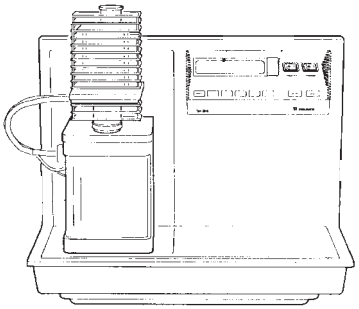
TA Instruments

Thermal Analysis & Rheology

A Subsidiary of Waters Corporation



TMA 2940

Thermomechanical Analyzer

Operator's Manual

PN 925615.001 Rev. D (Text and Binder)
PN 925615.002 Rev. D (Text Only)
Issued November 1999

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 validity and suitability must be obtained from TA Instruments, Inc. Otherwise, TA Instruments does not guarantee results and assumes no obligation or liability. This publication is not a license to operate under nor 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 under the guidelines described below:

NOTE: Highlights important information about equipment or procedures.

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

Marks a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Safety

Instrument Symbols

The following labels are displayed on the TMA 2940 instrument for your protection:

Symbol	Explanation
	This symbol, which appears on the furnace enclosure and on the stage can of the TMA 2940, 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 the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *TMA 2940 Operator's Manual* contains cautions and warnings that must be followed for your own safety.

Electrical Safety

Voltages exceeding 110 Vac are present in this system. Always unplug the instrument before performing any maintenance.

! WARNING

Because of the high voltages in this instrument, untrained personnel must not remove the cabinet cover unless specifically directed to do so in the manual. Maintenance and repair of internal parts must be performed by TA Instruments, Inc. or other qualified service personnel only.

! 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 2-11 for the method of drying out the equipment before use.

Handling Liquid Nitrogen

The TMA 2940 can use the cryogenic (low-temperature) agent, liquid nitrogen, for cooling in subambient experiments. Because of its low temperature (-195°C), liquid nitrogen will burn the skin. Use extreme caution when working with liquid nitrogen and other cryogenic materials to ensure that you do not burn yourself.

Personnel working with liquid nitrogen should take the following precautions.

! WARNING

Liquid nitrogen evaporates rapidly at room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent depletion of oxygen in the air.

1. Wear goggles or a face shield, gloves that are large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the shoe tops.

2. Transfer the liquid slowly to prevent thermal shock.

3. Use containers having adequate low-temperature properties. Ensure that closed containers have vents to relieve pressure; liquid nitrogen evaporates rapidly at room temperature.

4. The purity of liquid nitrogen alters as it evaporates. If much of the liquid in the container has evaporated, check the remaining liquid before using it for any purpose in which high oxygen content is dangerous.

IF BURNED BY LIQUID NITROGEN...

1. Flood the area (skin or eyes) with large quantities of cool water IMMEDIATELY; then apply cold compresses.

2. See a doctor IMMEDIATELY if the skin is blistered or if there is a chance of eye infection.

!WARNING

Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent LNCA container in confined spaces.

Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated.

The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use. Please refer to the "Safety" section of the *TA Instruments Liquid Nitrogen Cooling Accessory* manual for more detailed instructions regarding the use of the LNCA.

Lifting the Instrument

The TMA 2940 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	Introduces the TMA 2940 and lists its specifications.
CHAPTER 2	Describes how to install and assemble your TMA 2940.
CHAPTER 3	Describes routine operation of the TMA instrument, including subambient operations.
CHAPTER 4	Contains the procedures used to operate the TMA with the optional accessories.
CHAPTER 5	Explains the technical aspects of the TMA and its operation.
CHAPTER 6	Provides maintenance and diagnostic procedures.
Appendix A	Lists TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.
Index	Contains an alphabetical list of topics and page number references.

CHAPTER 1: Introducing the TMA 2940

Introduction

The Thermomechanical Analyzer (TMA) 2940 is an analytical instrument used to test the physical properties of many different materials.

The TMA 2940 works in conjunction with a TA Instruments controller and associated software to make up a themal 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.

Components

Your instrument consists of two major parts, the TMA cabinet and the TMA assembly (see Figure 1.1 on the next page). The following components make up the TMA assembly:

- The *balance enclosure* surrounds the TMA balance mechanism which exerts a specified force on the sample.

- The *probe assembly* is interchangeable for making several different measurements on various sample materials.

- The *stage* is an interchangeable component that supports the sample during measurement.

- The *furnace assembly* surrounds the stage to heat the sample; it contains the integral cooling container and the furnace monitor thermocouple.

- The *weight tray*, located behind the weight tray door, holds the weights to exert a known force on the sample.

- The Chromel/Alumel *sample thermocouple* senses the temperature of the sample and relays the reading to the instrument.

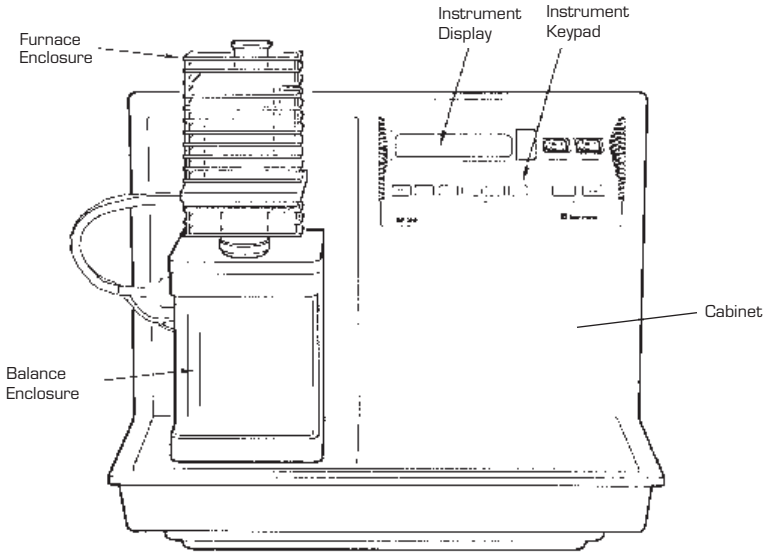

Figure 1.1
The TMA 2940
Instrument

The 2940 Instrument

The TMA 2940 instrument contains the electronics and software needed to perform experiments and store the results. The battery backed-up memory in the cabinet saves parameters vital to system operations if power is interrupted. Also contained in the cabinet is a GPIB interface for communication with the controller.

You can set up, start, stop, and reject the experiment using the keypad on the cabinet. The display provides valuable realtime information on the experiment in progress.

The TMA was developed by TA Instruments with the following features:

- operates over a temperature range of -150°C to 1000°C using heating rates up to 200°C/min
- determines changes in sample properties resulting from changes in four experimental variables: temperature, force, atmosphere, and time
- uses samples that can be in solid, film, fiber, or powder form
- employs interchangeable probes allowing you to measure the melting point, softening point, tensile modulus, compression modulus, glass transition, stress relaxation, creep, and expansion coefficient

- allows additional experiments in parallel plate rheometry, fiber tension, shrinkage force, flexure, and dilatometry with the optional accessories that can be used with the instrument.

The cabinet contains the keypad, used to control the mechanical movements of the instrument, and the display, used to observe the status of the instrument.

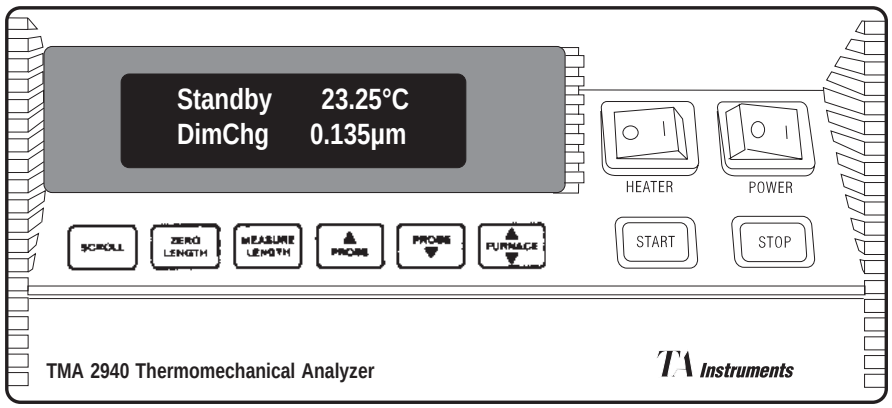

Figure 1.2
TMA 2940
Keypad and Display

2940 Display

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

During normal operation, the display is divided into three areas:

- Method status display—the eight-character line on the top left
- Sample temperature display—the nine characters on top right
- Realtime signal display—the whole bottom line.

2940 Keypad

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

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

Table 1.1
TMA 2940
Keypad Functions

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display.
ZERO LENGTH	Initializes the auto-measure system. This operation should be performed any time the probe or stage is changed.
MEASURE LENGTH	Measures your sample's length automatically; use this key *before* beginning the run. The measured length is transferred to the **Experimental Parameters** window and the value is recorded in the Sample Size field.
PROBE ▲	Used to "open" the probe, this key raises the probe 3 mm, the range of the LVDT, when pressed one time. When the key is pressed a second time, mechanical action occurs, moving the probe and LVDT coil to their maximum upward position.

(table continued)

Table 1.1 (continued)

Key/Function	Explanation
	If the probe is moving down and Probe ▲ is pushed, the probe will stop and reverse its direction.
PROBE ▼	Used to "close" the probe, this key lowers the probe 3 mm, the range of the LVDT, when pressed one time. When the key is pressed a second time, mechanical action occurs, centering the LVDT coil. If the probe is moving up and Probe ▼ is pushed, the probe will stop and reverse its direction.
▲ FURNACE ▼	Raises or lowers the furnace. You can stop the furnace movement by pressing this key a second time. If you press the key twice while the furnace is moving, you can halt and reverse the movement.
START	Initiates the experiment on the TMA after checking the program method against the mode type. This is the same function as **Start** on the controller.

(table continued)

Table 1.1
TMA 2940 Keypad Functions
(continued)

Key/Function	Explanation
STOP	If an experiment is running, this key ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions go into effect and the data that has been generated is saved. This is the same function as **Stop** on the controller. If an experiment is not running (the instrument is in a stand-by or method-end state), the STOP key will halt any activity (air cool, all mechanical motion, etc.).
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:	SCROLL operates normally (scrolls the text) until the STOP key is pressed. Then the display returns to the signal displayed on the screen before SCROLL was pressed. If an experiment is not running, SCROLL-STOP halts any activity as described for the STOP key.

HEATER Switch

The HEATER on/off switch turns the power to the instrument heater on and off (see Figure 1.2). The switch should be in the ON position before you start an experiment. If the HEATER switch is off, the run will not start.

NOTE: The light in the HEATER switch will glow only when an experiment is in progress or when the temperature is being controlled by a method-end condition.

POWER Switch

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

Accessories

The TMA 2940 can perform experiments on several different types of samples using both standard and optional accessories.

Standard Accessories

The accessory kit supplied with the TMA 2940 contains weights, sample holder (stage), a hex wrench, tweezers, samples for calibration, and standard probes.

The standard probes (see Figure 1.3) allow you to perform various basic analyses. These probes include the following:

- The *expansion probes* are used to measure the thermal coefficient of expansion and glass transition. The standard expansion probe is used for routine samples. The macro expansion probe covers a larger area of the sample surface and is therefore able to give a more representative reading for samples such as powders, materials with uneven surfaces, frozen liquids, and films.

- The *penetration probe* has a small tip that permits it to sink into the material as it is heated, and is used to measure softening and melting points.

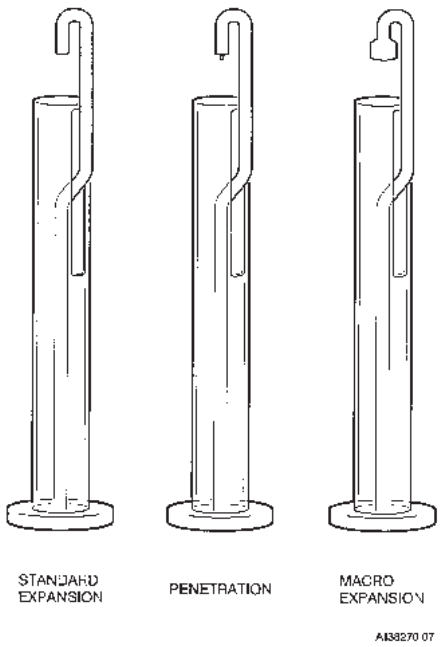

Figure 1.3
TMA Standard Probes

Optional Accessories

You can purchase extra items to use with your TA Instruments TMA. These optional accessories will allow you to perform experiments on different types of samples:

- Film/Fiber Accessory
- Dilatometer Accessory
- Parallel Plate Rheometer Accessory
- Flexure Accessory
- Hemispherical Probe.

Turn to Chapter 4, “Using Your Options,” for details on the installation and operation of these accessory kits.

Specifications

Tables 1.2 and 1.3 contain information about the TMA's specifications and temperature control.

Table 1.2 TMA 2940 Instrument Specifications

Dimensions	Depth 45.5 cm (18 in.) Width 58.5 cm (23 in.) Height 66 cm (26 in.)
Weight (approx.)	18 kg (60 lb)
Power	115 V ac 50/60 Hz 10 amps
Temperature Range	-150 to 1000°C
Sample Height	25 mm (1 inch) maximum
Sample Diameter	10 mm (0.39 inch) maximum
Sensitivity	100 nanometers
Displacement Range	±2.5 mm (±0.10 inch)
Linearity	±0.5%
Loading	0.001 to 1.0 Newtons (102 grams)
Atmosphere	Static or controlled flow with inert or reactive gases.

Table 1.3
Temperature Control

Programmed heating rate	0.01 to 200°C/min
Temperature reproducibility	±2°C

CHAPTER 2: Installing the TMA 2940

Unpacking/Repacking the 2940

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 TMA

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 this alone.**

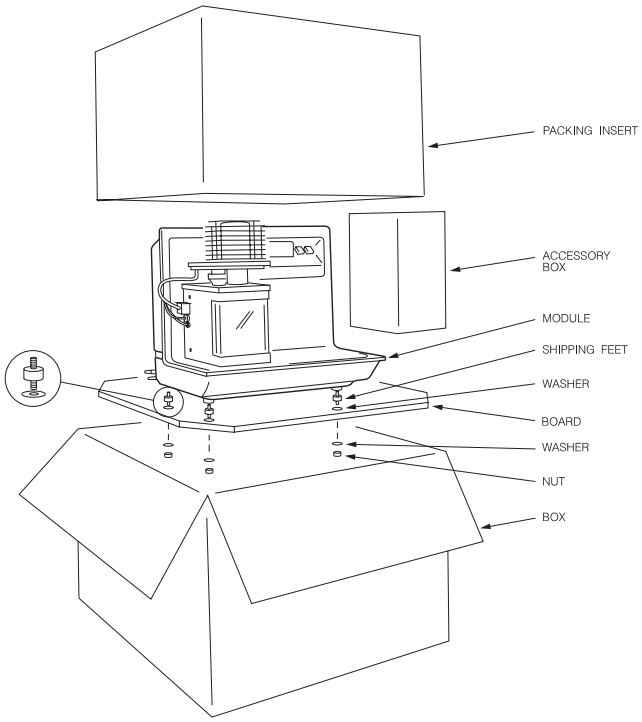

Figure 2.1
Shipping Boxes

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.**

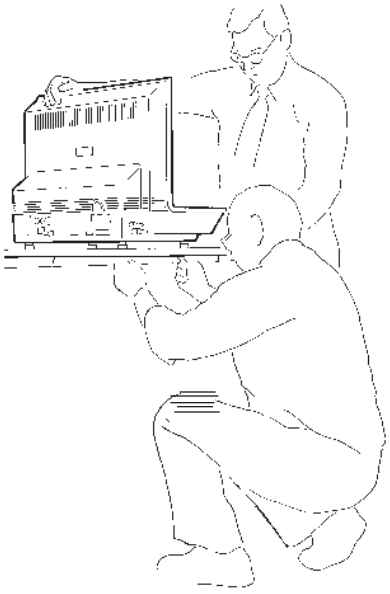

Figure 2.2 Removing the Plywood Board

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

7. Remove the ¼ x 20 screws and mounting feet from the accessory kit. Place the screw through the center of the rubber feet.

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

9. Have your assistant lift one side of the unit while you install two of the mounting feet (see Figure 2.3). Rotate the unit and install the two remaining mounting feet in the same manner.

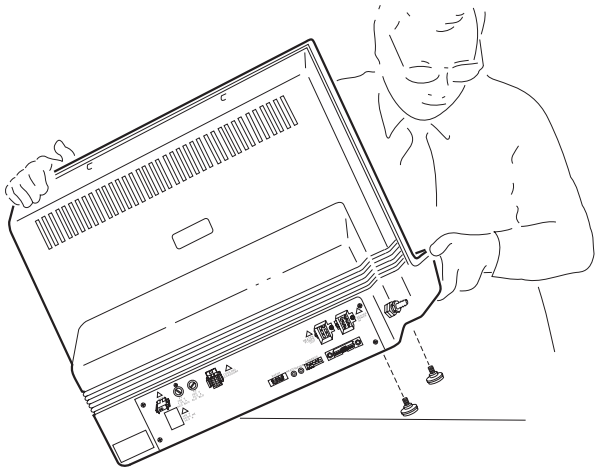

Figure 2.3 Installing the Mounting Feet

Removing the Shipping Material

The TMA 2940 has been specially packed to prevent damage to delicate parts. To unpack the instrument follow these steps:

♦ ***CAUTION:*** **Steps 1-9 may be completed by the customer. Only qualified service personnel should proceed with steps 10-19.**

1. Unscrew the black cap on the furnace post.
2. Slip off the cardboard cylinder, then replace the cap on the furnace post.
3. Raise the furnace and rotate it clockwise to move it off to the side.
4. Take the stage shield off and discard the packing foam.
5. Unscrew the large nut to remove.
6. Twist the stage retainer ring (with key slots) counter-clockwise and pull it up off the three posts.
7. Remove the shipping foam inside.
8. Get the hex wrench out of the accessory kit and remove the screws, two on each side, that hold the balance enclosure in place.
9. Slide the balance enclosure out and remove (see Figure 2.4).

! WARNING

Whenever you remove the balance enclosure, make sure that the power is off and the instrument is unplugged.

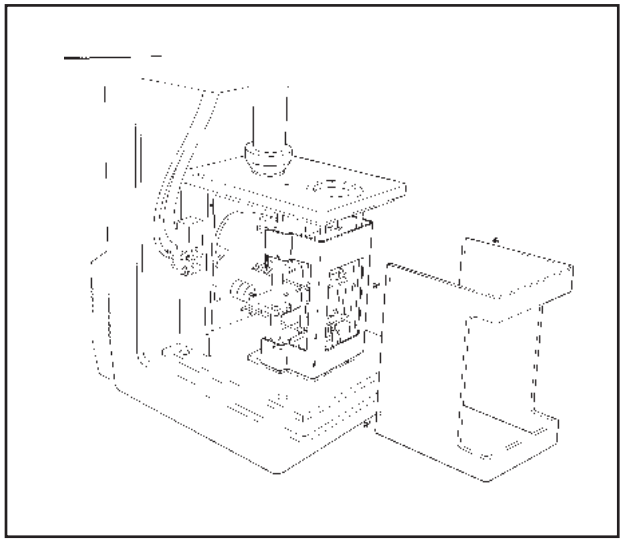

Figure 2.4 Removing the Balance Enclosure

♦ ***CAUTION:***

Only qualified service personnel should proceed beyond this point.

10. Using a 7/64" hex wrench, loosen and carefully remove the top and bottom clamping bars of the suspension shipping clamp.

11. Using the same wrench, remove the eight (8) screws from the front and rear clamping bars of the suspension shipping clamp. Carefully slide the clamping bars horizontally left or right to remove them.

12. Inspect the suspension components (wires and springs) for damage.

13. Remove the shipping foam at the rear of the balance assembly by pulling upward.

14. Locate the black plastic weight tray and 4-40 nylon screw which were packed in the top of the furnace. Using the nylon screw, install the weight tray into the front opening of the balance housing.

15. Verify the mechanical balance of the suspension assembly. Adjust if necessary by fine-tuning the positions of the weights.

16. Continue with installation of the thermocouple found on page 2-11.

17. Reinstall the balance enclosure and tighten the four screws.

18. Plug in and turn on the power to the TMA instrument. Allow it to warm up for at least 30 minutes.

19. Check the force calibration (see the instrument control online documentation for details).

Repacking the 2940

To pack and ship your instrument, use the hardware retained during unpacking and reverse the instructions found on pages 2-3 to 2-8.

Installing the Instrument

Before shipment the TMA 2940 instrument is inspected both electrically and mechanically, so that it is ready for operation after it has been installed. Installation involves the following procedures described in detail in this chapter:

- Unpacking the instrument components and accessory kit
- Inspecting the system for shipping damage and missing parts
- Assembling the TMA
- Connecting the TMA to the TA Instruments controller
- Connecting the purge gas and air line, accessories, and power cable.

If you wish to have your instrument installed by a TA Instruments Service Representative, call (302) 427-4000 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 TMA, 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 TMA experiments, it is important to choose a location for the instrument using the following guidelines:

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

On . . . a stable, vibration-free work surface.

Near . . . a power outlet (115 Vac, 50 or 60 Hz, 10 amps). A step up/down line transformer may be required if the unit is operated from a higher or lower line voltage.
. . . your TA Instruments thermal analysis controller.
. . . sources of compressed lab air and purge gas supply for use during cooling and subambient experiments.

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

NOTE:

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 (see page xiii) 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.

Installing the Thermocouple

Your TMA 2940 will arrive without a sample thermocouple installed. With the balance enclosure removed and the furnace raised, follow these steps:

1. Insert the two-pin, color-coded thermocouple connector into the fitting as shown in Figure 2.5.

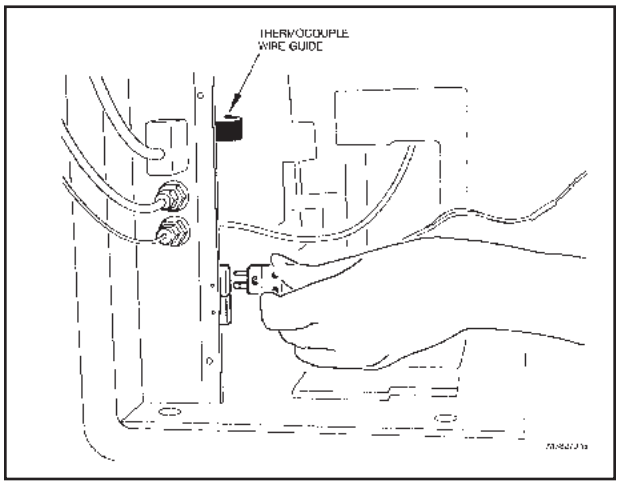

Figure 2.5 Plugging in the Thermocouple

2. Carefully thread the thermocouple, tip first, up through the hole in the top of the balance enclosure. (See Figure 2.6.)

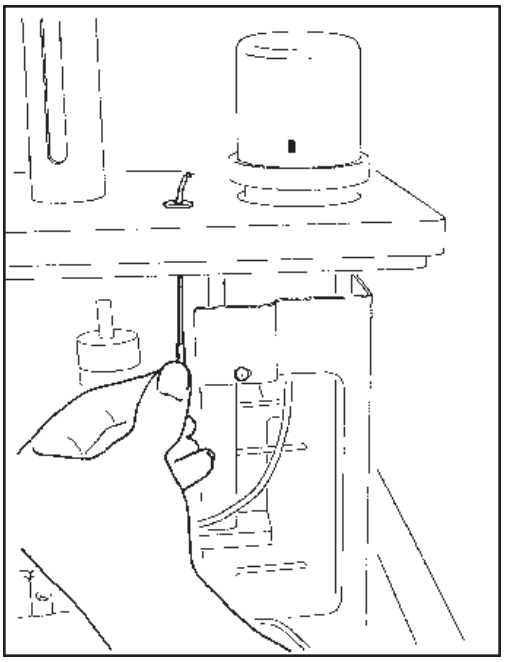

Figure 2.6 Threading the Thermocouple

3. Move the thermocouple off to the side of the stage platform.

4. Route the thermocouple wire through the wire guide to prevent interference with the balance mechanism.

5. Slide the balance enclosure into position.

6. Replace the screws, two on each side, that hold the balance enclosure in place.

7. Install the weight tray door as instructed in the next section.

Installing the Weight Tray Door

Locate the plastic weight tray door in the box with the accessory kit, wrapped in a sheet of shipping material. Remove the shipping material and follow these steps to install the door:

1. Locate the mounting holes on the top and bottom of the door.

2. Hold the door with these mounting holes to the left.

3. Put the lower hole over the small bottom post to the left of the weight tray.

4. Push down gently on the door as you guide the upper hole over the cone-shaped post at the top of the entrance.

5. Release the door and close it.

6. Install the stage as directed in the next section.

Installing the Stage

To install the stage on the TMA 2940, check to make sure the furnace is raised and off to the side, then follow these steps (refer to Figure 2.7 for illustration of the parts):

1. Remove the stage shield by lifting it straight up. (This is a friction fit.)

2. Turn the large stage nut counterclockwise to remove it.

3. Twist the stage retainer ring (with key slots) counterclockwise and pull it up off the three posts.

4. Remove the stage from the accessory kit.

5. Slide the wave washer (rippled edge washer) down over the top of the stage so that it fits on the flange.

6. Slide the stage retainer ring down over the top of the stage so that it rests on top of the wave washer.

7. Insert the whole assembly (stage, wave washer, and retainer ring) into the stage opening, lining up the key slots in the retainer ring with the posts.

8. Press down and turn the retainer ring clockwise to lock the assembly in position.

9. Replace the large stage nut, turning it clockwise to install it.

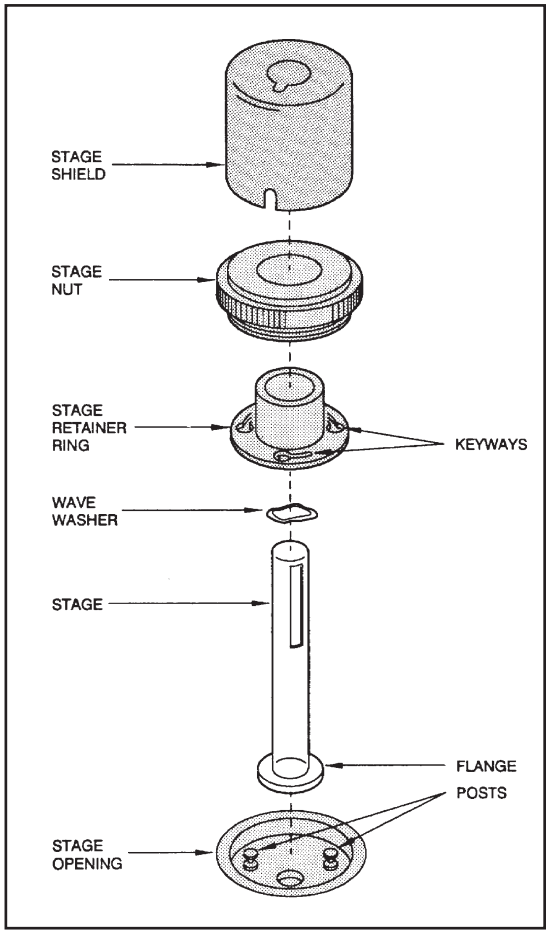

Figure 2.7
Exploded View
of TMA Stage Assembly

10. Attach the thermocouple to the stage as follows:

    a. Position the tip of the thermocouple so that it bends at a 90° angle and lies flat against the platform. It should be close to, but not touching the sample.

    b. Hold the thermocouple against the stage assembly and put on the spring clip to hold the thermocouple in place (see Figure 2.8).

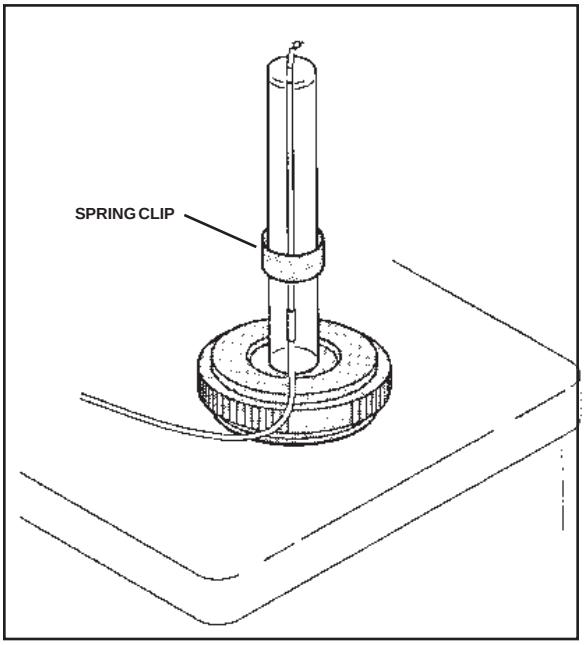

Figure 2.8 Attaching the Thermocouple to the Stage

11. Put the stage shield on the stage, aligning the slot in the bottom over the thermocouple wires (see Figure 2.9).

12. Rotate the furnace into position over the stage.

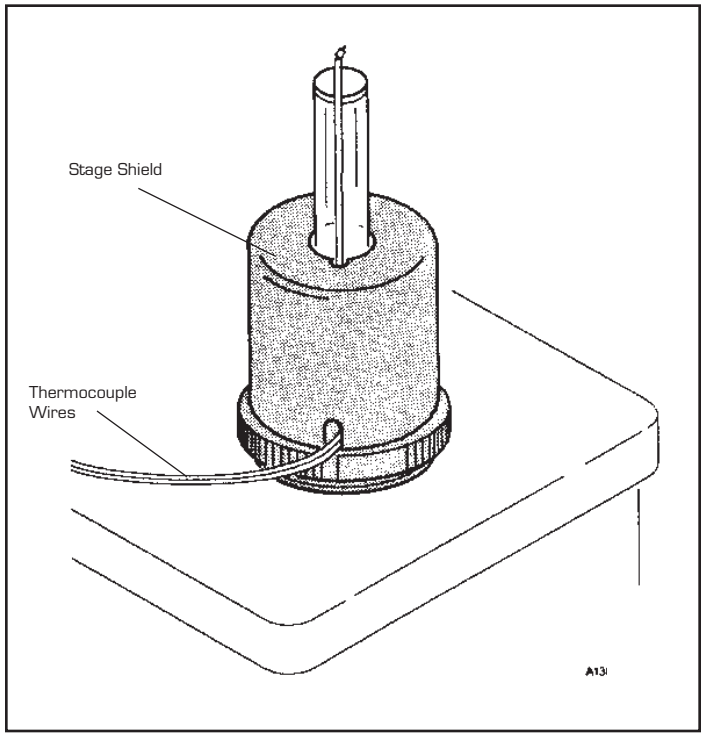

Figure 2.9 Assembled TMA with Stage and Thermocouple

13. Install one of the probes as directed in the next section. (Refer to Chapter 3 for guidelines in selecting a probe.)

Installing the Expansion/ Penetration Probes

When you first receive the TMA 2940, a probe will need to be installed. Later, if a different sample form is used, you can change to the appropriate probe for the experiment. (Refer to Chapter 3 for details on probe selection.) The procedures that follow explain the installation and removal of the expansion, macro expansion, penetration, flexure, dilatometer, and hemispherical probes.

NOTE: To learn how to install the film/fiber probe, turn to Chapter 4, "Using Your Options."

Installing a Probe

1. Raise the furnace and rotate it clockwise to move it off to the side.

2. Insert the core end of the probe carefully into the opening in the TMA stage (see Figure 2.10).

3. Loosen the probe-locking lever, which is the knurled post found behind the weight tray door, by turning it counterclockwise. Hold the probe-locking lever in the up position and continue lowering the probe into the stage until you can feel it seat in the locking mechanism.

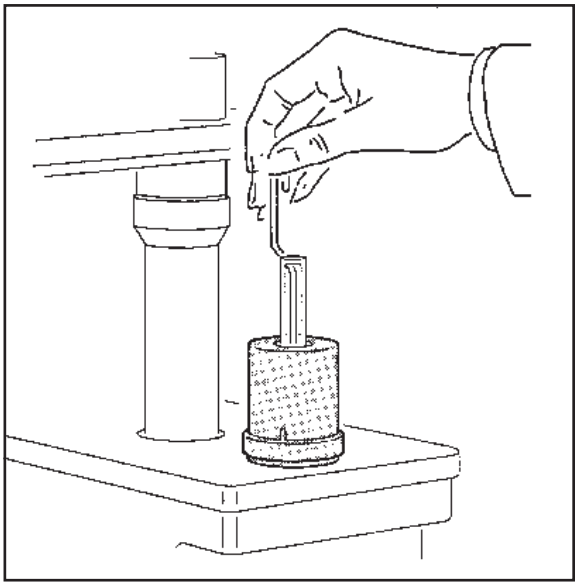

Figure 2.10
Installing the Probe

4. Tighten the probe-locking lever by turning it clockwise.

5. Calibrate the newly installed probe as directed in Chapter 3, “Running Experiments.”

Removing a Probe

1. Raise the furnace and rotate it clockwise to move it off to the side.

2. Grasp the top of the probe with one hand. Using the other hand, locate and hold the probe-locking lever, found behind the door that covers the weight tray.

3. Unscrew the locking lever by turning it counterclockwise approximately one turn.

4. Raise the probe gently and twist slightly to aid its removal from the stage opening.

Connecting Cables, Gas Lines, and Isolation Transformer

In order to connect the cables and gas lines, you will need to have access to the instrument's rear panel.*

NOTE: * 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:*** **When plugging or unplugging power cords, always 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 instrument (see Figure 2.11).
2. Connect the GPIB cable to the GPIB connector. The GPIB cable is the only cable that fits into this 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.

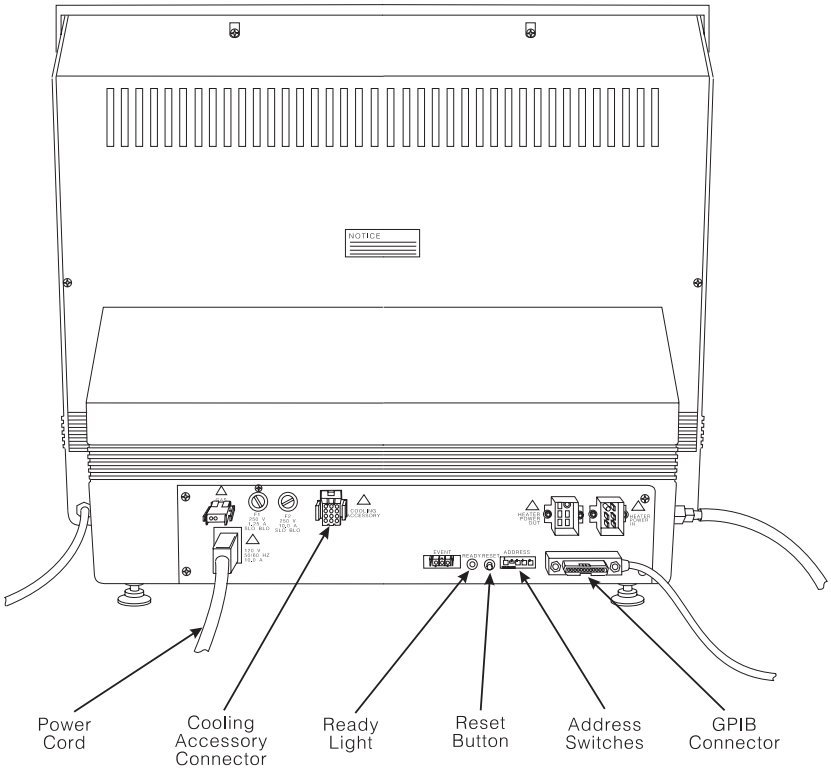

Figure 2.11
TMA Connector Panel

5. Select an address from 1 to 9. Use the binary address switches on the TMA connector panel to set the desired address (see Table 2.1). Figure 2.12 shows a instrument address of 7.

NOTE: If you have a multiple instruments system, each instrument must have a different address.

If you change the address after the TMA is powered on, you must press the TMA 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.

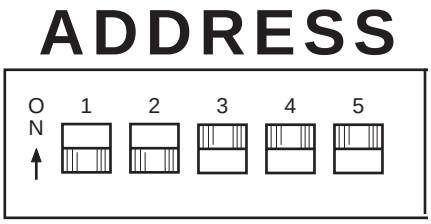

Figure 2.12
Binary Address Switches

Table 2.1
Binary Address Settings

Address	Switch Pattern
	1 2 3 4 5
1	0 0 0 0 1
2	0 0 0 1 0
3	0 0 0 1 1
4	0 0 1 0 0
5	0 0 1 0 1
6	0 0 1 1 0
7	0 0 1 1 1
8	0 1 0 0 1

* 0 = OFF; 1 = ON

Purge Line

1. Locate the PURGE fittings on the right side of the TMA instrument (see Figure 2.13).

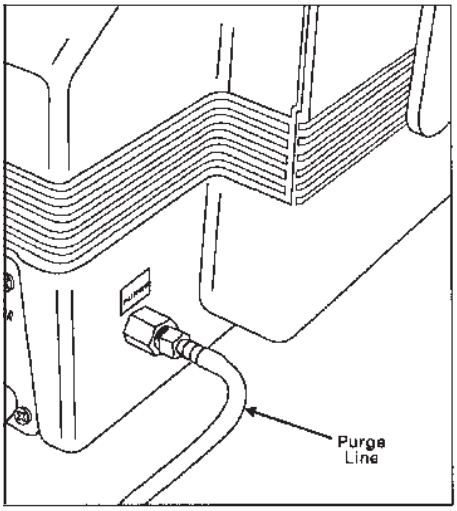

Figure 2.13
Purge Fitting

! WARNING

Use of an explosive gas as a purge gas is dangerous and is not recommended for the TMA instrument. For a list of the gases that can be used in the TMA, see Chapter 3.

♦ ***CAUTION:***

Use of corrosive gases will shorten the life of the instrument.

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

   The purge gas flows through the instrument and is channeled internally to the sample purge line shown in the figure on the next page.

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 (–100°C and above) and 200 mL/minute (–150°C and above).

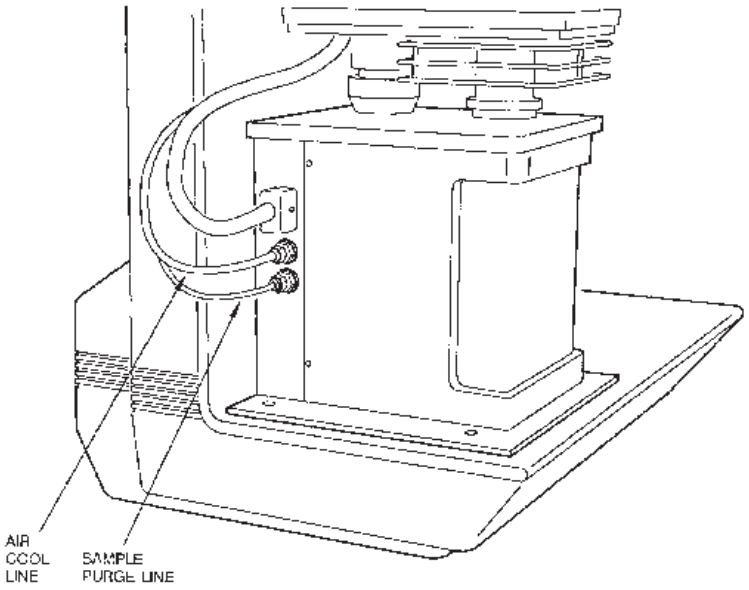

Figure 2.14
Air Cool and
Sample Purge Lines

Air Cool Line

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

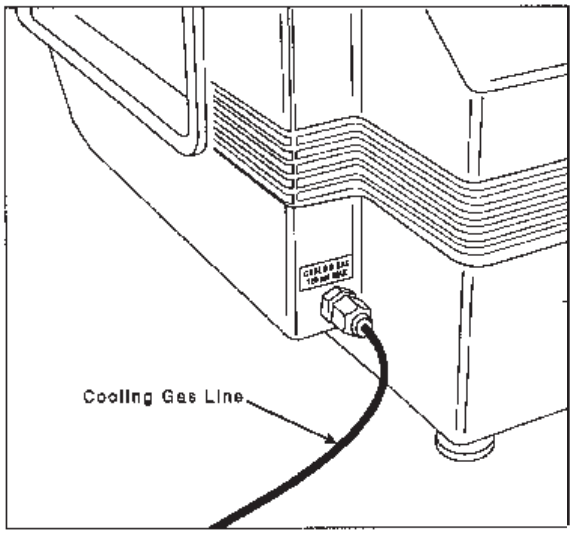

Figure 2.15 TMA COOLING GAS Fitting

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

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

Power Cable

NOTE: Connect all other cables and gas lines before connecting the power cable to a wall outlet.

1. Make sure the instrument POWER switch (see Figure 2.16) is in the OFF (O) position.

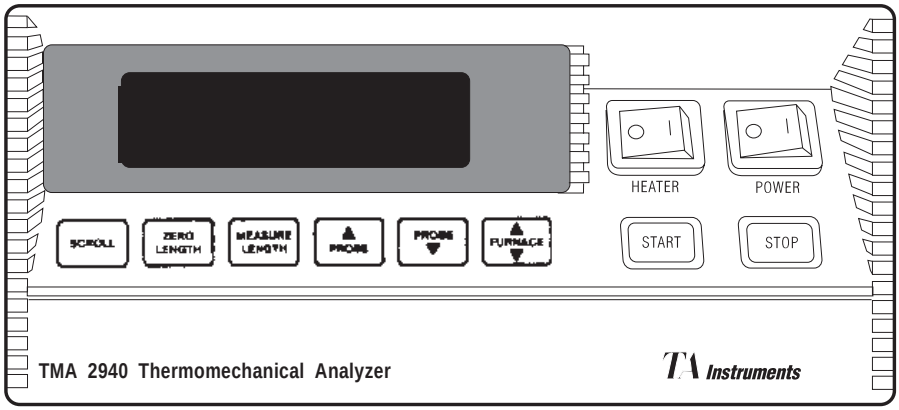

Figure 2.16
Front Panel of TMA
Showing POWER Switch

2. Plug the power cable into the TMA.

♦ ***CAUTION:*** **Before plugging the TMA 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.

Isolation Transformer

WARNING

The isolation transformer prevents any heater leakage current from reaching the instrument ground circuit. To eliminate a potential shock hazard, do not operate the TMA with the isolation transformer disconnected.

The isolation transformer must be connected to the back of your TMA. The number of cables that must be connected in sequence will depend upon the configuration of the connectors on the back of the module. All necessary cables will be provided, however, you may not need to use them all.

1. Place the isolation transformer near the heater power connections on the rear of the TMA cabinet.

2. Connect the isolation transformer to the heater power connections utilizing the appropriate cables provided. These cables accommodate different versions of the transformer and TMA cabinet and can only be connected one way for each combination. See the illustration on the next page.

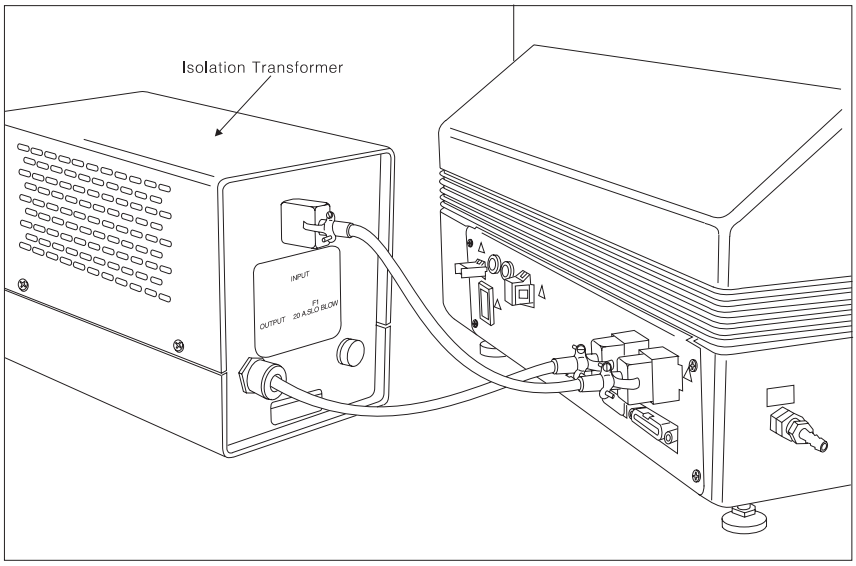

Figure 2.17
Isolation Transformer
Cable Connections

Starting the 2940

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

1. Check all connections between the TMA and the controller. Make sure each component is plugged into the correct connector, as described in this chapter.

2. Turn the instrument POWER switch to the ON (|) position. The instrument will run an internal confidence test each time you turn on the power.

3. Watch the instrument display during the confidence test for any messages that may be indicated. Confidence test definitions are given in Chapter 6.

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

4. Bring the instrument online with the TA controller.

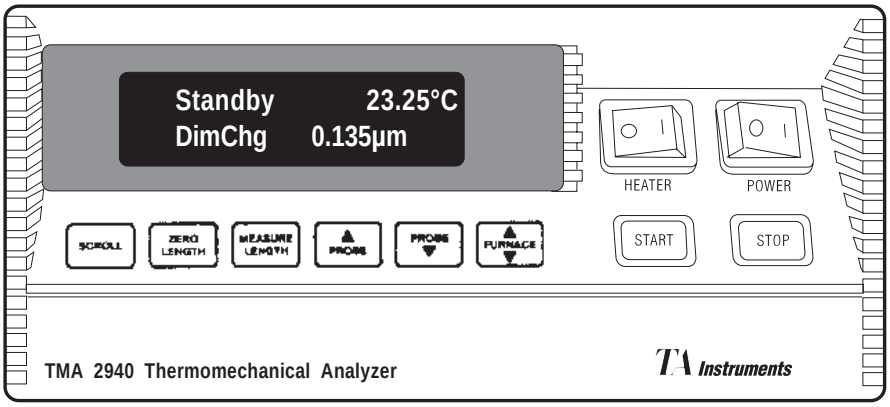

Figure 2.18
TMA Standby Display

Shutting Down the 2940

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 5 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 TMA experiments will have the following general outline. In some cases, not all of these steps will be performed.

- Selecting and calibrating the appropriate probe type
- 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)
- Zeroing the probe
- Preparing and loading the sample
- Adjusting the thermocouple position
- Measuring sample length
- Adding coolant to the furnace reservoir for subambient operation.
- Starting the experiment.

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

Before You Begin

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

- Made all necessary cable connections between the TMA 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
- Changed the instrument mode, if necessary
- Calibrated the instrument, if necessary.

Calibrating the TMA

To keep your TMA 2940 working to the highest level of performance possible, it is important that certain calibrations be performed on the instrument. Some procedures will need to be done on a regular basis and others only occasionally.

NOTE: Please note that this chapter provides guidelines on when to perform these calibrations, for details on how to perform each calibration procedure, refer to the *Thermal Solutions/Advantage User Reference Guide* or to the online documentation.

Force Calibration

Force calibration calibrates the force exerted by the probe on the sample during experiments using two weights and can be performed with any probe in place on the instrument. The first calibration point is 0 grams. It is suggested that the second calibration be performed with a 50 gram weight. You may use weights other than 50 grams (up to 100 grams is allowable). For example, you can use 10 grams for force calibration, if only low forces will be used in subsequent experiments.

When to Calibrate:

Perform the force calibration procedure in the following situations:

- When you first receive the instrument
- When you change software
- Periodically (approximately once a month).

NOTE: A force calibration performs the functions of a probe calibration (see the next section). Therefore, it is not necessary to perform a probe calibration immediately following a force calibration.

Probe Calibration

This procedure, sometimes called “initializing,” is used to calibrate zero force, the LVDT and the probe's compliance. This corrects for any difference in the different probes used on the TMA 2940 and should be done every time you change the instrument mode or when you change a probe on the TMA 2940. This calibration is performed from the TA controller.

Temperature Calibration

Temperature calibration is based on a run in which a temperature standard (*e.g.*, indium) is heated through its melting point. The recorded melting point of this standard is compared to the known melting point, and the difference is calculated for temperature calibration.

In addition, you can use up to four other standards to calibrate temperature. 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.

For all probe types, except the film/fiber accessory, small flattened pieces of standard metals are placed on the stage. To protect the stage from amalgamation with the metal, it is recommended that aluminum or platinum be placed between the stage and the metal standard. The end of the probe can also be wrapped with foil for added protection.

For the film/fiber probe, metal wires can be crimped into the aluminum balls and used for calibration.

When to Calibrate:

The sample thermocouple should be calibrated in the following situations:

- When the TMA is first installed

- When the sample thermocouple is changed
- When the TMA is serviced or repaired
- Periodically (approximately once a month)
- If you are changing the temperature range of interest
- If the run data obtained seems to be inaccurate.

NOTE:

Regardless of the size or shape of the sample that you are running on the TMA, position the tip of the thermocouple so that it bends at a 90° angle and lies flat against the platform. It should be close to, but not touching the sample.

Cell Constant

Cell constant calibration is based on a run in which a known sample (standard) is heated through its transition temperature and data is gathered for analysis. The cell constant is calculated by dividing the actual coefficient of expansion of the standard by the measured coefficient of expansion. The cell constant is then entered in the instrument control software for calibration of the instrument.

Using the default cell constant value of 1.000 is usually adequate; however, for greatest accuracy, calibration should be performed.

It is recommended that you follow the procedures of ASTM Standard Test Method E831 to perform the cell constant calibration. This method is summarized as follows.

1. Place your reference material (normally aluminum) on the stage (sample platform).

2. Using a 0.01 to 0.05 N force, with the standard expansion probe, measure the sample length.

3. Heat the sample from 20°C below the temperature range of interest to 20°C above the temperature range at 5°C/min heating rate.

4. Use a data analysis program to analyze the expansion of the aluminum over the temperature range of interest.

5. Calculate the cell constant using the following equation:

$$K_{cell} = \frac{\text{Expected (literature) expansion}}{\text{Observed expansion}}$$

Running a TMA Experiment

Experimental Procedure

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

- Zeroing the probe
- Selecting and preparing a sample. This involves preparing a sample of the appropriate size, and placing the sample in the sample stage.
- Entering experimental information through the TA controller.
- Creating and selecting the thermal method on the controller.
- Setting up any external accessories.
- Adding coolant to the furnace reservoir (for subambient operation).
- Starting the experiment.

Selecting a Probe

The type of probe that you use is dependent upon the kind of testing information desired. The table below lists the probes available, their specifications, and the type of testing yielded.

Table 3.1
TMA 2940 Probes

Probe Type	Contact Diameter mm (in.)	Pressure Exerted by 0.01 N Load	Types of Tests Yielded
Penetration	0.89 (0.035)	16 kPa	Softening point Melting point
Expansion	2.54 (0.100)	1.9 kPa	Expansion coefficient Compression modulus Tensile modulus Glass transition
Macro Expansion	6.07 (0.239)	0.34 kPa	Expansion coefficient Compression modulus Tensile modulus Glass transition

NOTE: Refer to Chapter 4, "Using Your Options," for details regarding the optional probes.

When choosing a probe to use for an experiment, follow these steps:

1. Select the appropriate probe for the analysis desired.

2. Check the instrument to see which probe is currently installed. If you change the probe currently on the TMA, turn to:

   - Chapter 2 to install the expansion and penetration probes.

   - Chapter 4 to install any optional probes.

3. Select the mode that is appropriate for the type of probe installed. Refer the the online documentation for information.

4. Calibrate a newly installed probe as described on page 3-5, or "zero" an already installed probe as described in the next section.

Zeroing the Auto Measure System

This procedure is used to initialize the auto-length measure system. It should be performed periodically to ensure accurate sample length measurements. To zero the TMA auto measure system, simply press the ZERO LENGTH key on the keypad.

Sample Preparation and Loading

Sample Preparation

Samples used for penetration and expansion studies should have the following characteristics:

- They should be as flat as possible, with parallel ends, to ensure stable placement on the stage.
- Samples should be long enough (5 to 10 mm for most materials) for adequate resolution, keeping in mind that large samples may experience temperature gradients during high heating rates.
- Thermoplastic samples can be heated and formed into suitable specimens and then cooled; however, this process may change important thermal history.

Sample Loading

After your sample has been prepared, follow these steps to load it on the TMA 2940:

1. Raise the furnace and rotate it clockwise to move it off to the side.
2. Remove any previously run samples from the stage and ensure that no residue remains.
3. Press ZERO LENGTH on the keypad to provide a zero reference point.
4. Open (raise) the probe. (Press PROBE ▲ key twice).

5. Place the sample on the stage under the probe tip (see Figure 3.1).

NOTE:

It is recommended that you place a quartz wafer or a piece of thin aluminum foil between the stage and any thermoplastic samples to prevent damage to the stage.

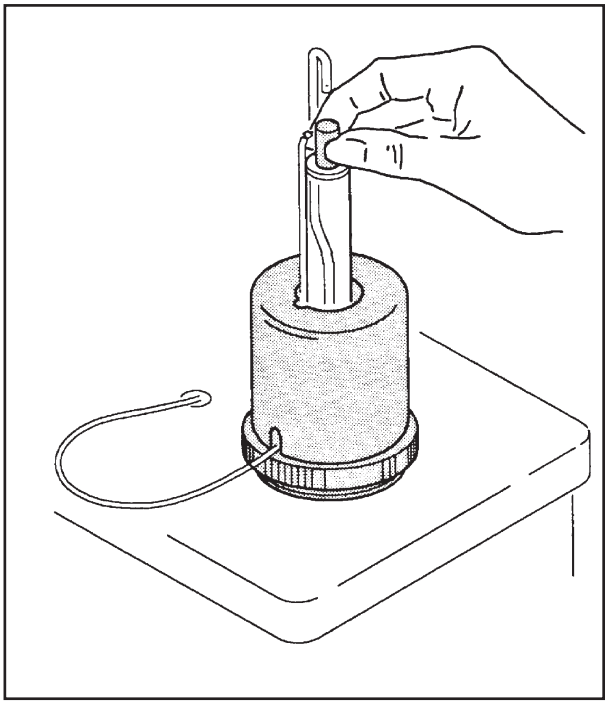

Figure 3.1
Loading a Sample

6. Adjust the sample thermocouple, if needed, as directed on the next page.

7. Program the force on the controller ,or add weight to the weight tray, if desired. Be sure to enter any static weight added to the weight tray on the instrument control program; see the online documentation for details.

NOTE: Low force levels may accentuate vibration-induced signal noise.

8. Measure the sample length by pressing the MEASURE LENGTH key.

9. Move the furnace back into position and close it.

Thermocouple Positioning

Follow these guidelines as you position the thermocouple on the stage with the sample:

Regardless of the size or shape of the sample that you are running on the TMA, position the tip of the thermocouple so that it bends at a 90° angle and lies flat against the platform. It should be close to, but not touching the sample (as shown here):

Measuring the Sample Length

Before you begin the experiment, it is important to take an initial measurement of the sample. To do this, simply press the MEASURE LENGTH key on the instrument keypad. This automatically measures and stores the sample length on the TA controller.

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 functions. Refer to the online 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 TMA 2940 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 TMA 2940:

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

Using Air Cool

Before you start an experiment that uses the air cool option or the "Return to temperature range" at method-end option, ensure that the supply valve from the air source is open and that the pressure is regulated to between 25 and 120 psig.

Using a Purge Gas

You can control the sample atmosphere during TMA experiments by connecting a purge gas to the system. You can purge the system using inert gases or reactive gases such as air and oxygen. The following gases can be used:

- Helium (recommended)
- Nitrogen
- Oxygen
- Dry, filtered air (to keep moisture, dirt, oil, and debris out of the system).

If an inert atmosphere is needed, nitrogen gas shoule be used. Close the furnace and purge the system with nitrogen for ten minutes. Then switch to helium gas for the experiment, if desired. To ensure an *inert* environment for your sample, a purge rate of 100 mL/min is suggested. Enter the purge gas and flow rate information using Instrument Control function, see the online documentation for details. The sample purge connection is located on the right side of the back of the TMA. See Chapter 2 for installation details.

Suggestions

The following guidelines are suggested when purging the system:

- Pass the purge gas through a dryer whenever you are working at subambient temperatures in order to remove any moisture in the gas itself.

- Because of its conductivity, helium is the purge gas recommended when performing TMA measurements at temperatures below ambient. Helium reduces the time required to cool the sample, provides a more stable sample environment, and reduces cold drafts on the system mechanism.

- Use the following helium purge rates for subambient operation:

  — 100 mL/min at -100°C and above
  — 200 mL/min at -150°C and above.

- If a denser purge gas than helium is used for subambient experiments, it is recommended that the silicon washer found in the accessory kit be attached to the probe. Raise the probe to the fully open position, then place the washer so it is just below the opening in the stage. This will minimize cold drafts into the lower balance enclosure.

- Let the purge gas flow for 3 to 5 minutes before adding coolant or starting the run; this allows a thorough exchange of sample atmosphere.

Starting an Experiment

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

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 TMA keypad or **Stop** on the controller. Another function that stops the experiment is Reject (SCROLL-STOP on the 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.**

Removing Samples

After the experiment has completed, allow the sample to cool down so that you can touch it; then the sample can be removed as follows:

1. Raise the furnace and move it off to the side.
2. Open the probe.
3. Use tweezers to remove the sample from the stage. If the sample is stuck to the stage or probe, do not attempt to mechanically remove the material (*e.g.*, by scraping or pulling) as this may cause damage.

- To clean off polymeric material, carefully apply heat to the stage and probe area, removing the sample as it softens. Take care not to overheat mineral- or glass-filled polymers as this will cause the filler to stick to the stage or probe.

- To remove metallic materials (*e.g.*, indium), immerse the probe and stage in an acid solution.

♦ ***CAUTION:*** **Use extreme caution when working with acid solutions.**

Heat is not recommended with metallic materials, as heat causes the metal to diffuse into the quartz and will change the properties of the stage and probe.

Subambient Experiments

The TMA can be used to run experiments on cooled samples with a cooling source such as liquid nitrogen. In order to perform subambient experiments the furnace must work against the coolant in the reservoir. Ideally, you should use the least amount of coolant necessary to keep the furnace drawing power over the entire subambient range.

NOTE: If the "Return to temperature range" method-end condition is activated in the instrument control program, press the STOP key to deactivate it before trying to cool the TMA.

Performing a Cooling Experiment

To run a subambient experiment perform the same operations as described in "Running a TMA Experiment" on pages 3-10 to 3-20. Once the sample is loaded and the furnace is lowered, conduct the following operations:

1. Turn on the purge gas at 100 mL/min and allow it to flow for 3 to 5 minutes to ensure a moisture-free environment.

2. Pour coolant carefully into the top of the furnace, refilling it as needed in order to cool the sample below the desired temperature.

♦ ***CAUTION:*** If using liquid nitrogen as a coolant, be sure to follow all of the safety precautions listed in the front of this manual for handling liquid nitrogen.

3. Start the run.

NOTE: Adjusting the coolant during the course of the run may cause nonlinear heating rates, which will be reflected in noisy or discontinuous curves.

Techniques for Improved Subambient Operation

In order to perform subambient experiments, the furnace must work against the coolant in the furnace reservoir. Ideally, you should use the least amount of coolant necessary to keep the furnace drawing power over the entire subambient range.

To improve the accuracy of your cooling experiments, the following guidelines have been suggested:

- Pass the purge gas through a dryer and use impermeable tubing whenever you are working at subambient temperatures in order to remove any moisture in the gas itself.

Between -20 and +20°C, false transitions may occur if moisture is present in the sample environment.

- Because of its conductivity, helium is the purge gas recommended when performing TMA measurements at temperatures below ambient. Helium reduces the time required to cool the sample, provides a more stable sample environment, and reduces cold drafts on the system mechanism.

- Use the following helium purge rates for subambient operation:

  — 100 mL/min at -100°C and above
  — 200 mL/min at -150°C and above.

- If a denser purge gas than helium is used for subambient experiments, it is recommended that the silicon washer found in the accessory kit be attached to the probe. Raise the probe to the fully open position, then place the washer so it is just below the opening in the stage. This will minimize cold drafts into the lower balance enclosure.

- If slow heating rates or isothermal holds are desired at subambient temperatures, it is recommended that the aluminum mass found in the accessory kit be inserted into the furnace reservoir. This adds a more stable cooling mass for the furnace to work against.

♦ ***CAUTION:*** **Do not program furnace temperatures greater than 600°C when using the aluminum thermal mass.**

CHAPTER 4: Using Your Options

Overview

This chapter explains the optional accessories that you can purchase to use on your TMA 2940, including the following information for each option:

- Description
- Operating procedures
- Accessory installation instructions
- Calibration
- Sample preparation.

Film/Fiber Accessory

Description

The TMA film/fiber accessory can be used to measure the physical properties of *fibers or films* as a function of force, temperature, or time.

Sample tensile stress is calculated using the following equation:

$$S_T = F / A$$

where:

S_T = stress at temperature T (Pascals)

F = force (Newtons)

A = cross-sectional area (meters^2).

The film/fiber accessory kit contains:

- Film/fiber probe
- Film/fiber stage
- Vial of cleaved aluminum balls
- Stainless steel clamps
- Clamp block
- Screwdriver
- Auto Measure gauge.

Operating Procedures

Film or fiber samples, held in tension during operation, are generally longer than expansion or penetration samples, therefore, the small temperature gradient normally present in the furnace is more significant. A faster heating rate serves to accent this gradient. For the best reproducibility, when creating a method, use a Ramp segment of 5°C/min or less for your heating rate. (See the *Thermal Solutions/ Advantage User Reference Guide* for details.)

NOTE: When comparing run data, make sure that all comparison scans are made with the thermocouple in the same position and at the same heating rate.

Accessory Installation

Because the configuration of the film/fiber accessory is different from the other types of probes, you will need to perform the following general steps to install the special film/fiber probe and stage:

- Remove the existing probe
- Remove the stage
- Change the instrument mode
- Install the film/fiber probe
- Install the film/fiber stage
- Calibrate the probe
- Calibrate temperature and cell constant.

Removing the Existing Probe

1. Raise the furnace and rotate it clockwise to move it off to the side.

2. Grasp the top of the probe with one hand and locate the probe-locking lever (behind the door that covers the weight tray) with your other hand.

3. Unscrew the locking lever by turning it counterclockwise approximately one turn.

4. Lift the probe gently and twist slightly to aid its removal from the stage opening.

Removing the Stage

1. Remove the stage shield from the base of the stage by lifting it straight up. (This is a friction fit.)

2. Take off the spring clip that holds the sample thermocouple in place.

3. Move the thermocouple off to the side of the stage.

4. Turn the stage nut counterclockwise to remove it.

5. Twist the stage retainer ring (with key slots) counterclockwise, pull it up off the three posts, and slide it up off the stage.

6. Take the wave washer up off the stage flange and remove it.

7. Remove the stage from the stage opening.

Changing the Instrument Mode

To learn how to change the instrument mode, refer to the *Thermal Solutions/Advantage User Reference Guide* for instructions.

♦ ***CAUTION:*** **The instrument must be placed into the film/fiber mode before installing the film/fiber accessory in order to prevent damage to the instrument or to the accessory.**

Installing the Film/ Fiber Probe

NOTE: The special configuration of the film/fiber accessory requires the installation of the probe before the stage.

1. Insert the core end of the probe carefully into the opening in the top plate.
2. Open the weight tray door and locate the probe-locking lever.
3. Loosen the probe-locking lever and hold it in the up position. Continue lowering the probe until you can feel it exerting downward pressure on the locking lever.
4. Align the slot in the top of the probe front to back and tighten the probe-locking lever by turning it clockwise.
5. Close the weight tray door.

Installing the Film/Fiber Stage

1. Lower the film/fiber stage down carefully over the probe onto the base.

2. Place the wave washer over the top of the stage and probe assembly and lower it onto the flange.

3. Slide the stage retainer ring carefully over the top of the stage and probe assembly so that it rests on top of the wave washer. Push down and turn clockwise to tighten the retainer ring.

4. Carefully align the stage slot with the slot in the top of the probe and position the stage opening to the front. (See Figure 4.1.)

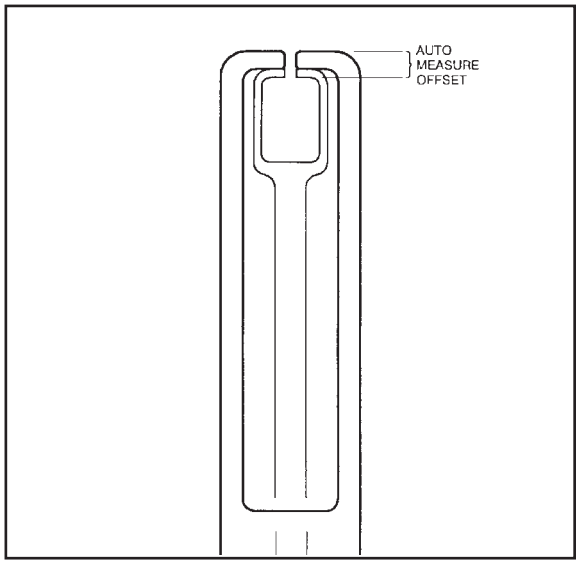

Figure 4.1
Film/Fiber Stage and Probe Assembly

5. Replace the large stage nut and turn it clockwise to insert it.

6. Attach the thermocouple to the stage as follows:

    a. Position the tip of the thermocouple approximately one half the length of the samples you will be measuring. The tip should be angled toward the sample location.

NOTE: After installing the thermocouple, tip placement may interfere with the zero length operation. Temporarily move the thermocouple when performing this operation.

    b. Hold the thermocouple against the stage assembly and put on the spring clip to hold the thermocouple in place.

7. Replace the stage shield on the base of the stage.

8. Move the furnace back into position and lower it. See Figures 4.4 and 4.7 for the correct probe setup with fiber and film samples.

Calculating the Offset

When you use the film/fiber accessory, analysis must compensate for the thickness of the stage platform and the upper portion of the probe. Perform the "Auto zero offset" procedure to compensate for the thickness. (See the *Thermal Solutions/Advantage User Reference Guide* for further instructions on performing this operation.)

The Auto Measure Offset will not need to be changed unless you:

- Reset the instrument parameters.
- Use a new film/fiber probe and stage assembly.

1. Record the value listed as Auto Measure Offset from the TA controller.
2. Use calipers to measure the gap in the Auto Measure Offset Gauge and record this value as the *Actual Size*.
3. Open the probe and insert the gauge as shown in Figure 4.2.

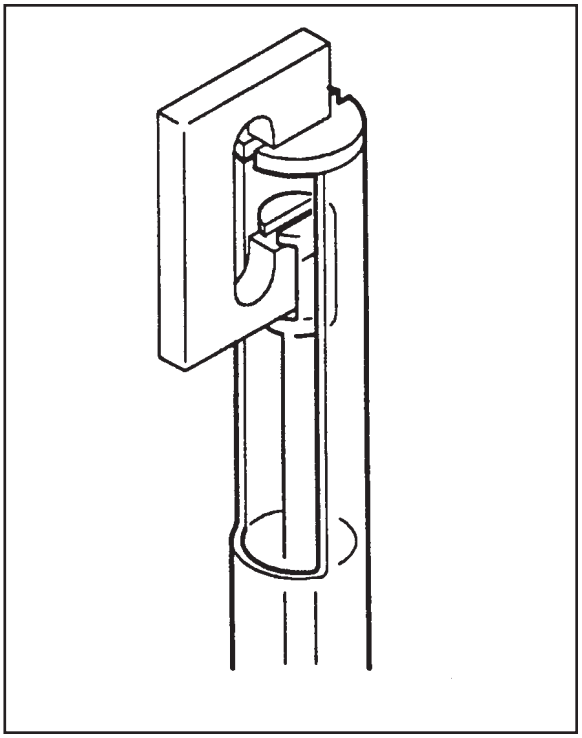

Figure 4.2
Inserting the
Auto Measure Gauge

4. Press the **Measure Length** key on the keypad. Record this value as the *Displayed Size.*

5. Calculate the new offset using the following equation:

$$Offset_{new} = (Actual\ Size - Displayed\ Size) + Offset_{current}$$

6. Enter the new Auto Measure Offset using the *Thermal Solutions/Advantage* Instrument Control functions.

7. Remove the Auto Measure Gauge and proceed with your experiment.

Calibration

You should perform probe, temperature, and cell constant calibration for the film/fiber accessory as you would normally calibrate any of the standard probes. Metal wires crimped in the aluminum balls should be used for temperature calibration. High quality aluminum foil clamped in the steel clamps should be used for cell constant calibration. See the *Thermal Solutions/Advantage User Reference Guide* for details.

Sample Preparation

The method used to prepare and load your sample varies according to the sample type. There are two methods available for the film/ fiber accessory: aluminum balls or stainless steel clamps.

Using the Aluminum Balls

The aluminum balls are generally used with fiber samples as follows:

1. Measure and cut the fiber sample slightly longer than desired length, typically 12 to 25 mm (0.5 to 1.0 inch).

2. Place two cleaved aluminum balls 12 to 25 mm (0.5 to 1.0 inch) apart.

3. Lay the fiber across the balls and press the fiber into the cleavage of each ball (see Figure 4.3).

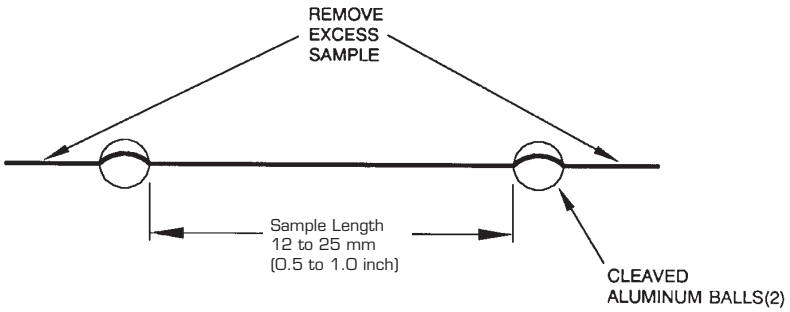

Figure 4.3
Fiber Sample Preparation

4. Crimp the two balls closed. The balls should not shift or slip when tested by hand.

5. Cut off any excess fiber extending beyond the aluminum balls.

6. Place one ball over the slot in the stage with the fiber hanging down through the slot and allow the other ball to hang freely.

7. Position the second ball below the slot in the film/fiber probe so that the fiber is suspended between the two balls as shown in Figure 4.4.

8. Measure the sample length.

9. Position the thermocouple close to, but not touching the sample.

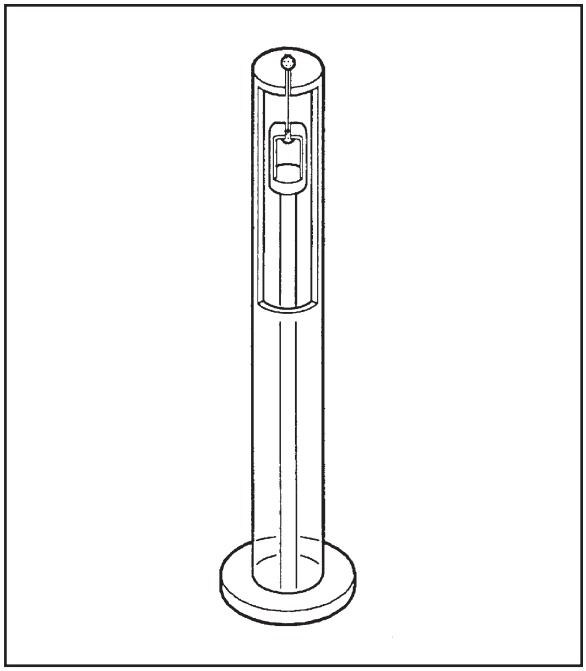

Figure 4.4
Probe Setup
Using Fiber Samples

Using the Stainless Steel Clamps

The stainless steel clamps may be used with either film or fiber samples as follows:

1. Measure and cut the sample to the desired length, typically 12 to 25 mm (0.5 to 1.0 inch). (Films should have a uniform width.)

2. Separate the jaws of one clamp. Position the clamp so that the clamp relief notch opening is at the top of the sample, then slide one end of the sample into the clamp and tighten the screws. (See Figure 4.5.)

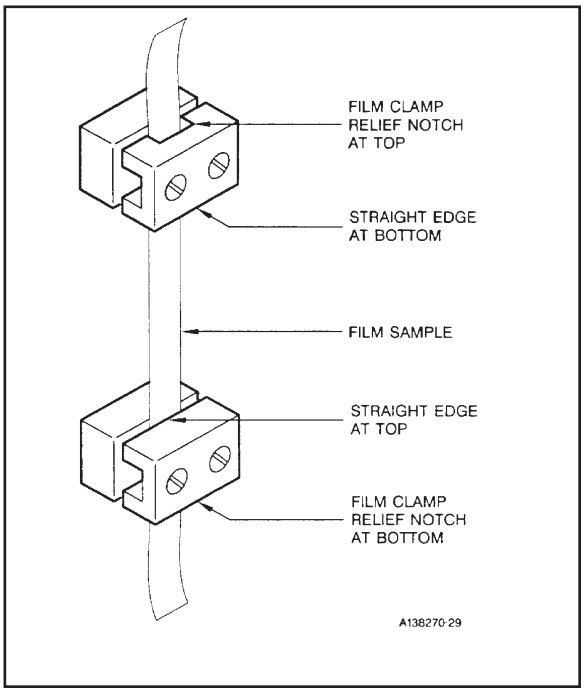

Figure 4.5
Stainless Steel Clamps and Sample

3. Lay the clamp and sample into the sample block so that the clamp fits into the notch on one end.

4. Lift up the free end of the film and slide the other clamp onto the end, positioning the clamp relief notch opening at the bottom of the sample (see Figure 4.5).

5. Move the clamp into the desired notch position (for either a 9, 13, or 25-mm sample).

6. Tighten the screws so that the sample is flat in the block and is positioned perpendicular to the clamps (see Figure 4.6).

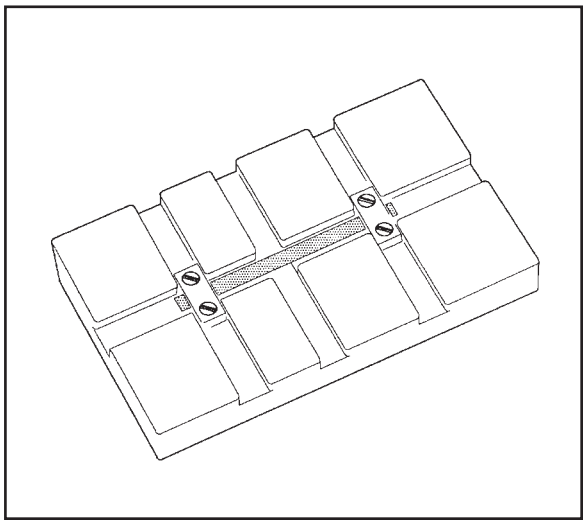

Figure 4.6 Film Sample Preparation

7. Remove the clamps and sample from the block and trim off the excess material.

8. Place one clamp on edge over the slot in the film/fiber stage so that the film hangs down into the slot.

9. Position the second clamp below the slot in the film/fiber probe so that the film is suspended between the two clamps as shown in Figure 4.7.

10. Measure the sample length.

11. Position the thermocouple close to, but not touching the sample.

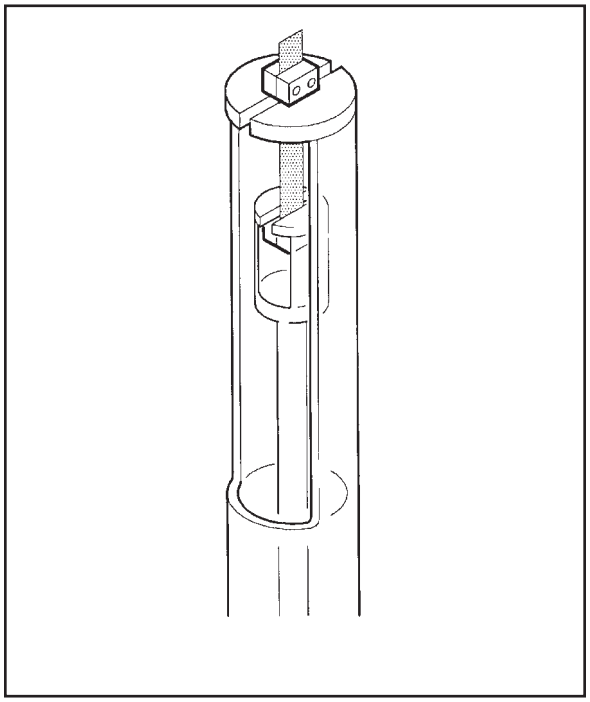

Figure 4.7
Probe Setup
Using Film Samples

Flexure Accessory

Description

The Flexure Accessory can be used on the TMA 2940 for *three-point bending studies* to measure the flexibility and strength of a variety of materials including: composites, plastics, PC boards, etc. It employs a knife-edged probe and a sample platform with two knife edges spaced 5.08 mm (0.2 inches) apart. This accessory permits the determination of deflection temperature under load.

Required loading is calculated using the following equation:

$$P = \frac{2\,Sbd^2}{3\,L}$$

Where:

P = calculated load (N)

S = maximum fiber stress (Pa) (1 psi = 6.895 x 10^3 Pa)

b = sample width (m)

d = sample depth (m)

L = width of span between the knife edge supports of the flexure platform (5.08 x 10^{-3} m).

The flexure accessory kit contains:

- Flexure probe assembly
- Flexure sample platform.

Accessory Installation

The flexure accessory uses a special flexure probe and sample platform, and the standard stage.

1. Remove the existing probe as instructed on page 2-19.

2. Install the standard stage, if it is not already in place. (For instructions to install the stage, refer to page 2-14).

3. Install the flexure probe in the same manner as the standard expansion and penetration probes as instructed on page 2-18.

4. Change the instrument mode, if necessary.

Calibration

The flexure probe should be calibrated as you would normally calibrate any of the standard probes. See the *Thermal Solutions/Advantage User Reference Guide* for details.

Sample Preparation

Samples used with the flexure accessory should have the following specifications:

- No wider than the flexure platform
- Uniform width and thickness
- Thick enough to be rigid when placed on flexure platform
- 7 to 10 mm long.

To load your flexure samples on the TMA 2940, follow these steps:

1. Raise the furnace and rotate it clockwise to move it off to the side.

2. Remove any previously run samples from the stage and ensure that no residue remains.

3. Open the probe.

4. Place the flexure platform on top of the stage

5. Place the sample on the flexure platform so that it is centered across the points.

6. Position the sample and platform on the stage under the flexure probe tip so that the probe knife edge is parallel to the platform points and is centered between them (see Figure 4.8).

7. Press ZERO LENGTH on the keypad to provide a zero reference point.

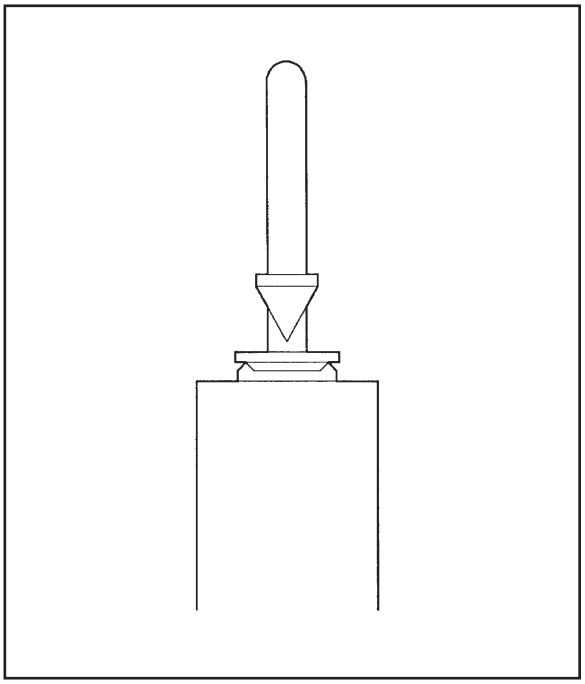

Figure 4.8
Probe Setup Using
Flexure Samples

8. Adjust the thermocouple position so that it is in close proximity to the sample.

9. Move the furnace into position and lower it.

Dilatometer Accessory

Description

The dilatometer accessory kit can be used to determine the volume coefficient of expansion. The cubic expansion of a sample is calculated using the following equation:

$$\beta = \frac{K \bullet (S - B)}{V}$$

where:

β = coefficient of expansion $\left(\frac{m^3}{m^3\ {}^oC}\right)$

K = vial constant ($cm^3/\mu m$)

S = slope ($\mu m/^oC$)

V = sample volume (cm^3). This volume may be obtained by dividing the sample mass in grams by its density in g/cm^3 at room temperature.

B = slope of baseline ($\mu m/^oC$).

To calculate the vial constant from a scan use the following equation:

$$K = \frac{\beta_{ref} \bullet V}{(S - B)}$$

where:

K = vial constant ($cm^3/\mu m$)

S = slope ($\mu m/^oC$)

β_{ref} = cubic expansion coefficient of standard material $\left(\frac{m^3}{m^3\ ^oC}\right)$

$\beta_{ref} = 3\ \alpha_{ref}$

Where α_{ref} is the coefficient of linear expansion of standard material, ($m/m \bullet {}^oC$), measured over the same temperature range.

V = sample volume (cm^3). This volume may be obtained by dividing the sample mass in grams by its density in g/cm^3 at room temperature.

B = slope of baseline ($\mu m/^oC$).

The dilatometer accessory kit contains:

- Dilatometer probe assembly
- Dilatometer sample vial
- Filling medium
- Aluminum calibration standards.

Operating Procedures

Considerations

The vial, filling medium, and sample assembly together make up a large thermal mass. Because of this large thermal mass, it is recommended that a slow heating rate be used to allow the sample to maintain the heating rate as sensed by the sample thermocouple. When creating a method, use a Ramp segment of 3 to 5°C/min or less for your heating rate.

NOTE: If you compare data runs, make sure that all comparison scans are made with the vial filled to the same level with filling medium, with the thermocouple in the same position, and heated at the same rate.

Accessory Installation

The dilatometer accessory uses the dilatometer probe, dilatometer sample vial and the standard stage.

1. Remove the existing probe as instructed on page 2-19.

2. Install the standard stage, if it is not already in place. (For instructions to install the stage, refer to page 2-14).

3. Insert the core end of the dilatometer probe carefully into the opening in the stage.

4. Hold the probe-locking lever in the up position and continue lowering the probe into the stage until you can feel it seat in the locking mechanism.

5. Tighten the probe-locking lever by turning it clockwise.

6. Position the sample thermocouple on the stage so that the tip is 13 mm (0.5 inch) above the stage.

7. Change the instrument mode, if needed.

Calibration

In addition to the normal probe calibration, the dilatometer accessory should be calibrated for baseline and displacement using the procedures described as follows.

Baseline Calibration

NOTE: The dilatometer filling medium must be kept dry. Store it in a desiccator when it is not being used. If the sample has become contaminated through long exposure to humidity, dry it in a vacuum oven at 200°C for one hour.

Run a thermal scan, under the same load that will be used when analyzing your samples (typically 0.01 N or less). Use a vial three-quarters full with filling medium only (no sample). Make sure the filling medium is well packed.

NOTE: Marking the vial at the fill level and filling future sample runs to this level will insure that the baseline effect due to the fill medium are minimized from run to run.

Displacement Calibration

Calibrate the dilatometer for cubic displacement using the following procedure:

1. Take three aluminum balls from the dilatometer accessory kit to use as a calibration sample.

2. Weigh the three aluminum balls and prepare the sample and vial as instructed on the following pages. Make sure that the aluminum balls do not touch the walls of the vial, and that they do not touch ech other.

3. Obtain a thermal scan under the same conditions of the baseline scan (*e.g.* same load and heating rate).

4. Calculate the vial constant from the scan by using the equation found on page 4-22.

Sample Preparation

The following method is used to prepare and load the dilatometer sample:

NOTE: The dilatometer filling medium must be kept dry. Store it in a desiccator when it is not being used. If the sample has become contaminated through long exposure to humidity, dry it in a vacuum oven at 200°C for one hour.

1. Fill the dilatometer vial one-third full with the filling medium found in the accessory kit.

2. Use a tamping rod to lightly pack the filling medium.

3. Select a sample that is small enough to easily fit inside the dilatometer vial *without* touching the walls.

4. Place the sample on the filling medium in the center of the vial. Make sure that the sample *does not* touch the vial walls.

5. Add more filling medium to the vial until it is approximately three-quarters full, then tamp down the filling medium hard with a tamping rod. The level will be compacted. Continue adding filling medium and tamp it hard until the vial remains three-quarters full. Mark this position on the vial.

6. Check the filled and packed dilatometer vial from all angles to make sure that the sample does not show (see Figure 4.9).

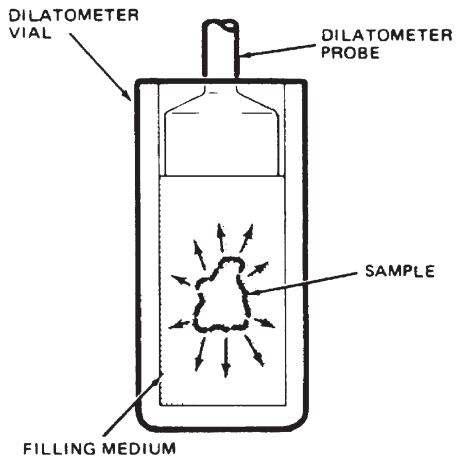

Figure 4.9
Dilatometer Sample Preparation

7. Raise the furnace and rotate it clockwise to move it off to the side.

8. Remove any previously run samples from the stage and ensure that no residue remains.

9. Open the probe.

10. Place the vial on the stage.

11. Close the probe until the probe tip is positioned above the filling medium.

12. Tap lightly on the upper probe assembly, the probe should oscillate freely within the vial.

13. Close the probe until it just rests on top of the filling medium. Gently tap the probe to seat it (see Figure 4.10).

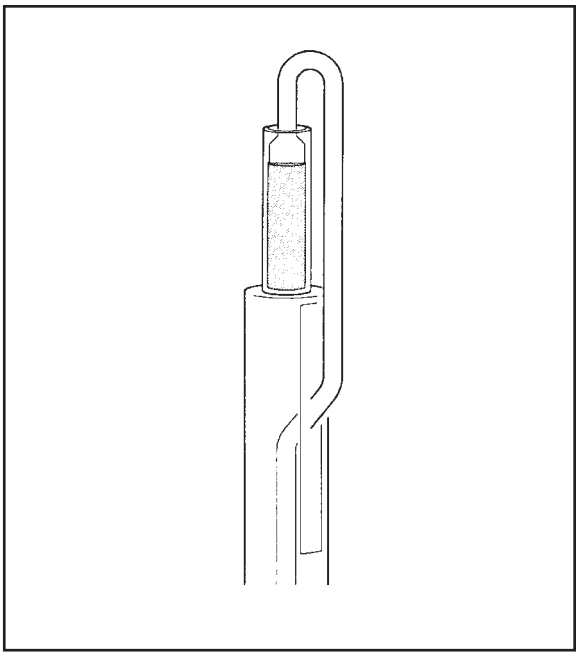

Figure 4.10
Probe Setup Using Dilatometer Samples

14. Press ZERO LENGTH on the keypad to provide a zero reference point.

15. Adjust the thermocouple position so that it is in close proximity to the vial.

NOTE: To avoid false thermocouple readings, make sure that the thermocouple tip does not touch the sample vial.

16. Move the furnace into position and lower it.

17. Program the force (typically 0.01 N or less) using *Thermal Solutions/Advantage*.

18. Check that the sample size is equal to zero before beginning your experiment.

Parallel Plate Rheometer Accessory

Description

The parallel plate rheometer accessory can be used to obtain viscosity-temperature or viscosity-time data on substances at low shear rates, over the range of 10 to 10^7 Pa-sec (1 to 10^6 Poise).

The parallel plate rheometer accessory kit contains:

- Sample-forming die set
- Parallel plates
- Alignment cage.

Accessory Installation

The parallel plate rheometer accessory uses the macro expansion probe, rheometer sample holder assembly, and the standard stage.

1. Remove the existing probe as instructed on page 2-19.

2. Install the standard stage, if it is not already in place. (For instructions to install the stage, refer to page 2-14).

3. Install the macro expansion probe in the same manner as the standard expansion and penetration probes as instructed on page 2-18.

4. Change the instrument mode, if needed.

5. Position the sample thermocouple on the stage so that the tip is 13.0 mm (0.5 inch) above the stage.

NOTE: To avoid false thermocouple readings, make sure that the thermocouple tip does not touch the alignment cages or plates. Exposed portions of the two thermocouple leads must not touch each other.

Calibration

The parallel plate rheometer accessory should be calibrated as you would normally calibrate any of the standard probes. See the *Thermal Solutions/Advantage User Reference Guide* for details.

Sample Preparation

The parallel plate rheometer employs cylindrical samples that are 9.53 mm (0.375 inch) in diameter and 0.25 to 1.00 mm (0.01 to 0.04 inch) in thickness. Three types of samples can be used with the rheometer accessory:

- Moldable powders
- Resinous materials
- Sheet materials.

In order to obtain a sample having the correct cylindrical dimensions, the material must be prepared according to the directions in this section. Moldable powders and resinous materials employ the use of the Sample Encapsulating Press (PN 900680.902) and a special set of dies, the *pellet press die set.*

To prepare samples follow the instructions found in these sections:

- Sample Encapsulating Press–see below
- Preparing Moldable Powders–page 4-35
- Preparing Resinous Materials–page 4-38
- Preparing Sheet Materials–page 4-40.

Sample Encapsulating Press

This accessory (see Figure 4.11) is used to form samples into a compact, cylindrical disc. In order to use it for parallel plate rheometer samples, you must first remove the Encapsulating Press hermetic sealing or non-hermetic crimping die assembly. After these dies have been removed, the pellet press die set can be installed.

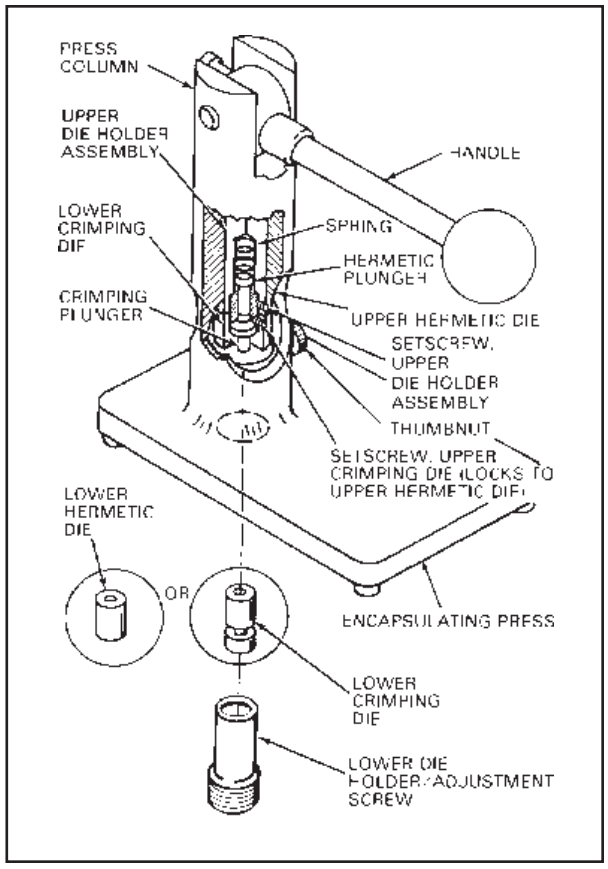

Figure 4.11 Sample Encapsulating Press

Removing the Existing Die Assembly

WARNING

The plunger in the upper hermetic die assembly is spring-loaded and should be removed with care.

1. Lower the Encapsulating Press handle. Then rotate the die-holder assembly and upper crimping die (if installed), so that the socket head setscrews are accessible through the hole in the Encapsulating Press housing.

NOTE: The upper crimping die is installed over, and locked to, the upper hermetic die.

2. Loosen only the setscrew in the upper crimping die (if installed), raise the press handle, and remove the die assembly (including the crimping plunger).

3. Lower the press handle and loosen the thumbnut located on the side of the press column.

4. Lay the Encapsulating Press on its side and screw in the lower die-holder/adjustment screw (located on the bottom of the press), until the lower die holder (either hermetic or crimping) just meets the upper hermetic die.

5. Loosen the setscrew in the Encapsulating Press upper die-holder assembly to unlock the upper hermetic die.

NOTE: The upper hermetic die is now held in position by either the lower hermetic or crimping die.

6. Unscrew and remove the lower die-holder adjustment screw. This allows removal of the lower hermetic or crimping die and upper hermetic die assembly (includes hermetic plunger and spring).

NOTE: As the lower die-holder adjustment screw is being removed, spring tension on the hermetic die plunger is relieved.

7. Set the press upright; it is now ready for the rheometer pellet press die installation.

Installing the Pellet Press Die Set

1. Remove the hermetic sealing or non-hermetic crimping die assembly as described in the preceding procedure.

2. Lower the Encapsulating Press handle and insert the pellet press piston into the upper die-holder assembly as far as it will go. Secure the piston in place by tightening the setscrew in the upper die-holder assembly.

3. Lay the Encapsulating Press on its side and screw in the pellet press base screw until it is nearly flush with the bottom of the press.

4. Place the pellet press cylinder head, cavity side down, on top of the pellet press base screw.

5. Lower the pellet press piston by bringing the handle forward until it is parallel with the press base. Adjust the pellet press base screw until the flat surface of the cylinder head meets the piston. Tighten the thumbnut, on the side of the press column, finger-tight.

6. Set the press upright. Install the pellet press cylinder into position on the cylinder head with the recessed portion of the cylinder fitting snugly onto the cylinder head (see Figure 4.12).

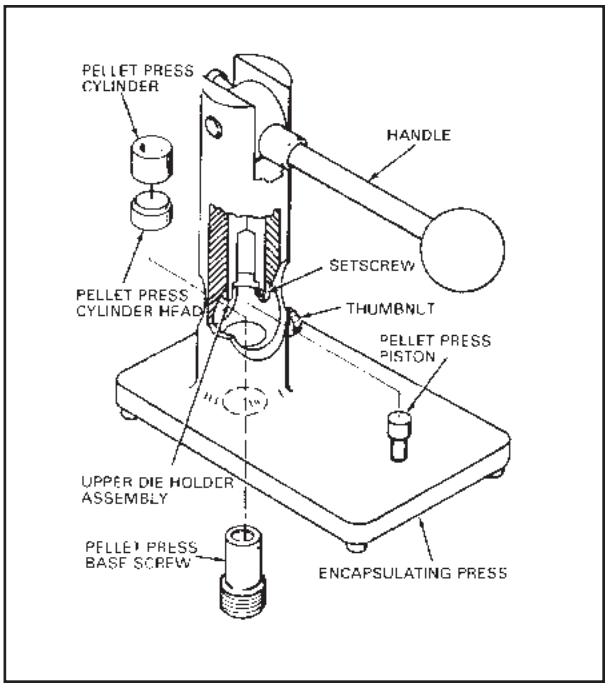

Figure 4.12
Encapsulating Press with Pellet Press Die Set

Preparing Moldable Powders

To prepare moldable powder samples for use with the parallel plate rheometer accessory, follow these instructions:

1. Install the pellet press die set in the Encapsulating Press.

2. Pour sufficient sample material (~30 to 50 mg) into the pellet press cylinder to produce a disc 0.25 to 1.00 mm in thickness.

3. Lower the pellet press piston by bringing the Encapsulating Press handle forward. Firmly press the handle down to form the sample disc.

4. Raise the Encapsulating Press handle and remove both the pellet press cylinder and cylinder head from the press.

5. Remove the cylinder head from the cylinder and reverse its position so that the cavity side of the cylinder head now faces the cylinder. Note the position shown in Figure 4.13.

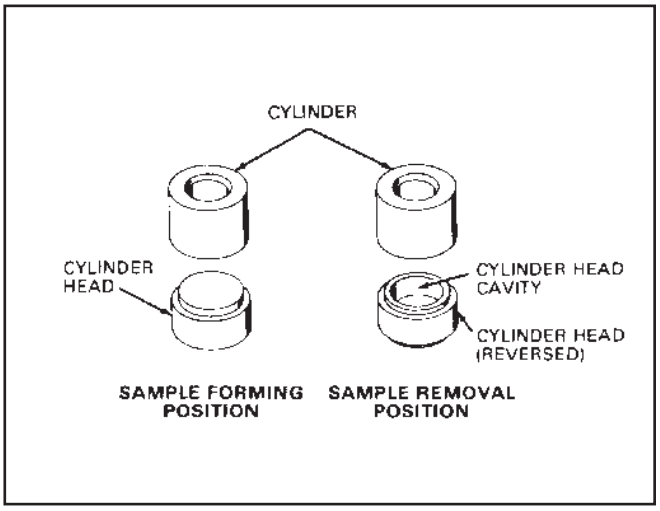

Figure 4.13 Cylinder Head Positions

6. Replace the cylinder head and cylinder in the Encapsulating Press. Lower the piston with the press handle. This will cause the sample disc to drop into the cylinder head cavity. Lift the piston, remove the cylinder, and collect the finished sample disc.

7. Measure the sample thickness, using calipers, to within $\pm$ 0.02 mm.

8. Place the sample between two parallel plates in the alignment cage as shown in Figure 4.14.

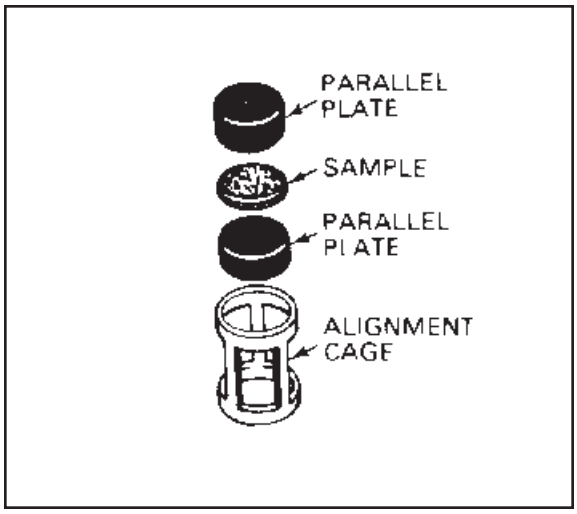

Figure 4.14
Parallel Plate
Sample Assembly

9. Load your sample according to the directions on page 4-41.

Preparing Resinous Materials

To prepare resinous materials for use with the parallel plate rheometer accessory, you will need the following parts to use as a molding form:

- Pellet press cylinder
- Cylinder head
- Piston.

1. Assemble the pellet press cylinder and cylinder head in the *sample-forming position* for use as a mold as shown (see Figure 4.15).

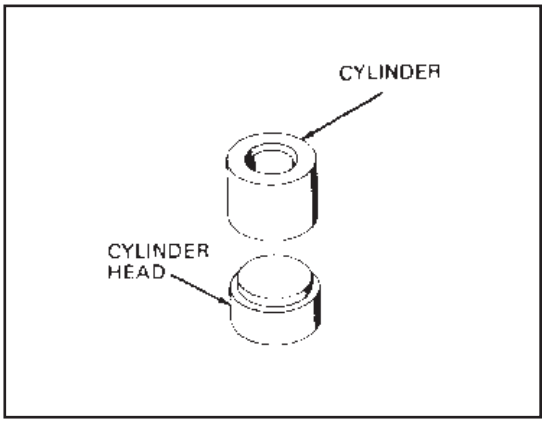

Figure 4.15 Sample-Forming Position

2. Heat up a hot plate to about 20ºC above the melting point of the sample material.

WARNING

This procedure involves heating up the sample and mold assembly. Handle with tongs to prevent burns.

3. Place the mold assembly on the hot plate and allow it to come to temperature.

4. Put enough sample material into the pellet press to yield a 0.25 to 1.00 mm thick disc (~30 to 50 mg).

5. Place the pellet press piston into the cylinder and add a suitable weight (50 g) on the top of the piston.

6. Note the position of the piston in the cylinder; as the sample starts to melt, the piston will settle.

7. Using tongs, remove the weight when the piston sinks to its maximum depth.

8. Remove the mold assembly from the hot plate using tongs, and allow it to cool to room temperature.

9. Disassemble the mold and remove the molded sample disc.

10. Measure the sample thickness, using calipers, to within $\pm$ 0.02 mm.

11. Place the sample between two parallel plates in the alignment cage as shown in Figure 4.14 on page 4-37.

12. Load your sample according to the directions on page 4-41.

Preparing Sheet Materials

To prepare sheet materials for use with the parallel plate rheometer accessory, follow these instructions:

1. Cut a sample from a sheet of material 0.25 to 1.00 mm in thickness using a standard 9.5 mm hole punch.

2. Measure the sample thickness, using calipers, to within ± 0.02 mm.

3. Place the sample between two parallel plates in the alignment cage as shown in Figure 4.14 on page 4-37.

4. Load your sample according to the directions in the next section.

Sample Loading

To load your parallel plate rheometer samples on the TMA 2940, follow these steps:

1. Raise the furnace and rotate it clockwise to move it off to the side.
2. Open the probe.
3. Remove any previously run samples from the stage and ensure that no residue remains.
4. Place the sample assembly on the stage under the probe tip (see Figure 4.16 on the next page).
5. Adjust the thermocouple position so that it is in close proximity to the sample.

NOTE: To avoid false thermocouple readings, make sure that the thermocouple tip does not touch the alignment cage, sample, or plates. Exposed portions of the two thermocouple leads must not touch each other.

6. Press Zero Length on the instrument keypad so that the probe rests on the top plate and provides a zero reference point.
7. Enter the Sample Size on the Experimental Parameters window on the controller.
8. Move the furnace into position and lower it.
9. Program the force on the Instrument Parameters window on the controller or add weight to the weight tray, if desired.

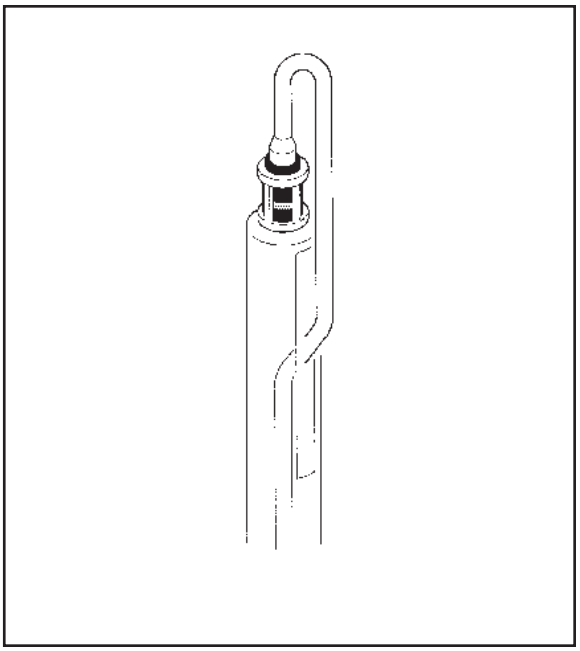

Figure 4.16
Probe Setup
Using Parallel Plate
Rheometer Samples

Cleanup

The heat used in parallel plate rheometer experiments often cements the parallel plates together inside the cage. If you want to reuse the parts, follow these directions:

♦ ***CAUTION:*** **These procedures involve the use of heat or chemicals; appropriate safety measures should be observed at all times.**

Thermoplastic material

1. Heat the plates and cages gently in a burner flame to separate them.

2. Allow the parts to cool, then scrape them clean with a small spatula.

Thermosetting material

Soak the plates and cages in a suitable solvent to separate and clean them.

or

Heat the parts to red heat in the oxidizing portion of a burner flame. The organic material should oxidize completely.

or

Heat the plates in a furnace to approximately 1000°C.

Calculations

Correction Calculations for Thermal Expansion of Parallel Plates

1. Calculate the apparent distance between the plates (h^*) at a given temperature (or time) by subtracting the decrease in distance between the plates (in mm) from the original sample height.

   Equation:

   $$h^* = \underset{(\text{mm})}{\text{sample thickness}} - \underset{(\text{mm})}{\text{dimension change}}$$

2. Correct the distance between the plates for thermal expansion.

   Each plate is nominally 5.08 mm (0.2 inch) stainless steel. Therefore, the total dimension for both plates is 10.2 mm (0.4 in.).

The expansion coefficient (α) for stainless steel as a function of temperature is shown in Table 4.1 on the next page.

a. Choose the appropriate coefficient of expansion (α) value for stainless steel at the desired temperature.

b. Multiply this a by the total plate thickness and the temperature change to obtain the thermal expansion correction factor.

c. Subtract the correction factor from the apparent distance between the plates (h^*) determined in step 1 to obtain the actual distance between the plates (h).

Where:

$h =$

$h^* - [\alpha\ (\mu m/m°C) \times \Delta T(°C) \times 10.2\ mm \times 10^{-6}\ (m/\mu m)]$

3. Calculate $1/h^2$.

4. Calculate a table of: $\frac{\Delta 1/h^2}{\Delta t}$ versus T

Where:

h^* = apparent distance between plates

h = actual distance between plates

t = time

T = temperature.

Table 4.1 Stainless Steel Coefficient of Expansion[1]

T (°C)	Coefficient of Expansion (α) (μm/m°C)
–18	14.8
93	16.0
149	16.6
204	16.7
260	16.9
316	17.3
371	17.5
427	17.6
482	17.8
538	18.0

[1]D.E. Furman, *J. Metals*, 188 <4>, 688 <1950>

Calculations for Viscosity and Wall Shear Rate Data

To calculate sample viscosity (η) and wall shear rate (γ), the experimental data is first scaled and corrected for expansion of the parallel plates (as previously shown on page 4-43). Then this corrected data is applied to equations developed by Dienes and Klemm (*J. Appl. Phys.*, 17,458, 1946) and Cessna and Jabloner (*J. Elast. Plast.*, 6, 103, 1974).

Equation:

$$\eta = \frac{4F}{3\pi r^4} \times \frac{\Delta t}{\Delta(1/h^2)} \times 10^6 \text{ mm}^2/\text{m}^2$$

$$\dot{\gamma} = \frac{3rh}{2} \times \frac{\Delta(1/h^2)}{\Delta t}$$

where:

F = applied force (N)

r = parallel plate radius (mm)

h = sample height (mm)

t = time (sec)

η = viscosity (Pa-sec)

$\dot{\gamma}$ = shear rate (1/sec)

Hemispherical Probe

The hemispherical probe (see Figure 4.16) is used with the standard quartz stage to obtain softening point data on substances. It is installed, operated, and calibrated using the same procedures as the standard expansion and penetration probes. Refer to Chapter 2 “Installing the TMA 2940” for information on installation.

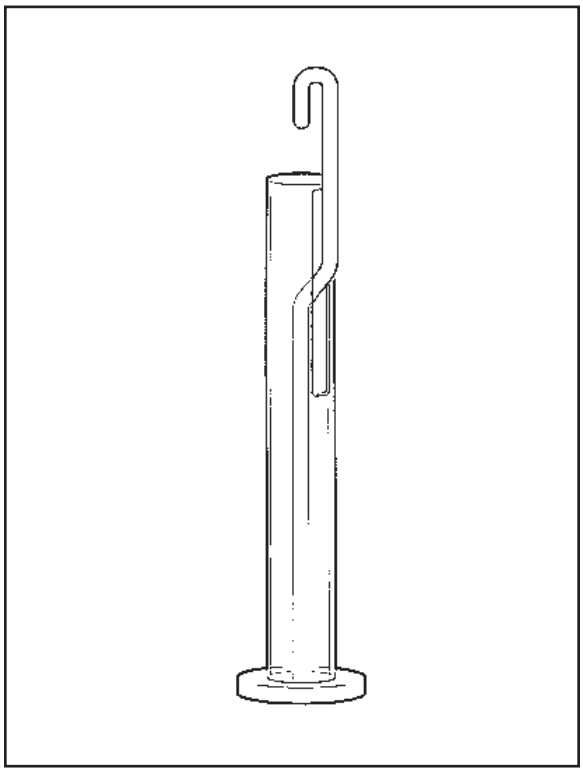

Figure 4.16
Hemispherical Probe

CHAPTER 5: Technical Reference

Description of the TMA 2940

The Thermomechanical Analyzer 2940 is capable of heating or cooling samples while applying a predetermined force on the material. A quartz probe, placed in contact with the sample, is used to determine any linear or volumetric changes in dimension, at the selected temperature and force. Valuable data on melting points, glass transition temperature, expansion coefficients, gel time and temperature, resin flow, and delamination temperature can be obtained by the sensitive measuring system of the TMA 2940.

Uses of Thermomechanical Analysis

Thermomechanical Analysis is an investigational tool used by various chemical, electronic, pharmaceutical, and manufacturing companies and labs. It is used for quality control studies, analysis of processing conditions, and performance characterization of samples. The samples analyzed using the TMA vary widely. They can include materials such as:

- polymers used in packaging materials
- optical and other types of fibers
- films of all kinds
- cured laminates such as those used in multilayer printed circuit boards
- composites
- elastomers
- epoxy prepregs
- resins, *etc.*

Calculating the Coefficient of Expansion

One of the analyses obtained using the TMA 2940 is an expansion profile of a sample. To calculate the coefficient of expansion (a experimental) from the expansion profile you can use the Data Analysis program or the following equation:

$$\alpha = \frac{\Delta L \times K}{\Delta T \times L}$$

Where:

α = measured coefficient of expansion (μm/m°C)

L = sample length (m)

ΔL = change in sample length (μm)

ΔT = change in temperature (°C)

K = cell constant.

Method Considerations

Everyone has their own methods to use when running experiments; however, the following items may prove useful:

- For measuring thermal expansion, low forces (0.005 to 0.05 N) are recommended.
- When searching for softening temperatures, studies of penetration temperature vs. force are helpful to eliminate false values.
- A heating rate of 5°C/min or less is best for polymeric and elastomeric materials.
- Temperature programs should be started well below the temperatures of any known transitions, since straight-line extrapolations are normally used to define transition temperatures. As a rule of thumb, start the run 2 to 4 minutes times the heating rate (°C/min) lower than the temperature of interest.

Status Codes

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

Table 5.1
Method Status Codes

Code	Meaning
Air Cool	The air cool solenoid is opened to allow cooling air flow to the furnace.
Balance	Force is being applied to balance the system at the center of the displacement range. This is used for force calibration.
Calib	The instrument is performing LVDT calibration or the instrument is in a calibration mode. No method is running.
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, a faulty control thermocouple signal.

(table continued)

Table 5.1 (continued)

Code	Meaning
Complete	The thermal method has finished.
Cooling	The heater is cooling, as specified by a Ramp segment.
Closing	The furnace and/or probe assembly is closing.
Equilib	The temperature is being equilibrated to the desired set point.
Err *xxx*	An error has occurred (*xxx* will appear as a two- or three-digit code). The local screen will give the Error Code number; the controller will 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. Press START to continue the run.

(table continued)

Table 5.1 (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 or by running a cooling ramp.
Initial	The temperature is equilibrating 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 set point is jumping to a new value.
Measure	Measurement of the sample length is in progress.
No Power	There is no power to the heater. Check the heater switch and fuse.
Opening	The furnace and/or probe assembly is opening.

(table continued)

Table 5.1 (continued)

Code	Meaning
Preheat	The furnace is equilibrating at a specified temperature. Initially, the furnace is in the open position. After the temperature has equilibrated, the furnace is closed and held isothermal for a specified time.
Ramping	The force is ramping to a specified value.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Press the START key to continue the method.
Reject	The experiment has been terminated and the data erased.
Repeat	The method is executing a repeat loop.
Set Up	The system is preparing to start the first segment of the method.
Standby	The method segments and end-of-method operations are complete.

(table continued)

Table 5.1
(continued)

Code	Meaning
Temp *	The temperature calibration is active.
Unstable	The Isostrain segment has been initiated, but the instrument has not or cannot meet the criteria specified.
Zeroing	The LVDT is being positioned to center the probe in the displacement range.

CHAPTER 6: Maintenance and Diagnostics

Overview

This chapter contains information needed for the efficient operation of the TMA as follows:

- Maintenance procedures needed to keep your instrument clean and in good working order
- Instructions on how to replace certain parts when needed
- Details on diagnosing fuse problems and dealing with power failures
- Descriptions of the TMA Confidence Tests.

Routine Maintenance

In order to keep your Thermomechanical Analyzer 2940 in good working condition, the maintenance procedures described here should be performed regularly. Periodic inspection and cleaning are recommended. Any further maintenance or repair should be performed by a representative of TA Instruments or other qualified service personnel.

WARNING

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

Inspection

Examine the instrument for good condition as follows:

- The furnace area should be clean; any residue should be removed before starting the next experiment.
- The stage, probe assembly, LVDT core, and sample thermocouple should be free of any sample material before the next sample is loaded.

Cleaning

Regular cleaning of your instrument helps to increase its longevity and maintain its efficiency. Taking a few minutes to clean various parts of the Thermomechanical Analyzer as directed, will be well worth the effort.

Cleaning the Keypad

You can clean the instrument keypad as often as desired. The keypad is covered with a silk-screened Mylar(R)* 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 keypad. Wet the towel, not the keypad, with the glass cleaner, and then wipe off the keypad and display.

Cleaning the Probe Assembly

After each experiment, check the probe assembly. If the probe is dirty, remove it using the procedures found in Chapters 2 (for standard probes) and 4 (for optional probes), then clean it as follows:

1. Use contact cleaner or acetone applied with a soft brush or cloth to clean the LVDT core and the upper probe.

2. Heat the end of the quartz probe with a Bunsen burner until the residue evaporates and the probe is clean. Heat the probe very slowly if the sample has a large amount of glass or mineral filler.

* Mylar(R) is a registered trademark of the of the DuPont Company.

Probes may also be cleaned in a nitric acid solution.

Cleaning the Stage

Dirt or sample residue left on the top of the stage may interfere with the next sample placed on the stage. To maintain proper experimental conditions, clean the stage as follows:

For small amounts of residue:

1. Use industrial cleaner or acetone on a soft cloth to wipe the top of the stage.

For thorough cleaning:

1. Raise the furnace and rotate it clockwise to move it off to the side.
2. Remove the stage shield.
3. Take off the spring clip that holds the sample thermocouple and move the thermocouple off to the side of the stage.
4. Turn the stage nut counterclockwise to remove it.
5. Twist the stage retainer ring (with key slots) counterclockwise, pull it up off the posts and slide it up off the stage.
6. Take the wave washer up off the stage flange and remove it.
7. Remove the stage from the stage opening.

8. Use industrial cleaner or acetone applied with a soft brush or cloth to clean the stage surface.

9. Heat the stage surface with a Bunsen burner until the residue evaporates and the stage is clean. Heat the stage very slowly if the sample has a large amount of mineral or glass filler.

   The stage may also be cleaned in a nitric acid solution.

Cleaning the Thermocouple

Sample residue and dirt may interfere with the accuracy of the thermocouple readings. To clean the thermocouple:

1. Raise the furnace and rotate it clockwise to move it off to the side.

2. Remove the stage shield.

3. Remove the spring clip holding the thermocouple in place.

4. Hold the thermocouple away from the probe assembly and clean gently with a low flame using a hand-held burner.

Replacements

Occasionally you may need to replace a broken or worn out part of the TMA. Any replacements needed, other than those discussed in this manual, must be supplied and installed by qualified TA Instruments service personnel. Call (302) 427-4050 for service.

Installing a New Thermocouple

You may find that the need arises for a new thermocouple (PN 944017.901) as a result of normal wear and tear, accidental breakage, contamination of the thermocouple, *etc*.

Before removing the thermocouple, you will need to take off the TMA balance enclosure, the stage shield, and the spring clip holding the thermocouple in place. Then carefully thread the thermocouple down through the opening in the platform and unplug it.

To replace the sample thermocouple, follow the directions found in Chapter 2, Installation.

Diagnostics

Fuses

The TMA contains several internal fuses; however, *not one is user serviceable*.

WARNING

Do not attempt to service or replace any internal TMA fuses. Call TA Instruments Service for repair if you suspect an internal fuse problem; a hazardous condition may be present in the instrument.

The only fuses that you should service yourself are the external fuses, located on the TMA control panel. Both of these fuses are housed in safety-approved fuse carriers labeled F1 and F2 (see Figure 6.1).

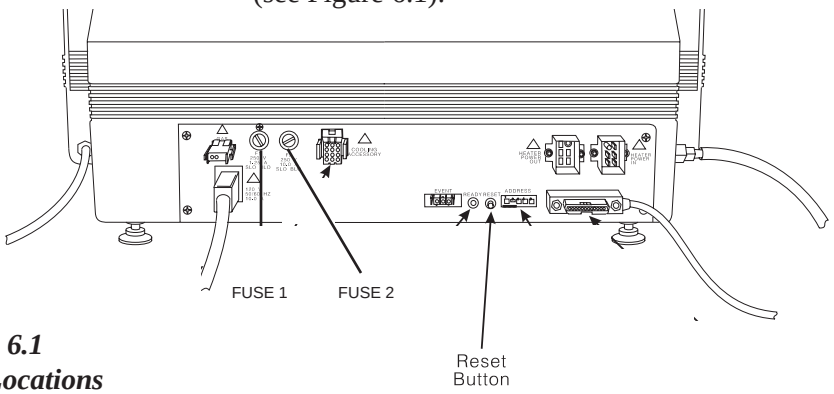

Figure 6.1
Fuse Locations

WARNING

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

Fuse F1 is in the circuit between the main electrical input and the rocker switch labeled POWER. 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 system.

Fuse F2 protects the heater coils in the oven. Because F2 does not power the internal logic, you may not know that this fuse is blown until you try to heat a sample, since the TMA instrument will pass its confidence test with this fuse open.

Fuse F2 is checked whenever a method is begun. Power supplied by this circuit is switched by a computer-controlled relay as well as the HEATER switch located on the TMA front panel. When both devices are active, the lamp in the HEATER switch turns on.

Power Failures

A power failure caused by a temporary drop in line voltage can result in one of two responses in the 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 instrument display.

In the latter case, the system is "hung." It has detected a power failure and shut down. The TMA will not restart until it is reset. To reset, press the Reset button on the instrument's back panel.

If the power fail error code ("Err F02") appears at startup and remains even after you have attempted to restart the instrument, the detection circuitry itself is probably at fault. Do not try to repair it yourself; call TA Instruments for service.

♦ ***CAUTION:*** **The TMA instrument requires a normal line voltage of 115 V AC, 50 or 60 Hz. Do not operate it at less than 105 V AC or greater than 130 V AC. Lower line voltage may result in poor instrument operation; higher line voltage may damage the instrument.**

TMA 2940
Test Functions

The TMA 2940 has three levels of test and diagnostic functions:

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

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

The TMA Confidence Test

The TMA runs a confidence test every time it is started or reset. The confidence test checks most of the computer and interface components in the system. Each of the tests that make up the confidence test provides status and error information on the instrument display.

When the confidence test is running, a two-digit hexadecimal test number is shown on the lower right of the instrument display (see Figure 6.2). The number changes each time a new test is started. Most of the tests are performed very quickly, so their test numbers may not be apparent.

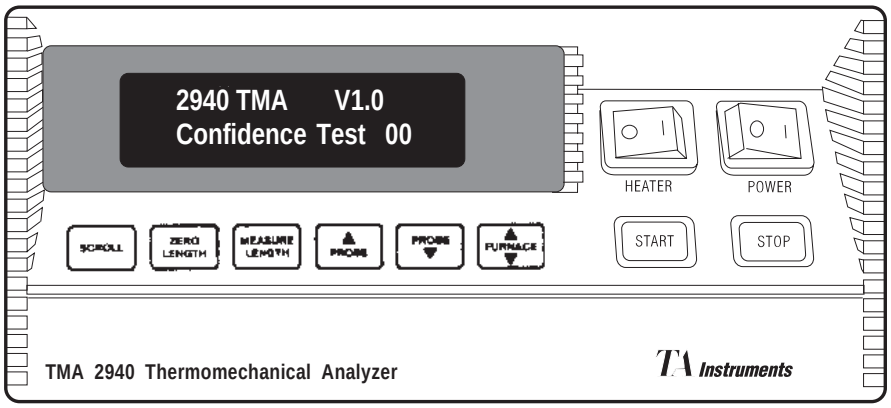

Figure 6.2
Instrument Display
During the Confidence Test

The length of time required to run the confidence test depends on the options installed. The base level system takes about 12 seconds. The longest tests are the RAM tests, which take about 6 seconds.

After the tests are completed, a sign-on message is displayed for three seconds. The system then starts normal operation, and the ready light on the back of the TMA is turned on.

If an error is detected during the confidence test, an error message is posted on the bottom line of the instrument display. If the error is *nonfatal*, the program holds the error display for three seconds and then goes on to the next test. If the error is *fatal*, the error message is posted, and the system halts. The ready light remains off. A fatal error means that a circuit essential to the operation of the TMA has failed the confidence test; the instrument cannot reliably perform any further functions.

Table 6.1 summarizes the primary confidence tests for the TMA 2940.

Table 6.1
Primary Confidence Test and Error Code Summary

Test Number	Test	Fatal (Y/N)
00	System failed start up	Y
30	CMOS RAM read/write test	Y
4*n*	PROM checksum, location, and version test	Y
5*n*	CPU board I/O function tests	Y
6*n*	Data storage memory (DRAM) read/write test	N
70	GPIB communications test	N
82	Keypad shorted key test	N
A*n*	Analog board tests	N
B*n*	Motor drive board tests	N
D0	CMOS saved memory checksum test	N
E0	Fatal error while running	Y

Replacement Parts

Tables 6.2 and 6.3 contain a list of the parts and the part numbers associated with the Thermomechanical Analyzer 2940.

Table 6.2
Standard TMA Items

Part Number	Description
944000.901	Automatic TMA
944004.001	Instruction Manual, TMA
944200.901	Standard TMA Accessory Kit consisting of:
944122.901	(1) Expansion Probe Assembly
944126.901	(1) Penetration Probe Assembly
944123.901	(1) Macro-Expansion Probe Assembly
944120.901	(1) Sample Stage
944017.902	(1) Sample Thermocouple - Type K with clip
900902.901	(1) Indium Calibration Standard
940070.000	(1) Aluminum Calibration Standard
942057.000	(1) Teflon (R) Demonstration Sample
259537.000	(1) Forceps
259522.000	(1) Set Weights
203947.005	(1) 3/32 Hex Wrench
269792.001	(2) Wave Washers
944073.001	(1) Washer disc silicon
944072.001	(1) Reservoir mass aluminum

(*table continued*)

Table 6.3
Optional Accessory Kits

Part Number	Description
944201.901	Film/Fiber Accessory Kit consisting of:
944121.901	(1) Film/Fiber Sample Stage
944128.901	(1) Film/Fiber Probe Assembly
941038.901	(1) Vial Cleaved Aluminum Balls
944047.901	(2) Film Clamp Assemblies
944144.001	(1) Film Clamp Fixture
269789.001	(1) Jeweler's Screwdriver (0.080 inch)
269793.001	(10)Film Clamp Screws (#0 - 80)
944202.901	Dilatometer Accessory Kit consisting of:
944124.901	(1) Dilatometer Probe Assembly
941143.000	(3) Dilatometer Sample Vials
941148.000	(2) Vials Filling Medium
941022.901	(1) Vial Aluminum Calibration Standards
944203.901	Flexure Accessory Kit consisting of:
944127.901	(1) Flexure Probe Assembly
941054.000	(1) Flexure Sample Platform

(*table continued*)

Table 6.3
(continued)

Part Number	Description
944204.901	Parallel Plate Rheometer Accessory Kit consisting of:
943147.901	(1) Rheometer Sample-Forming Die Set
943125.000	(3) Rheometer Alignment Cages
943126.000	(6) Rheometer Parallel Plates
944125.901	Hemispherical Probe Assembly
944011.901	Heater Assembly for use with the 2940 TMA

Appendix A: Ordering Information

For information or to place an order, contact:

United States:

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

Overseas:

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

TA Instruments GmbH
Siemenstrasse 1
63755 Alzenau
Germany
Telephone: 49-6023-30044
Fax: 49-6023-30823

TA Instruments Benelux
Ottergemsesteenweg 461
B-9000 Gent
Belgium
Telephone: 32-9-220-79-89
Fax: 32-9-220-8321

TA Instruments Japan
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1-3-12 Kitashinagawa
Shinagawa-Ku, Tokyo 140
Japan
Telephone: 813/3450-0981
Fax: 813/3450-1322

TA Instruments France
18 Rue Jean-Bart
Parc D'Activities De La Grande Ile
78960 Voisins-Le-Bretonneux
France
Telephone: 33-01-30489460
Fax: 33-01-30489451

TA Instruments Spain
Avienda Europe 21
Planta Baja
28100 Alcobenda
Madrid, Spain
Telephone: 34(9) 16618448
Fax: 34(9) 16610655

For technical assistance or service in the United States:

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

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

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