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Thermal Analysis & Rheology
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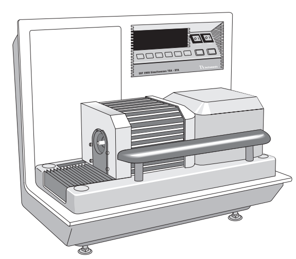

SDT 2960

Simultaneous DSC-TGA

Operator's Manual

PN 925605.001 Rev. F (Text and Binder)
PN 925605.002 Rev. F (Text Only)
Issued July 2000

Notice

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

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Helplines

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

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.**

Safety

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

- IEC 1010-1/1990 + A1/1992
- IEC 1010-2-010/1992
- EN 61010-1/1993 + A2/1995
- 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 SDT 2960 instrument:

- *Safety:*
  EN 61010-1/1993 + A2/1995 Installation Category II
  EN 61010-2-010/1994
- *Emissions:*
  EN 55022: 1995, CISPR 22:1993
  Class B (30–1000 MHz) Radiated
  EN 55022: 1995, CISPR 22:1993
  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.
  — IEC 801-3: 1984, 27–500 MHz, 3V/m. No response above 3.5 µg sample weight, 1.0°C sample temperature, and 0.01°C ΔT.
  — IEC 801-4: 1988, Fast transients common mode 1 kV AC power. No change of state.

Safety

(continued)

Instrument Symbols

The following label is displayed on the SDT 2960 instrument for your protection:

Symbol	Explanation
	This symbol, on the front panel between the furnace and balance assemblies, indicates a pinch-point caution. Before the furnace is closed, be sure this area is clear.
	This symbol, located in two places on the SDT 2960—on the furnace cage and on the "EGA" end of the furnace—indicates that a hot surface may be present. Take care not to touch these areas or allow any material that may melt or burn to come in contact with these hot surfaces.

Please heed these warning labels and take the necessary precautions when dealing with the furnace. The *SDT 2960 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. Hazardous voltages 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 WARNING on page 2-10 for the method of drying out the equipment before use.

Chemical Safety

Use only the purge gases listed in Table 1.3 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 corrosive or explosive gases in the SDT furnace. Oxygen can be used as a purge gas in the SDT. However, the furnace must be kept clean of volatile hydrocarbons that might combust.

Safety

(continued)

! WARNING

The SDT 2960 furnace assembly contains a layer of refractory ceramic fiber (RCF) insulation. This insulation is completely encapsulated within the furnace can, which is not meant to be disassembled.

! WARNING

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

Thermal Safety

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

! WARNING

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

Safety

(continued)

Lifting the Instrument

The SDT 2960 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 SDT 2960 module and its specifications.
Chapter 2	Describes how to connect the SDT to the rest of your system and how to unpack the balance.
Chapter 3	Describes how to run SDT experiments.
Chapter 4	Provides technical information and explains principles of SDT operation.
Chapter 5	Describes how to perform routine maintenance and diagnose power problems; also provides an explanation of the confidence test.
Appendix A	Lists worldwide TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.
Index	Lists the page numbers of important topics for your reference.

CHAPTER 1: Introducing the SDT 2960

Introduction

Your Simultaneous Differential Techniques instrument, or SDT 2960, is an analysis instrument capable of performing both differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) at the same time.*

The Simultaneous DSC-TGA works in conjunction with a controller and associated software to make up a thermal analysis system.**

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.

*In the "uncalibrated" mode, the SDT 2960 provides simultaneous TGA and differential thermal analysis (DTA) results.

**Use of the SDT 2960 as a DSC-TGA requires *Thermal Solutions* for Windows NT® Software (Version 2.4 or higher) to be loaded on the Thermal Analyst Controller and SDT Module Software (Version 3.0 or higher) to be loaded on the SDT 2960.

The 2960 Instrument

The SDT 2960 measures the heat flow and weight changes associated with transitions and reactions in materials over the temperature range ambient to 1500°C. The information provided differentiates endothermic and exothermic events which have no associated weight change (*e.g.* melting and crystallization) from those which involve a weight change (*e.g.* degradation). Furthermore, performing both DSC and TGA measurements at the same time, on the same instrument and same sample, offers greater productivity and removes experimental and sampling variables as factors in the analysis of data.

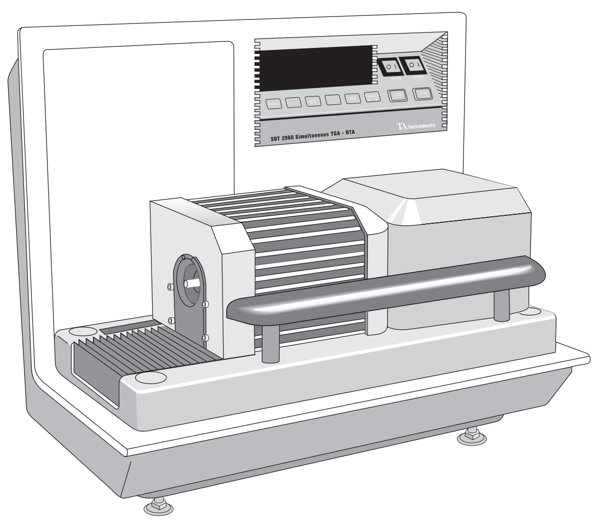


Figure 1.1
SDT 2960
Simultaneous DSC-TGA

Components

The SDT 2960 has three major components, illustrated in Figure 1.2:

- The **sample** and **reference balance assemblies**, which provide the precise measurement of heat flow and sample weight (See Chapter 4 for details.)
- The **furnace**, which controls the sample atmosphere and temperature.
- The **cabinet**, where the system electronics and mechanics are housed.

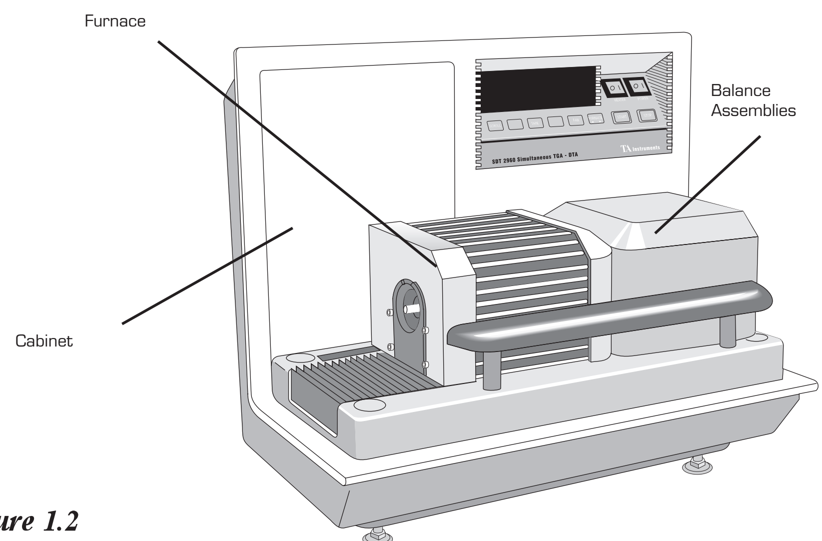


Figure 1.2
SDT 2960 Components

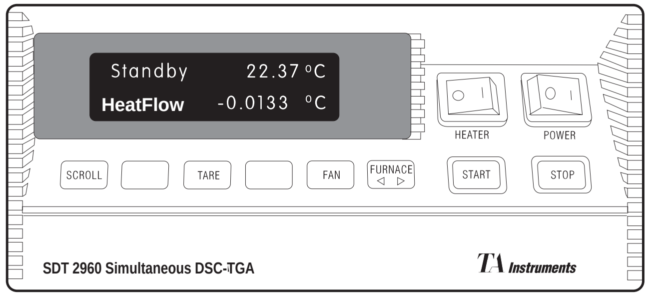


Figure 1.3
SDT 2960 Keypad and Display

SDT Display

The SDT display is the lighted area of the keypad (Figure 1.3). 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.*, HeatFlow). Status codes and realtime signals are described in the Technical Reference chapter of this manual.

A pound sign (#) in front of the weight signal indicates that the balance reading has not yet stabilized. When the weight stabilizes, the pound sign disappears.

SDT Keypad

The instrument keypad (see Figure 1.3) contains the keys described 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 SDT Keypad Function Keys

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display.
TARE	Zeros the displayed weight of the empty sample and reference cups and stores the weight as an offset.
FAN	Works like a toggle to turn the cooling fan on or off at any time before, during, or after a run.
FURNACE	Toggles between furnace closed (right) and open (left) functions, depending on where the furnace is when you press this key. This key can be used to reverse the direction of movement left to right.

(table continued)

Table 1.1 SDT Keypad Function Keys (cont'd)

Key/Function	Explanation
START	Begins the experiment. This is the same function as **Start** on the controller.
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 halts 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. Then the display returns to the signal displayed before SCROLL was pressed. If an experiment is not running, SCROLL/ STOP works like the STOP key.

HEATER Switch

The HEATER switch (see Figure 1.3) 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 whenever the power control circuits are enabled. This occurs when either a method is running or an end of method function is selected and active. (See “Heater Indicator Light” in Chapter 5 for more information.)

POWER Switch

The POWER switch (see Figure 1.3) turns the power to the instrument on and off.

SDT 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 SDT experiments.

Other Accessories

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

Specifications

Table 1.2 Operating Parameters

Temperature range	Ambient to 1500°C*
Control thermocouple	Platinum-Platinum/ 13% Rhodium Type R
Sample and reference thermocouple	Platinum-Platinum/ 13% Rhodium Type R
Heating rate	100°C/min to 1000°C 25°C/min to 1500°C
Cooling time	30 minutes; 1500°C to 50°C

♦***CAUTION:***

*** Leaving the furnace at high temperatures for extended periods of time may shorten the life of your furnace. Do not heat the furnace to high temperatures to clean the cups, clean them outside the furnace with a heat source such as a Bunsen burner.**

Table 1.3 Operating Parameters (cont'd)

Heat Flow Accuracy	Better than ± 2% (based on metal melting standards)
Heat Flow Precision	Better than ± 2% (based on metal melting standards)
Temperature Accuracy	±1°C (based on metal melting standards)
Temperature Precision	±0.5°C (based on metal melting standards)
Weight Sensitivity*	0.1µg
Weight Accuracy*	±1%
ΔT sensitivity (DTA)	0.001°C
Sample Cups	90 µL Alumina (DSC-TGA studies) 40 µL, 90 µL Alumina 40 µL, 110 µL Platinum (TGA-DTA studies)
Furnace atmosphere purge gases	Helium, nitrogen, oxygen**, air, argon
Purge rate	0–1000 mL/min initial rate 0–200 mL/min during experiments

*The SDT balance mechanism is sensitive to changes in the surrounding room temperature. For optimum accuracy, you must regulate the ambient temperature.

**Oxygen may be used. However, the furnace tube must be kept clean of volatile hydrocarbons.

! WARNING

Do not use hydrogen or any corrosive or explosive gases in the SDT furnace. Oxygen can be used as a purge gas in the SDT. However, the furnace must be kept clean of volatile hydrocarbons that might combust.

Table 1.4
Instrument Specifications

Dimensions	Depth 45.7 cm (18 in.) Width 58.4 cm (23 in.) Height 49.5 cm (19.5 in.)
Weight	29.4 kg (65 lb)
Operating line voltage	120 Vac, 50/60 Hz
Energy consumption	1.1 kVA
Processor data collection rate	0.5 to 1000 sec/pt
Communications port	GPIB (IEEE-488)

CHAPTER 2: Installing the SDT

Unpacking the SDT

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 for use in repacking and shipping your instrument.

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

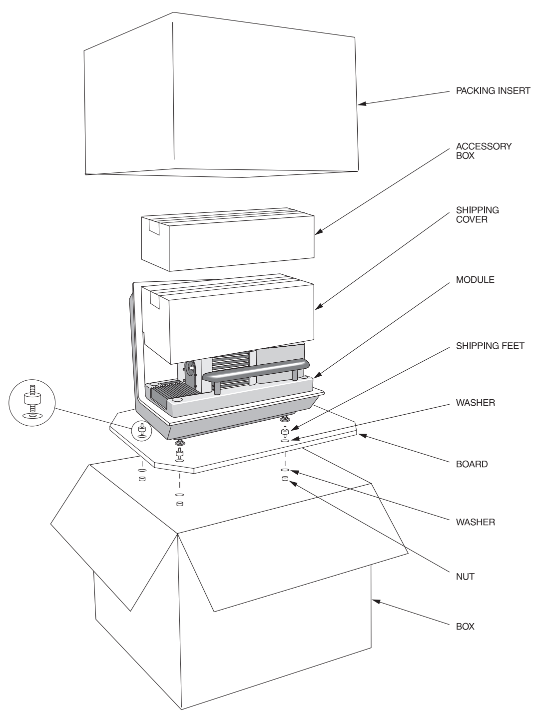


Figure 2.1
Shipping Boxes

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

2. Remove the cardboard packing insert.

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

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

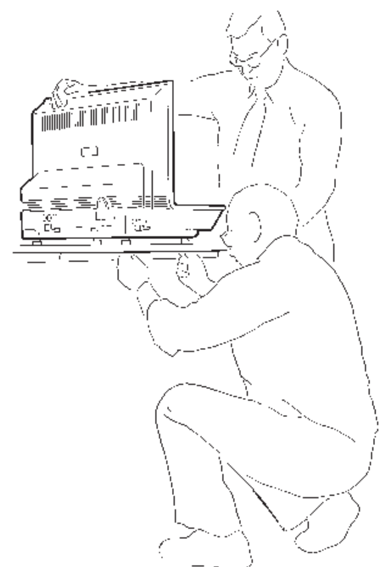


Figure 2.2 Removing the Plywood Board

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 hold onto the unit at all times while it is in**

this position. While your assistant holds the unit, remove the two nuts and washers from the other side.

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

7. 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.

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.

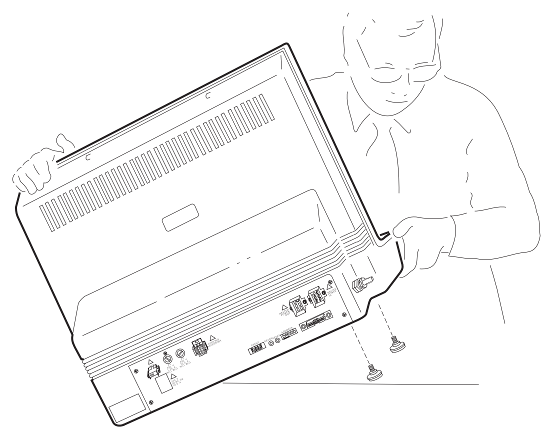


Figure 2.3 Installing the Mounting Feet

9. Remove the shipping cover and plastic shipping wrap.

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

If you need to repack the SDT 2960 for shipment, turn to Chapter 5 for directions.

Installing the Instrument

Before shipment, the SDT 2960 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
- Connecting the SDT to your TA Instruments controller
- Connecting the purge gas lines, accessories, and power cable
- Unpacking the balance.

If you wish to have your SDT installed by a TA Instruments Service Representative, contact your local representative (see Appendix A) 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 SDT, 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 offices can be found in Appendix A of this manual.

Choosing a Location

Because of the sensitivity of SDT experiments, it is important to choose a location for the instrument using the following guidelines. The SDT 2960 should be:

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

On
- . . . a stable, fire-resistant 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 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.
- . . . flammable materials.

! 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 moisure, causing leakage currents to exceed those specified in the applicable standards until the 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 "Running Experiments" for further information).

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

Connecting Cables and Gas Lines

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

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

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

! WARNING

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

GPIB Cable

1. Locate the GPIB connector on the right rear of the SDT instrument (see Figure 2.4).

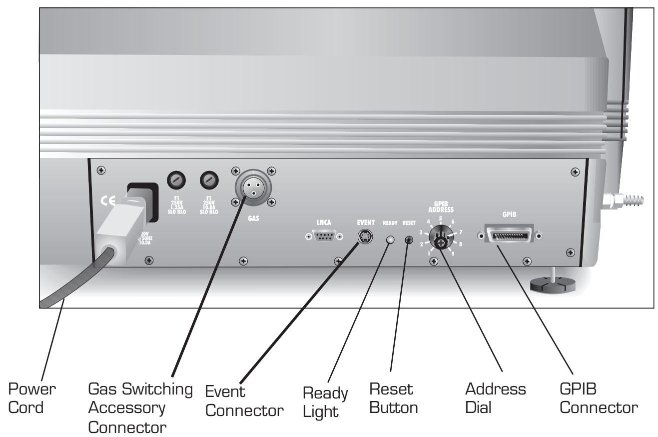


Figure 2.4
SDT Connector Panel

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

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

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

5. Select an address from 1 to 9 (one that is not used by any other instrument connected to your controller). Then use the address selector dial on the SDT connector panel to set the desired address. Figure 2.5 shows a instrument address of 7.

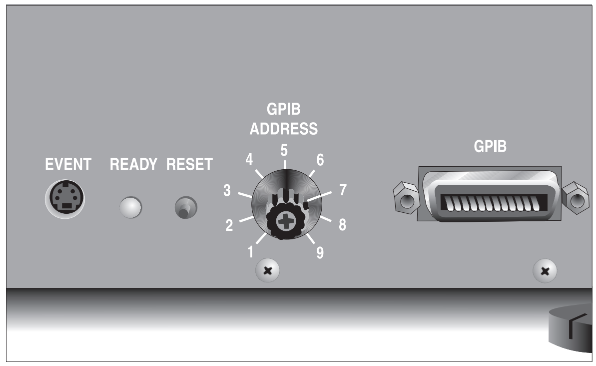


Figure 2.5
Address Selector Dial
(Showing an Address of 7)

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

If you change the address after the SDT is powered on, you must press and release the SDT's Reset button to enter the new address. Wait 30 seconds after releasing the Reset button; the green Ready light should begin to glow steadily. Reconfigure the instrument with the controller to bring the instrument back online.

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

Purge and Cooling Gas Lines

Purge Line

!WARNING **Do not use any liquid in the purge line.**

1. Locate the PURGE fitting on the left side of the SDT instrument back (Figure 2.6).

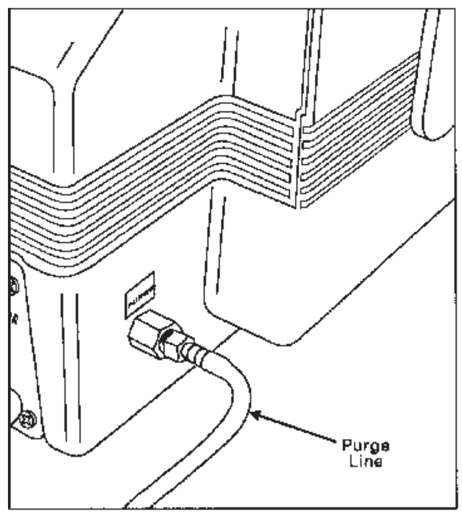


Figure 2.6
PURGE Fitting

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:***

Use of corrosive gases will shorten the life of the instrument. NEVER use a corrosive gas in the purge line.

! 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 SDT instrument, see Table 1.3.

3. Connect a length of 6.2-mm (1/4-inch) I.D. flexible tubing from the PURGE fitting to a flowmeter (consult your compressed gas vendor for specific requirements). Then connect the flowmeter to the purge gas source.

4. The recommended setting for the purge rate is 100 mL per minute.

Cooling Gas Line

1. Locate the COOLING GAS fitting, a 6.2-mm (1/4-inch) compression fitting on the left side of the SDT cabinet back, marked with a maximum pressure warning label (120 psi [840 kPa]) (Figure 2.7).

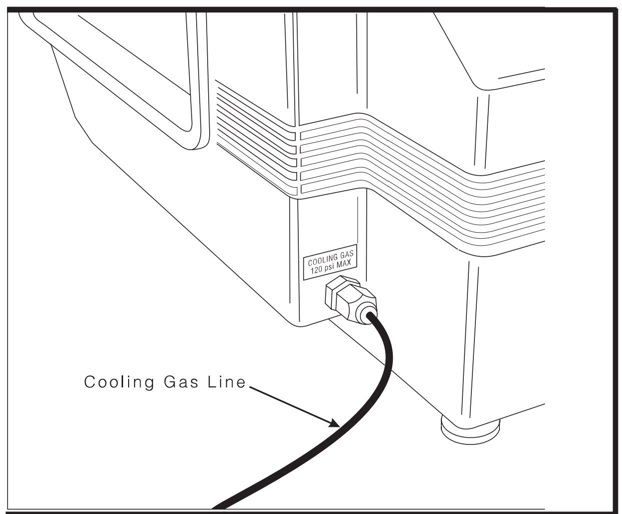


Figure 2.7 SDT COOLING GAS Fitting

2. Make sure your compressed air source is regulated to between 25 and 120 psi (175 and 840 kPa).

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

NOTE: Nitrogen may also be used as a cooling gas. Whichever gas is chosen as a cooling gas, it should be clean and dry.

NOTE: Air Cool will operate only if the furnace temperature is below 600°C.

Power Cable

1. Make sure the SDT POWER switch (Figure 2.8) is in the OFF (0) position.

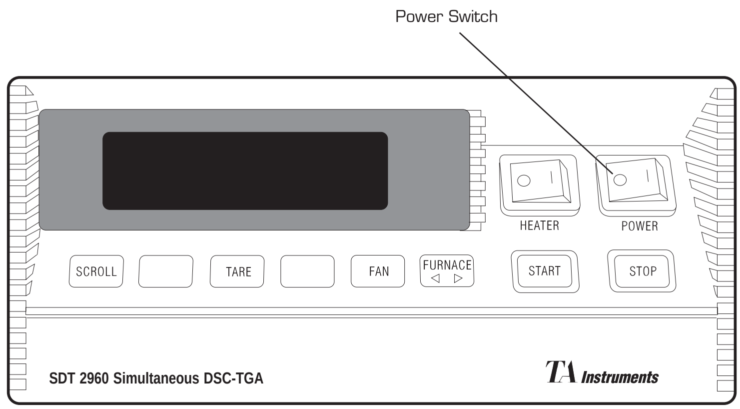


Figure 2.8
SDT POWER Switch

2. Plug the power cable into the SDT.

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

3. Plug the power cable into the wall outlet.

Unpacking the Balance

♦ ***CAUTION:*** **When unpacking the balance, follow these procedures carefully to prevent damage to the two balance beams.**

The SDT 2960 is shipped with a balance dress cover, shipping foam, and some mechanical parts locked in position in order to protect the inner mechanisms. Follow these procedures to unpack and prepare your instrument for use:

1. Using both hands, grasp the balance dress cover, and work it back and forth slightly as you pull it up. When you have enough clearance, place your fingers under the cover, as shown in Figure 2.9. Then pull out on the right bottom edge of the dress cover to clear the balance housing lid as you slide the dress cover up and off.

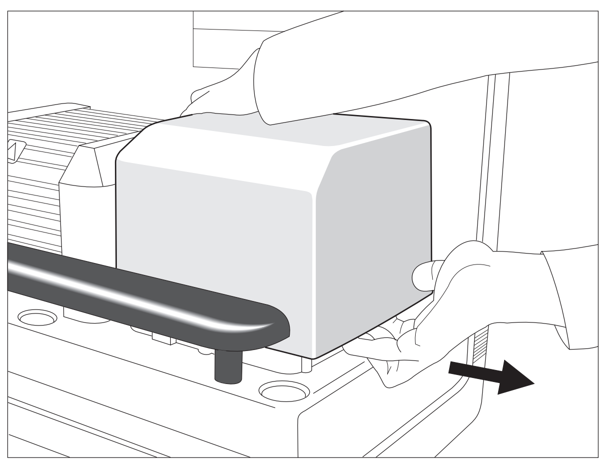


Figure 2.9 Removing the Balance Dress Cover

2. Locate the three screws on the shipping bracket positioned on the left side of the furnace (see Figure 2.10 below). This bracket is used to hold the furnace in place during shipping.

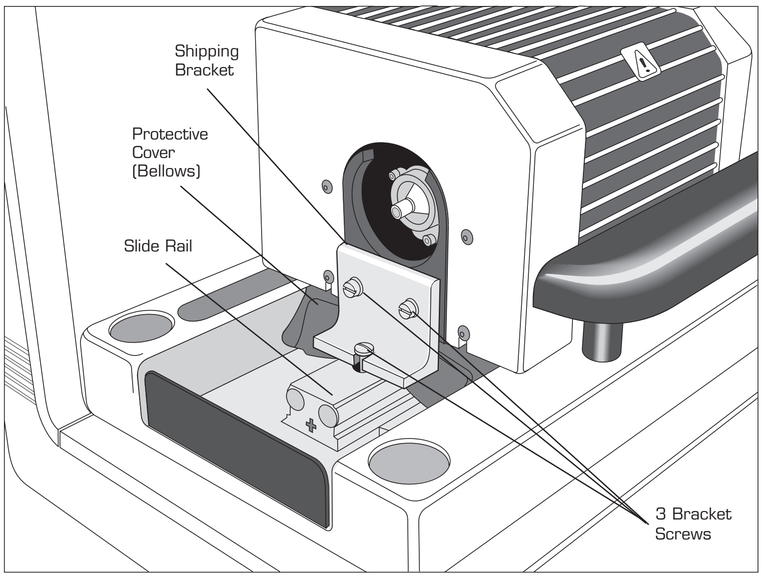


Figure 2.10
Location of the Shipping Bracket Assembly

3. Use a flathead screwdriver to completely remove the three (3) screws (shown in Figure 2.10) and their washers (each screw has one split washer and one flat washer). Then remove the bracket, but do not discard it. Retain the bracket in case you need to ship your SDT.

4. Replace all three screws and their washers in the same positions, two on the left side of the furnace and one in the slide rail.

5. Pull the rubber protective cover (bellows) out, and stretch it across to the left. Hook the end of the cover over the black metal plate.

6. Use a 3/32 Allen wrench to remove the six (6) screws holding the balance housing lid in place.

7. Place one hand on each side of the balance housing lid, and *carefully* lift the right side of the lid, as shown in Figure 2.11:

   a. Pivot the right side of the lid upwards first to clear the inner mechanisms of the balance assembly. Then lift up the left side of the lid, and remove the lid entirely.

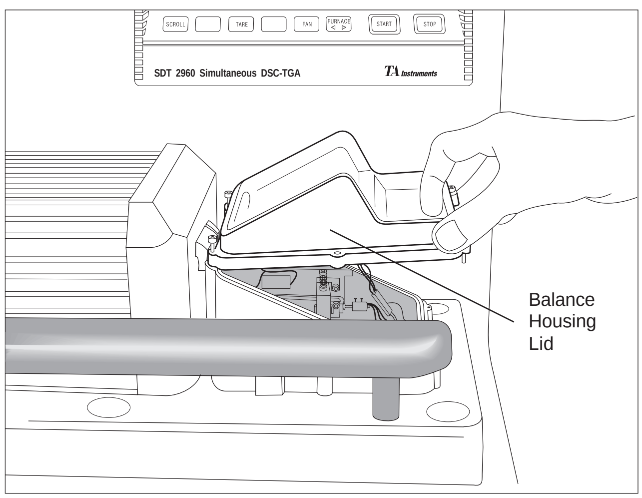


Figure 2.11 Removing the Balance Housing Lid

8. Inspect the balance assembly for signs of visible damage. If any damage is found, contact your TA Instruments representative before proceeding further.

9. Turn on the SDT's POWER switch.

10. Press the FURNACE key on the instrument keypad to start the furnace in motion. Stop the furnace by pressing the FURNACE button again (or using the STOP button) when the furnace has opened 2 to 3 inches. This allows enough room to reach the shipping foam around the balance beams, as shown in Figure 2.12 on the next page.

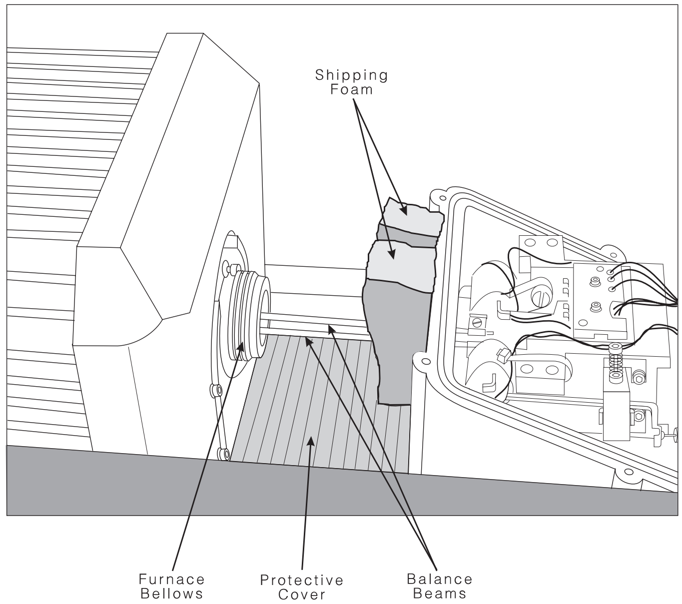


Figure 2.12
Location of Shipping Foam

11. Carefully grasp the tops of the foam with both hands, as shown in Figure 2.13, and separate the two top portions of the Y-shaped foam.

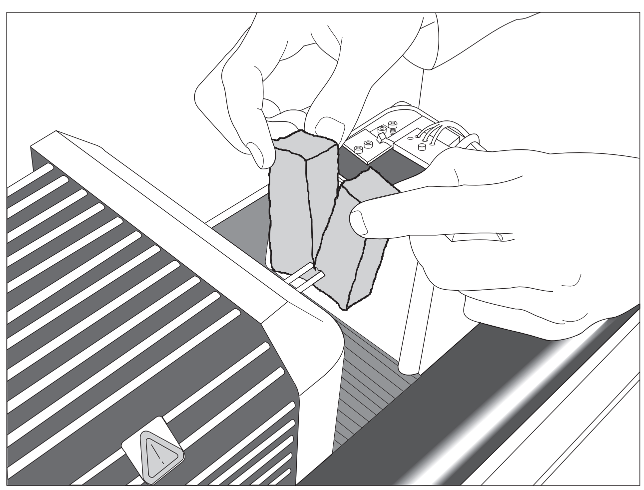


Figure 2.13
Removing the Shipping Foam

12. Push the back portion of the foam gently under the beams, and remove the foam *very carefully* to avoid breaking the sample and reference beams.

13. Locate the two screws on the front (sample) beam stop that are holding the tare beam in a locked position for shipment. Refer to Figure 2.14.

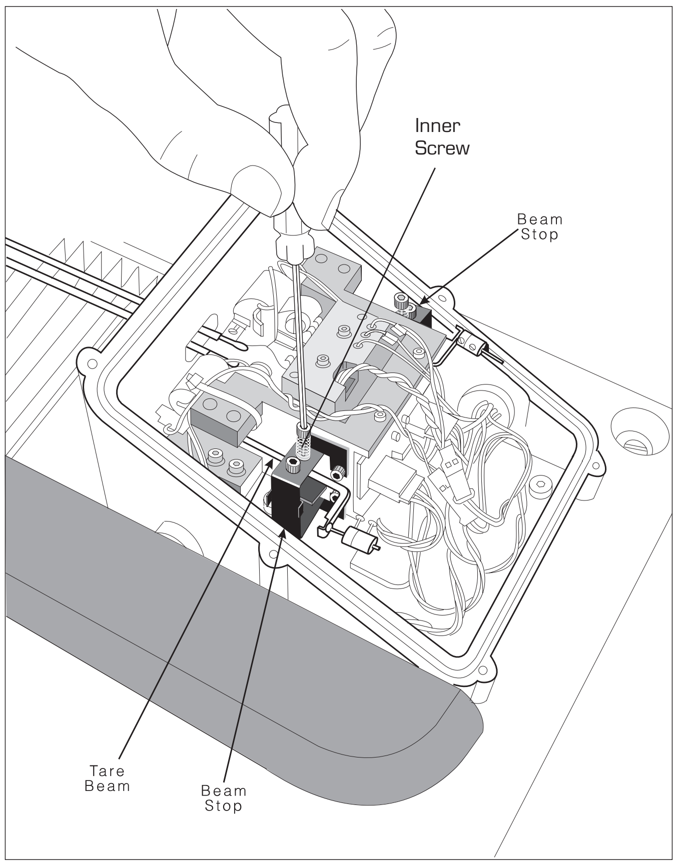


Figure 2.14 Releasing the Tare Beam—Inner Screw

14. Using a 3/32 Allen wrench, turn the inner screw (see Figure 2.14) counterclockwise a few turns to raise it up off the tare beam.

15. Turn the outer screw (see Figure 2.15) counterclockwise to loosen the beam stop assembly. This screw should be loosened until the tare beam has freedom of movement in both directions.

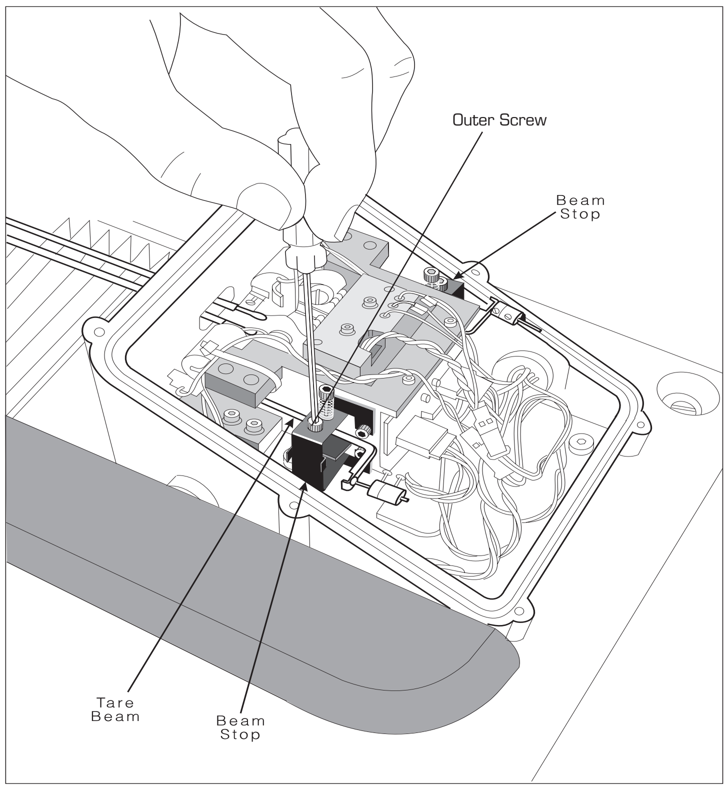


Figure 2.15 Releasing the Tare Beam—Outer Screw

16. Carefully turn the inner screw back down (clockwise) so that it is now very close to, but *not touching*, the tare beam. Do the same for the outer screw, so that the bracket is now very close to, but *not touching*, the tare beam. This readjustment of the beam stop ensures that the tare beam does not travel excessively.

17. Repeat steps 13 through 16 for the back (reference) beam stop.

18. Replace the balance housing lid, and re-install the six (6) screws to hold it in place.

19. Replace the balance dress cover.

NOTE: You must be sure to calibrate the instrument before beginning operation.

Turn to Chapter 5 for information on repacking the SDT 2960 for shipping, if required.

Starting the SDT 2960

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

1. Check all connections between the SDT and the controller. Make sure each component is plugged into the correct connector. (For installation information, turn to the beginning of this chapter.)

2. Press the instrument POWER switch 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. This is normal.

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 has finished, 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 in Figure 2.16.

4. Bring the instrument online with the TA controller.

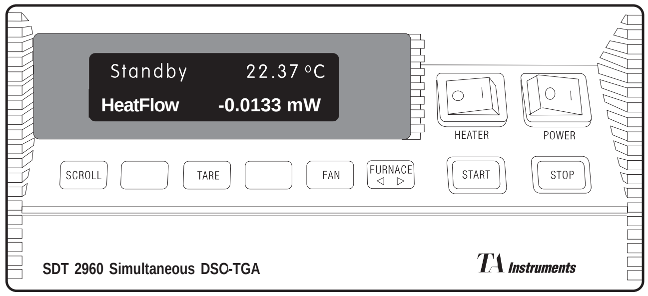


Figure 2.16
SDT Standby Display

Shutting Down the SDT 2960

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

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

- Selecting the desired mode of operation (DSC-TGA or TGA-DTA).
- Selecting the appropriate cup type and material for your sample.
- Entering experiment information through the TA controller (sample and instrument information).
- Creating and loading the thermal method on the controller.
- Attaching and setting up external accessories as required (*e.g.*, purge gas, air cool, gas switching accessory).
- Loading and taring two empty sample cups on the sample and reference beams.
- Loading the sample and reference, (if used).
- Starting the experiment.

To obtain accurate results, follow the procedures carefully and check calibration periodically (once a month). Consult the *Thermal Solutions* User Reference Guide for details on calibrating the SDT.

Before You Begin

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

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

Calibrating the SDT

To obtain accurate experimental results, you should calibrate the SDT 2960 when you first install it, and periodically thereafter. As a minimum, the SDT should be recalibrated anytime the beam set, experimental heating rate, or purge gas is changed.

The SDT can be operated as a DSC-TGA or as a TGA-DTA. In the former case, the TGA weight calibration, temperature calibration, and DSC heat flow calibration must be completed before beginning experiments. For TGA-DTA operation, the TGA weight calibration, DTA baseline calibration, and the temperature calibration are necessary. These calibrations should be performed in the order shown in the following sections.

TGA Weight Calibration

This is the initial calibration which should be performed. The TGA-DTA Calibration mode is used. TGA Weight Calibration is based on two runs: one using calibration weights and one using no weights (empty beams). The data from both runs is analyzed, and beam and weight correction factors are calibrated.

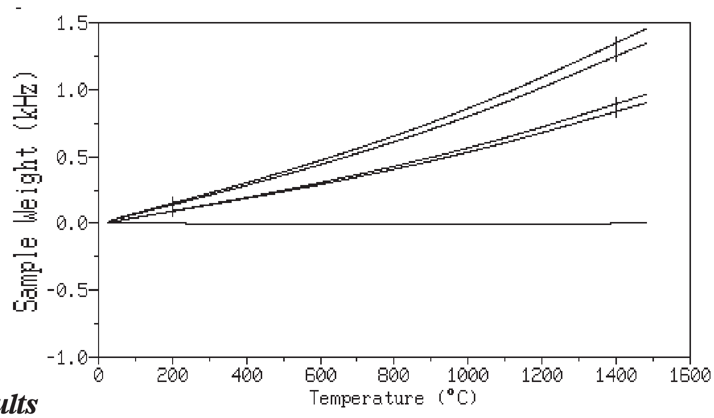


Figure 3.1 TGA Weight Calibration Results

DTA Baseline Calibration

DTA Baseline Calibration is based on analyzing data collected from a baseline run (see Figure 3.2) conducted over the temperature range expected in subsequent TGA-DTA mode experiments. (This experiment usually utilizes the same baseline run obtained for TGA Weight Calibration.)

The DTA baseline is corrected by a linear (slope and offset) function of the sample temperature. This results in shifting and rotating the baseline so that the calibrated portion is near 0°C.

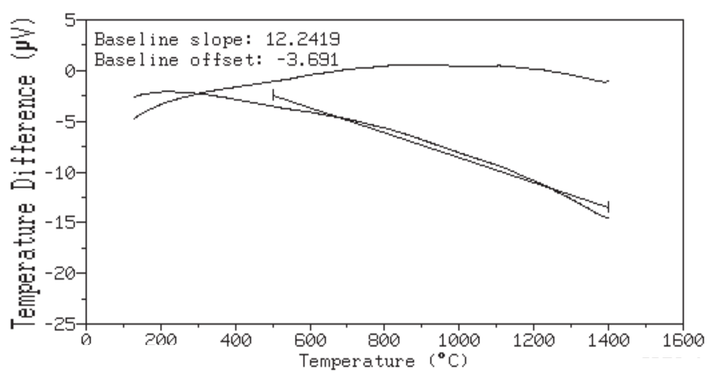


Figure 3.2 Baseline Calibration Results

NOTE:

The DTA baseline calibration is not required when using the SDT as a DSC-TGA. This is because the DSC Heat Flow Calibration includes a baseline subtraction. However, the shape of the DTA baseline is valuable as a quick verification that the SDT beams are correctly positioned and that DSC heat flow calibration can be successfully completed.

Temperature Calibration

Temperature calibration is based on evaluation of the melting endotherm(s) of a high purity metal standard(s). The recorded onset of melting of this standard(s) is compared with the literature value and the difference is calculated for temperature calibration. Up to five standards may be used. Zinc (419°C) is supplied in the SDT accessory kit. Other suitable standards are tin (232°C), aluminum (660.5°C), silver (961°C), gold (1064°C) and nickel (1455°C). If you use one pair of known and observed points, the entire curve is offset, or shifted, to the actual melting point. If you use multiple standards, the temperature is corrected by a cubic spline fit. The multiple-point temperature calibration is more accurate than the one-point calibration.

DSC Heat Flow Calibration

This calibration is available only for DSC-TGA mode and is required when running in this mode. SDT heat flow calibration is based on analyzing the heat capacity curve for sapphire over the range 200 to 1500°C (see the figure on the next page) and the heat of fusion of high purity zinc metal. Three experimental runs are required: one using an empty alumina cup (90µL) for both the reference and sample (baseline run) and one using a sapphire standard (supplied in the SDT Accessory Kit) as the sample. The measured heat capacity for sapphire is compared with the literature value at multiple temperatures across the range and mathematically fitted to generate an E-curve.

This E-curve may be further refined by a third experiment that measures the heat of fusion of high purity zinc wire (supplied in the accessory kit). The heat of fusion is measured and the cell constant is calculated using the known value of the heat of fusion of zinc (108.7 J/g) and the equation "Cell Constant = Known Value/ Measured Value." The calculated value of the cell constant is then entered into the software.

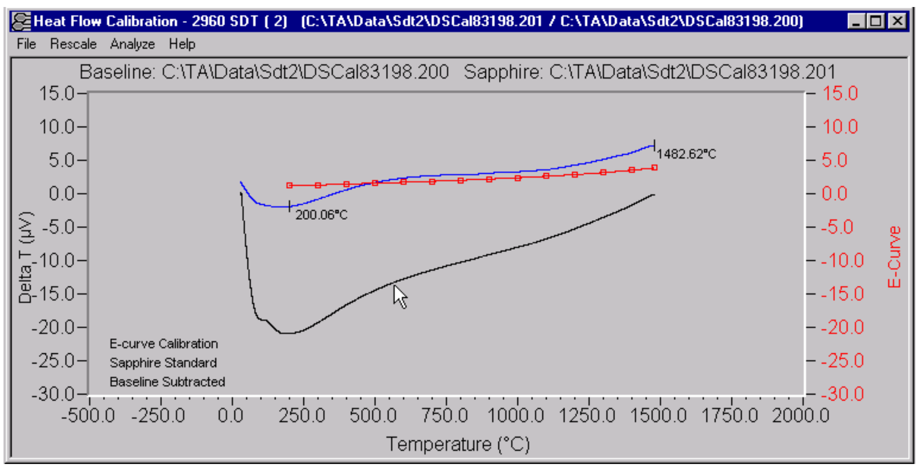


Figure 3.3
DSC Heat Flow Calibration

Running an SDT Experiment

Experimental Procedure

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

- Selecting the desired mode of operation (DSC-TGA or TGA-DTA).
- Selecting and preparing a sample. This involves preparing a sample of the appropriate size, selecting the sample cup, and placing the sample in the cup.
- Loading and taring the sample cup (and a reference cup) on the balance.
- Entering experimental information through the TA controller.
- Creating and selecting the thermal method on the controller.
- Setting up any external accessories.
- Starting the experiment.

Mode

The SDT can be operated as a DSC-TGA or as a TGA-DTA. The desired signals (heat flow or temperature difference) determines which operating mode to use. To change the mode, use the *Thermal Solutions* Instrument Control software (see the online documentation for information).

Preparing SDT Samples

Two kinds of specialized sample cups are available for the SDT 2960. In addition, these cups come in several sizes. The criteria for choosing a sample cup are simple:

- For DSC-TGA experiments, the ceramic (alumina) 90 µL cups must be used. These cups are compatible (*i.e.,* do not affect the results) with most high temperature materials. In addition, the sapphire DSC heat flow calibration standard has been specifically cut to fit the 90 µL cups.

- For TGA-DTA experiments, the ceramic (40 µL or 90 µL) or platinum (40 µL or 110 µL) cups are acceptable alternatives. Platinum is generally preferred for TGA-DTA work because it is easy to clean and does not react with most organics and polymers. Ceramic is a better choice for inorganics or corrosive materials.

All cup types are reusable. To clean a cup between experiments, use a Bunsen burner or a propane torch to burn out any residue.

♦***CAUTION:***

Do not heat the furnace to high temperatures to clean the cups, clean them outside the furnace as directed above. Leaving the furnace at high temperatures for extended periods of time may shorten the life of your furnace.

!WARNING

If platinum cups are used at high temperatures (*i.e.,* above 1300°C), they can stick to the platinum sensor in the sample and reference platforms, which can cause damage to the sensor when a cup is removed. This may result in the need to replace the sample and reference beam assembly.

(continued on next page)

! WARNING

To prevent this from occurring, you can (1) use a ceramic cup or (2) place some fine-grain alumina powder between the platinum cup and the sensor before you start a TGA-DTA experiment.

NOTE: Alumina absorbs moisture. Before using alumina, you will need to dry it. Store alumina powder in a desiccator.

Taring and Loading the SDT Cups

Taring the SDT cups ensures that the weights measured by the balance reflects the weight of the sample only. You should tare the SDT cups before each experiment with the furnace closed, even if you use the same cup in consecutive experiments.

When you tare a cup, the SDT reads the weight of the empty cup 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. When you use the automatic tare procedure, the SDT will determine when the weight reading is sufficiently stable. Because the SDT 2960 has two balance assemblies, taring is done for both the reference and sample SDT cups.

NOTE: Always use tweezers to handle SDT cups.

1. Press the FURNACE key on the instrument keypad to open the furnace.

2. Place an empty SDT cup on the platform for the front sample balance assembly, making sure that it is seated properly (see Figure 3.4).

3. Place an empty SDT cup on the platform for the back reference balance assembly, making sure that it is seated properly (see Figure 3.4).

4. Press the FURNACE key to close the furnace and protect the cups from air currents.

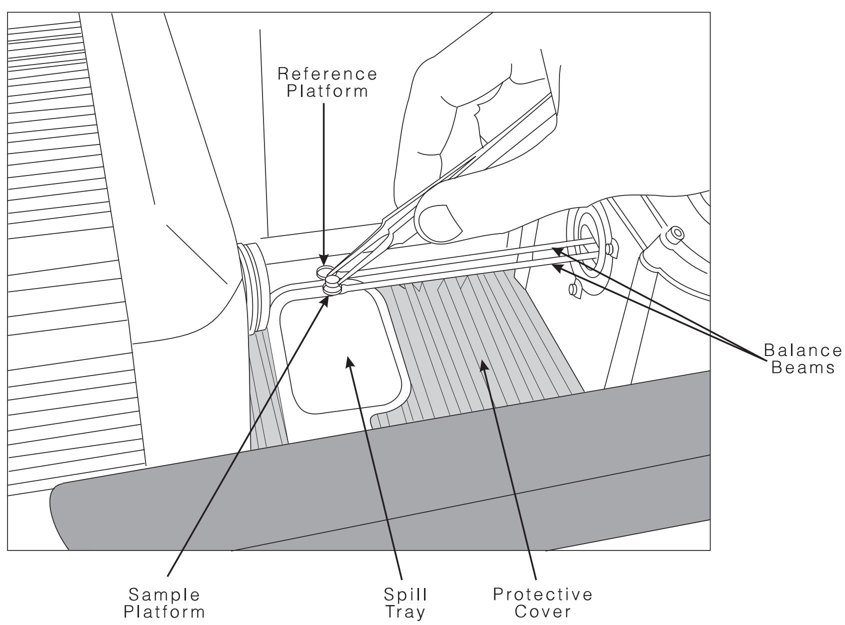


Figure 3.4
Loading the SDT Sample Cup

5. Press the TARE key on the instrument keypad. The SDT will automatically weigh the cups and store the weights as the offset.

NOTE: The instrument will not tare if the furnace is open, or if the temperature is changing too rapidly (*i.e.*, if the temperature causes the weight to change by more than 3.0 µg in a 10-second period).

6. Press the FURNACE key to open the furnace.

   After taring the SDT cups, you can load your reference material (such as aluminum oxide), if desired, by following steps 7 through 11. If you do not plan on using a reference material, skip to step 12.

NOTE: A reference material (such as aluminum oxide) is recommended for TGA-DTA experiments as a way to minimize the difference in heat capacity between the reference and sample pans; thereby improving the baseline. Reference material should not be used for DSC-TGA experiments. Rather, calibration and subsequent sample experiments should be performed with an empty alumina cup as the reference.

♦ ***CAUTION:*** **Spilling sample material on the platform could cause permanent contamination of the platform. If this occurs, both balance beams would need to be replaced. Therefore, remove the cups from the beams when loading samples.**

7. Rotate the spill tray into position under the beams.

8. Remove the reference cup from the rear (reference) platform.

9. Place the same amount of reference material inside the reference cup as you are planning to use for your sample.

10. Replace the filled cup on the reference platform.

11. Press the SCROLL key on the instrument keypad until the reference weight is displayed. If the weight is acceptable, go on to step 12. If it is not acceptable, repeat steps 8 through 11 as needed.

12. Remove the sample cup from the front (sample) platform.

13. Place the sample material in the sample cup. If a reference material was used, measure out approximately the same amount of sample material.

14. Position the cup on the sample platform, making sure that the sample cup is seated correctly on the platform.

NOTE: Reproducible positioning of the sample and reference cups on the sample and reference platforms (beam sensors) is critical to obtaining optimum SDT performance, particularly for DSC-TGA calibration and experiments. Because "centering" the cup on the platform is difficult to do repeatedly, it is recommended that the cups be positioned so that they are always located all the way to one side of the platform (see Figure 3.4). In addition, the rotational placement of the cups should be consistent. This can be accomplished by scribing a small mark on the cup and always facing it in the same direction on the platform.

15. Press the SCROLL key on the instrument keypad until the sample weight is displayed. If the weight is acceptable, go on to step 16. If it is not, repeat steps 12 through 15 as needed.

16. Return the spill tray to its position under the armrest.

17. Press the FURNACE key on the instrument keypad to close the furnace around the sample material.

NOTE: If the weight is out of range (<Range or > Range), the furnace will not close. This feature prevents damage to the balance beams.

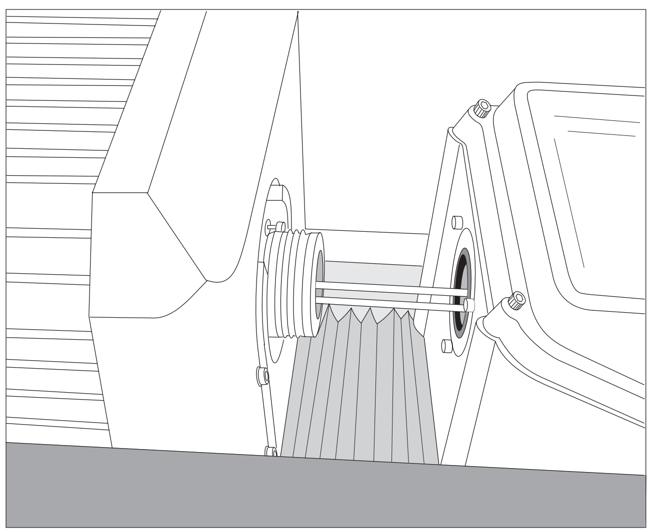


Figure 3.5 Furnace Closing

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 *Thermal Solutions User Reference Guide* 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 SDT 2960, 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.

♦CAUTION: **Leaving the furnace at high temperatures for extended periods of time may shorten the life of your furnace.**

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 SDT 2960:

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

The SDT can also be used with other accessories, such as vacuum, mass spectrometers, FTIR, gas chromatographs, and other 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 this option from the TA controller. The air cool option uses two techniques to achieve rapid cool down. Selecting the air cool option activates both techniques:

- The first technique uses a fan that blows air around the outside of the furnace. This fan will come on at the end of the experiment, but it will be effective only if the furnace is closed. (The fan can also be turned on or off at any time from the instrument keyboard.)

- The second technique is an air cool ring that uses compressed air (supplied by the cooling gas line) to blow air around the furnace tube. This technique is used when the furnace has cooled down below 600°C.

NOTE: Keeping the furnace closed is the quickest way to cool the furnace.

After the air cool option is activated, it will continue to run until the furnace reaches the desired temperature.

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 (175 and 840 kPa).

Using a Purge Gas

You can control the sample atmosphere during SDT experiments by connecting a purge gas to the system.

The balance purge maintains a positive pressure in the balance housing to prevent decomposition products from contaminating the sensitive balance mechanism. The balance purge flows into the right side of the balance housing, directly through to the furnace, and exits via the exhaust nozzle (see Figure 3.6) on the left side of the furnace. The purge flows through the furnace horizontally across the sample (Figure 3.7), permitting rapid removal of decomposition products from the sample environment.

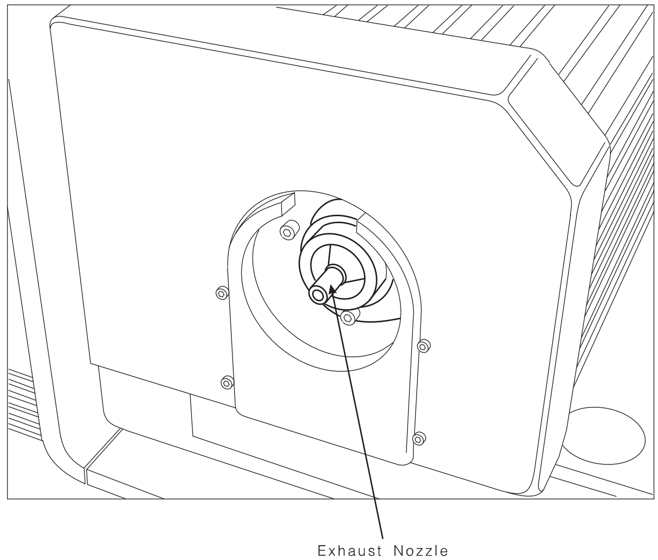


Figure 3.6
Furnace Exhaust Nozzle

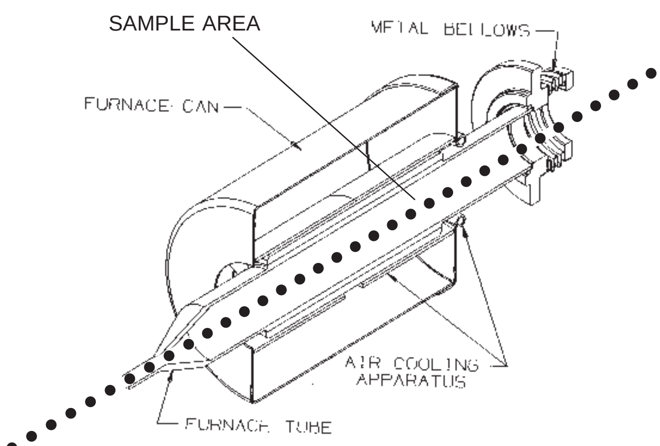


Figure 3.7
Furnace Purge

You can choose nitrogen, oxygen, helium, air, or argon for your purge environment. Do not use any other gases in the SDT 2960.

WARNING

Do not use hydrogen or any corrosive or explosive gases in the SDT furnace. Oxygen can be used as a purge gas in the SDT. However, the furnace must be kept clean of volatile hydrocarbons that might combust.

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 SDT.

The recommended setting for the purge rate is 100 mL per minute. (An initial rate of up to 1000 mL per minute may be used to prepurge the system before starting the experiment.)

To maintain this flow rate, you will need to connect a flowmeter to the purge fitting on the back of the SDT instrument. Set the purge gas flow rate by adjusting this meter.

Before you start the purge gas, make sure that the desired gas is connected to the purge port, that the line is clear, and that your supply of purge gas is sufficient for the experiment. Always maintain a constant purge flow rate throughout your experiment; changing the purge rate 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 an SDT experiment. Before starting an experiment that uses the Gas Switching Accessory, make sure its power switch is on, and make sure the necessary gas sources are properly connected.

The Gas Switching Accessory can be controlled with the Gas segment in the method (see the *Thermal Solutions User Reference Guide*) or with the **Gas** option.

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

Starting an Experiment

You can begin the experiment using either the START key on the SDT keypad or **Start** on the controller.

NOTE: Once the experiment is started, operations are best performed at the controller keyboard. The SDT 2960 is very sensitive to motion and might pick up the vibration caused by pressing a key on the instrument keypad.

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 lose a significant amount of weight during the setup period (*i.e.*, samples with volatile solvents). When a forced start is initiated, the current sample weight is stored as the initial weight, data collection is started immediately, and the instrument status changes from "Set Up" to "Started." The method begins when the normal setup procedures are completed

To use forced start, press the START key again on the instrument keypad *while* the instrument status 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 SDT instrument keypad or **Stop** on the controller). Another function that stops the experiment is **Reject** (SCROLL-STOP on the instrument keypad) or **Reject** on the controller. However, the Reject function discards all of the data from the experiment; the Stop function saves any data collected up to the point at which the experiment was stopped.

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

♦ ***CAUTION:*** **When removing hot SDT cups at the end of an experiment, be sure to place them in the catch pan provided in the accessory kit. This catch pan can be placed on top of the plastic meter housing cover to prevent a hot SDT cup from melting the plastic cover.**

Chapter 4: Technical Reference

Description of the Simultaneous DSC-TGA

The SDT 2960, as a simultaneous device, can perform the functions of two different thermal analysis techniques at the same time. The sample is placed under identical environmental conditions for both analyses, thus allowing you to produce data that can be correlated and closely compared.

TGA Functions

The TGA-functioning portion of the instrument 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 position sensor flag blocks an equal amount of light to each of the photodiodes (the light is supplied by a constant current infrared LED). As sample weight is lost or gained, the beam becomes unbalanced, causing an unequal amount of light to strike the photodiodes. The unbalanced signal, called the error signal, is acted upon by the control circuitry and reduced to zero, or nulled. This is accomplished by an increase or decrease in the current to the meter movement, causing it to rotate back to its original position (null position). The change in current necessary to accomplish this task is directly proportional to the change in mass of the sample. This current is converted to the weight signal. (See page 4-12 for the theory of operation.)

DSC Functions

The SDT accomplishes DSC measurements, along with TGA measurements, by employing a single heat source and two symmetrically located and identical sample platforms at the end of two parallel beams. Thermocouples, welded at the center of the sample platforms, measure the differential heat flow to the sample and reference as both are heated at a uniform rate by the furnace. Sample temperature is also monitored directly by the thermocouple in the sample platform. With proper calibration, the heat flow associated with endothermic and exothermic transitions in materials can be measured to a high degree of accuracy and precision (± 2%). Without calibration, the heat flow results obtained are qualitative (DTA). (See page 4-12 for the theory of operation.)

During normal operation of the SDT, the sample or reference may evolve gases as heat is applied. To prevent back-diffusion of these liberated gases to the balance housing, the balance housing is purged with an inert gas regulated by the flowmeter. An inert gas must be used to prevent contamination or corrosion of the balance.

Components

The SDT 2960 has three major components, illustrated in Figure 4.1:

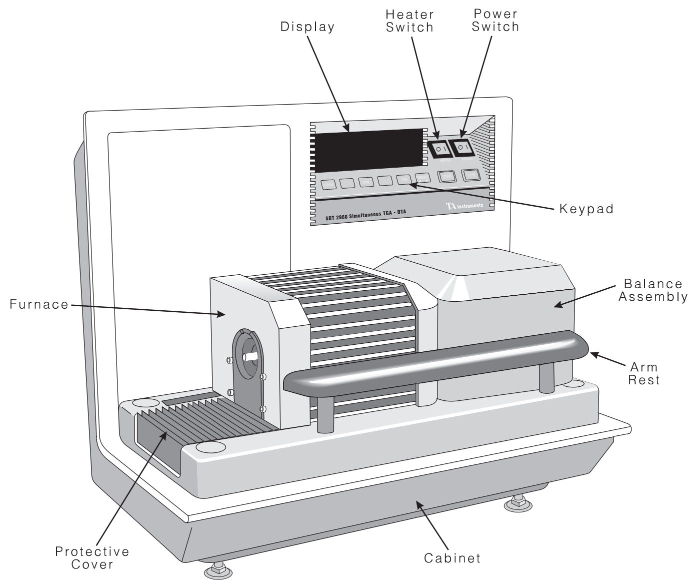


Figure 4.1
SDT 2960 Components

- The **sample** and **reference balance assemblies**, which provide the precise measurement of the sample weight and the DSC/DTA measurement.

- The **furnace**, which controls the sample atmosphere and temperature.

- The **cabinet**, where the system electronics and mechanics are housed.

Sample and Reference Balance Assemblies

The sample and reference balance assemblies consist of the individual meter movements, balance beams (including sample platform, platinum liners, and thermocouple wires), tare beams and tare weights, position sensor beams, and sensors, all fastened to a meter mount. Both meter mounts are, in turn, fastened to a *y*-axis adjustable platform. Figure 4.2 illustrates the parts of the balance assemblies.

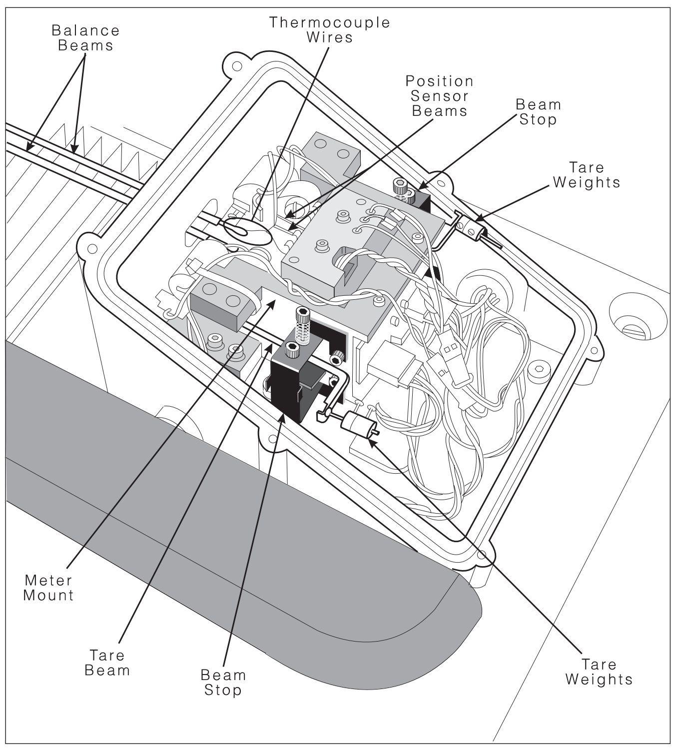


Figure 4.2
Balance Assemblies

Because the sample and reference balance assemblies are identical, but mirror images of each other, the following description applies to both assemblies (refer to Figure 4.2 for an illustration of these parts):

- The **balance meter movement** is a taut-band meter movement to which the balance beam, position sensor beam, and tare beam are attached.

- The **balance beam** is a ceramic alumina beam with the sample platform liner on one end, thermocouple wires running the length of the beam, and a metal bracket mounted on the other end. The metal bracket mounts the beam to the meter movement. The thermocouple wires are very fine at the ends and become thick before terminating in a reference junction copper block assembly.

- The **tare beam** is an aluminum sheet metal bracket with a screw fastened on one end. A counterweight (tare weight) slips over the screw and provides a semipermanent means of taring the balance assembly.

- The **position sensor beam** works with the position sensor printed circuit board assembly to detect the null position of the meter movement. They are used in conjunction with the analog circuitry to maintain a null position.

Furnace

The SDT furnace (Figure 4.3) consists of a furnace tube, a furnace can, an air-cooling apparatus, and a metal bellows, all of which are mounted to a cradle that moves the furnace to an open (left) or closed (right) position.

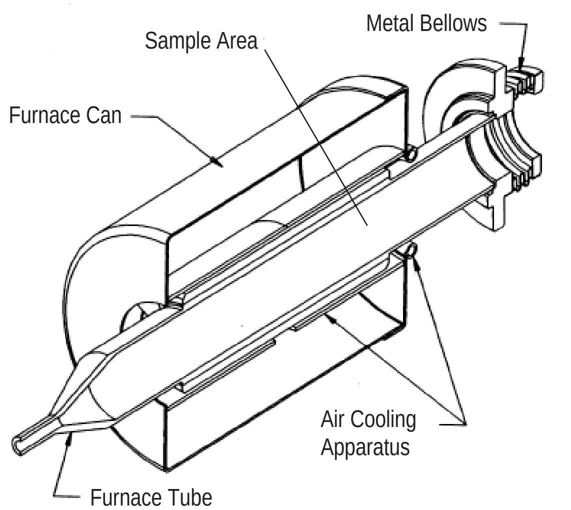


Figure 4.3
SDT 2960 Furnace

The **furnace tube** is a one-piece alumina sample tube/heater core arrangement that minimizes mass by winding the platinum rhodium heater wire directly on the sample tube. Controlled heating rates of up to 100°C/min can be obtained to 1000°C, and of 25°C/min up to 1500°C.

If desired, cooling air can be introduced and directed between the furnace tube and an air-cool sleeve via a metal tube at the completion of test runs.

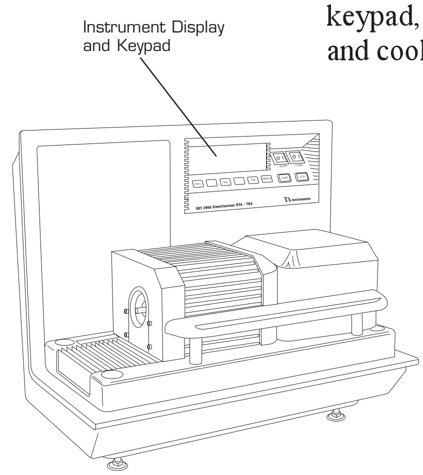
The cylinder-shaped **metal bellows** fits over the end of the **furnace tube** with an O-ring seal and provides a low thermal conductivity means of sealing the furnace tube to the balance housing. This minimizes heat flow into the balance housing.

The **furnace cradle** moves the furnace assembly around the sample and reference platforms when the furnace closes.

SDT cups are available in platinum in 40 and 110 µL sizes and alumina ceramic in 40 and 90 µL sizes. All pans are 0.258 inch in diameter. All four types can be used for TGA-DTA studies. Only the 90 µL alumina cups should be used for DSC-TGA evaluations.

Cabinet

The SDT cabinet (Figure 4.4) consists of the cabinet housing, the instrument display and keypad, the electronics compartment, the purge and cooling gas fittings, and the rear panel.



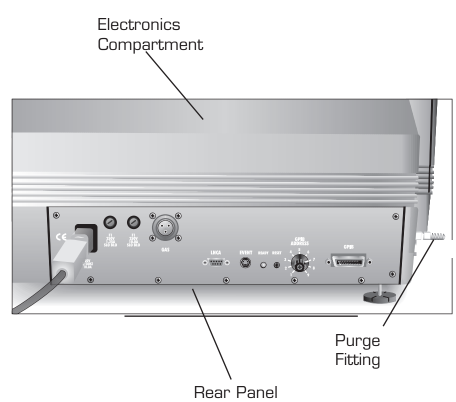


Figure 4.4
SDT Cabinet Components

The SDT **cabinet housing** consists of a base casting and a rear cover. The base casting is one-piece casting of heavyweight aluminum, designed to provide a stable platform for the SDT instrument parts. The rear cover is injection molded using a heavy-gauge thermoplastic material, designed for easy cleaning.

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

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

The **purge fitting** is located on the right side of the instrument back (see Figure 4.5).

The **cooling gas fitting** is located on the left side of the instrument back and is for furnace cool-down air (shown in Figure 2.7).

The **rear panel** (Figure 4.5) has the signal, event, GPIB, gas switching accessory, and power connections for the instrument, as well as the fuses, the Ready light, the address dial, and the Reset button.

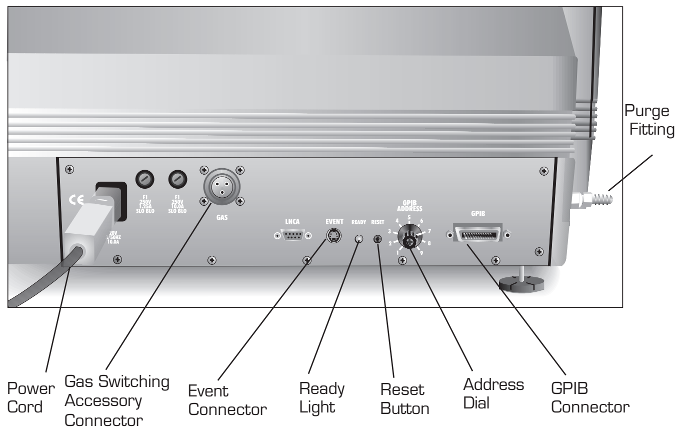


Figure 4.5
Rear Panel

Theory of Operation

The Simultaneous DSC-TGA instrument (SDT 2960) combines two thermal analysis techniques: (1) measuring the amount and rate of change in sample mass as a function of temperature and time—thermogravimetric analysis, or TGA—and (2) measuring the heat flow differences (enthalpic changes) as a sample is uniformly heated as a function of temperature and time—differential scanning calorimetry, or DSC.

The TGA analysis 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.

The DSC portion of the analysis works on the principle that when a sample and an inert reference are heated at a known rate in a controlled environment, the increase in the sample and reference temperature will be the same unless a heat-related change takes place in the sample. If this change takes place, the sample temperature either leads or lags the reference temperature (*i.e.*, the sample either evolves or absorbs heat). With a well defined heat flow, the (ΔT) which develops can be converted to heat flow using the thermal equivalent of Ohm's Law. This is the same "heat flux" approach widely used in most stand-alone DSC's.

Status Codes

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

Table 4.1
Method Status Codes

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

(table continued)

Table 4.1 Method Status Codes (cont'd)

Code	Meaning
Equilib	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 also shows 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. Press Start to continue the run.
Hot	The temperature is beyond the set point, and the instrument cannot remove heat fast enough to follow the thermal program. This is usually caused by a large ballistic jump to a lower temperature.

(table continued)

Table 4.1 (continued)

Code	Meaning
Initial	The temperature is being equilibrated to the desired set point. When the temperature has reached equilibrium, the status will change to "Ready."
Iso	The thermal program is holding the current temperature isothermally.
Jumping	The heater is jumping ballistically to the set point temperature.
No Power	No power is being applied to the heater. Check that the heater switch is in the ON (1) position. (See "Heater Indicator Light" in Chapter 5.)
Opening	The furnace assembly is opening.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Press Start to continue the method.
Reject	The experiment has been terminated and the data erased.

(table continued)

Table 4.1 Method Status Codes (cont'd)

Code	Meaning
Repeat	The method is executing a repeat loop that does not involve temperature control segments.
Stand by	The method and method-end operations are complete.
Tare	The SDT is measuring the weight of the empty sample and reference cups. The reference cup can be empty or can contain a reference material during taring. 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 standby mode, and the experiment has been terminated.
Temp *	Temperature calibration is in effect. The heater is in standby mode, and the experiment has been terminated.
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 Furnace

To remove any contaminants that have accumulated inside the furnace, it is recommended that you clean the furnace after every 10 runs by heating it to approximately 1000°C in air at a ramp rate of 20°C/minute. Keep the time allowed for this cleaning to the minimum needed. Leaving the furnace at high temperatures for extended periods of time may shorten the life of your furnace.

! WARNING

If you are using oxygen as a purge gas, it may be necessary to clean the furnace tube more frequently to remove combustible hydrocarbon deposits.

Cleaning the Instrument

Keypad and Display

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

*Mylar is a registered trademark of the Du Pont Company.

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

Protective Cover (Bellows)

♦ ***CAUTION:*** **DO NOT REMOVE the protective cover at any time.**

Remove dust from the folds of the cover (see Figure 5.1 or 5.2) with compressed air. To clean up any spills onto the cover, wipe gently with a soft rag or paper towel moistened with water and mild soap.

Y-Adjustment

To enhance the DTA baseline between ambient and approximately 500°C, you may find it necessary to adjust the *y*-axis setscrew. The setscrew should be moved if the DTA signal changes direction twice before reaching a peak at approximately 500 to 800°C. In other words, the DTA signal must approach 500°C in one direction. The effect of the setscrew on delta (Δ) T is approximately 1°C per turn of the setscrew. Use the following guidelines to help you determine which way to turn the setscrew:

- If the ΔT has an initial positive slope, then reverses to a negative slope at 500°C, turn the setscrew **clockwise**.

- If the ΔT has an initial negative slope, then reverses to a positive slope at 500°C, turn the setscrew **counterclockwise**.

Perform the procedures described on the following pages to adjust the *y*-axis setscrew.

1. Use a flathead screwdriver to remove the *y*-adjustment sealing screw located on the balance assembly just below the arm rest (see Figure 5.1). This leaves the *y*-adjustment slot uncovered.

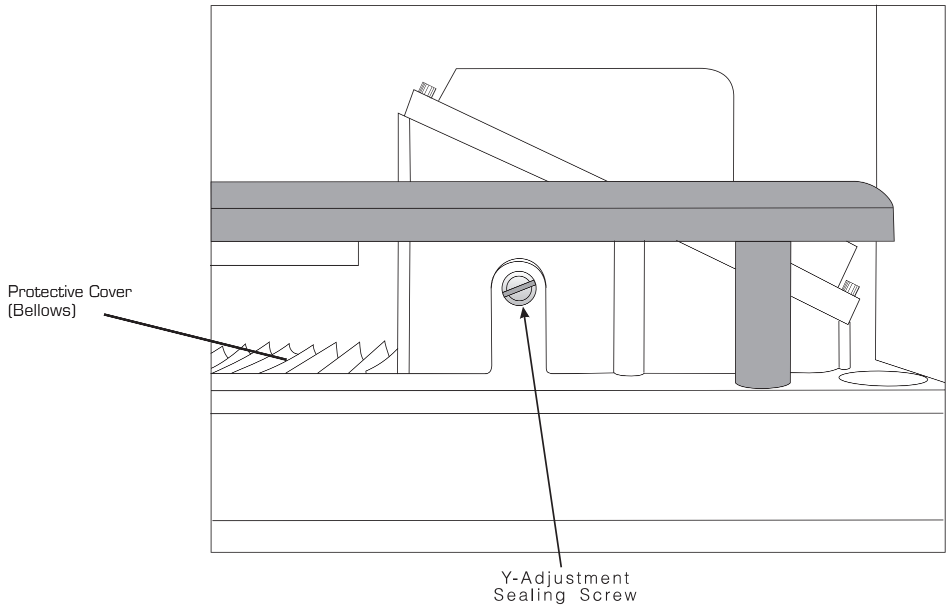


Figure 5.1
Location of the
Y-Adjustment Sealing Screw
(Y-adjustment Allen
screw is behind it)

2. Remove the balance dress cover, then remove the six screws holding the balance housing lid using a 3/32 Allen wrench. Follow the procedures described on pages 5-11 to 5-12 to remove the lid.

3. Loosen the two shoulder screws on the meter mount baseplate using a 3/32 Allen wrench (see Figure 5.2 on the next page for location of the shoulder screws).

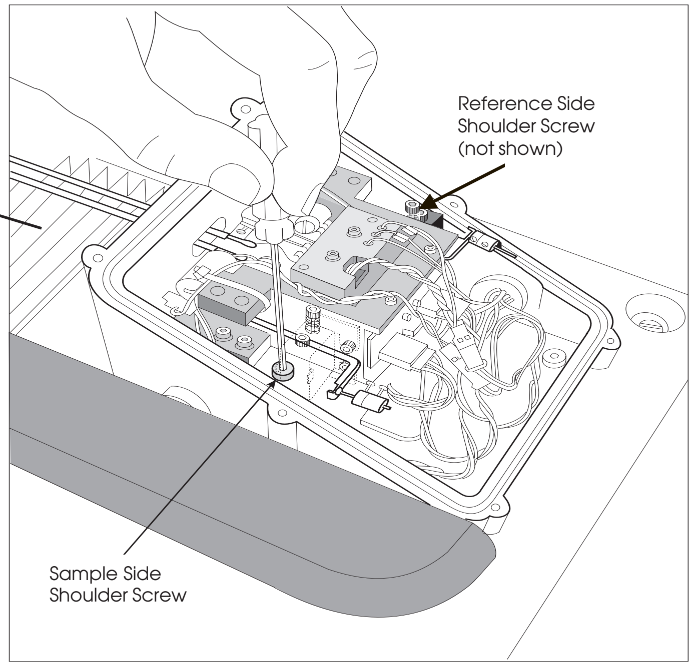


Figure 5.2
Moving the Meter Mount

4. Use a .050 Allen wrench on the *y*-adjustment Allen screw (see Figure 5.1 for location), and turn the wrench either clockwise to move the meter movement back or counterclockwise to move the meter movement towards the front.

5. Retighten the shoulder screws.

6. Replace the balance housing lid and the six screws that hold it in place.

7. Run a DTA-specific experiment, and check the baseline for acceptability.

8. Replace the *y*-adjustment sealing screw (see Figure 5.1) and the balance dress cover when the procedure is completed.

Repacking the SDT

If you need to pack up and move your SDT 2960 for any reason, you will need to take certain precautions to protect the instrument while it is in transit. The balance assembly and furnace must be placed in position and secured by the procedures described here.

Packing the Balance

♦ ***CAUTION:*** **When packing the balance, follow these procedures carefully to prevent damage to the two balance beams.**

To ship the SDT 2960, you must have the shipping foam, and some mechanical parts locked in position in order to protect the inner mechanisms. Follow these procedures to pack your instrument.

1. Using both hands, grasp the balance dress cover, and work it back and forth slightly as you pull it up. When you have enough clearance, place your fingers under the cover as shown in Figure 5.3. Then pull out on the right bottom edge of the dress cover to clear the balance housing lid as you slide the dress cover up and off.

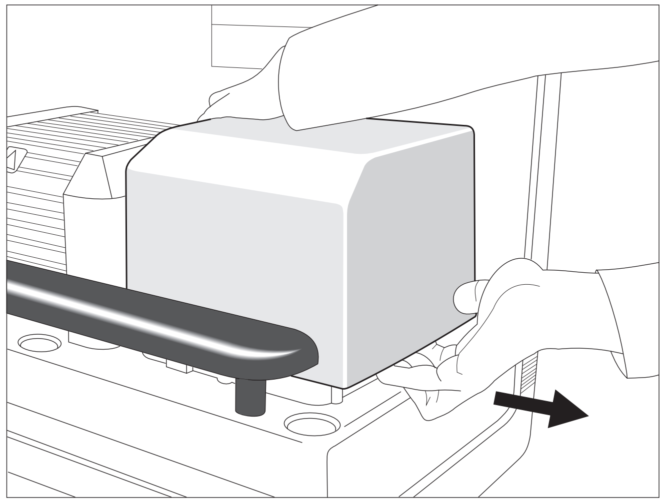


Figure 5.3
Removing the
Balance Dress Cover

2. Use a 3/32 Allen wrench to remove the six (6) screws holding the balance housing lid in place.

3. Place one hand on each side of the balance housing lid, and *carefully* lift the right side of the lid, as shown in Figure 5.4 on the next page.

   a. Pivot the right side of the lid upwards first to clear the inner mechanisms of the balance assembly, then lift up the left side of the lid and remove the entire lid.

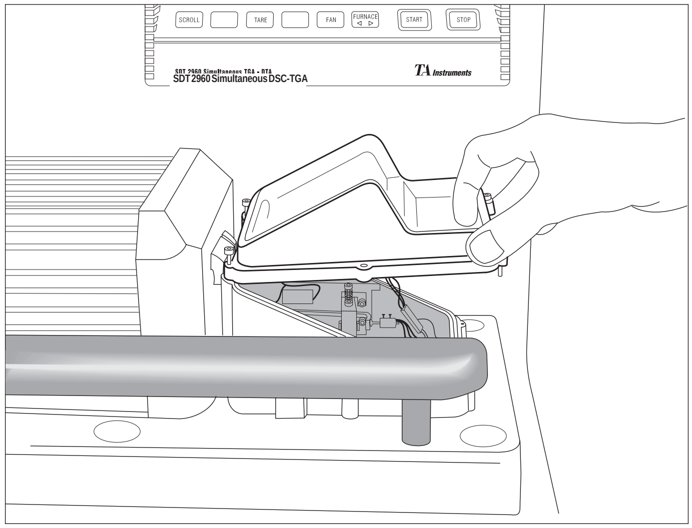


Figure 5.4
Removing the
Balance Housing Lid

4. Turn on the SDT POWER switch.

5. Press the FURNACE key on the instrument keypad to start the furnace in motion. Stop the furnace by pressing the FURNACE key again (or using the STOP key) when the furnace has opened 2 to 3 inches.

6. Locate the two screws on the front (sample) beam stop. You will use these to hold the tare beam in a locked position for shipment. Refer to Figure 5.5 on the next page.

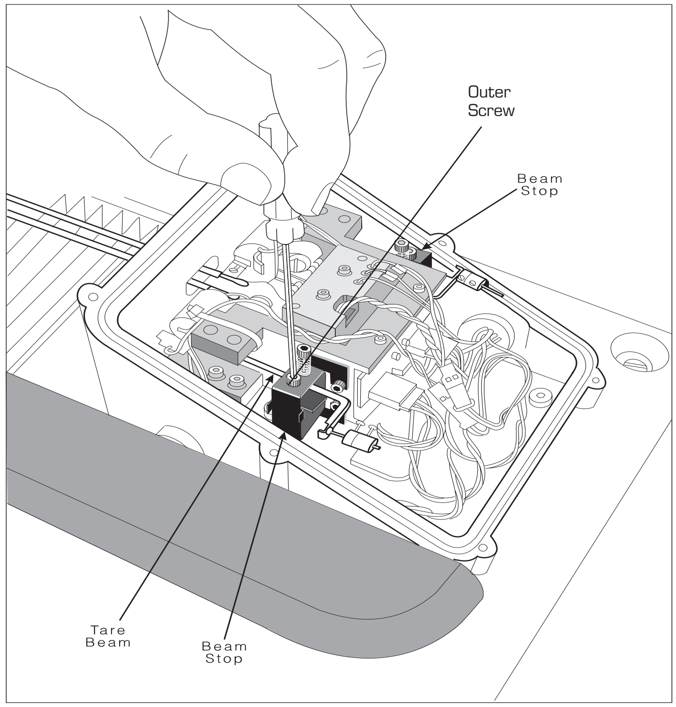


Figure 5.5
Locking the Tare Beam—Outer Screw

7. Press the SCROLL key on the keypad until the display indicates the sample weight.

8. Using a 3/32 Allen wrench, turn the outer screw (see Figure 5.5) clockwise to raise the tare beam stop until the rubber foam just barely touches the tare beam. This can be seen either by visually watching the tare beam or by watching the sample weight change until it is out of range.

9. Using a 3/32 Allen wrench, turn the inner screw clockwise until it makes the tare beam snug against the bracket. (See Figure 5.6).

♦ ***CAUTION:*** The screw should be tightened just enough to hold the beam securely, but not to damage or bend the beam.

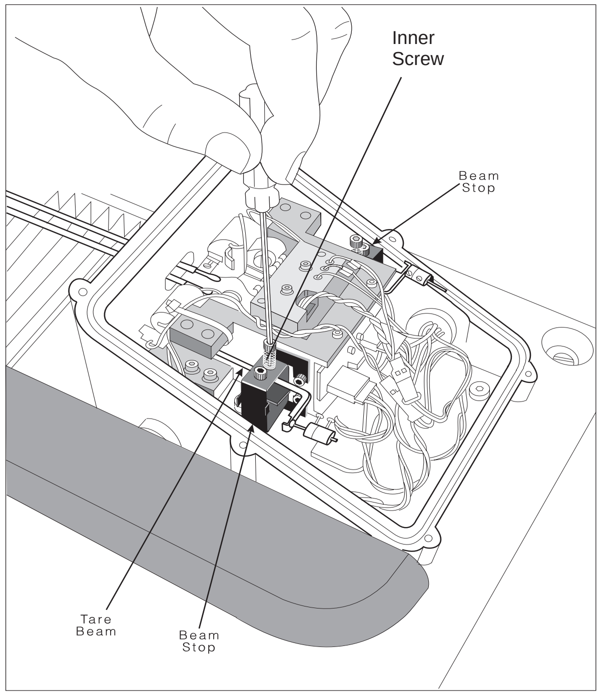


Figure 5.6 Locking the Tare Beam—Inner Screw

10. Repeat steps 7 through 9 for the back (reference) beam stop.

11. Carefully grasp the tops of the balance beam shipping foam with both hands, and separate the two top portions of the foam. Install the foam around the beams as shown in Figure 5.7.

Make sure the beams are still approximately centered in the furnace and that they will not be damaged when you close the furnace. If they are not centered, go back to step 7.

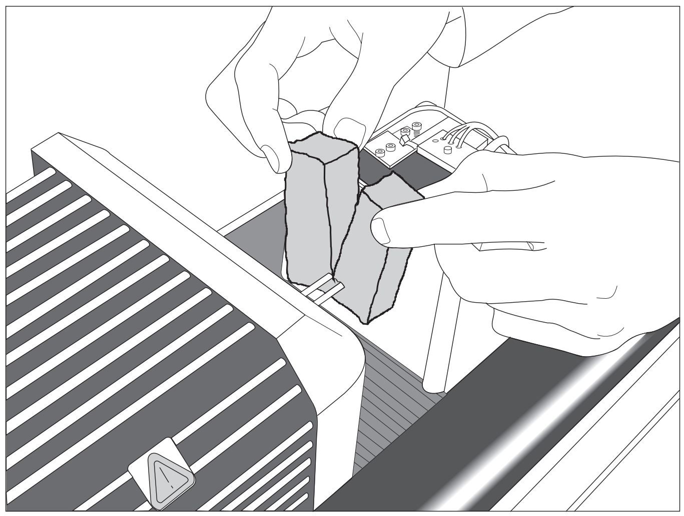


Figure 5.7 Replacing the Shipping Foam

12. Replace the balance housing lid, and reinstall the six (6) screws to hold it in place.

13. Press the SCROLL and FURNACE keys simultaneously to close the furnace on the foam. It is helpful to hold the top of the foam together as the furnace closes.

♦ ***CAUTION:*** **Be very careful not to pinch your fingers when the furnace closes!**

14. Replace the balance dress cover, then turn off the power to the SDT.

15. Locate the metal L-shaped shipping bracket (removed when balance was unpacked).

16. Push the rubber protective cover (bellows) to the right under the furnace assembly, exposing the screw located in the slide rail.

17. Using a flathead screwdriver, remove three screws: one from the slide rail and two from the left side of the furnace (refer to Figure 2.6 for their locations).

18. Position the metal bracket as shown in Figure 5.8 below. Pick up the three (3) screws you removed in step 17, and insert them through the bracket. Screw them in place with a flathead screwdriver.

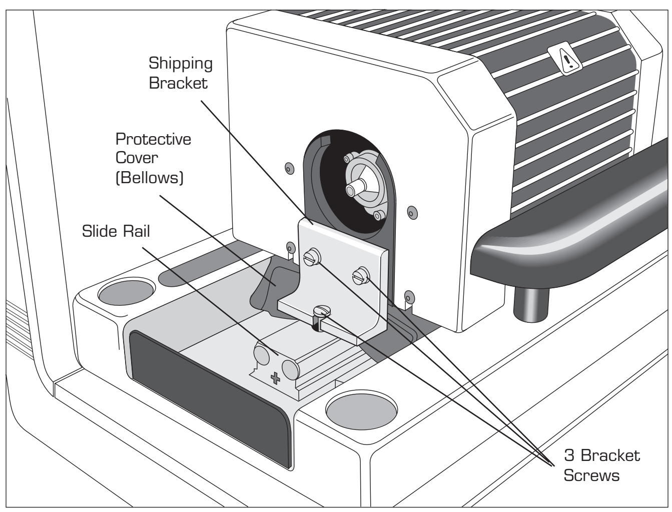


Figure 5.8 Replacing the Shipping Bracket

The SDT 2960 is now ready to pack up by reversing the unpacking instructions found in Chapter 2.

Diagnosing Power Problems

Fuses

The SDT contains several 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 SDT's rear panel. Both are housed in safety-approved fuse carriers, labeled F1 and F2 (see Figure 5.9). Replace these fuses with the same type and rating only.

! WARNING

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

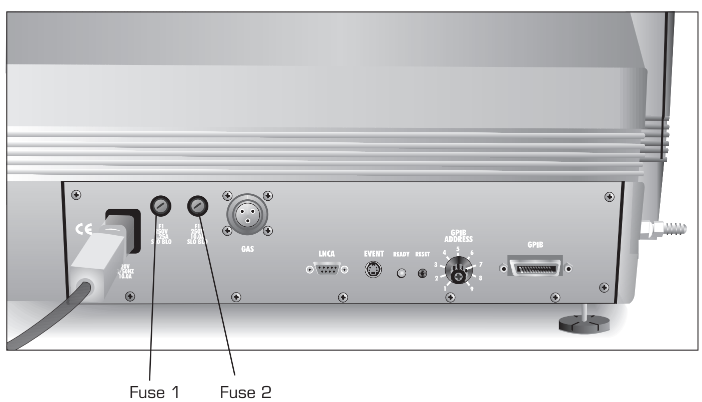


Figure 5.9
Fuse Locations

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. 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 instruments's front panel. TheHEATER switch must be ON (1) to start a method.

NOTE: The light in the HEATER switch will glow only after an experiment is initiated. 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.

Heater Indicator Light

The indicator light in the HEATER switch on the front panel of the SDT 2960 glows whenever the power control circuitry is enabled. If the light does not come on when the method is started, the indicator light may be defective or a hardware problem may exist in the SDT 2960 (call your TA Instruments Service Representative).

The heater light may also remain on after a method has terminated. This can happen if the method-end condition "Return to temperature range" function is chosen; see the *Thermal SolutionsUser Reference Guide* for further details.

Pressing the instrument STOP key after the completion of a method manually overrides the post-experiment heater power conditions.

Power Failures

A power failure caused by a temporary drop in line voltage results in one of two responses by the SDT 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 SDT’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 SDT is designed for a nominal line voltage of 120 volts AC ($\pm$ 10%), 50 or 60 Hz. It should not be operated outside this range. Low line voltage may result in poor instrument operation; high line voltage may damage the instrument.

SDT 2960 Test Functions

The SDT 2960 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 tests and drift runs.

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

The Confidence Test

The SDT 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 SDT 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 the error is nonfatal, the error message is displayed so that you can note the error and decide whether you need to correct it before performing an experiment. After 3 seconds, the error code disappears, and the confidence test continues. If no fatal errors are detected during the rest of the test, the SDT opening display appears, and the Ready light on the rear panel glows.

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. If the error is fatal, the confidence test is halted at that point, and the error code remains on the display. Contact your TA Instruments service representative if you see a fatal error code.

Table 5.1 summarizes the primary confidence tests for the SDT.

Table 5.1
SDT 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

Table 5.2
List of SDT Parts

Part Number	Description
992132.902	Gas Switching Accessory
270134.001	Flowmeter
270134.002	Flowmeter, dual tube
960020.901	Furnace assembly (includes ceramic furnace tube)
960017.901	SDT Dual Beam Kit
960148.901	Platinum sample cups, 40 µL; pkg of 3 (for TGA-DTA studies)
960149.901	Platinum sample cups, 110 µL; pkg of 3 (for TGA-DTA studies)
960072.901	Alumina sample cups, 40 µL; pkg of 3 (for TGA-DTA studies)
960070.901	Alumina sample cups, 90 µL; pkg of 3 (for DSC-TGA studies)
960239.901	Alumina sample lids; pkg of 3 (for DSC-TGA studies)
915079.903	Sapphire DSC Heat Flow Calibration Standard
960034.901	Aluminum oxide reference material (for TGA-DTA studies)

(table continued)

Table 5.2 (cont'd)

Part Number	Description
900905.901	Calcium oxalate
952183.901	Vial of aluminum wire for temperature calibration
960014.901	Calibration weights, set
259508.000	Brass tweezers
270226.022	O-ring, furnace tube
925605.001	SDT 2960 Operator's Manual
890035.901	Power cord
270711.001	GPIB cable
890014.901	Gas switching cable
205220.021	Fuse, 1.25 amp, 250V, 1/4 in., cer, slow
205220.041	Fuse, 12 amp, 250V, 1/4 in. x 1 1/4 in., cer, slow

Appendix A: Ordering Information

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.

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Leatherhead, Surrey KT22 7UQ
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Fax: 44-1372-360135

TA Instruments GmbH
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Fax: 49-6023-9647-77

TA Instruments Benelux
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Telephone: 32-9-220-79-89
Fax: 32-9-220-83-21

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