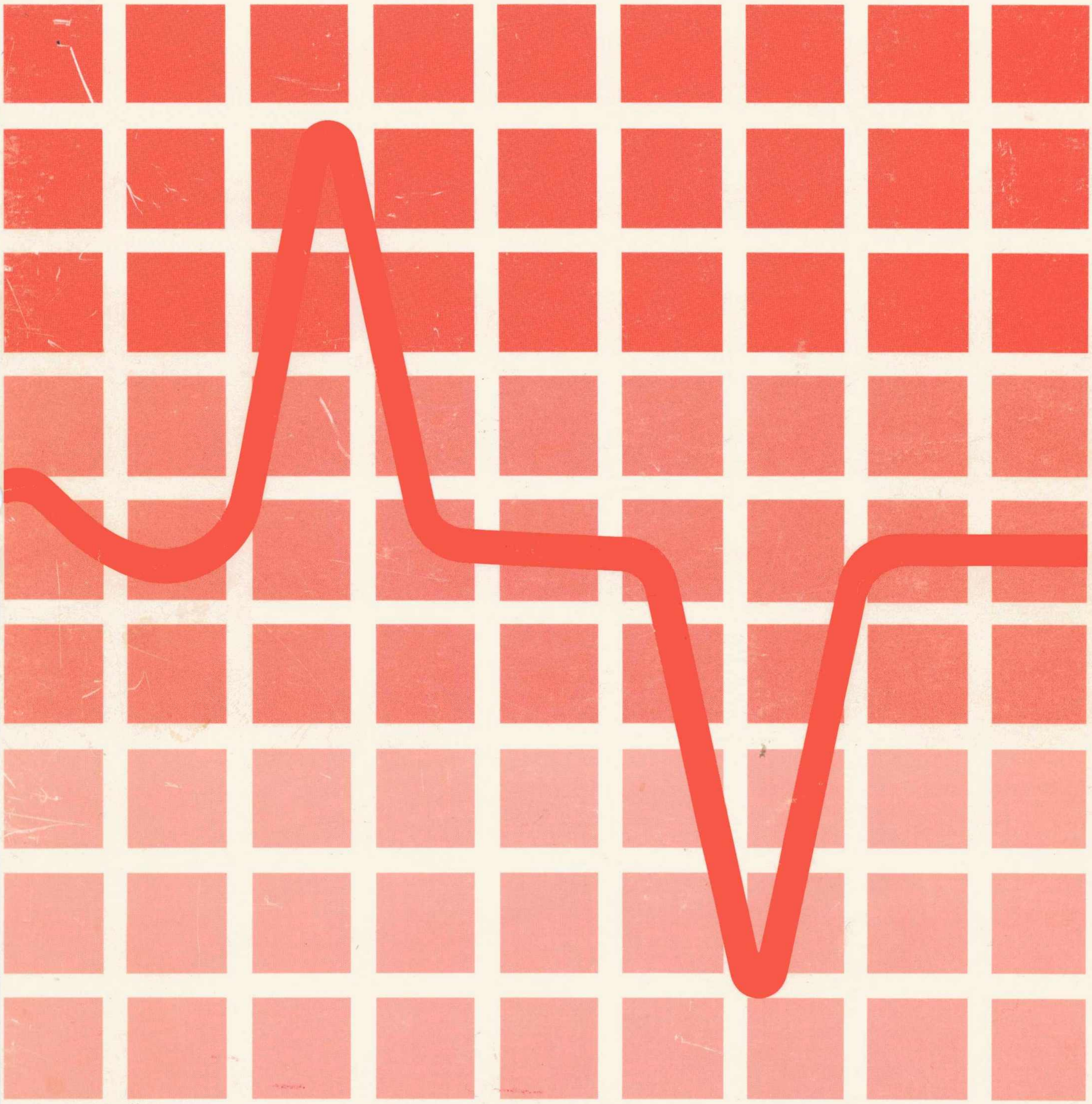
943 TMA Thermomechanical Analyzer

Operator's Manual



DU PONT

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Operator's Manual

943 Thermomechanical Analyzer

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Thermal Analyzers
Instrument Systems

DU PONT
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Part 1

INTRODUCTION

Introducing the 943 TMA

The Du Pont 943 TMA is a plug-in module that can be used with any of the Du Pont Thermal Analyzer control consoles to make up a system for collecting and reporting data from thermal experiments. The system measures changes in sample properties resulting from changes in three experimental variables: temperature, atmosphere, and time.

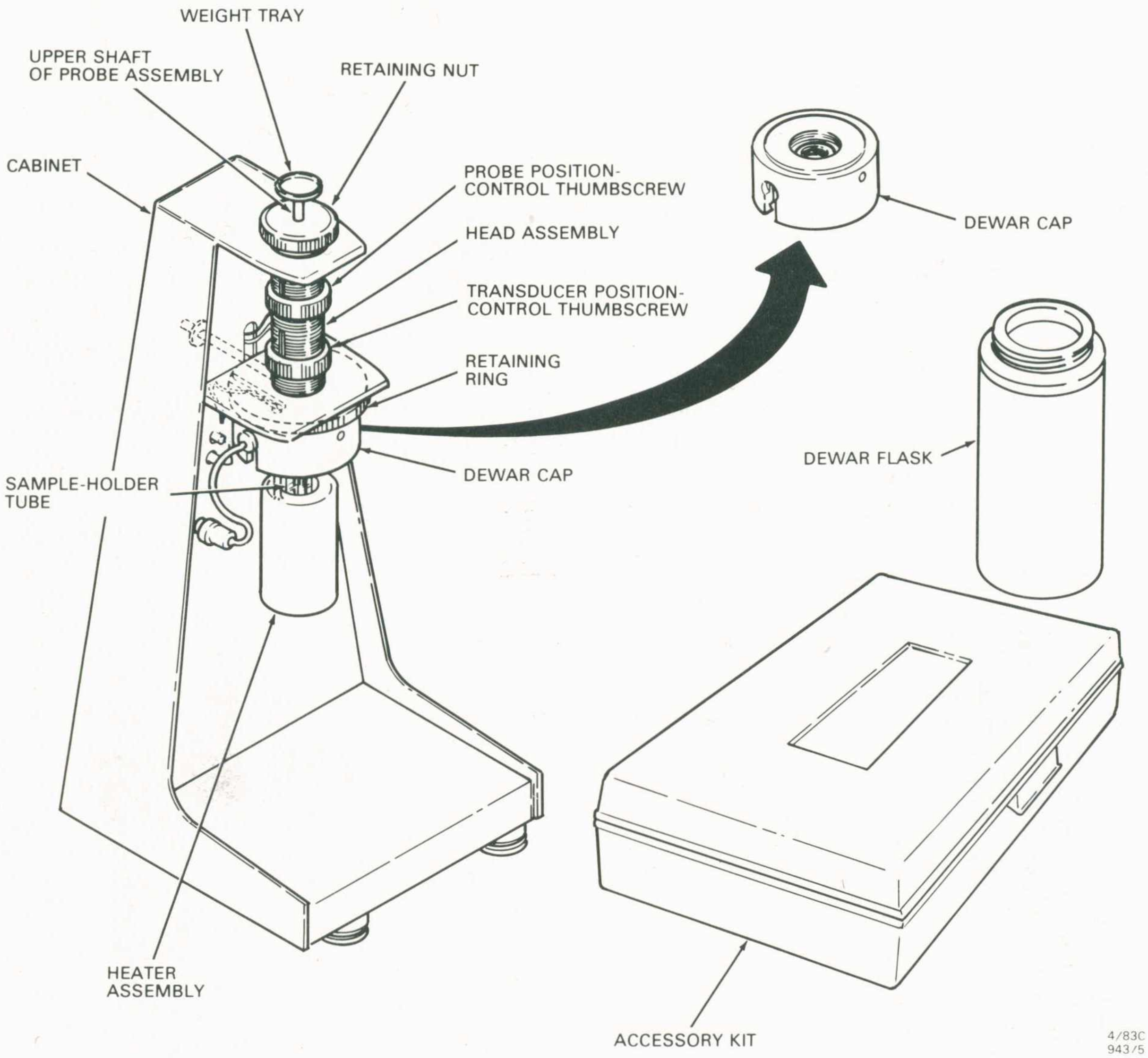
In the TMA, a sample, which can be formed as a plug, film, powder, or fiber, is compressed or held in tension by a probe assembly. Movement of the probe is translated electrically by a linear variable differential transformer (LVDT) attached to the probe, into a signal that appears on the Y axis of the plotter.

The TMA uses interchangeable probes that allow you to make a number of important measurements, including softening point, tensile modulus, compression modulus, glass transition, and expansion coefficient. With the optional accessories for the system, you can do experiments in parallel plate rheometry, fiber tension, stress relaxation, and dilatometry.

943 Thermomechanical Analyzer

SYSTEM COMPONENTS

The TMA cabinet holds the head assembly (containing the LVDT), the probe assembly, the sample holder, the heater, and the Dewar flask.



- head assembly — Attaches to the cabinet by a retaining nut and ring. Contains the LVDT for measuring vertical movement.
- probe assembly — Interchangeable for making several different measurements. Holds a weight tray for loading weights onto the sample. Positioned vertically on the sample by the probe position-control thumbscrew.
- sample holder — Interchangeable for making several different measurements. Contains the sample thermocouple.
- heater assembly — Surrounds the sample holder; contains the Platinel II control thermocouple.
- Dewar flask — Used as a temperature chamber below ambient, a gas chamber, and a container for holding coolants. Slips over the heater assembly and screws into the Dewar cap.

ACCESSORY KIT

The accessory kit supplied with the TMA contains weights, a weight tray, sample holders, a level, an Allen wrench, tweezers,samples for calibration, and compression probes.

The probes provided allow you to determine expansion coefficients and to examine films and coatings without removing them from the substrate.

Probe Descriptions

Description	Contact Diameter mm (in.)	Load Exerted by 1 g Load (kPa)
Penetration Probe	0.89 (0.035)	16
Expansion Probe	2.54 (0.100)	1.9
Macro Expansion Probe	6.07 (0.239)	0.34

Optional probe assemblies are described in the appendix to this manual, beginning on page 000.

SPECIFICATIONS

- Temperature Ranges .... -180°C to 800°C (Standard heater)
  Ambient to 1200°C (with optional 1200°C heater)
- Sample Height .... 25 mm (1 in.) maximum
- Sample Diameter .... 9.5 mm (.375 in.) maximum
- Sensitivity .... 0.5 to 500 μm/cm of chart
- Displacement Range .... ±1.3 mm (± 0.05 in.)
- Linearity .... ±0.5%
- Loading .... 0 to 100 grams
- Baseline .... Less than 1.3 μm total displacement, from -100°C to 600°C

Part 2

INSTALLATION

Installing the 943 System

When you receive the 943 TMA, inspect the instrument and shipping container for signs of shipping damage, and check the parts received against the shipping list. If the instrument is damaged, notify the carrier and Du Pont Instruments immediately. If the instrument is intact but parts are missing, notify Du Pont Instruments. You will find a list of Du Pont offices on the back cover of this manual.

ASSEMBLING THE TMA

1. Holding the Dewar cap in one hand, unscrew the Dewar flask from the cap. Refer to the figure on page 7 for parts identification.
2. Remove the Dewar cap from the TMA head assembly.

CAUTION

The sample-holder tube is fragile and should be handled with care.

3. Select a sample-holder tube from the accessory kit and insert it through the top of the Dewar cap. Two teflon washers are used between the holder and cap.
4. Replace the Dewar cap on the head assembly; hand tighten. The hole in the Dewar cap should face the thermocouple connectors on the front of the TMA cabinet. If necessary, loosen the setscrews in the Dewar cap with the Allen wrench supplied in the accessory kit. Turn the Dewar cap until the hole faces the thermocouple connectors and tighten the setscrews.
5. Using the tweezers from the accessory kit, thread the thermocouple leads through the hole in the Dewar cap (see detail A on page 7).

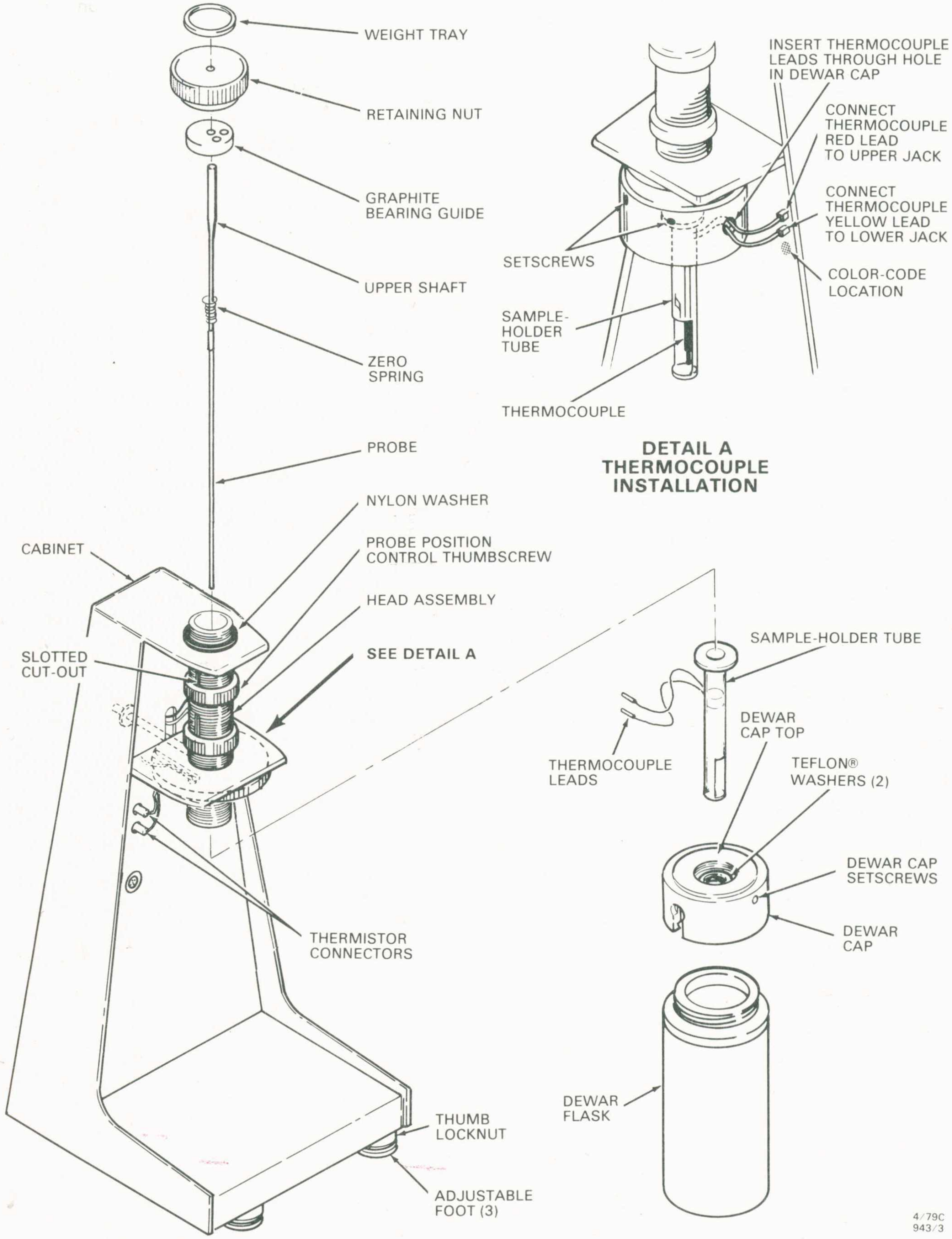
NOTE

Make sure that the bare thermocouple wires do not touch, as this could cause erroneous readings.

6. Connect the red thermocouple lead to the upper connector and the yellow lead to the lower connector.
7. Remove the retaining nut from the top of the TMA head assembly. Do not remove the nylon washer beneath the nut.
8. Use tweezers to remove the bearing guide. Two holes are provided in the guide for easy removal.
9. Make sure that the probe position-control thumbscrew is above the half-way point on the slotted cutout in the head assembly. Turn the thumbscrew counterclockwise to move it up.

CAUTION

- Do not drop the probe assembly into position; the delicate sample-holder tube could be broken.
- Try not to move the probe assembly from side to side while installing it. Side movement may bend or break the probe.



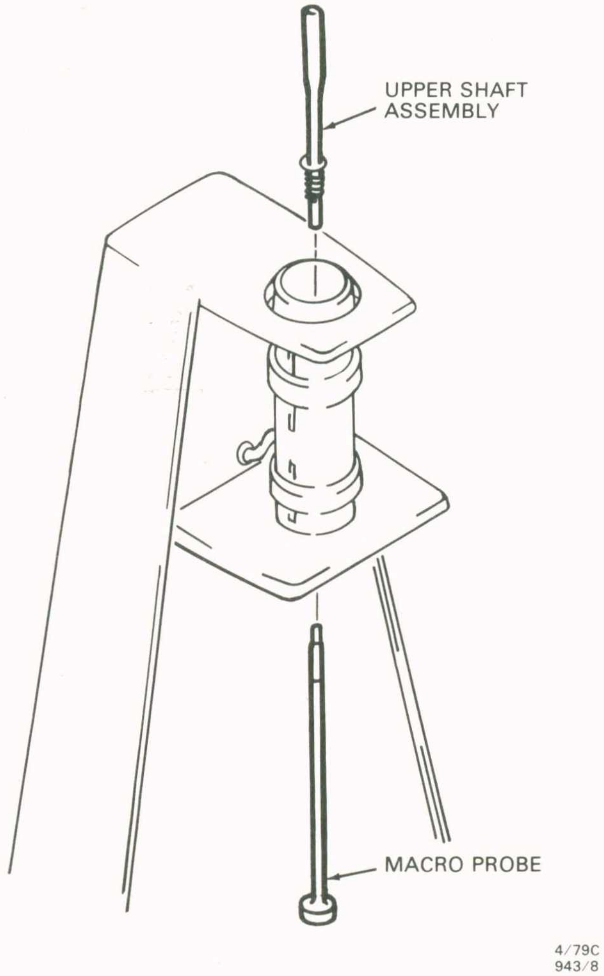
943 TMA Parts Identification

10. Insert the expansion probe assembly from the accessory kit into the head assembly as shown on page 7.

NOTE

The penetration probe is installed in the same manner as the expansion probe.

The macro probe is installed in two pieces as shown here.



11. Replace the bearing guide and the retaining nut.
12. Tap the top of the probe assembly gently to make sure that it moves freely and is spring-loaded. Then, turn the probe position-control thumbscrew clockwise to lower the probe until its end rests on the bottom of the sample-holder tube.
13. Place the retaining nut and weight tray on top of the probe assembly.

14. Place the TMA next to the programmer. The instrument should be on a vibration-free bench or marble benchtop. Observe the following precautions:
    - Do not locate the TMA near air conditioning or heating vents.
    - Avoid locations where there are high levels of corrosive gases (such as sulfur dioxide or hydrogen chloride), or where ventilation is poor.
    - Avoid dusty locations and places where vibrations occur.
15. Place the level from the accessory kit on top of the weight tray. Level the TMA by adjusting the three leveling feet on the base of the cabinet. To adjust the feet, loosen the thumb locknut for all three feet and screw the feet into or out of the cabinet. When the instrument is level, tighten the thumb locknuts and remove the level from the weight tray.
16. Raise the probe approximately 2 cm (1 in.) from the bottom of the sample-holder tube by turning the probe position-control thumbscrew counterclockwise.
17. Remove the weight tray, retaining nut, and bearing guide from the TMA.
18. Check probe alignment by looking down into the head assembly and observing the gap between the lower bearing and the probe. If the probe appears to be touching the side of the head assembly, rotate it until the gap is uniform around the probe.
19. Reinstall the bearing guide, retaining nut, and weight tray. Lower the probe until it rests on the bottom of the sample-holder tube.

CAUTION

Do not load the weight tray unless the probe is touching either the sample-holder tube bottom or a sample.

20. Check the thermocouple connections for tightness.

HOOK-UP

To connect the 943 TMA to the programmer:

1. Make sure that the programmer power is off, and that the programmer power cord is unplugged from the wall outlet. The 943 connector (marked ACCESSORY) is located on the back of the unit. The cable and connector are keyed so that they can only be connected one way.
2. Connect the accessory cable from the programmer to the TMA.
3. Connect the programmer power cord to the wall outlet.
4. If you wish to use inert or reactive gases for testing, attach the external gas supply tube to the TMA gas purge connection on the back of the instrument. The TMA is now ready for calibration. Calibration procedures appear in part 3 of the manual.

Part 3

OPERATION

Performing Routine Experiments

This section contains routine operating procedures for the 943 TMA. The section is divided into the following parts:

- 943 Controls
- Heater Installation
- Dewar Flask Installation
- Applications Notes
- Calibration Procedures
- Routine Operating Sequence
- Subambient Operation

Specific instructions for operating the TMA with each of the optional accessories appear in the appendix of the manual, beginning on page 000.

Before operating the TMA, you should have the unit installed and connected to a TA programmer.

943 CONTROLS

Controls for the 943 TMA are located on the front and rear panels of the cabinet. They are described in the tables that follow.

FRONT PANEL CONTROLS

(1) Baseline Control

The baseline control is used to obtain a level baseline over a temperature range. The baseline calibration procedure appears on page 000.

(2) Calibrate Control

This control is used to calibrate the output signal from the TMA so that the Y axis reads directly in $\mu m/cm$.

(3) Zero Switch

The zero switch is used when positioning the LVDT for electrical zero. Use the down position only with the Series 99 programmer. In the down position, the switch shorts output to the Y axis of the recorder, establishing a zero reference. In the up position, the output of the LVDT goes to the recorder.

(4) Heater Switch

The heater switch is normally kept in the HIGH position. Use the LOW position for low temperatures (below 200°) and slow heating rates (less than 5°C/min).

(5) Probe Position-Control Thumbscrew

The position-control thumbscrew is used to raise or lower the probe for positioning on the sample.

(6) Transducer Position-Control Thumbscrew

This control adjusts the position of the transducer to the probe for zero output.

(7) Derivative Reset Switch

(Series 99 only)

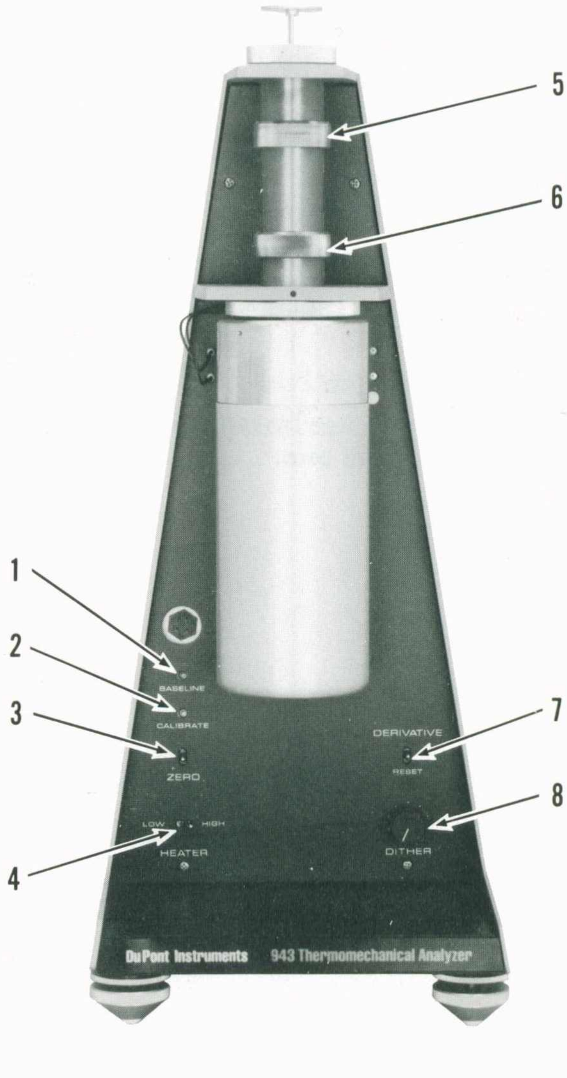
This switch has three positions:

Up — OFF
Middle — ON
Down — RESET

During normal operation, the derivative reset switch is kept in the middle position. The up and down positions are used to zero the output of the derivative. The up position is a locking position, and is normally used while loading samples or making adjustments. The down position is a momentary (nonlocking) position and is used to check the zero reference of the derivative.

(8) Dither Switch

This switch operates a small vibrator in the TMA cabinet that reduces static friction around the probe assembly. It may be used to remove pen stepping during recorder tracings; however, too high an adjustment can cause pen vibration.



REAR PANEL CONTROLS

(9) Control Reference Junction

The control reference junction is used to calibrate the control thermocouple.

(10) Sample Reference Junction

The sample reference junction is used to calibrate the sample thermocouple.

(11) Derivative Zero

This screwdriver adjustment zeroes the Y axis during calibration.

(12) Derivative Gain

The derivative gain control is a screwdriver adjustment that is used to calibrate derivative gain as a time-base function.

(13) Ramp Out Jacks

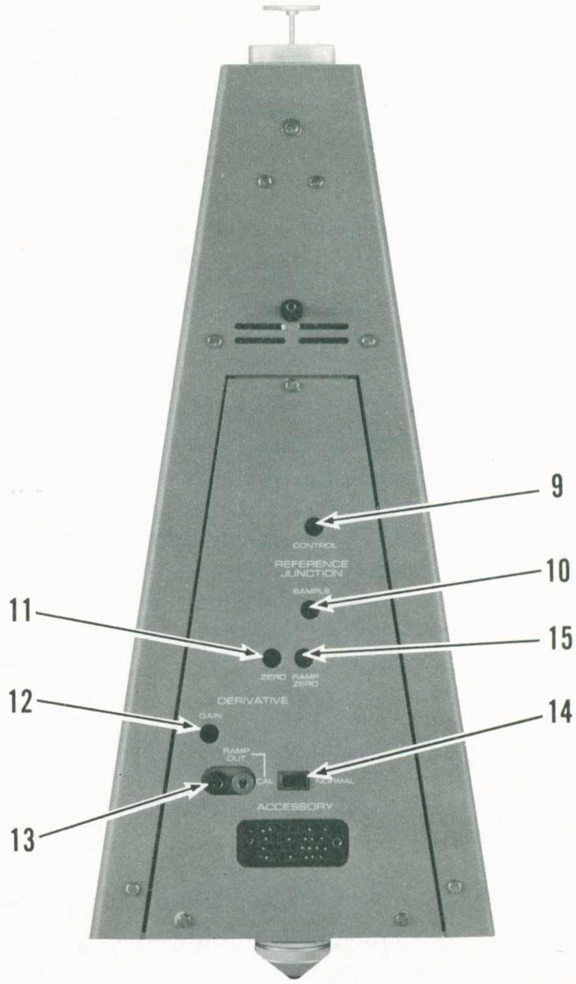
These jacks are used to connect the TMA to a recorder or oscilloscope.

(14) Cal-Normal Switch

The normal position is used for routine operation. The cal position is used during calibration.

(15) Derivative Ramp Zero

This control is used to zero the derivative output to the recorder zero.



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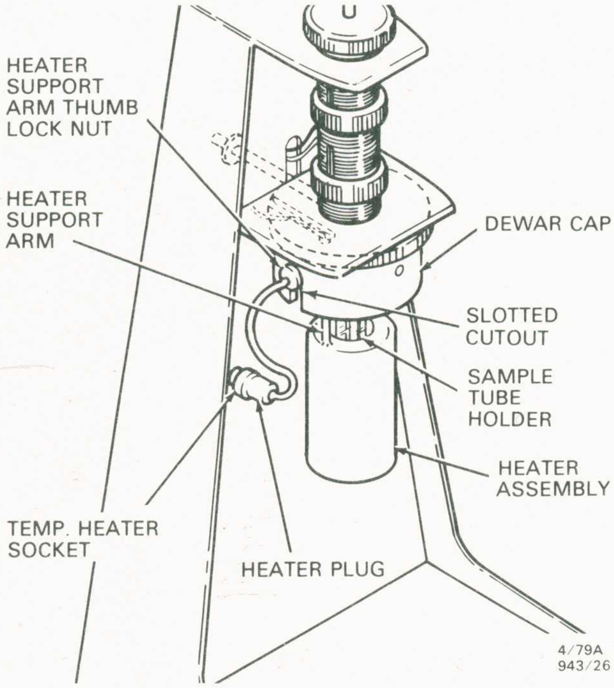
HEATER INSTALLATION

1. Slip the heater over the sample holder. Insert the heater support arm all the way into the slot in the Dewar cap.



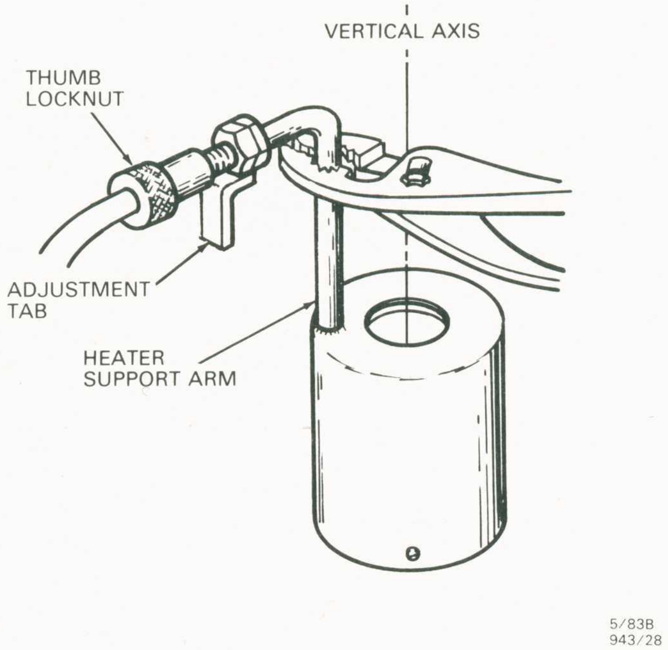
2. Make sure that the heater does not touch the sample holder. If it does, adjust the heater as follows:

   a. Vertical Adjustment

CAUTION

When performing this adjustment, hold the support arm rigid to prevent the arm from breaking at the weld joint.

To adjust the support arm, hold the vertical part of the arm rigid and gently bend the horizontal part. Bending downward will rotate the heater's vertical axis clockwise; bending upward will rotate the heater's vertical axis counterclockwise.



b. Horizontal Adjustment

To adjust the support arm horizontally, loosen the adjustment tab locknut and screw the tab in or out as needed. Tighten the locknut.

NOTE

Check heater alignment whenever the sample-holder tube is replaced.

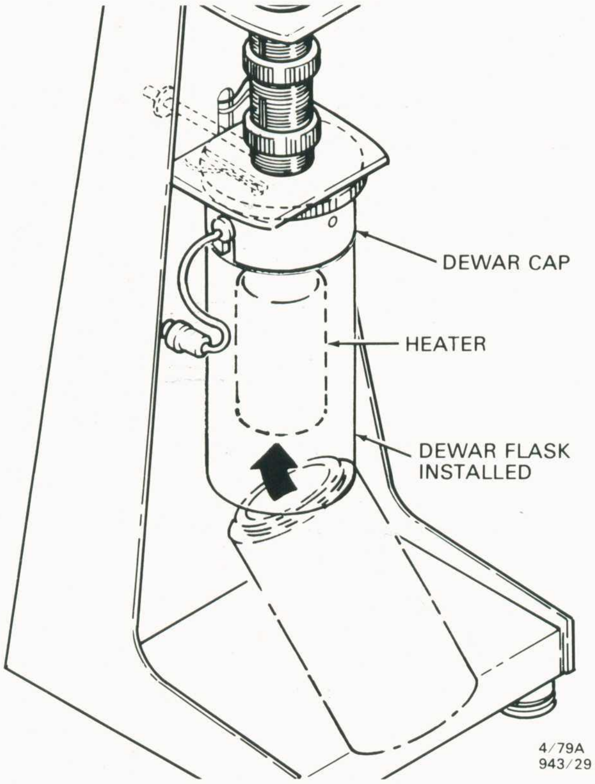
3. Attach the heater to the Dewar cap by tightening the support arm locknut.
4. Insert the heater plug into the TMA heater socket.

DEWAR FLASK INSTALLATION

CAUTION

The Dewar flask seals will melt at temperatures above 500°C.

To install the Dewar flask, tilt the flask and slip it over the heater. Then, while holding the Dewar cap in one hand, screw the flask into the cap.



APPLICATION NOTES

The following are some notes and precautions related to applications of the 943 systems.

WARNING

Wear protective gloves when handling the heater. The outside surface of the heater becomes hot during operation.

CAUTION

- The Dewar flask seals will melt at temperatures above 500°C.
- Do not leave the heater switch on low when heating to a final temperature above 200°C, or at a rate faster than 5°C/min.

NOTES

- Between -40°C and 0°C, false penetration may occur due to ice.
- To remove water from the system, thoroughly rinse the probe and sample-holder tube with acetone and allow drying time for evaporation.
- When adjusting the thumbscrews for the TMA probe position control and the transducer position control, maintain approximately the same distance between them. Otherwise, a false zero setting of the Y'-axis pen may occur, or full downward travel of the probe may be hampered.

  Check the zero setting, by moving the transducer above and below the zero point, to see if the output moves equally above and below the set point.
- Make the final adjustment of the probe assembly at a temperature below that of the lowest transition expected in the material under investigation.
- Samples for penetration and expansion should be as thin as possible to minimize temperature gradients in the sample. A height of 2.5 to 5 mm (100 to 200 mils) is normal for making expansion measurements on samples having expansion coefficients in the order of 100 μm/m°C.
- Film samples should be as flat as possible to prevent false penetrations due to buckling or relaxation.
- Powdered silica can be used both above and below the sample to level the sample and to prevent lateral movement of both probe and sample. Powdered or finely divided silica can be purchased from most chemical supply houses.
- Samples can be heated and formed into cylinders and then cooled; however, doing so may change important thermal history.
- A heating rate of 5°C/min is best for polymeric and elastometric materials.
- When searching for softening temperatures, studies of loading versus penetration are helpful to eliminate false values of Tg.
- Temperature programs should be started well below the temperatures of any known transitions, since straight-line extrapolations are normally used to define transition temperatures.

CALIBRATION PROCEDURES

The 943 system must be calibrated at installation and periodically thereafter. The calibrations described in this section include:

- Baseline Calibration
- Temperature Calibration
- Y′-Axis Static Calibration
- Y-Axis Derivative Calibration
- Y-Axis Dynamic Calibration

Baseline Calibration

The baseline should be checked as part of the initial set-up procedure and adjusted if necessary. Afterward, it should only need adjustment after servicing.

To adjust the baseline:

1. Prepare the TMA with a sample holder, probe and weight tray.
2. Adjust the probe position control so that the probe just rests on the sample-holder platform.
3. Place a 5 g weight on the weight tray.
4. Attach the heater to the TMA.
5. Set the Y(Y′)-Axis RANGE to its maximum sensitivity and adjust the Y(Y′)-Axis ZERO SHIFT so that the pen is in the center of the chart paper. 0,5 mm/cm
6. Heat at 5°C/min from ambient to desired upper limit.
7. If the baseline deviates from the horizontal more than 2.5 cm, adjust the BASELINE control on the front of the TMA to bring the pen back toward the center of the chart.
8. Repeat steps 6 and 7 until the desired baseline is achieved.

Temperature Calibration

The temperature calibration procedure is used to adjust the temperature readings of the recorder and programmer to standard with a known melting point.

SAMPLE THERMOCOUPLE

To calibrate the sample thermocouple:

1. Prepare the TMA with a sample holder, probe, and weight tray.
2. Place an indium sample on the sample-holder platform.
3. Adjust the probe position control so that the probe just touches the sample.
4. Place a 5 g weight on the weight tray.
5. Attach the heater to the TMA.
6. Zero the electrical signal from the TMA, as described in the Y′-Axis static calibration on page 20.
7. Heat the indium sample from ambient to 170°C. Record the scan.
8. Note the difference in inflection point from the standard (156.6). For example, 158.0°C - 156.6°C = 1.4°C. If the inflection point is not 156.6°C, proceed to Step 9.

9. Set the system to hold isothermally at 150°C. Allow the system to equilibrate, then adjust the SAMPLE reference junction on the back of the TMA by the difference between the actual inflection point and the standard. For example, if the inflection point recorded is 158.0°C, adjust the SAMPLE reference junction back 1.4°C. On the 1090, you can watch the adjustment on the plasma display; on the Series 99, you will use the recorder.

   When the sample thermocouple has been corrected, leave the system set to hold isothermally at 150°C and proceed to the Control Thermocouple Calibration Procedure.

CONTROL THERMOCOUPLE

To set the control thermocouple:

Turn the CONTROL reference junction on the back of the TMA until the programmer indicates 150°C.

Y'-Axis Static Calibration

1090 PROGRAMMER

The 1090 programmer allows direct read-out of electrical zero from the 1090 display using the DISPLAY AXIS key. To set static calibration using the 1090:

1. Prepare the TMA with a sample-holder tube, expansion probe, and weight tray.
2. Turn the programmer on and set it to standby.
3. Adjust the TMA probe position control so that the probe just rests on the empty sample-holder platform.
4. Place a 5 g weight from the accessory kit on top of the weight tray.
5. Set the DISPLAY AXIS to read signal 3 on the last line of the display. Adjust the TMA transducer position control until signal 3 reads zero. When you get close to zero, you can use SIGNAL ZERO to bring the 1090 reading to zero.
6. Remove the 5 g weight from the weight tray.
7. Raise the probe with the probe position control. Place the 380μm (15 mil) calibration shim from the accessory kit on the sample-holder tube platform. Then, lower the probe gently until it rests on the shim.
8. Replace the 5 g weight on the TMA weight tray.
9. Adjust the TMA CALIBRATE control so that the display axis reading is equivalent to the shim thickness.
10. Remove the weight and shim. The system is now statically calibrated.

SERIES 99 PROGRAMMER

To set static calibration using the Series 99 programmer:

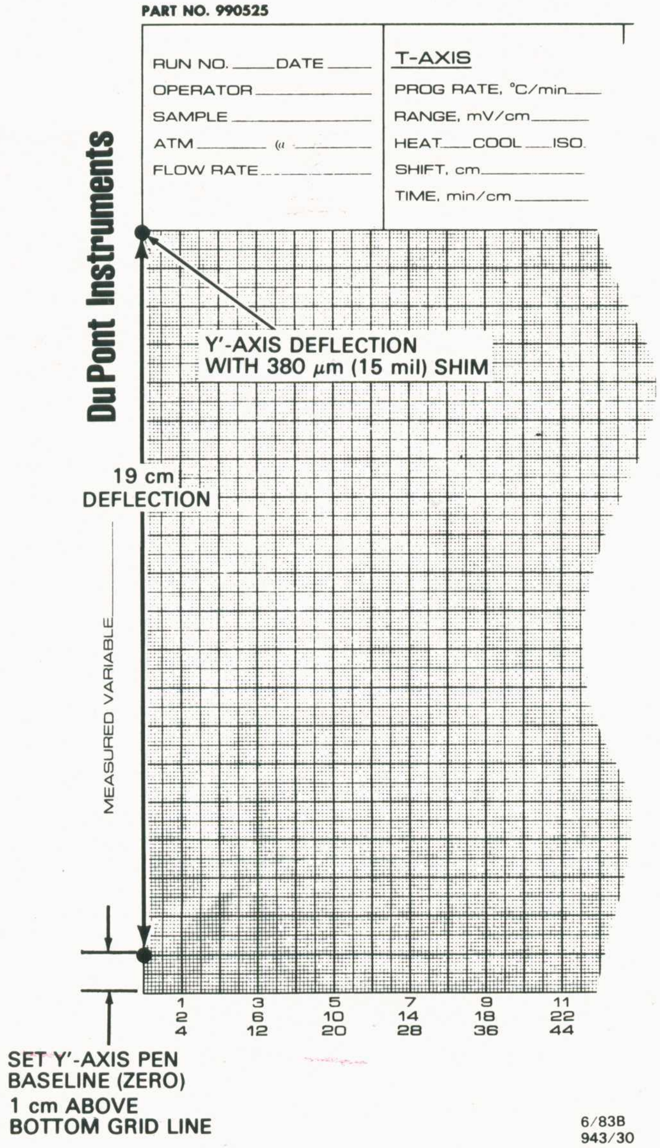
1. Prepare the TMA with a sample-holder tube, expansion probe and weight tray.
2. Push the programmer POWER switch to on.
3. Set the programmer to standby.
4. Set the recorder PEN selector to LOAD.
5. Place a sheet of linear chart paper (PN 990525) on the recorder platen.
6. Set the recorder PEN selector to UP.
7. Set the recorder T-axis ZERO SHIFT selector to 0 (zero).

8. Set the recorder T-axis RANGE selector to TIME.
9. Set the recorder Y′-axis RANGE selector to 20 mV/cm.
10. Adjust the TMA probe position control so that the probe just rests on the empty sample-holder tube platform.
11. Place a 5 g weight from the accessory kit on top of the weight tray.
12. Push and hold the recorder Y′-axis ZERO switch up. Adjust the ZERO SHIFT selector so that the Y′-axis pen is positioned as a line-zero reference point, 1 cm above the bottom grid line of the chart paper. Release the ZERO switch.

NOTE

When ZERO switch is released, the pen will change its position.

13. Adjust the TMA transducer position control so that the Y′-axis pen is positioned to the previously established line-zero reference point, 1 cm above the bottom grid line of the chart paper.



14. To check the accuracy of the settings performed in steps 12 and 13, push the recorder Y′-axis ZERO switch up momentarily, and while releasing it, observe the pen. If pen movement occurs, readjust the transducer position control slightly to raise or lower the pen. Repeat this procedure until no pen movement is detected.
15. Remove the 5 g weight from the TMA weight tray.
16. Obtain the 380 μm (15 mil) calibration shim from the accessory kit. Raise the probe with the probe position control and place the shim on the sample-holder tube platform. Lower the probe gently until it rests on the shim.

NOTE

The Y′-axis pen will move upward.

17. Replace the 5 g weight on the TMA weight tray.
18. Adjust the TMA CALIBRATE control so that the Y′-axis recorder pen deflection is equivalent to the shim thickness shown. For example, the pen deflection for a 380 μm (15 mil) shim is 19 cm, starting from the baseline, which is 1 cm up from the bottom grid line.
19. Set the recorder PEN selector to LOAD, and remove the chart paper.
20. Remove the weight from the weight tray, and the shim from the sample-holder tube platform. The system is now statically calibrated.

Y-axis (Derivative) Calibration

NOTE

The following procedure is written for the Series 99 programmer. If you have a 1090 programmer, you should not be concerned with this calibration unless you are interested in the analog derivative signal, signal 5. To perform this procedure on the 1090, use the DISPLAY AXIS to zero the electrical signal.

The TMA's derivative printed circuit board provides its own calibration signal. Two multiposition controls, the function switch (CAL/NORMAL) on the rear panel and the DERIVATIVE (off/on/RESET) on the front panel, are used selectively to control both the ramp and the derivative signals.

When the function switch is in the NORMAL position, the derivative board circuit is placed in parallel with the LVDT output; this results in the output signal from the derivative board being the derivative of the LVDT signal. If the function switch is placed in the CAL(IBRATE) position, the output from the internally generated ramp signal is connected to the input of the derivative board for calibration purposes. The two RAMP OUT jacks on the rear provide an output ramp signal.

The DERIVATIVE switch, located on the front panel, provides two functions. When the DERIVATIVE switch is moved to the on (center) position, the signal is the derivative function of the input signal. In either the off (up) or RESET position (down), the switch shorts both the ramp function and the derivative output.

Setup Procedure:

1. Prior to performing the actual derivative calibration procedure, perform the Y′-axis static calibration described on page 20.
2. Remove the probe assembly.
3. Place the function switch on the rear panel of the TMA to the CAL position.

4. Power on the programmer and set it to standby.
5. Set the PEN selector to the LOAD position.
6. Place a sheet of linear chart paper on the recorder (PN 990525).
7. Set the PEN selector to the UP position.

Calibration Procedure:

1. Place the DERIVATIVE/RESET switch in the up (off) position.
2. Using the recorder ZERO switch, adjust the Y′-axis pen to the 2 cm line from the bottom of the chart paper. This is a reference zero for the RAMP ZERO adjustment on the rear panel of the TMA.
3. Using the recorder ZERO switch, adjust the Y-axis pen to the 20 cm line of the chart paper. This will be the top line of the chart paper and represents the reference zero for the derivative output.
4. Observe the movement of the Y′-axis (lower) pen as the RAMP ZERO potentiometer (15), on the rear panel of the TMA, is adjusted. Rotate the screwdriver-adjustable potentiometer clockwise to move the pen down. Adjust this zero to coincide with the previously set recorder zero.
5. Observe the movement of the Y-axis (upper) pen as the derivative ZERO potentiometer (11), on the rear panel of the TMA, is adjusted. Turning the screwdriver-adjustable potentiometer clockwise moves the pen up and counterclockwise moves the pen down. Adjust this zero to coincide with the previously set recorder zero.

NOTE

Proper adjustment of the RAMP ZERO and ZERO potentiometers should result in virtually no pen movement (<1 mm) when the recorder ZERO switch is pressed for Y′ and Y.

6. After the zeroing procedure is complete, proceed to set up the derivative output calibration by setting the recorder and TMA controls as follows:

a. TMA

Function Switch:	CAL position

b. Recorder

T-axis:	TIME BASE
Y-axis:	50 mV/cm (5 mil/min/in.)
Y′-axis:	50 mV/cm (5 mil/in.)
TIME BASE:	0.5 min/cm (0.5 min/in.)
T-AXIS ZERO SHIFT:	0
PROGRAM:	STANDBY Position

7. Start the plot by pressing the START switch on the recorder. Allow the pens to plot for approximately 1 cm distance; then place the DERIVATIVE switch in the center (on) position.
8. Allow the program to run for 7 to 10 minutes; a typical plot, as shown on the following page, results.
9. Choose an arbitrary point, P_1, on the Y′ curve. Drop a vertical line to intersect a horizontal line drawn from P_3 to P_2.
10. Measure line segments P_1P_2 and P_2P_3. Substitute these values into the relationship.

$$Y_1 = \frac{20\ \overline{P_1P_2}}{\overline{P_2P_3}}$$

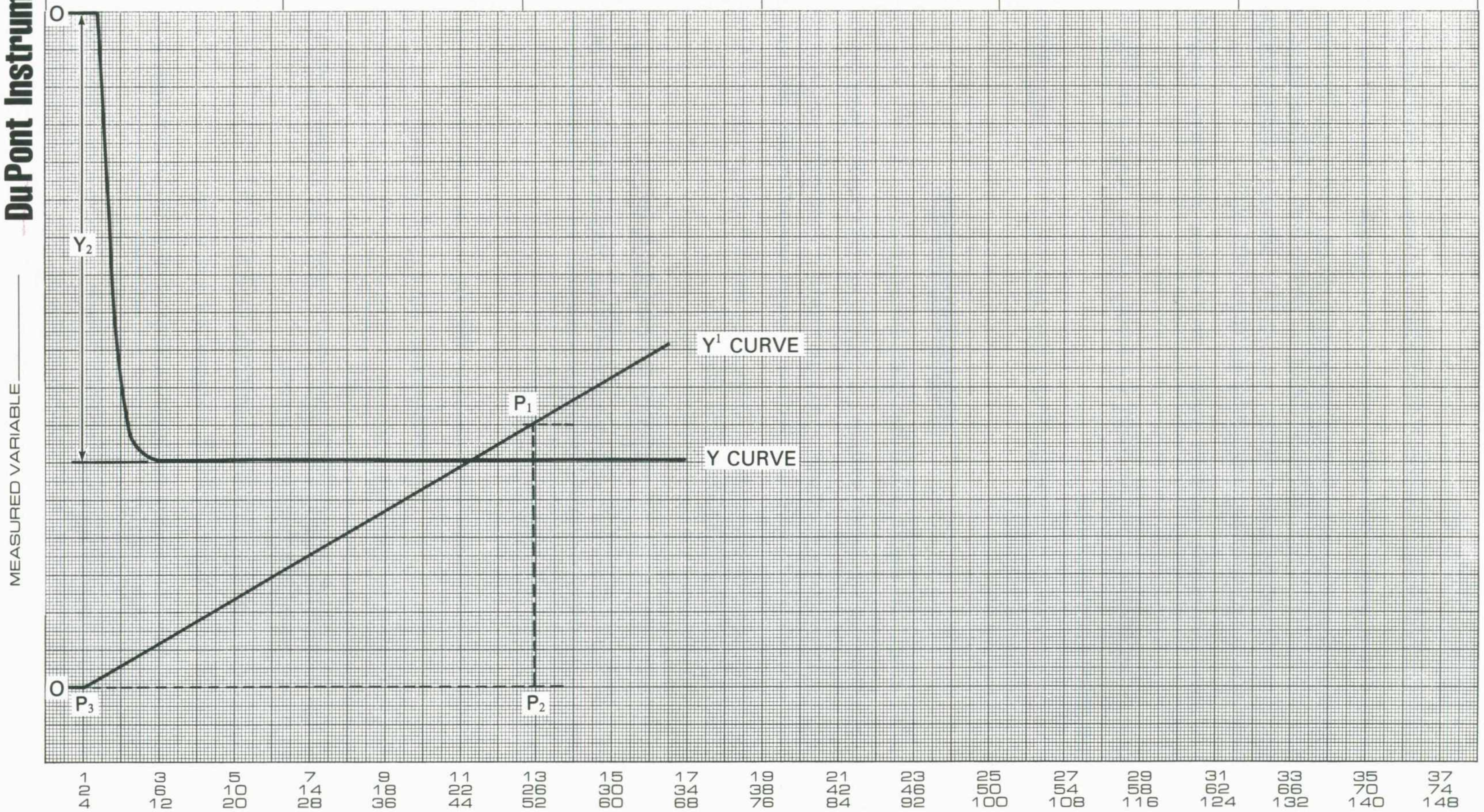
11. Measure the distance Y_2 on the Y curve. Compare it with the calculated value Y_1, which should be equal to Y_2, (to 3 significant figures).
12. If the calculated value, Y_1, is not equal to Y_2, adjust the GAIN potentiometer (on the rear panel of the TMA) clockwise to increase deflection (Y_2) and counterclockwise to decrease deflection (Y_2).

Y-axis (Derivative) Static Calibration

PART NO. 990525

DuPont Instruments

RUN NO.____ DATE ____	T-AXIS	DSC 200 µW/mV	TMA 1 µm/mV	DMA 0.05 Hz/mV 0.05 dB/mV	
OPERATOR ____	PROG RATE, °C/min ____	DTA 50 mK/mV	DTG 0.1 µm/(min mV)	FREQ, mV/cm ____	
SAMPLE ____	RANGE, mV/cm ____	RANGE, mV/cm ____	MODE ____	DAMPING, mV/cm ____	
ATM ____ @ ____	HEAT __ COOL __ ISO __	WEIGHT, mg ____	RANGE, mV/cm ____	OSC AMP, mm ____	
FLOW RATE ____	SHIFT, cm ____	REFERENCE ____	SAMPLE SIZE ____	A/Z GAIN, % ____	
	TIME, min/cm ____		LOAD, g ____	SAMPLE SIZE ____	



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Y′-Axis and Y-Axis (Derivative) Dynamic Calibration

For high precision dynamic measurements (e. g., determination of the coefficient of expansion), perform the following calibration procedures:

1. Prepare the TMA with a sample-holder tube, expansion probe, and weight tray.
2. Perform Y′-axis static calibration procedures described on page 20.
3. Obtain the calibration standard (aluminum cylinder) from the TMA accessory kit. Measure its length within±0.02 mm (±mil), using a micrometer calipers (not supplied), and note the result. Place the aluminum cylinder on the TMA sample-holder tube platform. The aluminum cylinder for our example measured 7.62 mm (300 mil).
4. Adjust the TMA probe position control so that the probe just rests on top of the aluminum cylinder.
5. Obtain a 5 g weight from the TMA accessory kit and place it on top of the weight tray.
6. Attach the heater assembly.
7. Set the programmer to standby. (For 1090 operation follow steps 8 through 11. For Series 99 operation skip to step 12.)

1090 PROGRAMMER

8. Zero the electrical signal using the transducer position control and the display axis.
9. Set up a thermal run from ambient to 300°C at 10°C/min. (Prepare to store the run.)
10. Start the calibration run. Then, press and hold the RESET switch on the TMA until the 1090 display axis returns to zero. Release the switch and complete the run.
11. Playback signals 3 and 5 vs. time. Then, proceed to Calculations of Y′ Axis and Y Axis for Determining Coefficient of expansion on page 27.

SERIES 99 PROGRAMMER

12. Place a sheet of thermocouple-corrected chart paper on the recorder platen. Our example uses 0°C to 360°C (PN 9900529) chart paper.
13. Set the recorder PEN selector to UP.

NOTE

The Y′-axis pen will move upward.

14. Turn the recorder T-axis RANGE and ZERO SHIFT selectors to the settings indicated on the chart paper under the heading, "T axis."
15. Turn the recorder Y′-axis RANGE selector to 2 mV/cm.
16. Turn the recorder Y-axis RANGE selector to 2 mV/cm.
17. Adjust the TMA transducer position control so that the Y-axis pen is 1 cm above the bottom grid line of the chart paper.
18. Push and hold the recorder Y-axis ZERO switch up. Adjust the ZERO SHIFT selector so that the Y-axis pen is postitioned as a line zero reference point on the top grid line of the chart paper. Release the ZERO switch.

NOTE

When ZERO switch is released, the pen may change its position slightly.

19. Set up a thermal run from ambient to 300°C at 10°C/min.

NOTE

The following settings are nominal. You may choose other chart papers and instrument settings that correspond more closely to your desired experimental conditions.

20. Start the calibration scan. Then, press and hold the RESET switch on the TMA until the pen returns to zero. Release the switch and allow the scan to proceed until the pen lifts and starts to return to ambient temperature.
21. Perform Y′-axis and Y-axis (derivative) calculations as follows:

Calculations of Y′-Axis and Y-Axis for Determining Coefficient of Expansion

Calculate the Y′-axis and Y-axis (derivative) coefficient of expansion from the expansion profile by using the following equations. Example calculations are based on the expansion profile shown on page 29.

a. Calculate Experimental Value (α experimental) for the Coefficient of Expansion on the Y′-axis.

Equation 1:

$$\alpha = \frac{\Delta L \times Y' \times ETMA \times K}{\Delta T \times L}$$

Where:

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

ETMA = static calibration coefficient = 1 μm/mV [$10^3\mu$ in/min(in.)]

L = sample length in m(in.)

Δ L = change in sample length in cm of chart

Δ T = change in temperature in °C (°C)

Y′ = Y′-axis sensitivity in mV/cm

K = dynamic calibration coefficient (nom. 1.1 to 1.3)

Metric example:

$$\alpha = \frac{(10.3\ \text{cm})\ (2\ \text{mV/cm})\ (1\ \mu\text{m/mV})\ \times\ 1}{(127°\text{C})\ (7.62\ \times 10^{-3}\text{m})} = 21.2\ \mu\text{m/m°C}$$

1. Select the temperature range of interest in the experiment.The range selected for our example is 100°C to 250°C.
2. Use the table on page 28 to select the temperature (T°) value closest to (but not above) the upper limit of the selected temperature range. For our example, the closest T°C value is 227°C.
3. Draw a horizontal line from the expansion curve at the lower limit of the selected temperature range (100°C) until it intersects with the T°C value selected from the table (227°C). Calculate the degrees centigrade between these points, and apply the result to equation 1 as Δ T. In our example, Δ T = 127°C.
4. Draw a perpendicular line from the intersection point, determined in step 3, up the T°C (227°C) line to the Y′-axis expansion curve. Measure its length in cm (in.). Apply this measurement to equation 1 as ΔL. In our example, Δ L = 10.3 cm.

ALUMINUM COEFFICIENT OF EXPANSION (AMERICAN INSTITUTE OF PHYSICS HANDBOOK, [2nd edition], 4-66 [1963])

T (°C)	μm/m°C (μin./in.°C)
-73	20.0
-23	21.9
27	23.2
77	24.1
127	24.9
227	26.4
327	28.3
427	30.7
527	33.8

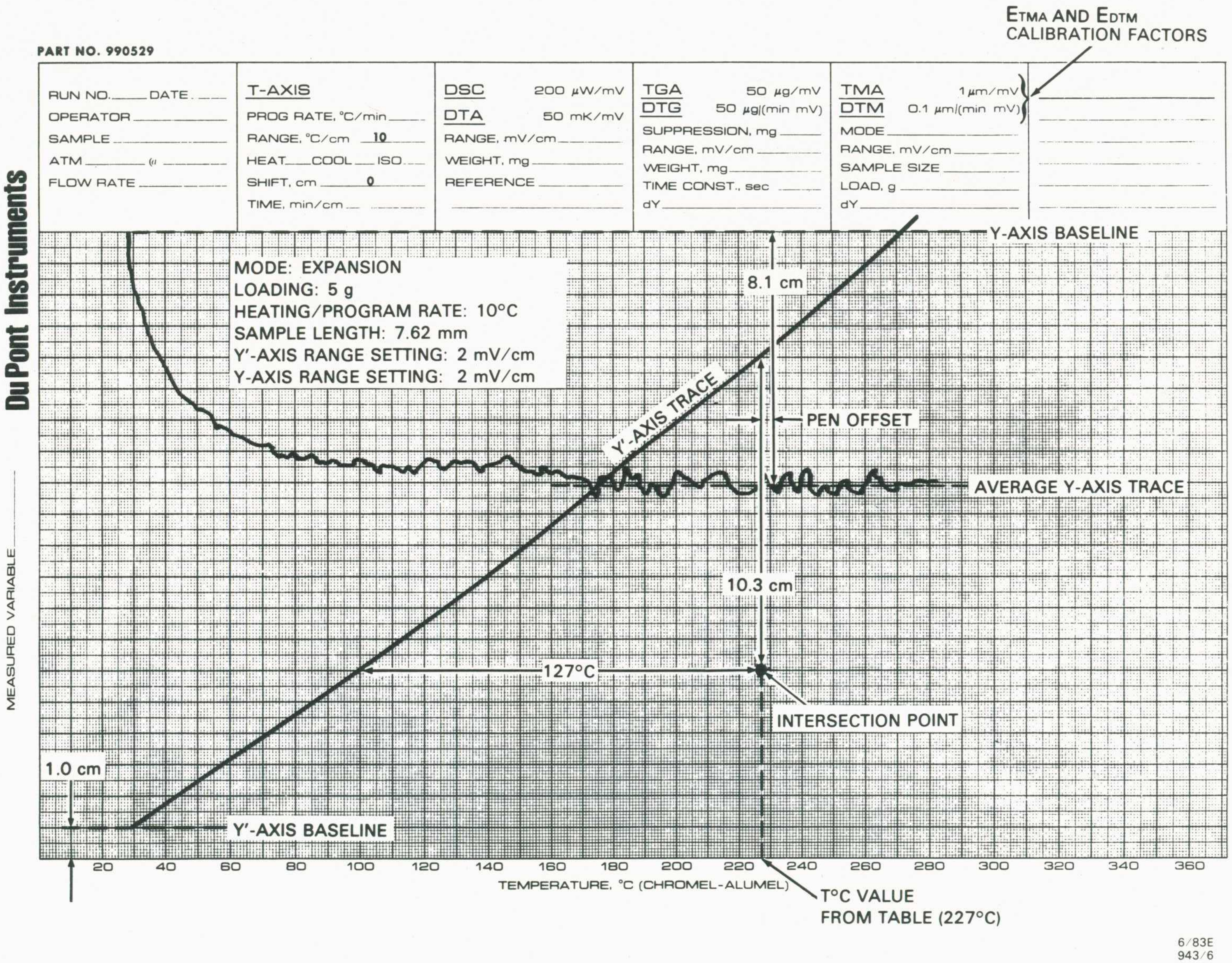
NOTE

Calculate the specific temperature and coefficient of expansion values under investigation by means of interpolating the known values listed in the table.

5. Apply the calibration standard (aluminum cylinder) length measurement to equation 1 as L. Our example measures 7.62 mm.

6. Use the Y′-axis RANGE setting at which the sample scan was run, and apply this to equation 1 as Y. Our example used a setting of 2 mV/cm.

Coefficient of Expansion Profile



7. Use the TMA calibration coefficient factor specified on the chart paper, and apply this to equation 1 as ETMA. The ETMA factor for our example is 1 μ/mV.
8. Assume that the value of K (dynamic calibration coefficient) is equal to 1 and apply this to equation 1 as K.
9. Solve equation 1 and record the result as α experimental for the Y′-axis. The result of our example is 21.2 μm/mm°C.

b. Calculate Experimental Value (α experimental) for the Coefficient of Expansion Y-axis (derivative).

Use the following equation to calculate the experimental coefficient of expansion value for the Y-axis (derivative).

Equation 2:

$$\alpha = \frac{dY \times Y \times EDTM}{R \times L}$$

Where:

α = coefficient of expansion in μm/m°C (μ in./in.°C)

EDTM = static derivative calibration coefficient = 0.1 m/min mV ($10^3 \mu$ in./mil)

L = sample length in m (in.)

R = program rate in °C/min (°C/min)

dY = Y-axis measurement of derivative

Metric example:

$$= \frac{(8.1 \text{ cm})\,(2 \text{ mV/cm})\,(0.1\ \mu\text{m/min mV}) = 21.2\ \mu\text{m/m°C}}{(10\text{°C/min})\,(7.62 \times 10^{-3}\text{m})}$$

1. Apply the calibration standard (aluminum cylinder) length measurement to equation 2 as L. Our example measures 7.62 mm.
2. Use the PROGRAM RATE setting at which the sample scan was run, and apply this to equation 2 as R. Our example uses a setting of 10°C/min.
3. Measure in cm (in.) the Y-axis (derivative) signal at the same T°C (227°C) value, less pen offset, from the derivative baseline to the Y-axis expansion curve. Apply this measurement to equation 2 as dY. In our example, dY = 8.1 cm.

NOTE

The dY (derivative) signal reads the coefficient of expansion directly as displacement from the derivative baseline.

4. Apply the Y-axis RANGE setting at which the sample scan was run to equation 2 as Y. Our sample uses a setting of 2 mV/cm.
5. Use the DTM calibration coefficient factor specified on the chart paper, and apply this to equation 2 as Y. Our sample uses a setting of 2 mV/cm.
5. Use the DTM calibration coefficient factor specified on the chart paper, and apply this to equation 2 as EDTM. The EDTM factor for our example is 1 μm/min mV.
6. Solve equation 2 and record the result as α experimental for the Y-axis. The result of our example is 21.2 μm/m°C.

c. Determine Actual Value (α Actual) for the Coefficient of Expansion

Equation 3:

$$K = \frac{\alpha \text{ actual}}{\alpha \text{ experimental}}$$

Where: K = dynamic calibration coefficient

α actual = actual value of coefficient of expansion in μm/m° (μin/in°C)

α experimental = experimental value of coefficient of expansion in m/m°C (μin/in°C)

Example Y′-axis:

$$K = \frac{26.4}{21.2} = 1.24$$

Example Y-axis (derivative):

$$K = \frac{26.4}{21.2} = 1.24$$

1. Refer to the table and find the μm/m°C value that corresponds to the T°C value (227°C) previously selected. Apply this value to equation 3 as α actual. Our example is 26.4 μm/m°C.
2. Apply to equation 3 the α experimental value for the Y′-axis previously recorded from equation 1 step 9. The experimental value previously from our example was 21.2 μm/m°C.
3. Solve equation 3 to determine the dynamic calibration coefficient (K) value for the Y′-axis.
4. Apply to equation 3 the α experimental value for the Y-axis (derivative) previously recorded from equation 2 step 6. The α experimental value previously recorded from our example was 21.2 μm/m°C.
5. Solve equation 3 to determine the dynamic calibration coefficient (K) value for the Y-axis (derivative). Calculations for the coefficient of expansion are completed. Use the determined K values as a calculation factor in subsequent experiments.
6. Detach the heater assembly, remove the weight from the weight tray, and remove the aluminum cylinder from the sample-holder tube.

Routine Operating Sequence

1. Prepare the TMA with a sample holder and probe.
2. Load the sample into the sample holder.
3. Turn programmer power on.
4. Adjust the probe position control so that the probe almost touches the sample.
5. Zero the electrical signal from the TMA using the transducer position control. The 1090 will allow you to do this using the display axis. Refer to the Y-Axis Static Calibration Procedure, page 20, for more information.
6. Connect the heater to the TMA.
7. Use the TA programmer to set up the thermal run.

Subambient Operation

The TMA can be used when programming from -100°C to 800°C (ambient to 1200°C with optional heater). When an experiment requires heating from subambient temperatures, perform the following steps:

CAUTION

Before operating the TMA at subambient temperatures, read the safety precautions for handling cryogenic material on page iv of this manual.

1. Remove the TMA Dewar flask and heater assembly.
2. Prepare the TMA with a weight tray, the desired probe assembly, and the corresponding sample-holder tube.
3. Place a sample on the TMA sample-holder tube.
4. Set the programmer to STANDBY.
5. Adjust the probe position control so that the probe almost touches the sample.
6. Zero the electrical signal.
7. Connect the heater plug to the TMA heater socket.
8. Set the programmer starting temperature to 30°C below the start temperature required for the experiment.
9. Set the programmer to hold isothermally.
10. Place the Dewar flask next to the TMA and fill it with 5 cm (2 in.) of liquid nitrogen.
11. Lower the heater into the liquid nitrogen until the heater support arm rests on the top of the Dewar flask. Replenish the Dewar flask until the programmer indicates that the start temperature has been reached.

NOTE

To prevent frost from accumulating inside the heater well, **do not** remove the heater assembly when replenishing the Dewar flask with liquid nitrogen. Care should be taken to ensure that as little liquid nitrogen as possible enters the heater well. This will reduce the amount of power applied to the heater to maintain the initial temperature.

12. Remove the heater from the liquid nitrogen, empty any liquid from the heater well, and attach the heater to the Dewar cap.
13. Remove liquid nitrogen from the Dewar flask, then screw the Dewar flask into the Dewar cap.
14. Enter the program parameters for the experiment.
15. Lower the probe until it touches the sample.
16. Adjust the transducer position control back to zero.
17. Press down and release the TMA derivative reset switch.
18. Start the thermal run.

Part 4

MAINTENANCE

Keeping the 943 Working

Maintenance procedures should be performed periodically to keep the 943 in good working condition.

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

WARNING

Do not open the electronics compartment. Because of the high voltages in this instrument, repair procedures requiring access to the electronics compartment must be performed by Du Pont or other qualified service personnel.

CLEANING THE BEARING GUIDES

The surfaces of the bearing guides must be kept clean for the probe assembly to operate properly. If the probe is sticking or moving sluggishly, clean the bearings with Freon® TF degreasing solvent (PN 102401) using a pressurized spray can. This solvent will not harm any internal components.

CAUTION

Remember quartz parts are fragile and should be handled carefully.

Clean the bearing guides as follows:

1. Remove the weight tray, Dewar flask, and heater assembly.
2. Disconnect the thermocouple leads and remove the Dewar cap with the sample-holder tube.
3. Place a piece of cloth on the TMA cabinet directly below the head assembly to catch any Freon® drippings.
4. Remove the retaining nut from the top of the head assembly to expose the upper bearing guide.
5. Spray Freon® TF down into the head assembly at the point where the probe shaft meets the bearing guide. Spray for 10 to 15 seconds. Gently move the shaft up and down while spraying to dislodge any hidden dust particles.
6. Wait about one minute to allow any solvent between the shaft and bearing guide to drain and evaporate.
7. Spray up into the head assembly at the point where the probe shaft meets the lower bearing guide. Again, move the shaft while spraying and allow sufficient time for the solvent to penetrate.
8. Make sure that the probe is centered in each of the bearing guides to prevent binding.
9. Install the retaining nut, weight tray, sample-holder tube, Dewar cap, heater assembly, and Dewar flask.

NOTE

Replace the dust cover when the TMA is not in use. This will prevent dust from settling into the bearing guides.

CLEANING THE PROBE ASSEMBLY

At the end of each experiment, check the probe assembly and the LVDT core for grease or other residue. If the probe is dirty, clean it as follows:

1. Clean the LVDT core and the upper probe with Freon® TF spray, or with acetone applied with a soft brush or cloth.
2. Clean residue from the end of the quartz probe by heating it with a Bunsen burner until the residue evaporates.

NOTE

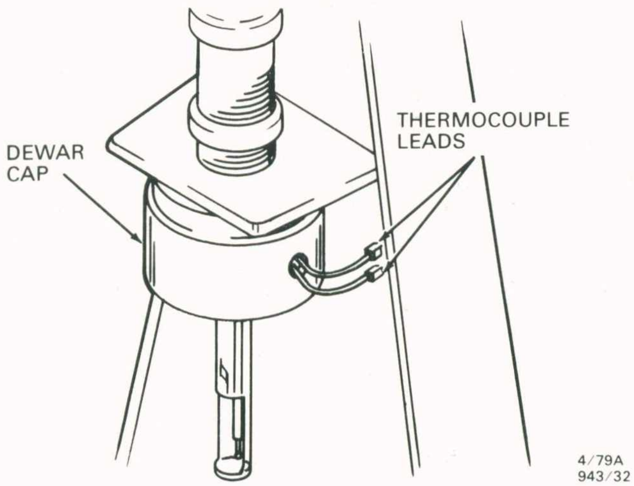
Small amounts of residue on the probe may be removed with acetone.

REPLACING THE SAMPLE THERMOCOUPLE

Remove and replace the sample thermocouple as follows:

a. Removal

   1. Remove the Dewar flask from the Dewar cap.
   2. Disconnect the thermocouple leads and remove the Dewar cap from the head assembly.

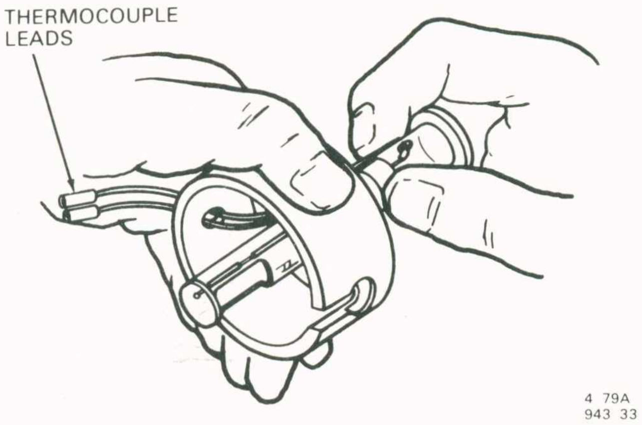


3. Hold the Dewar cap in one hand, and remove the sample-holder tube with the thermocouple from the Dewar cap as shown here.

   Make sure that the thermocouple leads are properly aligned with the hole in the Dewar cap for easy removal.

NOTE

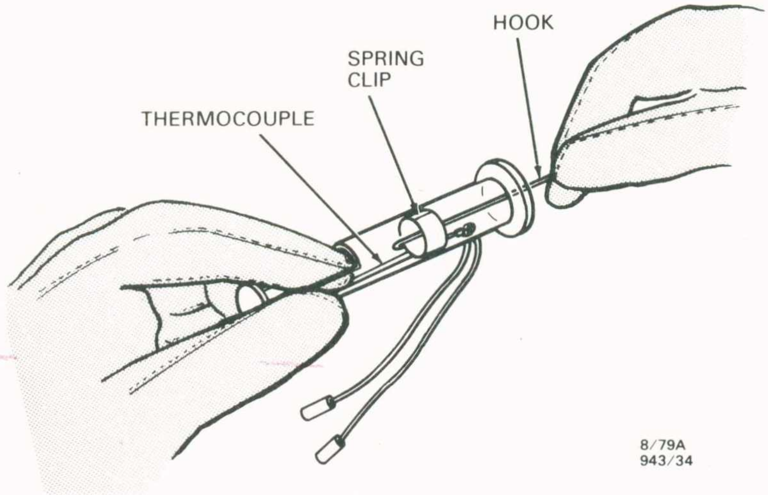
You do not have to remove the two Teflon® washers in the Dewar cap.



4. Holding the sample-holder tube in one hand, insert a hook (which can be fashioned out of a piece of stiff wire) into the top of the sample-holder tube. Remove the spring clip while holding the thermocouple in position.

WARNING

Excessive pressure can break the sample-holder tube; wear protective gloves when removing or installing the spring clip.

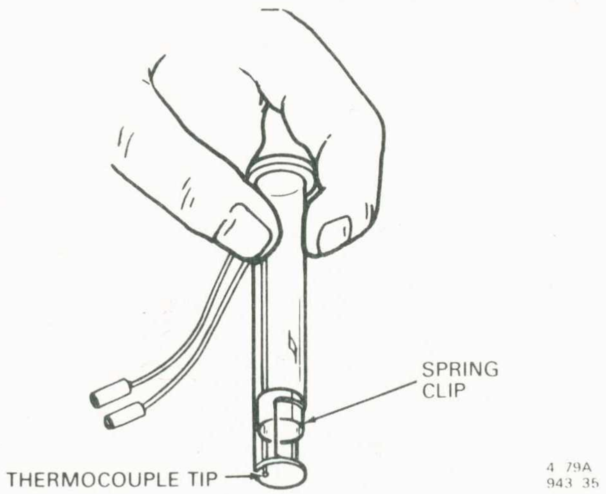


5. Use tweezers to pull the thermocouple out through the bottom of the sample-holder tube. Make sure that the thermocouple leads are properly aligned with the hole in the side of the sample-holder tube when removing the thermocouple.

b. Replacement

1. Insert the thermocouple leads up through the bottom of the sample-holder tube. Then, using tweezers, pull the leads through the hole in the side of the sample-holder tube.

2. Position the thermocouple so that the thermocouple tip is approximately 1.5 mm (0.06 in.) from the bottom of the sample-holder tube. Hold the thermocouple in position and insert the spring clip into the bottom of the sample-holder tube.



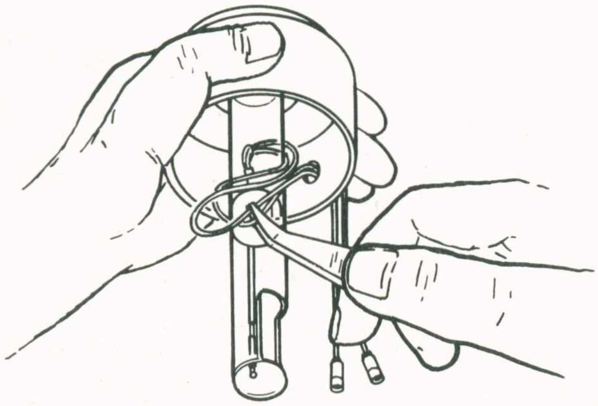
3. Hold the thermocouple in position and insert the hook into the top of the sample-holder tube. Pull the spring clip upward to approximately 6 mm (0.25 in.) from the hole in the side of the tube.

4. Check the position of the thermocouple. If it moves upward while you are installing the spring clip, insert a wooden dowel in the top of the sample-holder tube and carefully push the spring clip down (the thermocouple will also move down) until the thermocouple is in its proper position.

NOTE

To avoid false thermocouple readings, make sure that the thermocouple tip does not touch the sample holder-tube. Exposed portions of the two thermocouple leads (usually at the hole of the holder) must not touch each other.

5. Insert the sample-holder tube and thermocouple leads through the hole in the top of the Dewar cap. Use tweezers to thread the thermocouple leads through the hole in the side of the Dewar cap.



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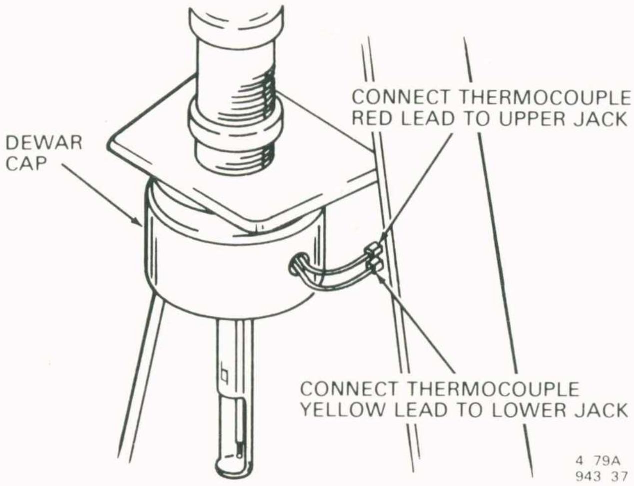
6. Replace the Dewar cap.

7. Connect the red thermocouple lead to the upper jack on the TMA and the yellow lead to the lower jack.



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8. Attach the heater assembly and check alignment. If adjustment is required, refer to page 15.

Part 5

PARTS LIST

Part 5

PARTS LIST

Parts for the 943 System

This section lists replaceable parts for the 943 and its accessories.

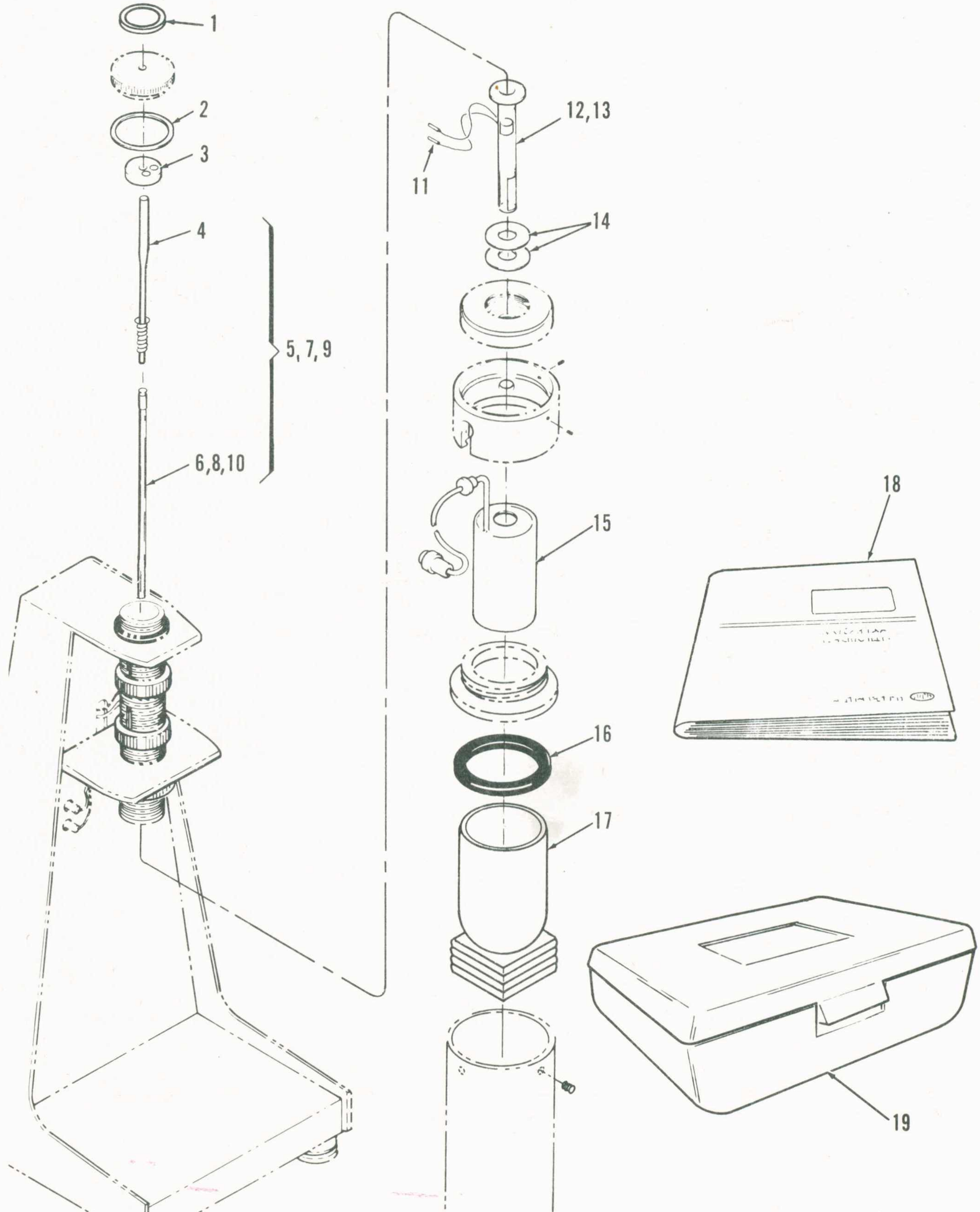
To order replacement parts, contact the nearest Du Pont district office. A list of offices appears on the back cover of this manual.

To ensure that you receive the correct part for your unit, be sure to include the part number and description, as well as the instrument type, model number and serial number.

943 TMA PARTS LIST

Item No.	Part Number	Description
1	940020	WEIGHT TRAY*
2	940078	NYLON® WASHER
3	941024	UPPER BEARING GUIDE
4	941128	UPPER SHAFT ASSEMBLY
5	941082	PENETRATION PROBE ASSEMBLY
6	941078	• Lower Shaft Assembly*
7	941086	EXPANSION PROBE ASSEMBLY*
8	941079	• Lower Shaft Assembly
9	941150	MACRO EXPANSION ASSEMBLY
10	941147	• Lower Shaft Assembly*
11	941136	SAMPLE THERMOCOUPLE, CHROMEL-ALUMEL WITH SPRING RETAINER
12	941099	SAMPLE-HOLDER TUBE WITH THERMOCOUPLE
13	941097	SAMPLE-HOLDER TUBE WITHOUT THERMOCOUPLE
14	940053	TEFLON® WASHERS (2)
15	942050-901	HEATER ASSEMBLY (Standard 800°C)
16	942062	NEOPRENE GASKET
17	942058	DEWAR ASSEMBLY INSERT
18	943145	INSTRUCTION MANUAL
19	941112	TMA ACCESSORY KIT
	942057	• Teflon® Demonstration Sample
	259534	• Circular Level
	940070	• Aluminum Calibration Standard
	259522	• Balance Weights
	259537	• Tweezers
	259521	• Allen Wrench
	259073	• Accessory Kit Case
	942056	• Calibration Shim

*Item included in TMA accessory kit.



Exploded View, 943 System

943 TMA OPTIONAL ACCESSORIES PARTS LIST

Part Number	Description
941131	FILM TENSION PROBE ACCESSORY KIT
941128	• Upper Shaft Assembly*
941139	• Film Tension Lower Probe Assembly
941145	• Film Tension Sample-Holder Assembly
941151-901	• Film Clamp Assembly Set
941133	FIBER TENSION ACCESSORY KIT
941128	• Upper Shaft Assembly*
941140	• Fiber Tension Lower Probe Assembly
941144	• Fiber Tension Sample-Holder Assembly
941038-901	• Vial of Cleaved Aluminum Balls
943010-902	FIBER TENSION SPECTROMETER ACCESSORY KIT
943105	• Load Cell Carrier
943106	• Load Cell Adapter
943107	• Load Cell Weight Tray
943013-902	• Load Cell Assembly, Low Range
943015-901	• Fiber Tension Lower Probe Assembly
943012-901	• Fiber Tension Upper Shaft Assembly
941144-901	• Fiber Tension Sample-Holder Assembly
941038-901	• Vial of Cleaved Aluminum Balls
943118	• Load Cell Key
943010-901	STRESS RELAXATION ACCESSORY KIT
943105	• Load Cell Carrier
943106	• Load Cell Adapter
943107	• Load Cell Weight Tray
943013-901	• Load Cell Assembly, High Range
943014-901	• Stress Relaxation Probe Assembly
943011-901	• Stress Relaxation Upper Clamp Assembly
943016-901	• Stress Relaxation Lower Clamp Assembly
203947-005	• Allen Wrench, 2.38 mm (3.32 in.)
943118	• Load Cell Key
941137	DILATOMETER PROBE ACCESSORY KIT
941128	• Upper Shaft Assembly*
941142	• Dilatometer Lower Probe Assembly
941099	• Sample-Holder Tube with Thermocouple*
941022	• Aluminum Calibration Standard
941148	• Vials of Filling Medium (2)
941143	• Dilatometer Sample Vials (2)

*Item included in TMA accessory kit.

943 TMA OPTIONAL ACCESSORIES PARTS LIST (continued)

Part No.	Description
943138-901	PARALLEL PLATE RHEOMETER ACCESSORY KIT
943136-901	• Rheometer Shaft Assembly (2)
943133-901	• Rheometer Sample-Holder Assembly (2)
943147-901	• Rheometer Sample-Forming Die Set (Pellet Press)
943124	— Pellet Press Cylinder
943122	— Pellet Press Cylinder Head
943123	— Pellet Press Piston
943121	— Pellet Press Base Screw
943125	• Rheometer Alignment Cages (3)
943126	Rheometer Parallel Plates (6)
900902-901	• Indium Metal Standard
900680-901	SAMPLE ENCAPSULATING PRESS
943022-901	1200°C TMA HEATER ACCESSORY
943153-901	• Heater Assembly
943139-901	• Water Cooling Jacket
940053	• Teflon Washers (3)

*Item included in TMA accessory kit.