and so on up to 8.0 (an eighth). In general, higher sensitivity settings bring the furnace to the transition temperature more quickly but then tend to keep it at that temperature longer. In other words, higher sensitivity settings bring the control closer to stepwise isothermal heating. (See "Stepwise Isothermal Heating" in the section on using Abort Segments for more information.)

The recommended procedure for adjusting the sensitivity setting for dynamic rate mode is as follows: Start with a sensitivity setting of 1.0, and adjust resolution setting to obtain the best separation possible in the desired time frame. Then increase sensitivity to 2.0, 4.0, and 8.0 to see whether a useful improvement in resolution results.

It is possible that no improvement in resolution will result. This situation is usually caused by overlapped transitions that are weakly temperature-dependent and strongly time-dependent. In this case, no matter how precisely you control at a specific temperature, the individual components of the sample material will all decompose more or less together in the range of the decomposition temperatures you have selected using the resolution setting. All you can do in such a situation is lengthen or shorten the total time of decomposition by selecting larger or smaller resolution settings.

Figure B.14 (page B-62) shows the effect of changing sensitivity settings in dynamic rate mode.

Adjusting Sensitivity in Constant Reaction Rate Mode

In constant reaction rate Hi-Res™ mode (negative resolution settings), the sensitivity setting is used to adjust the heater control system to minimize transition temperature overshoot and heating control fluctuation. Higher sensitivity settings result in decreased percent/minute overshoot and tighter control at the beginning of transitions. Lower settings have the opposite effect.

In this mode, sensitivity setting is used to adjust the response of the heater control system to changes in the rate of weight change (percent/minute). For materials that react gradually, low sensitivity settings are generally preferred, because they help dampen noise and greatly reduce the possibility of control cycling. However, when it is very important to avoid percent/minute overshoot, or if the sample is highly reactive, you need to use higher sensitivity settings. The problem with too high a sensitivity setting is that control cycling or heating rate "ringing" may occur (see Figure B.11 on page B-50).

The recommended procedure to adjust sensitivity setting for constant reaction rate mode is as follows: Start with a sensitivity setting of 1.0, and observe the transition for percent/minute overshoot and control cycling. If overshoot is acceptable, no further adjustment is needed. If overshoot is excessive, increase the sensitivity setting by 1.0, and recheck for overshoot and cycling. Continue increasing the sensitivity setting until the results are acceptable or control cycling becomes excessive.

If you cannot find a satisfactory sensitivity setting, either the resolution setting is too low or the heating rate is too high. Try a higher resolution setting (larger negative number) to reduce the percent/minute set point, and rerun the experiment. Set points in the range of 0.1 to 1.0 %/minute (resolution settings –5.0 to –3.0) generally give the best results.

If the percent/minute overshoot is primarily associated with the first transition in a group of overlapped transitions, the maximum heating rate may be too high. Try reducing the heating rate of the Hi-Res™ ramp segment by half, and rerun the experiment. Heating rates in the range of 1.0 to 5.0 °C/minute generally give the best results.

When you use heating rates higher than 5.0 °C/minute, proper adjustment of sensitivity setting becomes critical to maintaining smooth heating control. The default sensitivity of 1.0 is usually too low at these higher heating rates and typically results in significant transition temperature overshoot and heating control "ringing," as shown in Figure B.9. At high heating rates, a sensitivity setting of 3.0 or 4.0 gives better results for most materials. The improvement can be seen in Figure B.10. If sensitivity setting is adjusted too high, however, continuous control cycling may result, as shown in Figure B.11.

With some experimentation, you can usually find an optimal sensitivity setting. When you use very high heating rates (greater than 10 °C/minute), you may be able to completely eliminate control ringing. However, this should not affect the quantitative measurement of weight loss for the transition.

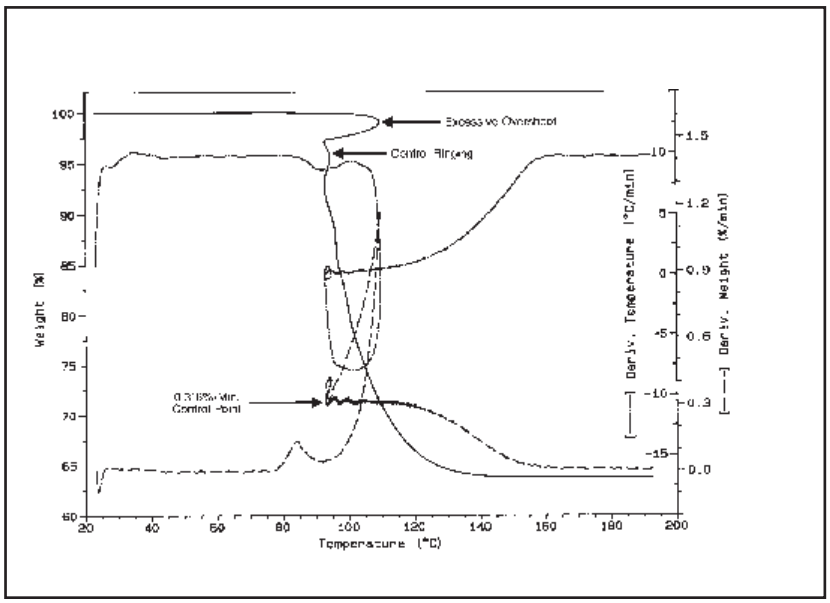


Figure B.9
Sensitivity Setting Too Low

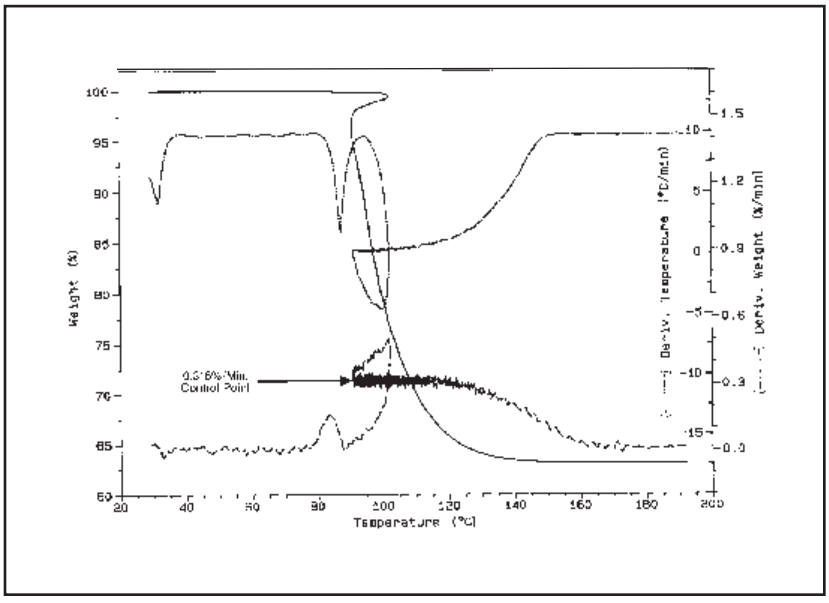


Figure B.10
Correct Sensitivity Adjustment

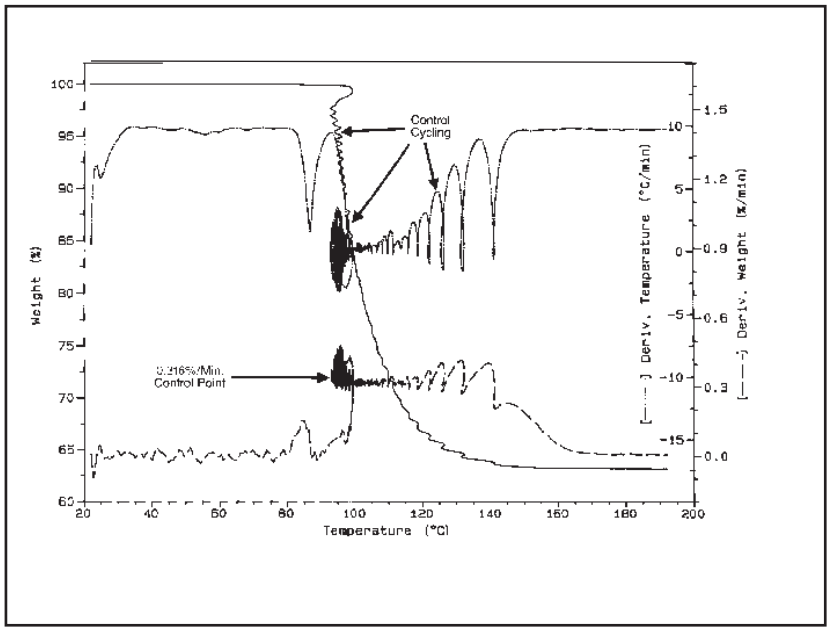


Figure B.11
Sensitivity Setting Too High

Abort Segment

The abort segment provides a mechanism to skip over or terminate other method segments once specific weight change conditions are met. This segment has the following format:

Abort next seg if <signal> <condition> <value>

where:

<signal> is the real-time measurement used to decide whether to abort or not (weight % or weight %/minute)

<condition> is the limit condition for aborting ("<" or ">")

<value> is the abort limit that is compared with the real-time signal.

Example:

Abort next seg if % < 20.0

Abort segments execute immediately when encountered in a method and simply establish conditions for testing the next segment. The specified limit, designated by the <signal>, <condition>, and <value> parameters, is tested before and during the execution of the next method segment. If the limit is reached at the beginning of a segment, that segment is skipped, and method execution continues with the next segmentin the method. If the segment following the abort segment is equilibrate, initial temperature, ramp, Hi-Res™ ramp, isothermal or step,

and the limit has not been reached yet, the limit will be tested at the rate of 2 times per second until the segment terminates normally or the limit is reached. If the limit is reached during the execution of a segment, the remaining portion of the segment is skipped. Method execution then continues with the next segment in the method.

The conditions for determining whether limits have been reached for each type of limit are as follows:

%: If the condition operator is "<" and the sample weight percent is less than or equal to the limit percent, or if the condition operator is ">" and the weight percent is greater than or equal to the limit percent, then the limit is reached. Values greater than 100% are permitted to accommodate weight gains.

%/min: If the condition operator is "<" and the derivative of sample weight percent is less than or equal to the limit percent/minute, or if the condition operator is ">" and the derivative of weight percent is greater than or equal to the limit percent/minute, then the limit is reached. Negative values indicate weight gain, and positive values indicate weight loss.

Abort segments are very versatile, because they can be used in front of any method segment (including other abort segments) to dynamically change the execution of a method.

You can use an abort segment in the following ways:

- To control the switching of a purge gas or activation of an event relay according to the rate of reaction or the amount of weight loss
- To terminate a loop prematurely by putting the abort segment in front of the repeat segment
- To customize a method for a particular material by allowing different ramp and/or isothermal segments to be used for each transition region without regard to specific temperature limits
- To control data storage so that file size is minimized by turning off storage or increasing sampling interval during baseline sections of a scan.

Abort segments also provide a convenient mechanism for shortening experiments after the data of interest has been collected. For example, if you are using a heating ramp to analyze a material that has two weight losses and the only data of interest is the percent weight loss during the first transition, you can put an abort segment in the method to terminate the ramp after the beginning of the second weight loss by specifying a "%" limit condition. This is particularly useful for Hi-Res™ ramps, because the reduced heating rate during a weight loss causes the majority of the transition to occur within a very narrow temperature range, making termination by final temperature difficult to predict.

Stepwise Isothermal Heating

Another useful TGA technique that can be implemented with abort segments is transition-controlled stepwise isothermal heating. This process consists of heating a sample via a ramp segment until a certain rate of weight change is detected, and then switching to an isothermal segment to hold constant temperature until the transition has completed. Then the sample heating is continued until the next transition is detected, whereupon isothermal holding is again initiated, and so on, until the final temperature is reached.

You can easily implement the stepwise heating technique by placing a ramp segment followed by an isothermal segment into a "repeat to final temperature" loop, and preceding the ramp and isothermal segments with abort segments. Set up the abort segment preceding the ramp to terminate the ramp when the percent/minute is greater than a specified limit. Set up the abort segment preceding the isothermal segment to terminate the holding period when the percent/minute is less than a second limit. An example of this type of method is shown below:

1: Abort next seg if %/min > 0.5
2: Ramp 10 °C/min to 700 °C
3: Abort next seg if %/min < 0.05
4: Isothermal for 500 minutes
5: Repeat segment 1 til 700 °C

Guidelines for stepwise thermal heating with abort segments are as follows:

- Select a "%/min" limit for the isothermal

segment that is equal to the baseline rate of weight change encountered during the onset of the transition of interest in a normal constant heating rate scan of this material. (Be sure to use the same ramp rate as that selected for stepwise heating.)

- Make the percent/minute limit for the ramp segment about an order of magnitude greater than that selected for the isothermal segment (but not more than the maximum rate of weight change encountered during the transition of interest).

- Set the ramp final temperature and the repeat final temperature to the final experiment temperature.

- For the isothermal time, select an arbitrary time that is long enough that the segment will not terminate until the percent/minute limit has been reached.

Stepwise heating often improves transition resolution, because transitions are time-dependent as well as temperature-dependent. Stepwise heating gives transitions more time to complete, thereby reducing overlap with neighboring transitions.

To get the maximum benefit from stepwise heating, you will have to run several TGA scans to properly "tune" the reaction rate thresholds used to start and stop heating. Relatively slow heating rates are generally required to prevent transition overshoot. A rough guideline is to use a heating rate about one tenth of the difference between the transition temperatures of the transitions being resolved.

For example, if the transitions are separated by 10 °C, use 1 °C/min as the heating rate prior to, between, and following the transitions. If precise reaction temperatures are important, you should always specify slow heating rates prior to encountering transitions of interest, even though they may be well separated in temperature, and set the percent/minute limit for the ramp segment closer to the limit for the isothermal segment. To avoid excessively long experiments, you can use higher heating rate ramps or equilibrate segments to skip over baseline portions of the scan.

Stepwise heating has several disadvantages:

- Most experiments take much longer in total time to complete than a conventional constant heating rate scan.

- The decision to leave isothermal mode and continue heating is somewhat arbitrary and may lead to incorrect assumptions about the number and size of transitions. This is particularly true for materials with transitions that are overlapped even at very slow heating rates (such as the sodium/potassium bicarbonate mixture in the "Examples" section). As a general rule, stepwise isothermal heating cannot be used to reliably separate transitions that cannot be separated by conventional TGA at very slow heating rates.

- There may be anomalies in the weight loss versus temperature curve. These appear as small, unexpected secondary weight losses following a larger transition. The anomalies can be caused by the following two factors:

(1) Using too high a heating rate in the ramp that follows the transition. Any small amount of sample material that did not finish decomposing during the transition will now quickly decompose because of the rapid elevation of the furnace temperature, causing the decomposition rate (percent/minute) to rise substantially. If the rising decomposition rate crosses the abort threshold for the ramp segment, a second isothermal period is introduced, which will appear on the weight loss versus temperature plot as a small unexplained transition.

Generally, it is best to use the same heating rate on both sides of a transition. You can change to a higher heating rate later in the method, after the furnace temperature has risen away from the transition enough to avoid this problem.

(2) Leaving the isothermal holding period prematurely during a transition because the percent/minute threshold for aborting the isothermal segment was set too high. This situation leaves a significant amount of undecomposed sample material, which now accelerates its rate of decomposition. The rising temperature causes the remaining sample to quickly decompose, raising the rate of weight loss (percent/minute) to a high level. This event either triggers another isothermal hold period or appears as a backside “shoulder” on the weight loss curve.

An example using stepwise isothermal heating can be found in the next section.

Hi-Res™ TGA Examples

This section contains example TGA scans of common materials. These examples can be used to compare the results of Hi-Res™ TGA heating control and conventional constant heating rate TGA. Where possible, the effects of using the different Hi-Res™ modes and parameters are shown.

Mixture of Bicarbonates

A mixture of potassium bicarbonate (potassium hydrogencarbonate) and sodium bicarbonate (sodium hydrogencarbonate) was chosen to demonstrate the effects of different Hi-Res™ techniques and parameter settings. The individual bicarbonates decompose to carbonates between 100 and 200 °C with the simultaneous release of CO_2 and H_2O. The decomposition temperature for potassium bicarbonate is approximately 50 °C higher than that for sodium bicarbonate. When the two bicarbonates are mixed together, however, their decompositions are overlapped in temperature and very difficult to resolve.

You can easily make this sample yourself by thoroughly mixing equal parts (by weight) of finely powdered potassium bicarbonate ($KHCO_3$) and sodium bicarbonate ($NaHCO_3$). Inadequate mixing or large granule size will reduce weight loss reproducibility. Note that potassium bicarbonate is very hygroscopic.

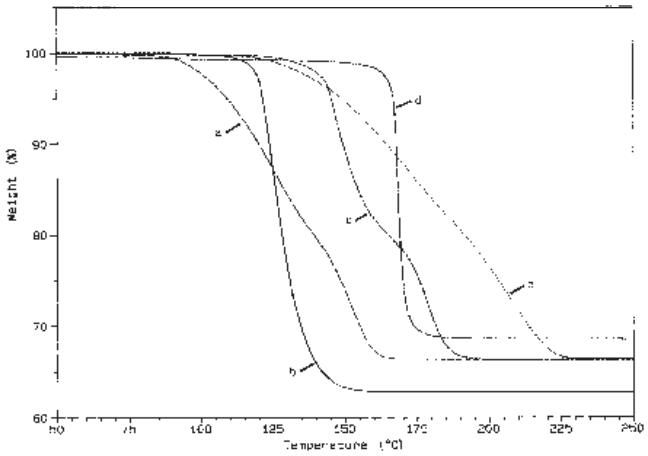
The mixture must not be exposed to ambient humidity for long, or a significant surface water transition will become evident between 70 and 100 °C, which will affect the overall weight loss percentages. When mixing and using this sample, be sure to keep the sample supply containers tightly capped.

Load the TGA quickly, and use a dry purge gas (air, nitrogen, or argon). Dry air purge at 100 mL/min was used for all example scans shown. Sample sizes varied from 20 to 40 mg. Small variations observable in weight loss reproducibility in the example scans are largely due to the nonhomogeneity of the sample mixture and variations in ambient humidity from run to run.

Dynamic Rate Scans

In Figure B.12 (on the next page), we have overlaid the individual bicarbonate decompositions (curves *b* and *d*) along with the decomposition of the mixture by conventional TGA (curves *a* and *e*) and by dynamic rate Hi-res™ TGA (curve *c*). Reducing the heating rate of the mixture from 20 °C/minute (curve *e*) to 1 °C/minute (curve *a*) gave a very slight improvement in resolution.

Comparing these results to the 50 °C/minute Hi-res™ scan, we observe a significant improvement in resolution with Hi-res™ TGA in about twice the time of the 20 °C/minute scan, and about one tenth the time of the 1 °C/minute scan. The method used for all scans was a single ramp segment.



(a) 1°C/min Normal Ramp of Mixture; (b) 20°C/min Res 4.0 Hi-Res™ Ramp of $NaHCO_3$; (c) 50°C/min Res 4.0 Hi-Res™ Ramp of Mixture; (d) 20°C/min Res 4.0 Hi-Res™ Ramp of $KHCO_3$; (e) 20°C/min Normal Ramp of Mixture.

Figure B.12
Decomposition Curves for Potassium and Sodium Bicarbonates and Mixture

Varying the Resolution Setting

In Figure B.13, we have overlaid the conventional constant heating rate decompositions of the bicarbonate mixture (curves *a* and *b*) with dynamic rate Hi-Res™ scans at eight different resolution settings (curves *1* through *8*). All of the Hi-Res™ scans were run at 50 °C/minute with the default sensitivity setting of 1.0.

Note that increasing the resolution setting improves the resolution of each transition and simultaneously reduces the transition temperature. The initial weight losses of approximately 1% on each curve are due to the evaporation of surface water absorbed by the mixture.

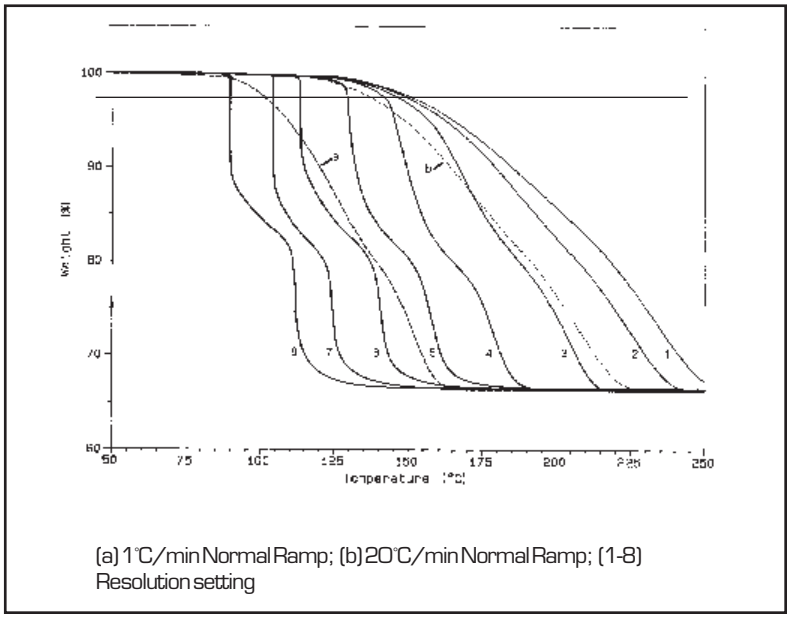
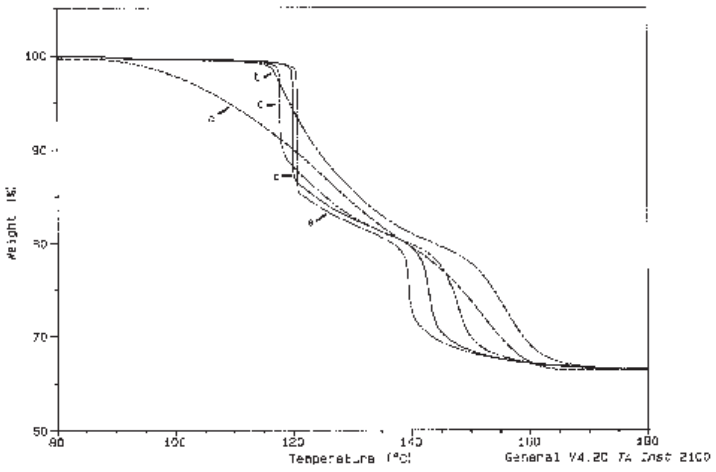


Figure B.13
Decompositions Curves at Different Resolution Settings

Varying the Sensitivity Setting

In Figure B.14, we have overlaid the 1 °C/minute conventional constant heating rate decomposition of the mixture (curve *a*) with a dynamic rate Hi-Res™ scan at four different sensitivity settings (curves *b* through *e*). All of the Hi-Res™ scans were run at 20 °C/minute with a resolution setting of 5.0. Note that raising the sensitivity setting increases the sharpness of each transition but does not substantially change the transition temperature.



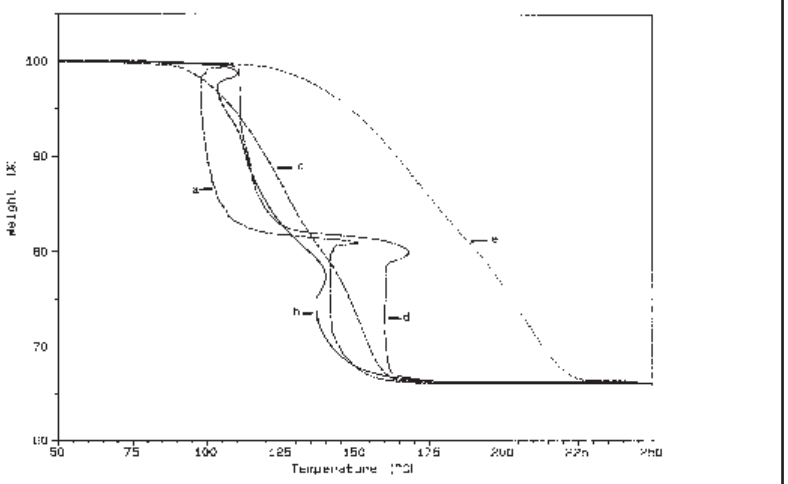
(a) 1°C/min Normal Ramp; (b) Sensitivity setting 1; (c) Sensitivity setting 2; (d) Sensitivity setting 4; (e) Sensitivity setting 8.

Figure B.14
Decomposition Curves at Different Sensitivitiy Settings

Constant Reaction Rate Scans

In Figure B.15 (on the next page), we have overlaid the decompositions of the bicarbonate mixture from conventional constant heating rate scans (curves *c* and *e*) with those from Hi-Res™ constant reaction rate scans at different resolution and sensitivity settings (cuirves *a*, *b*, and *d*). Curve *b* shows the Hi-Res™ scan run in an open sample pan at resolution setting –4.0 and sensitivity setting 1.0. For curve *d* (resolution –4.0) and curve *a* (resolution –5.0), the sample was contained in a hermetic aluminum DSC sample pan with a 0.1-mm pin hole in the top. All of the Hi-Res™ scans were run at 5 °C/min.

Comparing curve *b* with curves *a* and *d* reveals a significant improvement in resolution because of the vapor pressure buildup in the semihermetic sample container. Because the sample pan was open in curve *b*, there was no opportunity for a vapor pressure/reaction rate equilibrium to occur as the sample decomposed, resulting in only partial separation of the transitions. The pressure buildup in the closed container (curves *a* and *d*) retarded the potassium bicarbonate decomposition until the sodium bicarbonate decomposition had completed. As with dynamic rate mode (Figure B.13), a higher resolution setting (larger negative number) reduces the transition temperature.



(a) Res-5.0, Sens 2.0, Semi-hermetic Pan; (b) Res-4.0, Sens 1.0, Open Pan; (c) 1°C/min Normal Ramp; (d) Res-4.0, Sens 2.0, Semi-hermetic Pan; (e) 20°C/min Normal Ramp.

Figure B.15
Conventional vs. Hi-Res™
Constant Reaction Rate Scans

Stepwise Isothermal Scans

Figures B.16 and B.17 show the results of stepwise isothermal scans of the bicarbonate mixture using a conventional 1°C/minute ramp and the abort segment.

The following method was used for the scan shown in Figure B.16:

1: Abort next seg if %/min > 0.15
2: Ramp 1 °C/min to 300 °C
3: Abort next seg if %/min < 0.015
4: Isothermal for 500 minutes
5: Repeat segment 1 til 300 °C.

Although this method gives apparently excellent separation results, the quantitative value of the weight loss plateau between the two bicarbonate decompositions is questionable, because there is no inflection point in the plateau, and the rate of weight loss increases immediately after the isothermal segment is aborted. This indicates that the two decompositions are still overlapped and that holding for a longer isothermal period during the first transition would have resulted in a lower weight loss plateau between transitions.

In Figure B.17, the same stepwise isothermal method is repeated with the percent/minute limits for the abort segments set to smaller values (0.05 %/minute for the ramp abort and 0.005 %/minute for the isothermal abort). The following method was used for the scan shown in Figure B.17:

1: Abort next seg if %/min > 0.05
2: Ramp 1 °C/min to 300 °C
3: Abort next seg if %/min < 0.005
4: Isothermal for 500 minutes
5: Repeat segment 1 til 300 °C.

The scan results in Figure B.17 are a definite improvement over those in Figure B.16. The improvement occurred because the abort limit for the isothermal segment (0.005 %/minute) was chosen to be equal to the baseline percent/minute immediately preceding the sodium bicarbonate transition observed in a conventional constant heating rate scan of the mixture at 1°C/minute. The theory supporting this decision is that if the two transitions are separable, then the rate of weight loss should return to baseline between the transitions. The percent/minute limit for the ramp segment was then chosen to be ten times larger than that for the isothermal segment.

Although the weight loss result in Figure B.17 seems more reasonable, we are suspicious that the decomposition of the potassium bicarbonate (second transition) has already started, because the rate of weight loss immediately increases as heating is resumed at 88 °C. Another problem is the unreasonable1300-minute time frame of the experiment.

As can be seen from these results, you must always use caution when interpreting results from stepwise isothermal heating experiments. It is usually wise to run confirming experiments using other TGA techniques, particularly if the sample material is of relatively unknown composition.

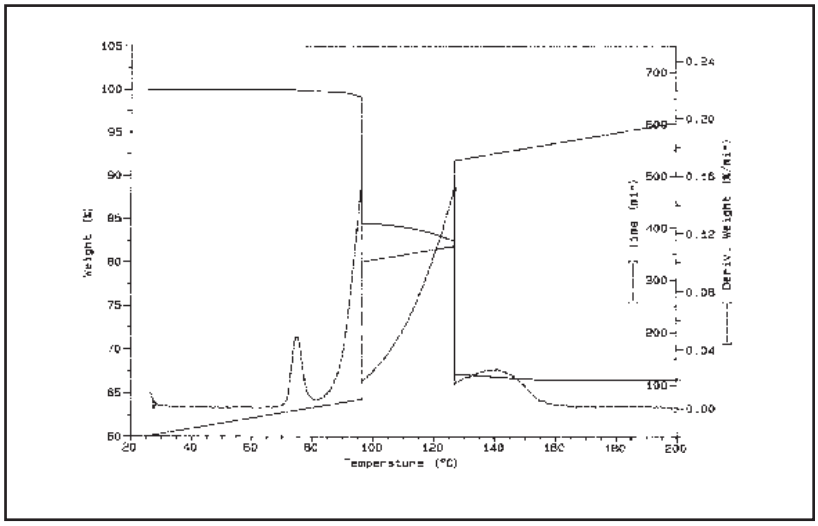


Figure B.16
Stepwise Isothermal Scan of Bicarbonate Mixture

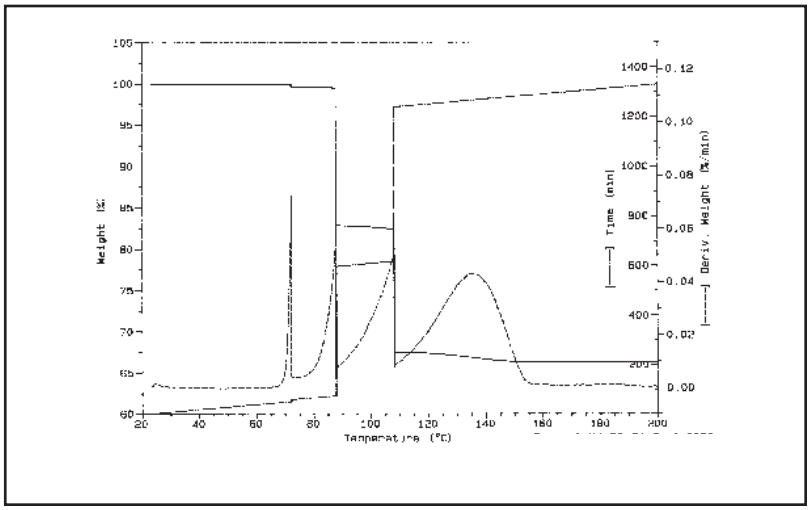
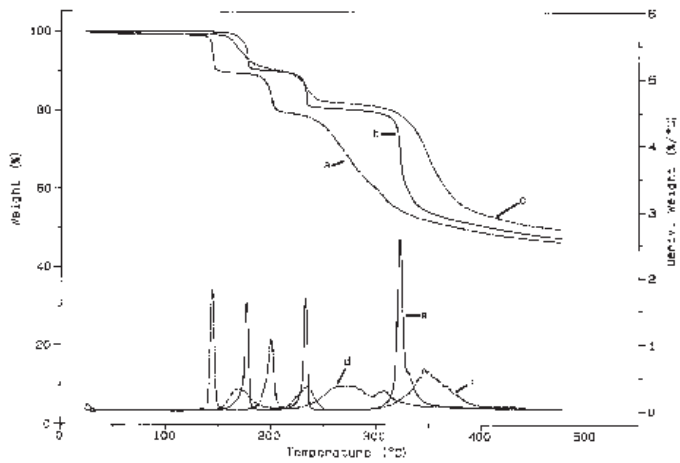


Figure B.17
Stepwise Isothermal Scan of Bicarbonate Mixture with Smaller Abort Segment %/Minute Limits

Monosodium Glutamate

To obtain the results shown in Figure B.18, we analyzed Accent® brand monosodium glutamate (MSG), a common salt used for seasoning foods, which has three well-resolved transitions below 500 °C. Curves *a* and *c* show the result of conventional constant heating rate scans of MSG at 1 and 20 °C/minute. Curve *b* shows the result of a dynamic rate Hi-Res™ scan at resolution setting 4.0 and sensitivity setting 1.0. As can be seen from the derivative of weight loss curves, the Hi-Res™ scan gives resolution comparable to the 1 °C/minute scan in a fraction of the time.



(a) 1°C/min Normal Ramp; (b) 50°c/min Hi-Res™ Ramp, Res 4.0, Sens 1.0; (c) 20°C/min Normal Ramp; (d) 1°C/min; (e) 50°C/min; (f) 20°C/min.

Figure B.18
MSG: Conventional Constant Heating Rate vs. Dynamic Heating Rate Hi-Res™ Scans

If you should decide to run this sample, be aware that MSG foams significantly at these temperatures. If you use a sample size larger

than about 10 mg, the sample material may rise high enough in the pan to touch the sample thermocouple. If MSG is heated to temperatures well above 500 °C, it leaves a residue that is difficult to remove from the sample pan.

Banana Taffy

Figures B.19 and B.20 show results of the analysis of a sample of artificial banana taffy (a common confectionery product composed primarily of water and sugar), which has a number of overlapped transitions between 100 and 500 °C. Figure B.19 shows the result of a conventional constant heating rate scan of taffy at 10 °C/minute. Figure B.20 shows the result of a 50 °C/minute dynamic rate Hi-Res™ scan at resolution setting 4.0 and sensitivity setting 1.0.

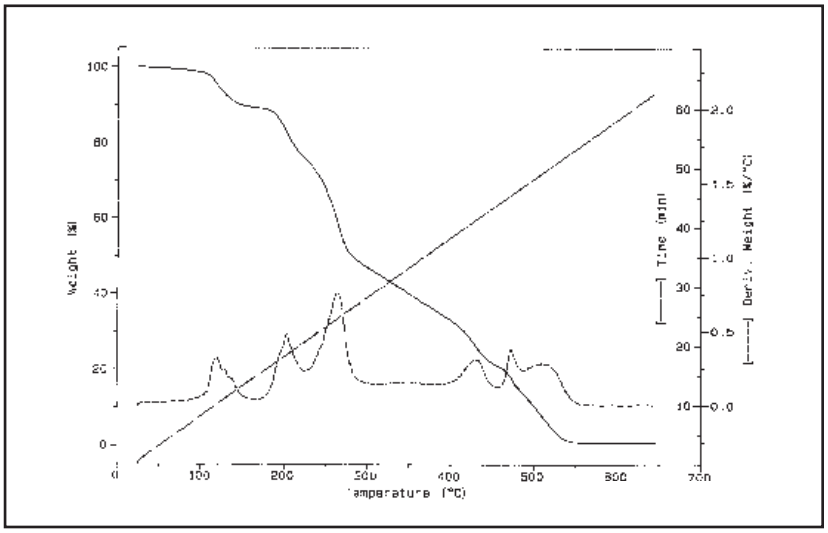


Figure B.19
Banana Taffy: Conventional Constant Heating Rate Scan

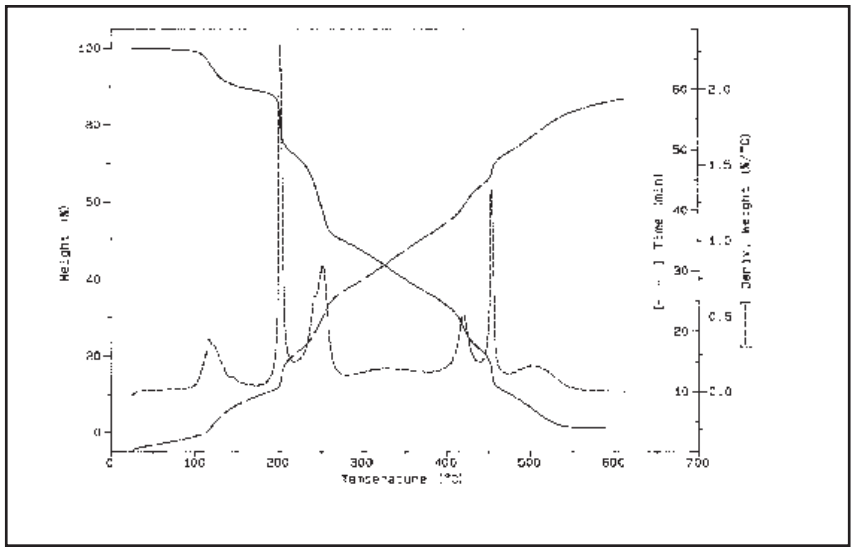


Figure B.20
Banana Taffy: Dynamic HeatingRate
Hi-Res™ Scan

As can be seen by comparing the derivative of weight loss curves in Figures B.19 and B.20, the Hi-Res™ scan gives better resolution in the same time as the conventional scan. This is possible because the Hi-Res™ TGA ramp heats the taffy sample rapidly during baseline portions of the scan and slowly during transitions, resulting in an average heating rate of about 10 °C/minute. A 200 °C/minute Hi-Res™ scan of this sample takes about the same time as a 20 °C/minute conventional scan and provides a resolution improvement similar to that observed with the 50 °C/minute Hi-Res™ scan.

Plastic Laboratory Tubing

To obtain the results shown in Figures B.21 through B.25, we analyzed a sample of Tygon® R-3603 plastic tubing, a polyvinyl chloride (PVC)–based clear flexible tubing commonly used in laboratories and industry. A number of overlapped transitions are evident between 100 and 350 °C, followed by two well-resolved transitions at approximately 400 and 500 °C. Of interest are the changes in resolution of the various transitions as maximum heating rate changes in both the conventional and Hi-Res™ scans.

Results of the two conventional scans show that the resolution of the initial overlapped transitions is good in the 1 °C/minute scan (Figure B.21) but poor in the 20 °C/minute scan (Figure B. 22). In contrast, the resolution of the two transitions at 400 and 500 °C is good in the 20 °C/minute scan and reduced in the 1 °C/minute scan. In both cases, however, the small backside transition at 25% to 35% weight loss is barely discernible. Comparing these conventional TGA results (Figures B.21 and B.22) with those in the 50 °C/minute Hi-Res™ scan (Figure B.23) shows that all of these transitions are better resolved in the Hi-Res™ TGA scan in a timely fashion.

Figures B.23, B.24, and B.25 demonstrate the result of changing the maximum heating rate of a dynamic rate Hi-Res™ scan. Maximum heating rates of 50 °C/minute (Figure B.23), 20 °C/minute (Figure B.24), and 10 °C/minute (Figure B.25) were used with a method consisting of a single Hi-Res™ ramp segment.

Sample size (approximately 10.45 mg), sensitivity setting (1.0), purge gas (100 mL/minute dry air), and sample pan (open platinum) were the same for all three runs.

The results are similar in the three scans, except that the measurement of transitions is moved to slightly lower temperatures as heating rate is reduced, and each run takes about 50% longer to complete when heating rate is reduced by 50%. Most noticeable, however, is that transition resolution is best in the fastest scan. This is because higher maximum heating rates allow the TGA furnace temperature to change more quickly between transitions, thereby reducing transition overlap and flattening weight loss baseline. As an added benefit, experiment time is shorter than with traditional resolution enhancement techniques. Here you can see the real beauty of dynamic rate Hi-Res™ TGA: better results in the same or less time compared with traditional constant heating rate TGA.

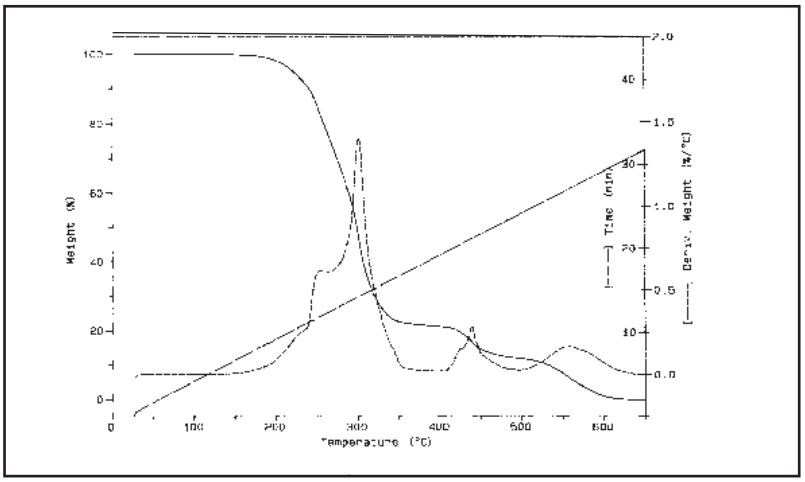


Figure B.21
Plastic Tubing: Conventional Scan at 1 °C/minute

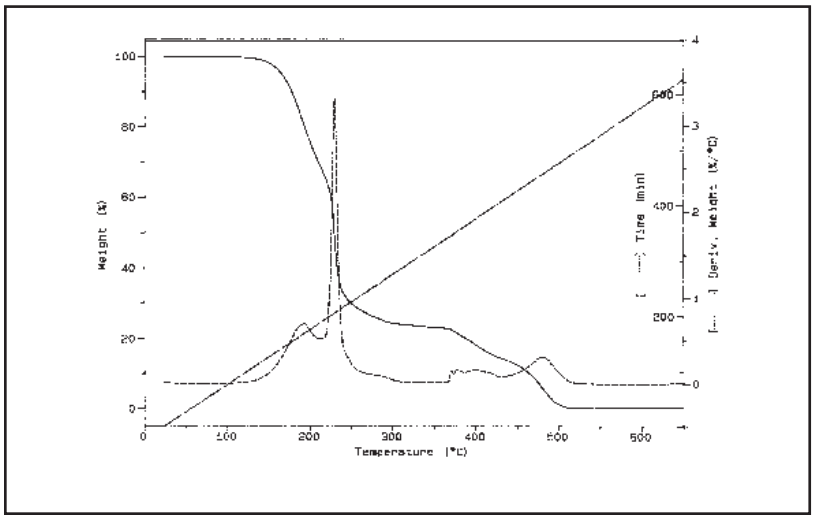


Figure B.22
Plastic Tubing: Conventional Scan at 20 °/minute

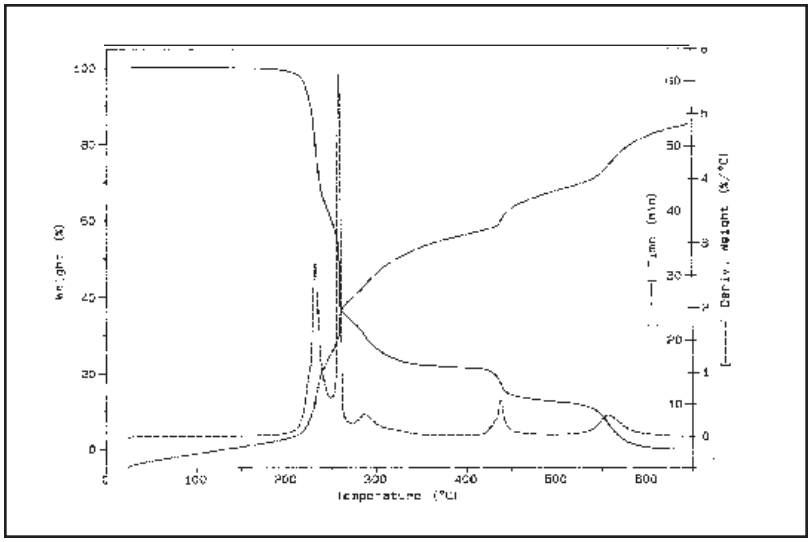


Figure B.23
Plastic Tubing: Hi-Res™ Scan at 50 °C/minute

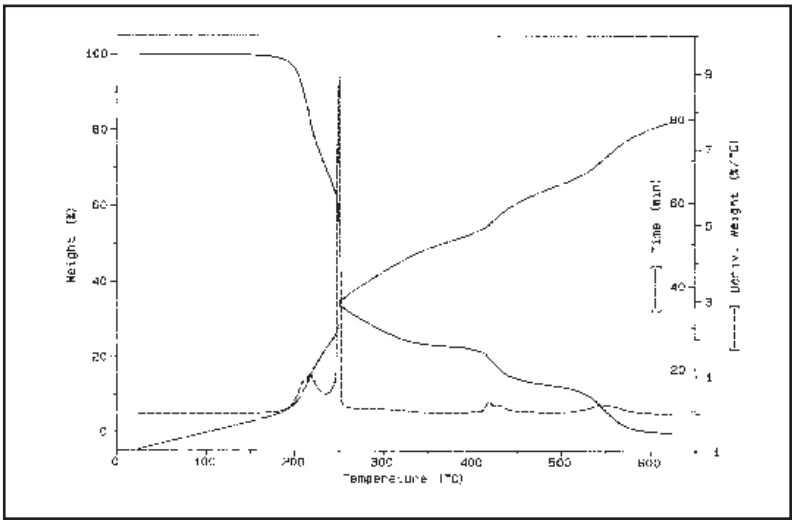


Figure B.24
Plastic Tubing: Hi-ResTM Scan at 20 °/minute

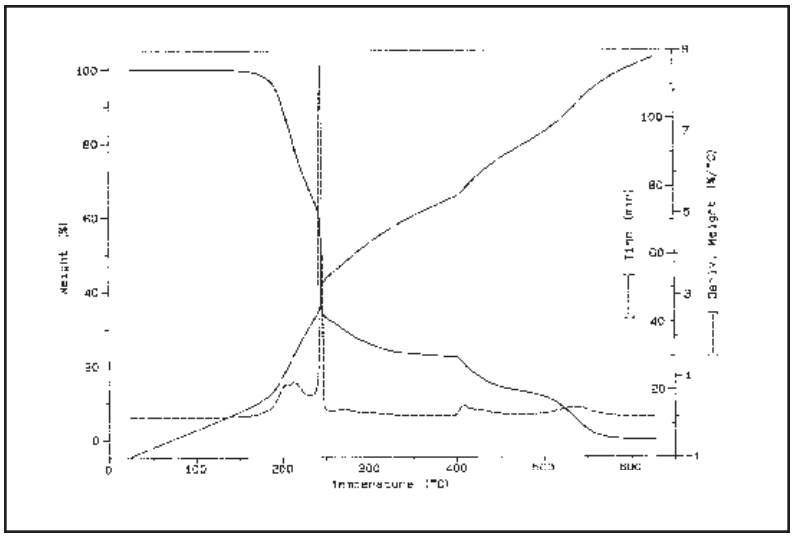


Figure B.25
Plastic Tubing: Hi-Res™ Scan at 10 °C/minute

References

Listed below are two excellent additional references for specific materials and techniques. We have chosen them because they give a broad review of the techniques involved and address the physical and chemical processes taking place, as opposed to a specific analysis of any one sample material. Each of these papers contains an extensive list of additional references for specific materials and techniques.

1. Thermoanalytical Examinations Under Quasi-Isothermal–Quasi-Isobaric Conditions, F. Paulik & J. Paulik, Thermochimia Acta, 100 (1986) 23–59.

2. Controlled Transformation Rate Thermal Analysis: The Hidden Face of Thermal Analysis, J. Rouquerol, Thermochimica Acta, 144 (1989) 209–224.

Appendix C: TGA 2950 CE Autosampler Option

Introducing the Auto TGA

The TGA Autosampler, known as the Auto TGA, is an accessory to the TA Instruments Thermogravimetric Analyzer (TGA) 2950 CE (see Figure C.1 on the next page). It allows you to place up to 16 samples at one time on the TGA instrument to measure the amount and rate of weight change in a material. Experiments are performed as they normally would be using the TGA—but now you can run samples on a continual basis and keep a log of the results using the Autosampler screens. The six (6) standard TGA pans listed below are used with the Auto TGA:

- 100 μL aluminum
- 50 and 100 μL platinum pans and
- 100, 250, and 500 μL alumina ceramic pans.

This appendix provides information on the setup of the Auto TGA. For instructions on the use of the Auto TGA through the Instrument Control software, please refer to the online help and documentation for further information.

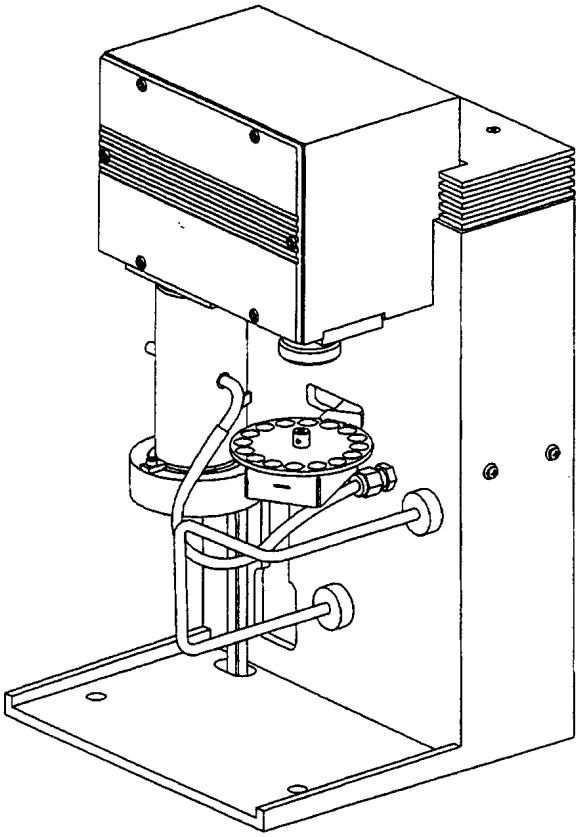


Figure C.1
TGA Autosampler

Getting Started

The Auto TGA, as an accessory to the TGA, does not alter the procedures used to start up and shut down the TGA instrument and the controller; refer to the procedures found in Chapter 3 of this manual when starting your instrument.

The TGA 2950 CE with Autosampler

When you receive your Auto TGA, you will notice that a key has been added to the TGA instrument keypad—the AUTO SELECT key, shown in the figure below.

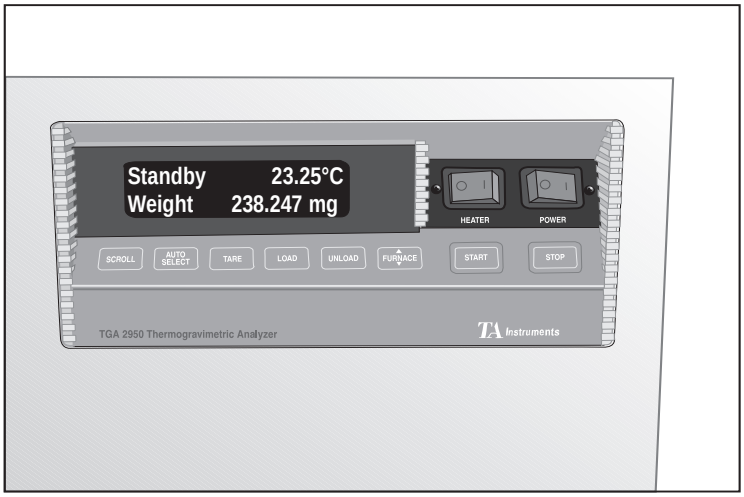


Figure C.2
TGA Instrument Display and Keypad with Auto TGA Accessory

Table C.1 on the next page explains the functions of the instrument keys when used in conjunction with the Auto TGA accessory.

Table C.1
Auto TGA
Instrument Keys

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display. For more information on the progress of the experiment, refer to theonline help and documentation. Auto TGA function: This key has an added function, to act as a shift key for the AUTO SELECT, TARE, and LOAD keys.
AUTO SELECT	Auto TGA function: Increments the next pan to be loaded when pressed alone.
SCROLL & AUTO SELECT	Auto TGA function: Decrements the next pan to be loaded when you hold down the SCROLL key while pressingAUTO SELECT. The selected sample is loaded or tared when the appropriate key is pressed.

(table continued)

Table C.1
Auto TGA
Instrument Keys
(continued)

Key/Function	Explanation
TARE	Auto TGA functions: Zeros the displayed 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. Performs a tare on the *selected* pan when pressed alone.
SCROLL/TARE	Auto TGA function: Tares *all* of the pans on the sample platform when you hold down the SCROLL key while pressing TARE.
LOAD	Loads the selected pan from the sample platform onto the balance. Auto TGA function: Loads the *selected* sample pan when pressed alone.

(table continued)

Table C.1
(continued)

Key/Function	Explanation
SCROLL/LOAD	Continuously loads and unloads *all* of the pans on the sample platform when you hold down the SCROLL key while pressing LOAD.
NOTE:	LOAD functions can be stopped by pushing the STOP key on the instrument keypad.
UNLOAD	Functions of these remaining keys do not change when used with the Auto TGA accessory; see Chapter 1.
Δ FURNACE ∇	
START	
STOP	
REJECT (SCROLL/STOP)	

Calibrating the Auto TGA

When using the Auto TGA accessory, the need for calibration remains the same—weight calibration and temperature calibration are recommended at least once a month or when replacing the thermocouple; and the platform adjustment procedure needs to be performed if the sample hang-down wire fails to pick up a sample pan. See the online help and documentation for all of the calibration procedures.

Running Experiments

Performing experiments with the Auto TGA is similar in many respects to the regular operation of the TGA instrument, the exceptions are explained in this section.

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

- Selecting the pan type and material
- Loading the pans
- Taring the empty sample pans
- Loading the samples into the pans
- Entering experiment information through the TA controller (sample and instrument information).
- Creating and selecting the thermal method on the controller.
- Attaching and setting up external accessories as required (*e.g.*, purge gas, Gas Switching Accessory).
- Starting the experiment.

Preparing the Samples

Selecting Sample and Tare Pans

The Auto TGA can utilize the same types of pans that are available for the TGA 2950 CE instrument.

When you are using the Auto TGA, it is possible to prepare from 1 to 64 different samples. Each sample platform can hold 16 numbered samples. The four sample platforms are numbered in the following ranges:

- 1-16 samples
- 17-32 samples
- 33-48 samples
- 49-64 samples.

Once you have selected the type of pan that you wish to use, you must use the *same type of pan* for all of the samples on the sample platform disk. The *same type of pan* that you use for experiments must also be used as a tare pan.

Tare Pan

1. Obtain a pan of the same type and size to be used for your experiments.

2. Remove the tare tube.

3. Use brass tweezers to hang the tare pan from the tare hook.

4. Mechanically tare the balance using the first step of the weight calibration procedure.

5. Replace the tare tube.

Before running any experiments on the TGA you must tare the sample pans to ensure that the weight measured by the balance reflects the weight of the sample only. You should tare the sample pans before each experiment, even if you use the same set of pans in consecutive experiments.

Taring the Sample Pans

When you tare the sample pans, the TGA reads the weight of the empty pans in their numbered order and then stores these weights as a set of offsets. These offsets are subtracted from subsequent weight measurements for each numbered sample. You can tare the pans manually or automatically as explained here.

NOTE: View the Autosampler log, found on the Instrument Control software, after taring to determine which pan, if any, does not tare during the procedure (Error 118). If a tare error occurs, replace the pan and manually retare it.

Automatic Tare

Because the TGA 2950 CE has two weight ranges, taring is done for both ranges. The tare weight is stored by the instrument for the appropriate weight range.

1. Place the platform on the sample arm as shown in Figure C.3. Make sure that the small pin is inserted in the hole in the platform. This will seat the platform correctly.

2. Using brass tweezers, place each sample pan in a numbered place on the platform, making sure it is stable.

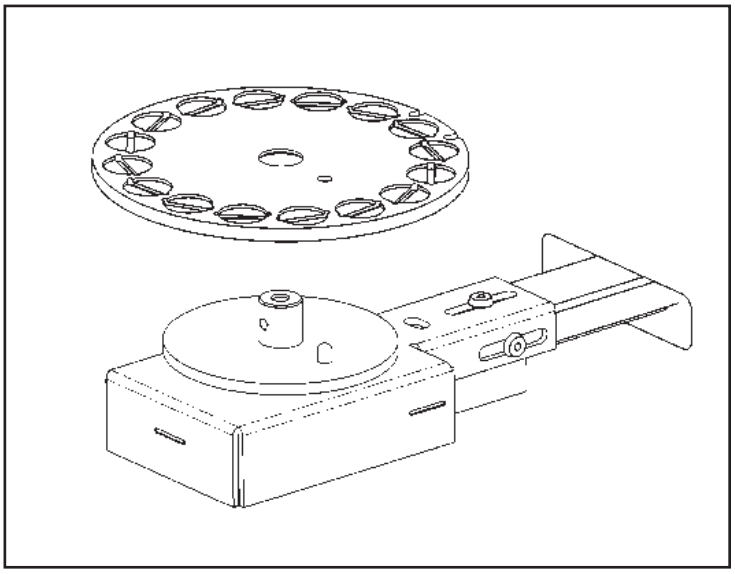


Figure C.3
Loading the Auto
TGA Sample Platform

3. Hold down the SCROLL key, then press the TARE key on the instrument keypad. The TGA will perform the following tare functions on each pan 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
- Unload the pan.

The Auto TGA will automatically tare each of the remaining pans in sequence. It should take approximately 45 minutes to tare all 16 pans on one sample platform. When the Auto TGA has completed taring each pan on the sample platform, the tare status message will disappear.

NOTE: The advantage to using the Auto TGA is to save time and effort; therefore, it would not be efficient to perform manual tare operations on each sample, as that would defeat the purpose of an automatic operation. If you wish to use manual taring, however, see Chapter 3 for the procedure.

Loading the Samples

After you have done the taring procedures for all of the empty sample pans, load the samples as follows:

1. Place each sample in the correct sample pan making sure that you do not switch around the pans on the sample platform (they have already been tared in the numbered position).

2. Place the sample pans on the sample platform in their original order. Make sure that the wire on the bottom of the sample pans 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 the brass tweezers to handle the sample pans.

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

3. Press the LOAD key. The TGA will automatically load the selected sample.

4. Position the thermocouple at the edge of the sample pan, rather than in the middle for the best results (see Figure C.4).

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

5. Press the UNLOAD key. The TGA will unload the sample and be ready for automatic sequencing.

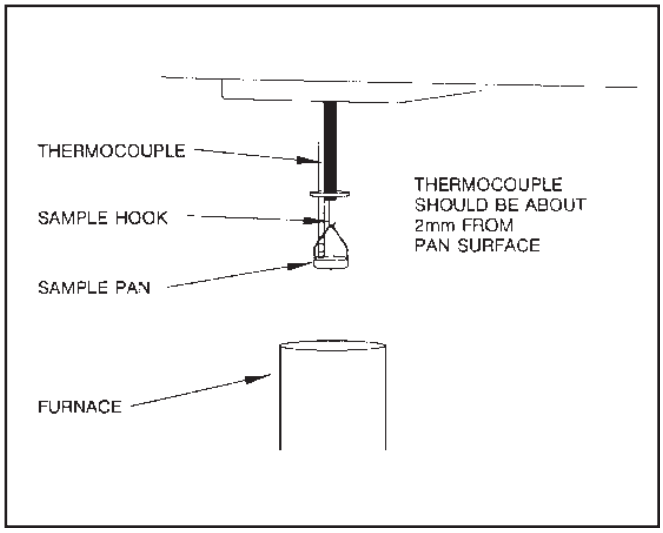


Figure C.4 Adjusting the Thermocouple

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. **Access the Autosampler Sequence**.

4. **Enter Sample Information.**

5. **Enter Instrument Information.**

NOTE:

If you are planning to run the Autosampler, be sure to specify furnace open at method end, as well as air cool, to cool down the furnace between samples.

6. **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. A different method may be selected for each sample.

Manual Operation

Use the AUTO SELECT and/or SCROLL/ AUTO SELECT to manually select the pan that you want to run, then follow the procedures found on in Chapter 3 to start, stop, and monitor an experiment.

For details setting up the methods consult the online help and documentation for further information.

Tracking the TGA Autosampler Status

To monitor the current run, observe the status of the Auto TGA , *etc.* you will need to access the Instrument Control software. Refer to the online help and documentation for further information.

Interrupting a Run

If you want to stop an Auto TGA run that is in progress, you can use one of the following keys on the TGA instrument keypad:

- Stop — Terminates the current method and run and causes the Auto TGA to unload the pan and start the next run. However, the data file for the interrupted run is saved.

- Reject — Terminates the current method and AS run and causes the Auto TGA to unload the cell and start the next run. The data file for the interrupted run is discarded.

- Other options are available through the controller functions, see the online help and documentation for further information.

Appendix D: TGA 2950 CE EGA Furnace Option

Introducing the EGA Furnace

The Evolved Gas Analysis (EGA) Furnace (Figure D.1) is an optional accessory that allows you to connect a spectrometer to the instrument so that the gases evolved by sample decomposition can be analyzed. The EGA furnace and the standard TGA furnace can be exchanged by a TA Instruments service representative. Version 3.3B (or higher) of the TGA 2950 CE instrument software is required for the EGA furnace.

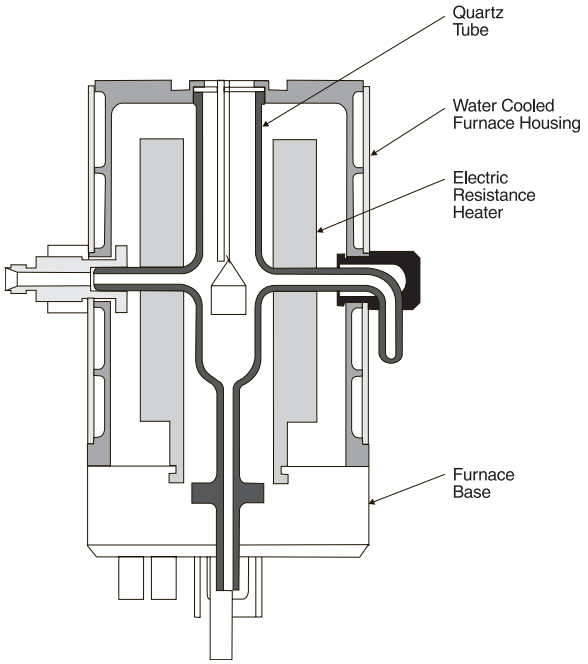


Figure D.1
TGA 2950 CE EGA Furnace

NOTE: The following description of the EGA furnace may also be found in Chapter 4 of this manual.

The EGA 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.

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 mass spectrometer. 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.

*Platinel II is a registered trademark of Engelhard Industries.

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.

EGA Furnace Specifications

Refer to Table D.1 below for the specifications of the EGA furnace. Some of these may be similar to the standard furnace for the TGA 2950 CE.

Table D.1 EGA Furnace Specifications

Temperature range	25°C to 1000°C
Thermocouple	Platinel II*
Heating rate	0.1 to 50°C/min

*Platinel II is a registered trademark of Engelhard Industries.

Installing the EGA Furnace

Contact a TA Instruments Service Representative for installation of the EGA furnace on the TGA 2950 CE instrument.

Connecting the Spectrometer

The TGA 2950 CE EGA furnace allows you to connect a spectrometer, such as a FTIR spectrometer, to the instrument. To connect any spectrometer, you will need to use a transfer line (supplied by the spectrometer manufacturer) to transport the gas evolved from the sample on the TGA to the spectrometer.

- The transfer line should be 1/8 inch in diameter to connect with a 1/8-inch Swagelok™ fitting on the exhaust gas connection.

- The transfer line should be made of heat-resistant alloy capable of resisting corrosion by the evolved gas and oxidation at temperatures up to 1000°C.

- The transfer line must pass through the exhaust gas fitting and a glass branch tube in the sample tube. It should end at a point just short of the inside diameter of the sample tube to ensure that the evolved gases do not condense before entering the transfer line.

- The transfer line must be long enough to allow flexible movement. It must accommodate movement of the EGA furnace up and down 3 5/8 inches to open and close for sample loading and unloading. (If the transfer line is not long enough, it must be disconnected and reconnected each time the furnace is opened and closed.)

To connect your spectrometer to the EGA furnace follow these steps:

1. Install a Swagelok™ nut, drive ring, and ferrule on the correct length of transfer line, leaving more than two inches of transfer line projecting beyond the ferrule.

2. Swage the ferrule, then cut the end of the transfer tube off so that two inches of the tube projects beyond the ferrule. See Figure D.2 below

NOTE: Extending the transfer line more than two inches beyond the Swagelok™ fitting may cause the TGA 2950 CE to operate improperly.

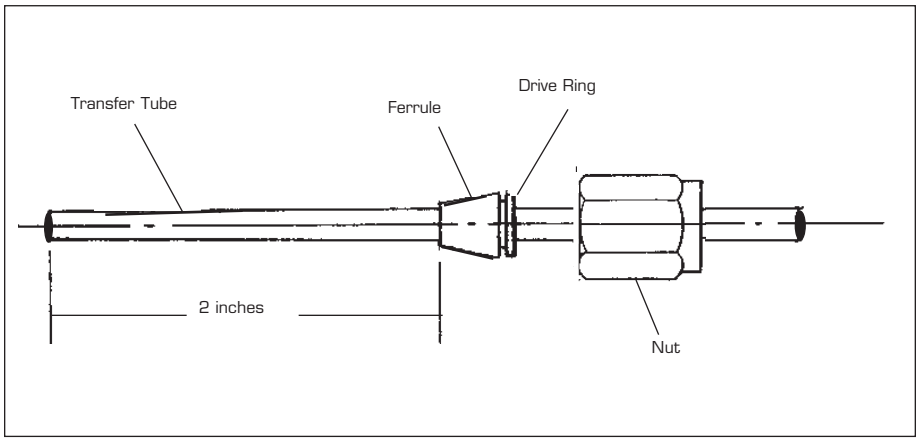


Figure D.2
EGA Transfer Line with Ferrule

3. Make sure that the end of the transfer line is straight and free of oxide deposits before you insert it into the exhaust gas connection.

4. Insert the transfer line and tighten the Swagelok™ nut to seal the connector. When you tighten the Swagelok™ nut, use a 3/8-inch wrench on the exhaust fitting flats to prevent them from turning.

WARNING

If the transfer line is not straight, or has heavy oxide deposits on it, the sample tube may be broken as the line is inserted.

Using the EGA Furnace

After proper installation, the EGA furnace can be used as you would normally use the TGA standard furnace. No special procedures must be followed when preparing TGA samples, setting up methods, or running experiments. Refer to the appropriate sections in the main manual for information. Follow the manufacturer's instructions for the use of your spectrometer when connected to the EGA furnace.

Cleaning the Quartz Furnace Tube

WARNING

If the TGA is used to evaluate materials using an oxygen purge, the furnace must 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. Do not insert metallic instruments inside the sample tube to scrape or chip contaminants from the sample tube as breakage may result.

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

WARNING

When cleaning the furnace, do not disturb the hang-down wire and furnace thermocouple, located directly above 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 D.3).

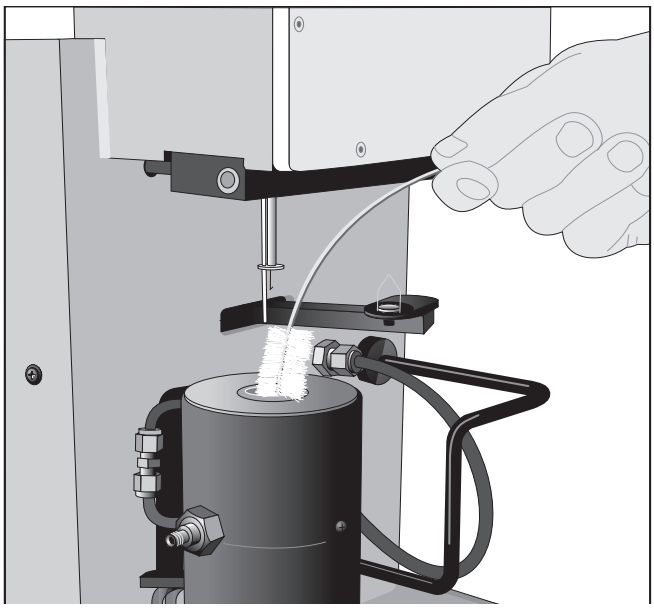


Figure D.3 Cleaning the Inside of the Furnace Tube

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 for one hour with nitrogen.

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

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