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Operating the DMA 7e 5

Controls and Indicators

Probe Control Keys

The probe control keys are located on the front panel of the DMA 7e analyzer. A light next to each key is illuminated when the key is selected.

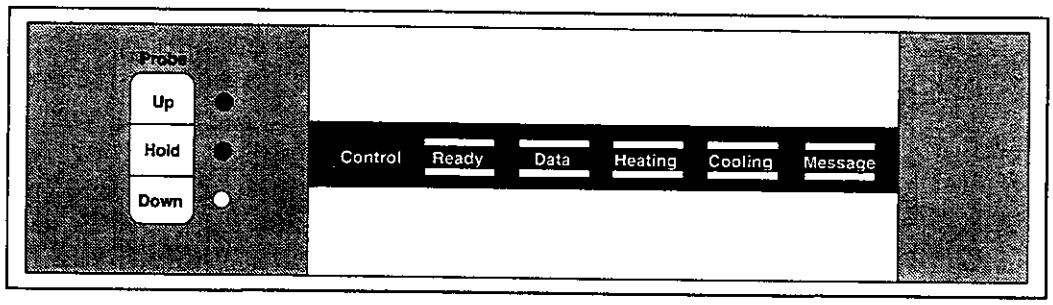


Figure 5-1. Status Indicators and Probe Control Keys.

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The function of each key is described below:

Probe Up — Causes the probe assembly to be raised and locked into place. Selecting **Probe Up** with a sample in place, engages the extension analysis accessory and applies the desired stress to the sample while using the Extension measuring system.

Probe Hold — This key holds the probe assembly at its current location between the Probe Up and Probe Down positions.

Probe Down — This key causes the probe assembly to be lowered to the base of the sample platform (or on the sample currently resting on the sample platform). The weight of the core rod plus the measuring system will be measured by the DMA 7e each time the probe is lowered (except when using the Extension measuring system). This allows accurate measurement of the system inertia.

Status Indicators

The temperature program that controls the DMA 7e is specified on the computer by use of the 7 Series/UNIX software. The current status of the DMA 7e will always be displayed in its status window at the top of the computer screen. To quickly inform you of the analyzer's status, six additional status indicators are located on the DMA 7e front panel:

Control — The DMA 7e temperature sensors are in control of the temperature; power is being supplied to the furnace to maintain the program temperature selected on the computer. The Control light should always be illuminated in conjunction with the Heating or Cooling and/or Data light during the main section of a temperature program run.

NOTE: The Control indicator may go out momentarily at the beginning of a run.

NOTE: The temperature at which the Control indicator lights and, therefore, the acceptable temperature range of the DMA 7e, is dependent upon the type of coolant or cooling accessory that you use. For example, with an ice water bath in the DMA 7e dewar, temperatures as low as 20 °C can be attained. With liquid nitrogen as the coolant, temperatures as low as -170 °C can be attained.

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Ready While blinking, Ready means that the program temperature of the DMA 7e has reached the temperature defined by the user. When the Control light is also lit, you are ready to begin the temperature program.

Upon startup of the 7 Series Thermal Analysis System, the Ready indicator should be the only indicator that is illuminated. (In some cases the Control indicator may also be illuminated upon startup depending on the heatsink and coolant temperature.)

Data While lit, Data means that data is being taken. The data light remains unlit at all other times.

Heating When illuminated, Heating indicates that the DMA 7e is heating under program control.

Cooling When illuminated, Cooling indicates that the DMA 7e is cooling under program control.

Message While blinking, Message means that an informative message must be acknowledged at the computer. Message is also an indication that the measuring system and sample may be stuck if the sample has melted and fused to the probe tip. In such a case, detach the probe tip from the probe holder (for a stainless steel measuring system), and remove the sample from the probe tip outside of the DMA 7e.

NOTE: Refer to Chapter 9 for a description of the use of the status indicators as diagnostic tools.

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Table 5-1. Summary of the DMA 7e Status Indicators

Indicator	Blinking	On	Off
Control	Does not blink	Power is being supplied to the furnace.	1. Analyzer is not in temperature control. 2. Power is not being supplied to the furnace.
Ready	Program temperature has reached Load Temperature.	1. Analyzer temperature has reached Load Temperature.	1. Analyzer temperature has not reached Load Temperature.
		2. Power is being supplied to DMA 7e.	2. Power is not being supplied to DMA 7e.
			3. The wrong power-on sequence was performed or cable is not connected properly.
Data	Does not blink	Data is being taken from the DMA 7e.	Data is not being taken from the analyzer.
Heating	Does not blink	Analyzer is heating under program control at the selected rate.	Analyzer is not heating.
Cooling	Does not blink	Analyzer is cooling under program control at the selected cooling rate.	Analyzer is not cooling.
Message	A message has been sent to the computer.		No message is waiting.

NOTE: If the indicators are blinking in sequence, the DMA 7e is calibrating. Do not touch the front panel of the analyzer.

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Using the Screen Display, Keyboard, and Mouse

Using the DMA Status Windows and Status Lines

The Status Windows and Status Line display analyzer specific information.

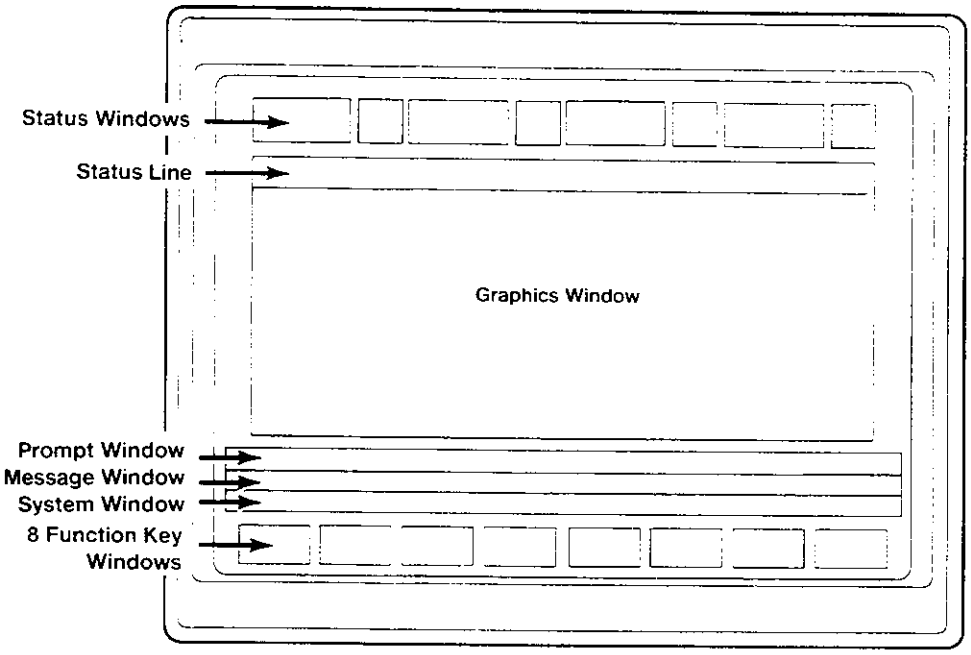


Figure 5-2. Location of the Status Windows and Status Line on the Screen.

Status Windows

When you select **Run System** from the Main menu you can choose the number of tasks such that a Status Window appears for each task. A task would be selected for each analyzer you wish to run as well as any unassigned, or "Free", tasks that you may use to do post-run analyses. For example, you can have up to 4 DMAs connected to the system (i.e., tasks 1-4) and while you are collecting data you can perform post run anlysis by selecting a "Free" task (task 5). The status windows indicate the current status of the task or analyzer that is connected to the task.

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The background color for a Status Window can be one of two colors:

- red - is the current task or the task that is currently using the main portion of the screen.
- blue - is the task currently operating independently in the background.

You can use the mouse or the **Ctrl** and numeric keys to select or change tasks. If you are using the mouse, point to the status window you wish to select and left-click or hold down the **Ctrl** key and press the appropriate numeric key (1-8, depending on the number of tasks entered in the Main menu). To change to Task 3, for example, hold down the **Ctrl** key and type **3**. To fully expose a partially hidden status window without making it active, point to the window and right-click.

There are two lines of information in each status window:

- Line 1 displays the task number (1=task 1, 2=task 2, etc.), the analyzer identification letter (A, B, C, etc. in case two or more of the same analyzer are being run from one workstation), and the current X-axis value.
- Line 2 displays the name of the analyzer being monitored (DSC, TMA, TGA, DMA or Free if an analyzer is not connected) and the current Y reading.
- If a task is free, the filename of curve 1 and curve 2 will appear on lines 1 and 2, respectively.

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Status Line

The status line displays all the important real-time program, calculated, and measured parameters. There are four status lines that appear on the screen, one at a time. To toggle through the four status lines, simultaneously press the **Ctrl** and = keys. The four status lines are shown below:

STATUS LINE 1

Heat, Hold, cool status and run time.

Temp Program Status | Temp | Freq | S.Stress | D.Stress | Strain | Height | Amplitude | Phase

STATUS LINE 2

Temp Program Status | F_s | F_d | F_t | f_o | Height | Amplitude | Phase

STATUS LINE 3

Temp Program Status | K_s | D_s | T_b | Height | Amplitude | Phase

STATUS LINE 4

Temp Program Status | K_o | D_0 | Height | Amplitude | Phase

Some of the parameters that appear on the status lines are defined below. Refer to Appendix 5, Operational Guidelines, and the Glossary for more detailed information on all of the parameters.

F_s	Program static force
F_d	Program dynamic force
F_t	Program total force
f_o	Sample/Analyzer resonant frequency
k_s	Sample spring constant
D_s	Sample damping
T_b	Block temperature
k_o	Analyzer spring constant
D_o	Analyzer damping
Height	Average probe position
Amplitude	Average probe amplitude
Phase	Sample phase lag

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Using the Keyboard Function keys

The keyboard function keys are located on the upper portion of the computer keyboard. All of the control function keys may not be active at all points in the program.

A description of each keyboard function key and its general use is described below. The names and locations of the keyboard function keys are printed on the keyboard overlay shipped with your system (see next figure).

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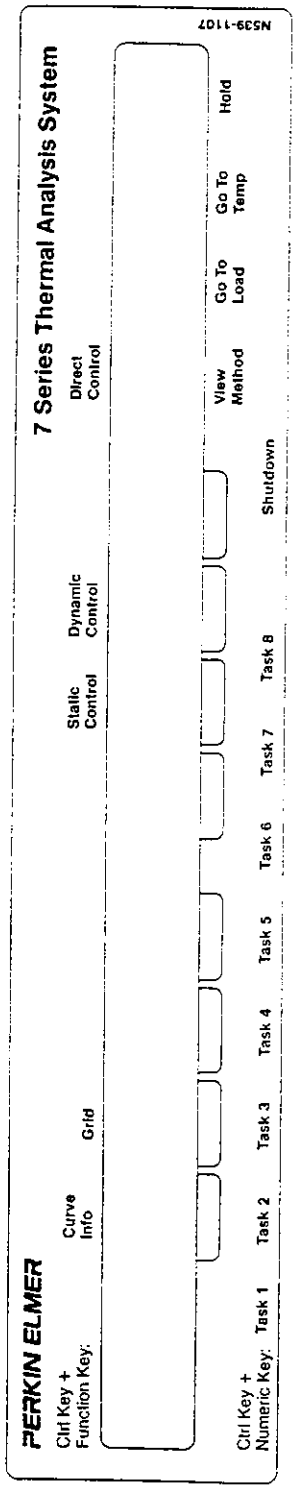


Figure 5-3. The 7 Series Keyboard Overlay.

Some functions are activated by simply pressing the function key. The **View Method**, **Go to Load**, **Go to Temp**, and **Hold** options, printed in blue directly below the overlay cutout only require the use of a function key (**F9 - F12**).

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Some functions require you to hold down the **Ctrl** (Control) key and then press a numeric key. If you wish to shut down the 7 Series software, for example, hold down the **Ctrl** key and type **0**. To select or change tasks, hold down the **Ctrl** key and type the appropriate number (1-8).

Other functions, such as **Curve Info**, **Grid**, and **Direct Control**, require you to hold down the **Ctrl** key and press a function key.

The color code for the keyboard overlay is as follows:

- Blue - Just press a function key to perform an action.
- Black - Press the **Ctrl** key and the function key, simultaneously.

The following table summarizes the use of the function keys:

Function Key 1-8

Keys F1 through F8 are menu-driven function keys that change their function as the menus change.

Menu-driven function keys appear in function key windows at the bottom of the computer screen. They are continually updated as you select options in the system. Select menu-driven function keys by either pressing the corresponding function key on the keyboard or pressing the left mouse button with the screen cursor positioned on the desired function key window.

When a menu-driven function key is selected, one of three actions will occur:

- The system will prompt you for information
- The system will perform an action.
- A new menu will be displayed with different options.

Curve Info (Ctrl–F1)

When a curve or curves are displayed on the graphics screen, the sample identification for the first curve is displayed at the top left corner of the screen above the Y1 axis, and the sample identification for the second curve, if there is one, is displayed at the top right corner of the screen above the Y2 axis.

When you select the **Curve Info** key, the curve descriptions for all recalled files will be redisplayed in a column on the right side of the graphics screen. Each curve description consists of two lines. The first line displays the curve number and the entire sample identification up to 40 characters. Also, the first line

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will contain the file name if the fileID and filename are ≤40 characters. The second line displays the ordinate unit of the curve and the segment number(s). If the combined length of bothordinate unit and segment number(s) exceeds 40 characters, only the ordinate unit will be displayed.

Selecting the **Curve Info** key toggles between the above curve information displays.

Grid (Ctrl-F2)

The **Grid** key displays a grid inside the frame used for display of the graphics. Use this key to turn the grid on and off.

Static Control (Ctrl-F6)

Use the **Static Control** key to turn on the applied static force necessary to maintain sample to probe contact without causing penetration (or buckling with the Extension measuring system) of the sample. The **Static Control** key toggles on and off. (Although the control value is set in the Modify Parameters menu, it does not become active until the **Dynamic Control** key is toggled on.) Refer to the *7 Series / UNIX DMA 7 Temp / Time Software Users Manual* (Part No. 0993-8679) or another appropriate software mode manual for details.

Dynamic Control (Ctrl-F7)

Use the **Dynamic Control** key to turn on the dynamic force control at a preset value. The **Dynamic Control** key toggles on and off. (Although the control value is set in the Modify Parameters menu, it does not become active until the **Dynamic Control** key is toggled on.) Dynamic Control is turned off when you press the **Dynamic Control** key. Refer to the *7 Series / UNIX DMA 7 Temp / Time Software Users Manual* (Part No. 0993-8679) or another appropriate software mode manual for details.

Direct Control (Ctrl-F9)

The **Direct Control** key is functional when a GSA 7 Gas Selector Accessory is connected to the DMA 7. The **Direct Control** key is used to select Gas A or B as the primary purge gas.

View Method (F9)

The **View Method** key displays the complete Method Form, which contains the parameters and variables used for a sample run.

While setting up a method to be run or recalling a method, use this key to toggle between the Set Up and Run values and the X-Y axis that will be graphically displayed on the screen when the method is run.

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While working with a data set that has been collected, use this key to toggle between the method that was used to collect the data set and the graphic display of the data.

Go to Load (F10)

Automatically heats or cools the analyzer to the load temperature and applies the load conditions (stress (or force) and frequency). If a run is in progress, (F10) will terminate the run and heat or cool the analyzer to the load temperature and apply the default stresses (or forces) and frequency. If your default stress or force is set to zero, this key can be used to "turn off" the motor. You can set the value of the load temperature in the top level **Change Defaults** menu.

Go to Temp (F11)

Use the **Go to Temp** key to heat or cool the analyzer to the temperature you enter. If a run is in progress, selecting **Go to Temp** will terminate the run and heat or cool the analyzer to the temperature entered. The stresses (or forces) and frequency will remain the same. If a run is not currently in progress, and **Go to Temp** is selected, then the analyzer is heated or cooled to the temperature entered.

Hold (F12)

Use the **Hold** key to automatically hold the analyzer at the current temperature program. If a run is in progress, and **Hold** is seleced, the run is terminated and the analyzer is held at the current temperature. The stresses (or forces) and frequency will remain the same. If a run is not in progress, and **Hold** is selected, then the analyzer is automatically held at the current temperature. The stresses (or forces) and frequency will remain the same.

Shutdown (Ctrl-0)

The **Shutdown** key shuts down the 7 Series software and returns you to the Main menu.

You should shut down the 7 Series software when you have completed your work. Make sure that you are finished collecting data before shutting down. Otherwise, you will lose the data you have started to collect.

When the shutdown has been completed, you can select another command, enter DOS or UNIX, or shut down UNIX.

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The Method Display

When you press the **Set Up And Run** function key to set up a DMA 7e method, the method display appears. Since the parameters cannot all fit on one screen, they are displayed on the screen as two pages. The figure below shows the two pages for a DMA 7e Temp/Time Scan Mode method.

(Sometimes you can position the pointer by using the **Tab** or **Shift-Tab** keys (e.g., in a Method display)).

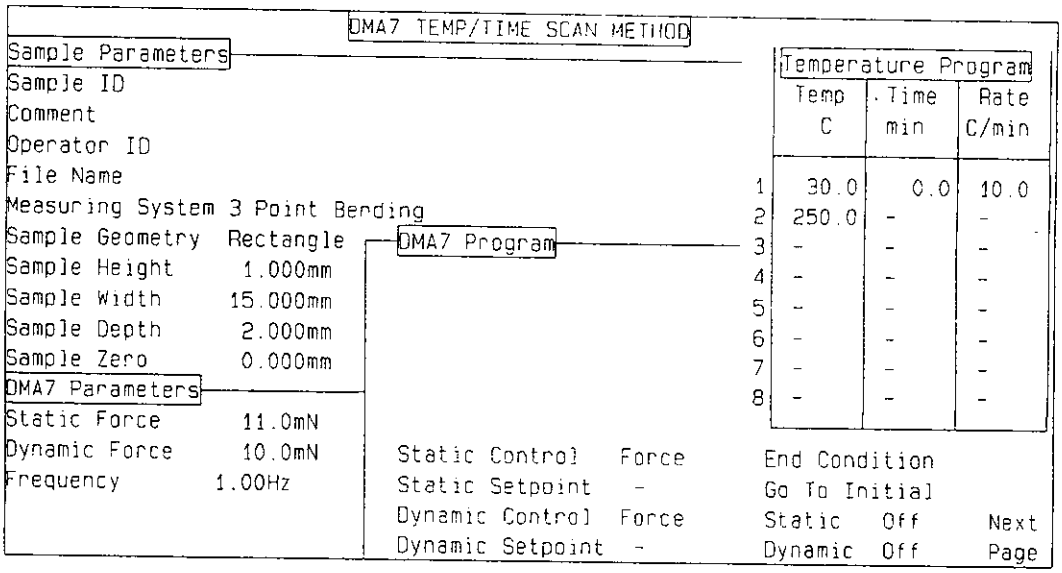


Figure 5-4. Page one of the DMA 7e Temp/Time scan mode method.

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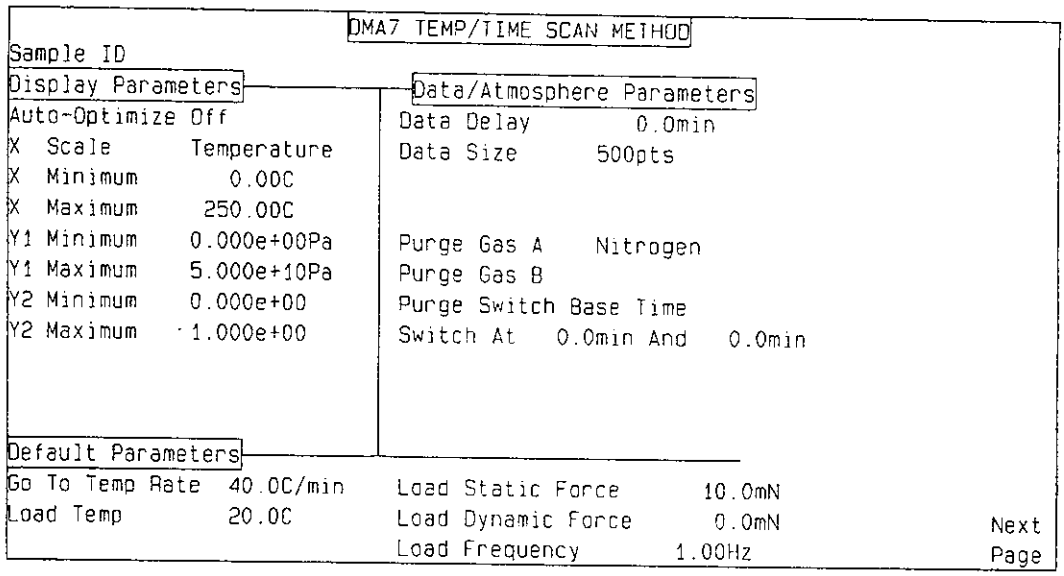


Figure 5-5. Page two for a DMA 7e Temp/Time scan mode method.

The parameters that appear on the method display are organized into the following blocks and are accessed in the sequence shown:

- Sample parameters
- DMA 7 parameters
- DMA 7 program
- Temperature program
- Display parameters
- Data/Atmosphere parameters
- Default parameters

Page 1 of the DMA 7 method display shows the DMA 7-specific parameters and programmable parameters that are changed most often. Page 2 shows the display and data parameters, as well as the default parameters. To move from one page to the other, press the **Page Up** or **Page Down** function key.

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NOTE: Some of the parameter blocks listed above do not appear in the method displays for all DMA 7 modes. Refer to the individual DMA 7 Mode manual for details.

Moving Around in the Method Display

When a parameter is selected, it becomes the active parameter. This section describes how to move around in the method display and activate different parameters.

To move:

- from one block of parameters to the next, press the **Tab** key.
- to the previous block in the sequence, simultaneously press the **Shift** and **Tab** keys.
- between columns in the Temperature Program block, use the **left and right cursor** keys.

To activate:

- the next parameter in a block, press the **down cursor** key.
- the previous parameter, press the **up cursor** key.
- the first parameter in the next block when the last parameter in the previous block is active, press the **down cursor** key.
- the last parameter in the previous block when the first parameter in the next block is active, press the **up cursor** key.
- the first parameter in the current block, press the **Home** key.
- the last parameter in the current block, press the **End** key.

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Entering Parameter Text and Values

Parameters require either text entries (for example, Sample ID) or numerical entries (for example, Sample Height). Some parameters have values that can be scrolled by pressing the space bar on the keyboard (for example, End Condition). When you enter text, it appears in the prompt window as you type.

Parameters Entered by Pressing Space Bar

- Measuring System
- Sample Geometry
- Static Control
- Static Setpoint
- Purge Switch Base
- Dynamic Control
- Dynamic Setpoint
- End Condition
- X Scale

If you enter an invalid number into a parameter entry field, the following message appears in the message window:

You have not entered a valid number. Please try again.

If you enter a number into a parameter entry field that is out of the acceptable range, the following message appears in the message window:

Your number must be in the range <low value> to <high value>. Please try again.

<low value> represents the lowest allowed value and *<high value>* represents the highest allowed value.

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Operating Variables

GSA 7 Gas Selector Accessory

The GSA 7 Gas Selector Accessory is a computer-controlled gas switching device. The GSA 7 permits the automatic switching of sample purge gases at an operator-selected time or temperature during an analysis. In the DMA 7e Method you can use the following parameters to specify the purge gas:

- **Purge Gas A** - Type the name of the gas connected to valve 1.
- **Purge Gas B** - Type the name of the gas connected to valve 2 (or bypasss this entry if you are using only one purge gas).

In the Method, **Purge Switch Base** can be toggled to *Temperature* or *Time* and simultaneously, **Switch At** (*x °C And x °C,* or *x.x min And x.x min*) is toggled to the appropriate units.

- For **Purge Switch Base** *Temperature* -

  Switch At *x °C And x °C* allows you to type the temperature, in *°C*, during the run when the first gas will be switched to the second gas. The next entry for temperature (following the *And*) specifies the temperature at which the second gas is switched back to the first gas (if you type, 0 *°C* this would indicate that the run will finish with the second gas and so will not switch back to the first gas until the end condition is met).

OR

- For **Purge Switch Base** *Time* -

  Switch At *x.x min And x.x min* allows you to type the time, in minutes, into the run when the first gas will be switched to the second gas. The next entry for minutes (following the *And*) specifies the time **after the first switch** that you wish to switch back to the first gas (if you type, 0.0min this would indicate that the run will finish with the second gas and so will not switch back to the first gas until the end condition is met).

Apply the following equation to calculate the number of minutes into the run when you wish the first gas to be switched to the second gas:

Temperature Span (°C) ÷ Scan Rate (°C/min) = Time (minutes)

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For example, if you wish to heat from 30°C to 130°C at 20°C/min in nitrogen (first gas) and then switch to oxygen (second gas):

(130°C - 30°C) ÷ 20°C/min = 5 min

Therefore, the Method parameters would be set as follows:

- **Purge Gas A** - *Nitrogen*
- **Purge Gas B** - *Oxygen*
- Press the spacebar to toggle **Purge Switch Base** to either *Time* or *Temperature*
    - If you select **Purge Switch Base** *Time* then **Switch At** *5.0 min And 0.0 min*

    OR

    - If you select **Purge Switch Base** *Temperature* then **Switch At** *130 °C And 0 °C*

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Measuring Systems

A wide variety of stainless steel (gold-colored coating) and quartz measuring systems are available for use with the DMA 7e to meet specific applications needs. Each measuring system is easily installed and removed to facilitate changing the measuring systems and cleaning them outside of the analyzer. A short description and an illustration of each measuring system is provided on the following pages. Refer to Appendix 2 for illustrated parts lists of all measuring systems.

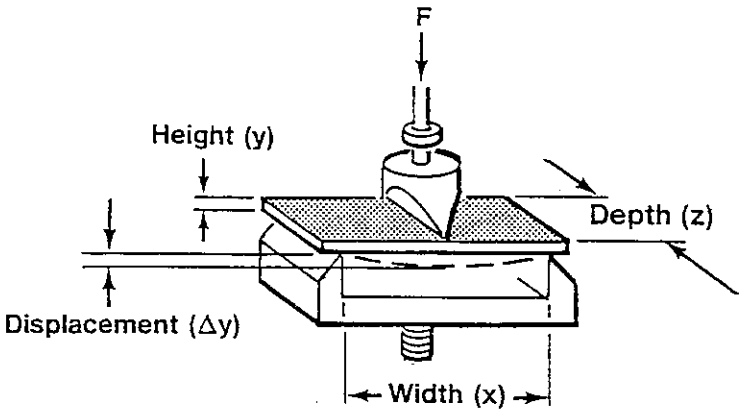
Three-Point Bending

The Three-Point Bending measuring systems are used to perform many of the classical mechanical testing applications. They are typically used with high modulus materials such as engineering thermoplastics, resins and composites. Sample geometries include solid materials, such as sheetstock, as well as wire, rods and tubes.

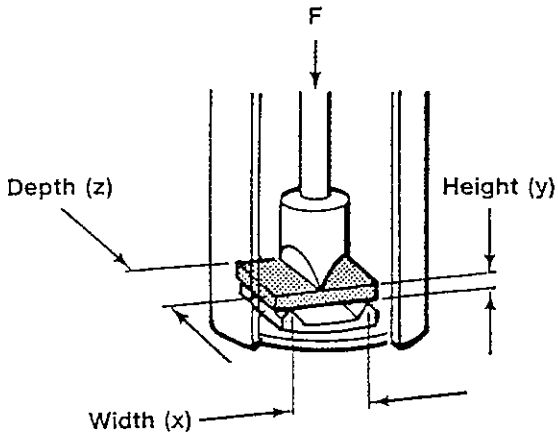
Three different Three-Point Bending Measuring System Kits are available. The Stainless Steel Three-Point Bending Kit (Part No. N539-0130) is provided with the DMA 7e analyzer. The kit contains: a 3-mm sphere probe tip, 5-mm and 10-mm knife probe tips, and 5-mm, 15-mm, and 20-mm bending platforms. An optional 10 mm bending platform is also available.

The Quartz Small Bending Kit (Part No. N539-0137) and Quartz Large Bending Kit (Part No. N539-0136) have a 1-mm round probe and 3-mm and 5-mm knife probes. The Small Quartz Bending Kit is used with analyzers that have a high-temperature DMA 7e furnace. The Large Quartz Bending Kit is used with analyzers that have a standard DMA 7e furnace.

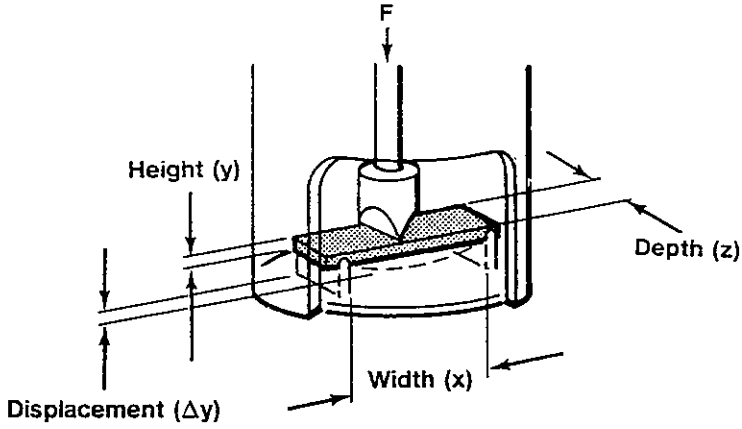
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A. Stainless Steel



B. Quartz Small Bending



C. Quartz Large Bending

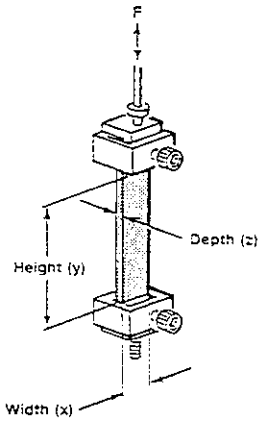
Figure 5-6. Three-Point Bending Measuring Systems.

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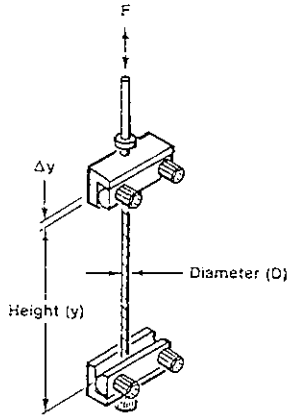
Extension Analysis

The Extension Analysis measuring systems are used to study low modulus materials such as thin films, textile and other fibers, and hair. The Stainless Steel Extension Kit (Part No. N539-0132) has two clamping fixtures. The Parallel Fixture provides flat parallel clamping surfaces for use when analyzing films and thin materials. The Tangential Fixture provides tangential clamping surfaces for analyzing materials such as fibers and wire.

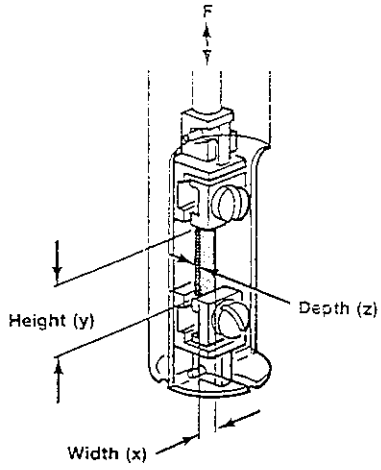
The Quartz Extension Kit (Part No. N539-0134) has a clamp assembly with top and bottom clamps for samples such as films that require flat, parallel clamping surfaces. Fiber pins are also provided and are recommended for very thin samples.



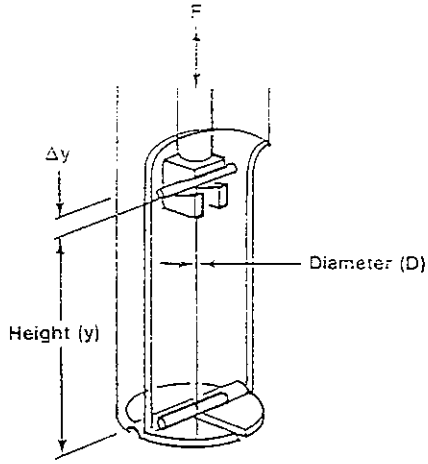
A. Stainless Steel Parallel Clamp



B. Stainless Steel Tangential Clamp



C. Quartz Film Clamp



D. Quartz Fiber Pin Clamp

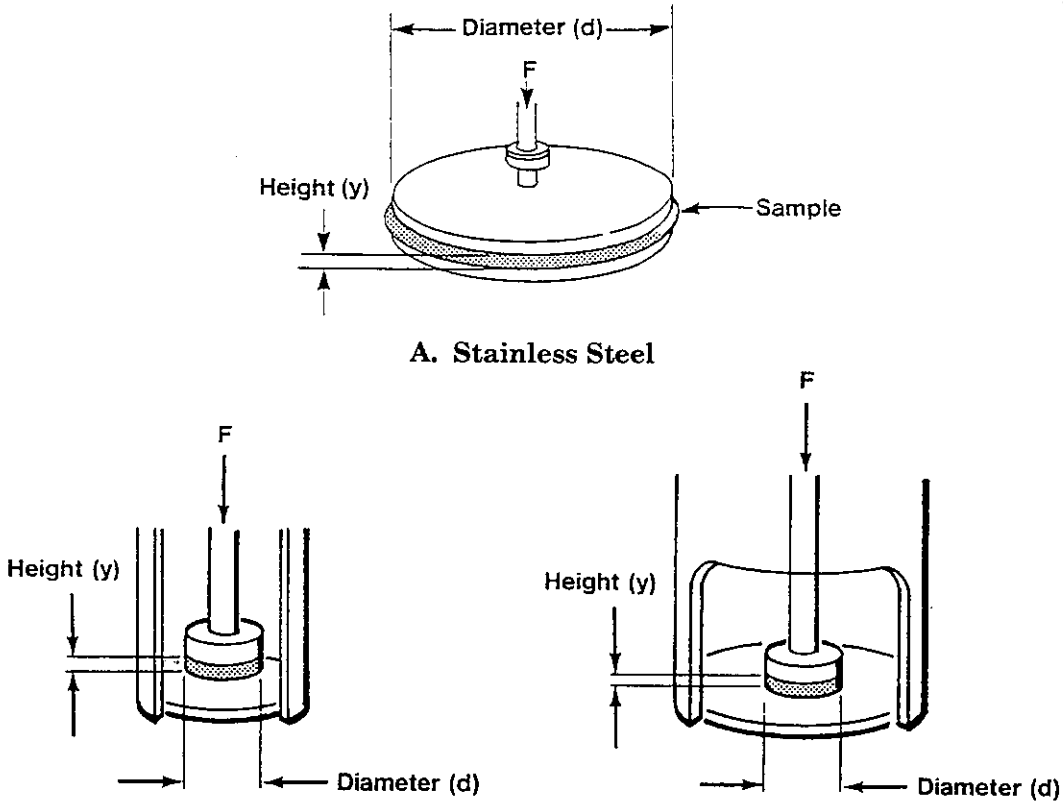
Figure 5-7. Extension Analysis Measuring Systems.

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Parallel Plate

The Parallel Plate measuring systems are used to study materials ranging from those with honey-like consistency to thermoplastics and rubbers above their glass transition temperature. Three different Parallel Plate Measuring System Kits are available. The Stainless Steel Parallel Plate Kit (Part No. N519-0133) has stainless steel parallel plates with diameters of 1, 3, 5, 10, 15, and 20 mm. With unique mounting fixtures, the parallel plates can be easily aligned to provide exact positional location for the most accurate measurements.

The Quartz Large Parallel Plate Kit (Part No. N519-0135) and Quartz Small Parallel Plate Kit (Part No. N519-0138) have quartz probe plates with diameters of 1, 3, and 6 mm. The Quartz Small Parallel Plate Kit is used with analyzers that have a high-temperature DMA 7e furnace. The Quartz Large Parallel Plate Kit is used with analyzers that have a standard DMA 7e furnace. Optional accessories available for the Parallel Plate Measuring System include: cup and plate accessories, parallel plate with tray accessories, and scintered parallel plates.



A. Stainless Steel

B. Small Quartz

C. Large Quartz

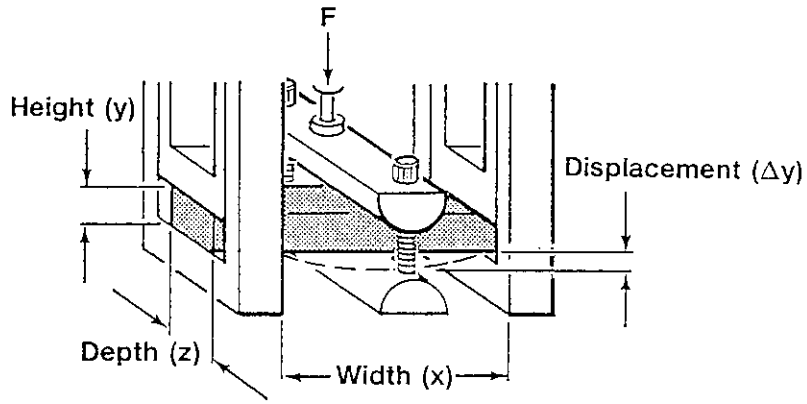
Figure 5-8. Parallel Plate Measuring Systems.

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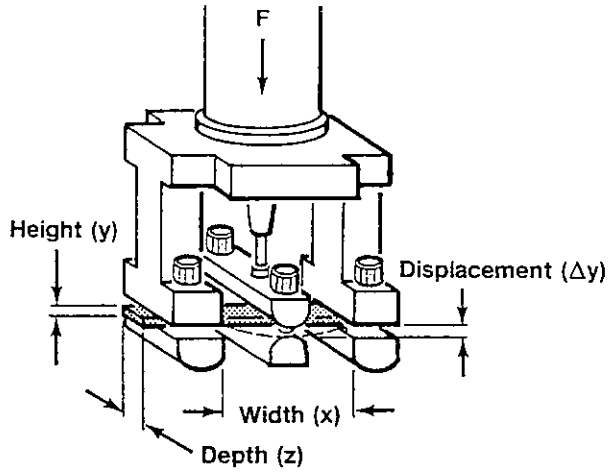
Dual Cantilever

The Dual Cantilever measuring system is used to study materials with mid-range modulus such as elastomers and materials above their glass transition temperature. This measuring system uses a clamping assembly with a hemispherical center clamp that increases effective sample size and reduces localized clamping stress.

The Dual Cantilever Kit (Part No. N539-0131) comes with two sample tube assemblies; one is recommended for high modulus samples and the other for low modulus samples.



A. Quick Clamp Sample Tube for Low Modulus Samples



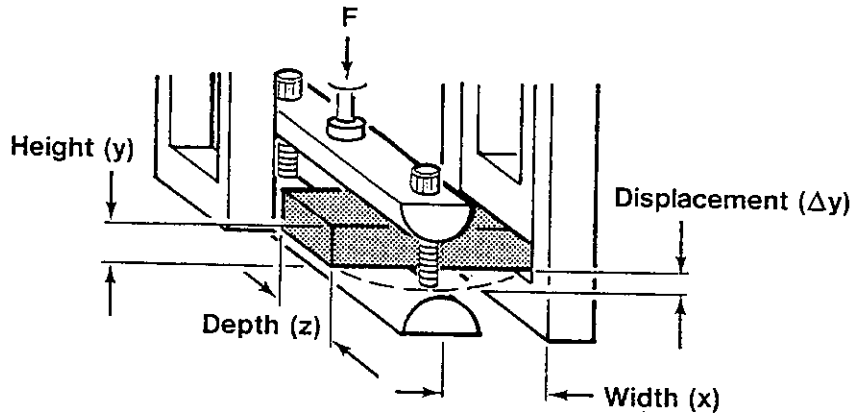
B. High Modulus Sample Tube

Figure 5-9. Dual Cantilever Measuring Systems.

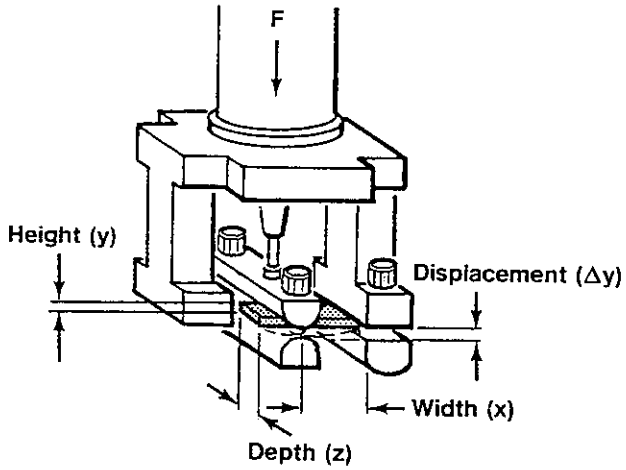
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Single Cantilever

The Single Cantilever measuring system is used for the same classical mechanical testing applications as the Dual Cantilever measuring system. When only one side clamp is attached, the Dual Cantilever can be used as a Single Cantilever measuring system.



A. Quick Clamp Sample Tube for Low Modulus Samples



B. High Modulus Sample Tube

Figure 5-10. Single Cantilever Measuring Systems.

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Positioning of the Sample Thermocouple

A small thermocouple "pocket" has been added to the inside side wall of the sample tube (Figure 1). The pocket allows the user to place the thermocouple in the optimum position every time with significant improvement of results. Although this change greatly improves sub-ambient operation when using liquid Nitrogen, it also improves thermal performance over the entire temperature range (-170 C to +500 C).

Route the thermocouple as you normally would (described on page 5-25). Rather than placing the thermocouple in the area near the sample (described on page 5-27, Step 8), place the tip of the thermocouple all the way down into the bottom of the thermocouple "pocket." Run the DMA 7e as you normally would.

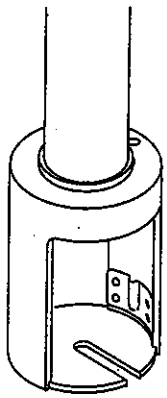


Figure 1. DMA 7e Sample tube with thermocouple pocket.

Hints for Optimum Thermal Performance

1. Be sure the control light is illuminated before beginning a run.
2. Check your purge gas regularly. Variations in the purge rate can cause nonuniformities in the furnace temperature.
3. Use Helium purge gas whenever possible. Helium provides optimum thermal conductivity at low, moderate and high temperatures. Nitrogen should be used when operating to very high temperatures (i.e. above 500 °C).
4. Heat at reasonable heating rates. Heating rates are typically 10 °C/min. for the TMA 7 (i.e., when using the quartz measuring system and small furnace) and 5 °C/min. for the DMA7e (i.e., when using the stainless steel measuring system and large furnace).
6. Avoid bending or kinking the thermocouple during use or installation.
7. Do not move the thermocouple after calibrations have been made.
8. When installing a new furnace, or changing a furnace, use heat sink compound. Apply it to the furnace mounting area, around the furnace leads at the heat sink, the base of the heat sink and to the heat sink mounting block.
9. Avoid overfilling of the dewar. If the dewar is overfilled, liquid can come in contact with the furnace. Liquid can also come in contact with the furnace by seeping between the walls of the dewar liner and the dewar insulation. If liquid enters the furnace, it can cause premature failure or damage.

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Installing the DMA 7e Thermocouple

Follow the instructions below when installing or replacing the DMA 7e sample thermocouple.

Although the calibration software can compensate for thermocouple placement, careful routing and placement of the thermocouple can improve thermal performance of the DMA 7e, especially when stainless steel measuring systems are used.

A thermocouple is a device that generates a differential voltage as temperature is increased or decreased. The voltage is generated by fusing wires made from two different metals at the tip. When this device is repeatedly flexed, the point at which the two wires are fused is stressed and can occasionally fail. Restricting the movement of a thermocouple can greatly improve its service life. The high temperature tape, provided in the Spares Kit, restricts the movement of the thermocouple when placed at the top and bottom of the sample tube. This tape also serves as an insulator.

IMPORTANT: The thermocouple should not touch any metal parts.

Installing the Thermocouple

1. Remove the furnace diffusion cap and carefully lower the furnace assembly to the base of the DMA 7e by pressing the furnace locking mechanism. Make sure that the furnace locks in place when it reaches the base of the DMA 7e.

2. Press the locking button that secures the coupling access covers and separate the doors.

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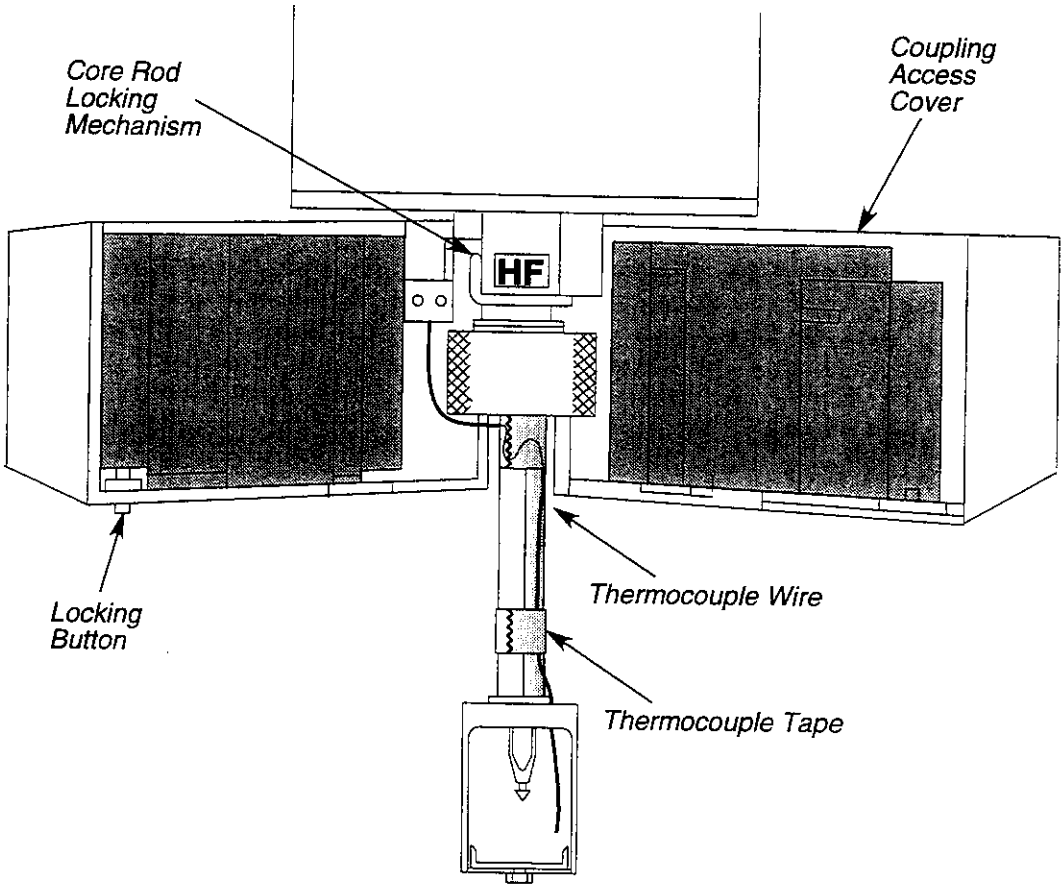


Figure 5-11. View of DMA 7e after opening the Coupling Access Covers.

3. Plug the new thermocouple into the receptacle at the back of the probe assembly. Observe the polarity (+ or –) as marked on the plugs.

4. Apply a piece of tape along the length of the sample tube as shown in the next figure. This will insulate the thermocouple from the sample tube.

5. Route the thermocouple along side of the sample tube on top of the tape as shown in the next figure, making sure to include the loop at the top to prevent tip movement.

   NOTE: Do not route the thermocouple through the mounting bracket. This arrangement will let you remove the sample tube from the analyzer without disturbing the thermocouple route or position. Remember, do not make any sharp bends in the thermocouple. This might damage the thermocouple.

6. Place the thermocouple insulator (a white ceramic sleeve about 2 mm long and 1 mm wide) in the hole in the top of the sample tube.

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7. Route the tip of the thermocouple through the insulator.

8. Ensure the thermocouple tip is placed 12 mm (± 1 mm) above the base of the sample tube and 2 mm (± 0.5 mm) from the sample tube wall.

9. Apply another piece of tape over the thermocouple and over the first piece of tape. This will hold the thermocouple in place.

10. Apply a piece of tape around the thermocouple at the top and bottom of the sample tube as shown below. This will secure the thermocouple in place.

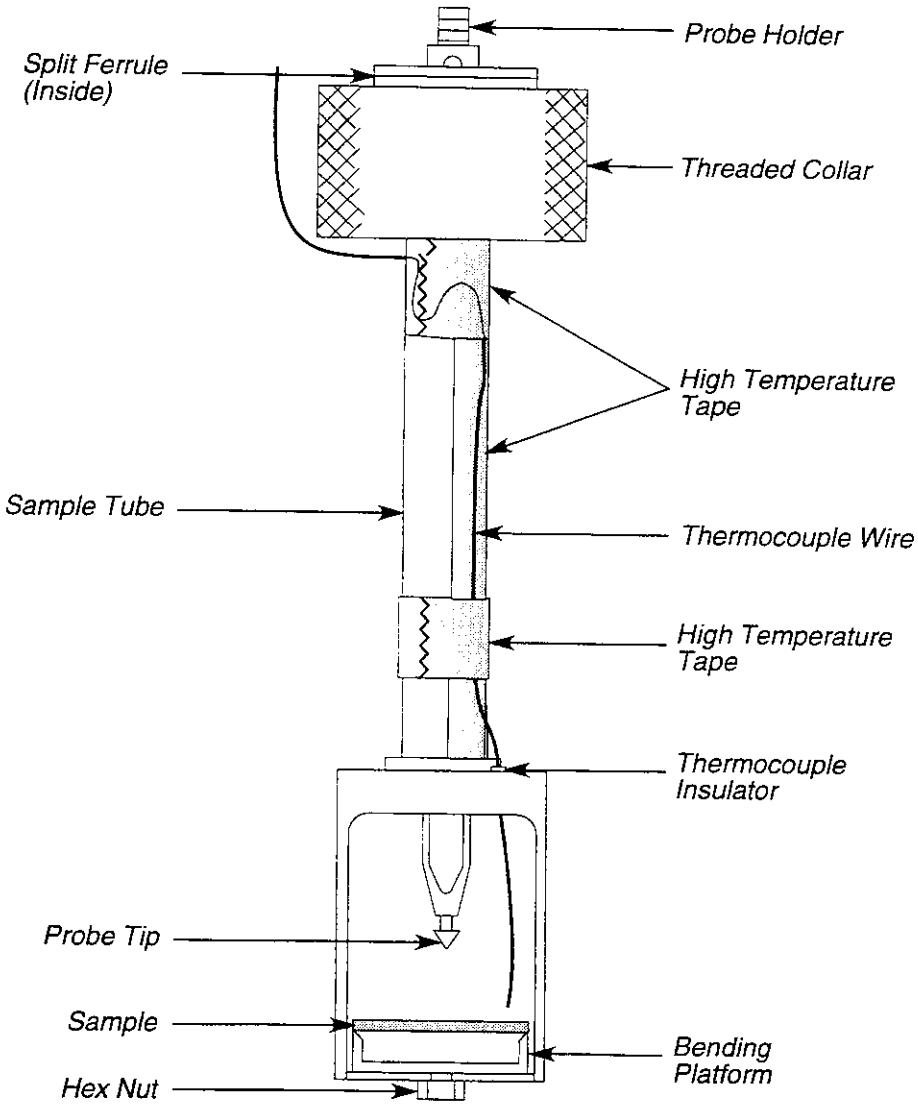


Figure 5-12. Routing the thermocouple.

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NOTE: The top of the sample tube does not reach extreme temperatures during normal use. However, the adhesive on the high temperature tape may age over time or if very high temperatures over long periods of time are used. Positioning the lower tape wrap approximately one centimeter from the thermocouple insulator should reduce the aging of the tape adhesive. Replace the tape if discoloration or other signs of wear appear.

11. Slowly close the coupling access covers, making sure that you do not pinch the thermocouple in the doors as they are closed.

12. The thermocouple should exit through the rear of the access covers. Notice that a space is present at the back of the access covers where the thermocouple can pass without being bent or pinched.

The thermocouple installation is now complete.

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Operating Modes

The Dynamic Mechanical Analyzer can be operated in many different modes. A mode is defined as a software state where a selected variable (or variables) is programmed and all other variables are held constant. These modes, which are briefly described in the following paragraphs, provide a broad range of instrument flexibility and performance. The DMA 7e is shipped with software for operation in temperature and time modes. Other software modes must be purchased separately. Part numbers for the various software packages are listed in Appendix 2.

Temperature Scan Mode

In the Temperature Scan mode, the temperature is programmed and the frequency and stress are held constant. Temperature-dependent behavior is characterized by monitoring changes in strain and phase. Alpha, Beta, and Gamma transitions may be identified as a function of temperature or time. Modulus and viscosity, as well as other standard reporting variables, may be readily quantitated for these materials as a function of temperature or time.

Time Scan Mode

In the Time Scan mode, the frequency, temperature and stress are held constant over time. Time-dependent behavior is characterized by monitoring changes in strain and phase. This mode is useful for examination of time-dependent curing behavior in materials such as coatings, adhesives, rubbers, and epoxies. Modulus and viscosity, as well as other standard reporting variables, may be readily quantitated for these materials as a function of temperature or time.

Frequency Scan Mode

In the Frequency Scan mode, the frequency of oscillation is programmed over a specific range. Temperature and stresses are held constant. Rate-dependent behavior is characterized by monitoring the change in strain and phase over time. The Frequency Scan mode is useful in the detection of subtle differences in the molecular structure of materials. This information can be used to predict material behavior or emulate processes.

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Stress Scan Mode

In the Stress Scan mode, the dynamic and static stress are linearly programmed up or down over time and the frequency and temperature are held constant. The stress-dependent behavior is characterized by monitoring changes in strain and phase. This mode is useful for conducting DMA analysis in the stress-dependent, linear, viscoelastic region. Stress Scan software is also useful for performing standard test methods and identifying stress-dependent differences in materials.

Creep-Recovery Mode

In the Creep-Recovery mode, a Creep or a Recovery analysis can be performed or both analyses can be performed during one experiment. In the Creep mode, a static stress is instantaneously applied and held at a constant value for a set time. In the Recovery mode, a static stress is instantaneously removed. The resultant sample displacement is measured. The Creep-Recovery mode is used to generate modulus and viscosity values at long times (very low frequencies) that are not within practical limits of instrument testing.

The temperature in these modes of operation can be held constant or changed during a run, and there is no dynamic stress applied. Time and stress-dependent behavior are characterized by monitoring the strain. Compliance, modulus and stress are calculated and displayed on the screen as a Retardation Spectrum in the Creep mode and as a Relaxation Spectrum in the Recovery mode.

Creep Ramp Mode

This mode is accessed through the Creep-Recovery mode. Normally, in a Creep-Recovery experiment, the creep stress is applied instantaneously. When the Creep Ramp is turned on, the user can program the rate at which the creep stress is applied. This mode is useful for characterizing stress dependent properties of the sample.

Constant Position Mode

In the Constant Position mode, only static forces are applied. The sample is programmed over a selected temperature range or held at a constant temperature versus time. Temperature or time-dependent behavior is characterized by monitoring changes in the sample dimensions. In this mode of operation, constant length experiments are performed. This mode is accessed through the Creep-Recovery mode.

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Constant Force (TMA) Mode

In the Constant Force (TMA) mode, only static forces are applied. The sample is programmed over a selected temperature range or held at a constant temperature versus time. Temperature or time-dependent behavior is characterized by monitoring changes in the sample dimensions. In this mode of operation, constant force experiments are performed, such as softening points and expansion coefficients.

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Running the System

This section gives an overview of how the DMA software is set up and summarizes how to set up a typical DMA 7e experiment using the features of the Thermal Analysis software. For detailed information about running samples with the DMA 7e, refer to the instructions provided with the specific mode software kit (for example, *Temp / Time Software Users Manual*). For detailed information about the use of DMA 7e measuring systems, refer to the instructions provided with the specific measuring system.

Setting Up a DMA 7e Experiment

The following steps summarize the set-up procedure for a typical DMA 7e experiment:

1. Start the system.
2. Make DMA the current task.
3. Select the appropriate DMA mode.
4. Select the type of curve(s) you wish to use for your method.
5. Create a new DMA method

   or

   recall an existing DMA method and modify it, if necessary.
6. Mount your sample onto the analyzer.
7. Start the run.
8. Save the run.

These steps are described in the following sections.

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Starting the System

1. Start the system (refer to **Starting the System** in Chapter 3).
2. Select **Run System** from the Main menu.
3. Select the number of tasks you want to run.

Making DMA the Current Task

When you enter the 7 Series/UNIX Thermal Analysis software display, task windows appear on the top of the screen. The number of task windows that appear depends on the number of tasks you specified.

To make DMA the current task, left-click the mouse on the DMA task window (the task window will turn red) or use the appropriate keyboard function key to select that task window (for example, press **Ctrl-2** to select Task 2 if that is the DMA task).

The Run System menu also appears on the screen with the following function keys:

F1 **Exit**

F2 **Set Up And Run**

F3 **Recall Data**

F7 **File Utilities**

F8 **DOS Shell**

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Changing Modes

The DMA 7e is designed so that you can easily change from one mode of operation to another. To change modes:

1. From the Run System menu, select **Exit**.

   The System menu is displayed:

   F2 **Change Defaults**

   F3 **Run System**

   F4 **Change Mode**

   F5 **Curve Selection**

   F6 **Restart Analyzer**

2. Select **Change Mode**.

   The Change Mode menu is displayed. The currently selected mode of operation is highlighted in red.

   The Change Mode menu contains the following function keys:

   F1 **Exit**

   F2 **Temp/Time Scan**

   F3 **Frequency Scan**

   F4 **Stress Scan**

   F5 **Creep Recovery**

   F6 **TMA**

3. Use the mouse or keyboard function keys to select the mode you want. In a few moments, a blank method for the mode you selected appears on the screen.

4. Select **Exit**. The newly selected option will be stored in memory and the System menu will appear.

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Selecting Curves

You can select the type of curve(s) you want to recall for a given mode of operation. You can select up to five curvetypes for each mode. Once you set the curvetypes, those curves will be recalled each time you complete a run or recall curves, until you delete or change the selections. The first curve can be changed but not deleted.

Refer to the Users Manual provided with each DMA 7e Mode Software Kit for detailed instructions on selecting curves.

Creating a New Method

Create a DMA method by entering parameter values on the DMA method display:

1. Select **Run System** on the System menu to display the Run System menu.

2. Select **Set Up And Run**. The Set Up And Run menu appears:

   F1 **Exit**

   F2 **Recall Method**

   F3 **Modify Parameters**

   F8 **Start Run**

3. Enter parameter values on the DMA method display using the mouse or the **Modify Parameter** function key as described in **Chapter 3, Software Basics**.

   For a complete description of all the parameters, refer to the instructions provided with the specific mode software you are using.

   Parameter values that are displayed in gray can only be modified using the **Change Defaults** function key in the System menu. Select **Exit** twice to return to the Run System menu.

Using the Change Defaults Function Key

To modify the parameters that are displayed in gray on the method display, use the **Change Defaults** function key:

1. In the Run System menu, select **Exit**.

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2. In the System menu, select **Change Defaults**.

   The Change Defaults menu appears with the following function keys:

   F1 **Exit**

   F2 **Go to Temp Rate**

   F3 **Load Condition**

   F6 **Forces/Stresses**

   F8 **Restore Defaults**

3. Select **Go To Temp Rate** to set the speed at which the analyzer will respond to a Go To Temp command. Type the new rate in °C/minute and press **Enter**.

4. Select **Load Condition** to set the Load Conditions for your experiment. When you select this function key, the Load Conditions menu appears:

   F1 **Exit**

   F2 **Temp**

   F3 **Static Stress**

   F4 **Dynamic Stress**

   F5 **Frequency**

   a. Select **Temp** to set a new Load Temperature. Type a new temperature in °C (typically 30 °C) and press **Enter**.

   b. Select **Static Stress** to set a new Load Static Stress. Type a new value in Pa (typically 100 Pa) and press **Enter**. If parameter entry is set for forces (see Step 5), this function key is labeled **Static Force**. Type a new value in mN (typically 10 mN) .

   c. Select **Dynamic Stress** to set a new Load Stress. Type a new value in Pa (typically 0 Pa) and press Enter. If parameter entry is set for forces (see Step 5), this function key is labeled **Dynamic Force**. Type a new value in mN (typically 0 mN) .

   d. Select **Frequency** to set a new Load Frequency. Type a new value in Hz (typically 1 Hz) and press **Enter**.

   e. Select **Exit** to exit from the Load Conditions menu.

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5. Select **Forces/Stresses** to toggle between force or stress entry of parameters.

   The DMA 7e uses Standard International (SI) units. If you select **Stresses**, the Run Parameters for Dynamic Stress and Static Stress will be displayed in Pa. If you select **Forces**, those Run Parameters will be displayed in mN.

6. To restore all parameters to their original settings, select **Restore Defaults**.

7. When you have finished setting the prameters in the Change Defaults menu, select **Exit** to return to the System menu.

Recalling an Existing Method

You can recall an existing DMA method and run it as is or modify it by performing the following procedure:

1. Select **Exit** from the System menu to display the Run System menu.

2. Select **Set Up And Run**.

3. Select **Recall Method** from the Set Up And Run menu. A listing of DMA method files appear on the screen and the Recall Method menu appears on the bottom of the screen with the following function keys:

   F1 Exit

   F2 Search

   F3 Page Up

   F4 Page Down

   F5 More Detail

   F6 Sort

   F8 Recall File

4. Use the mouse or cursor keys to scroll for the file you want to archive:

   Using the Mouse

   - Left-click on a filename to position it in the blue bar. (You may select the **More Detail** option when the filename is highlighted in blue.)

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- Left-click on the last file on the screen to scroll down one-half of the page.
- Left-click on the first file on the screen to scroll up one-half of the page.
- Right-click on the file to recall it.

Using the Cursor Keys

- Press the ↑ or ↓ cursor key on the right-hand keypad of the keyboard. Each time one of the keys is pressed, the directory listing will move one position on the screen.

The next several steps review the options in the Recall Method menu that are used with the mouse or cursor keys to simplify the selection of method files. If you have already located the file you wish to recall and have positioned it in the blue bar, go to Step 8.

Make sure you select a method file that uses the same DMA mode you are using.

5. Select **Search** to enter a filename, sample I.D, mode, or probe pattern you wish to search. After entering this pattern, all files containing the pattern you entered appear on the screen. You can then use the mouse or cursor keys to scroll through the files to find a method that you wish to recall.

   Refer to "Recalling Data" in Chapter 7, Analysis for more information.

6. Select **Page Up** to view the previous page of files.

   A new page of method files appear. These are the files immediately preceding the files shown on the screen. This option can be selected as many times as you wish. When you reach the beginning of the directory, paging will cease. **Page Up** is useful when you wish to quickly scan through your data file listings.

7. Select **Page Down** to view the next page of method file listings.

   A new page of method files appear. These are the files immediately following the files shown on the screen. This option can be selected as many times as you wish. When you reach the end of the directory, paging will cease. **Page Down** is useful when you wish to quickly scan through your data file listings.

8. Select **More Detail** to view information about the file that is currently highlighted in the blue bar.

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The parameters table for the file in the blue bar appear on the screen. You can review all of the major Set Up And Run parameters for this method file.

a. Select **Next File** to view the parameters table for the file listed below the one you are viewing.

b. Select **Previous File** to view the parameters table for the file listed above the one you are viewing.

9. Select **Sort** to sort the directory by file name, sample I.D. (alphabetical order), analysis date, mode, or probe type. When the files are sorted by date, the most recent analyses are listed first.

   Refer to "Recalling Data" in Chapter 7, Analysis for more information.

10. Up to this point you have been able to review the method files stored in the selected directory and search the current directory. To recall the file you have selected, select **Recall File**.

    If you have selected a file that uses a different DMA mode than the one you are using, the following message appears:

    You cannot recall a method from another analyzer type; try another method.

    Select another method file or change the DMA mode.

11. The method display for your selected method file will appear on the screen. Select **Exit** to return to the Set Up And Run menu.

Modifying an Existing Method

Once you have recalled an existing DMA method, you can either run it or modify it by changing parameter values.

1. Select **Set Up And Run** on the Run System menu.

2. Select **Modify Parameters** on the Set Up And Run menu.

3. Change the parameter values on the DMA method display using the mouse or the **Modify Parameter** function key as described in **Chapter 3, Software Basics**.

   For a complete description of all the parameters, refer to the instructions provided with the specific mode software you are using.

   Parameter values that are displayed in gray can only be modified using the **Change Defaults** function key in the System menu.

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Mounting Your Sample

Zero the DMA, measure your sample and mount it onto the measuring system as described in the instructions provided with the specific measuring system.

Starting the Run

To start the run:

1. Select **Start Run** on the Set Up And Run menu.

The analyzer will run according to the parameters in the method.

At the end of the run, the analyzer will ramp to or hold at the temperature you selected as the end condition and the Run System menu will appear.

Stopping the Run

To stop a run in progress, perform any one of the following steps:

- Select **Stop Run** in the Start Run menu.
- Press the **Go to Load** keyboard function key (**F10**) and, depending on the mode of operation, automatically change the temperature, stress and frequency to the load conditions.
- Press the **Go to Temp** keyboard function key (**F11**) to heat or cool the analyzer to the temperature you select. The forces (stresses) and frequency remain the same.
- Press the **Hold** keyboard function key (**F12**) to hold the analyzer at the temperature displayed in the status window and maintain the current stress and frequency.

Saving the Run

If a filename was specified during method setup, that filename is used when the run has been completed. If no filename is specified in the method, you can save the results by typing a 10-character filename, then pressing **Enter**. If you don't want to save the results in a file, just press **Enter** without typing a filename. In either case, the displayed curve will be automatically optimized (if Auto-Optimization is on) and displayed in the graphics window of the screen.

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At the end of the run you can do any of the following:

- Exit from the system.
- Set up another run.
- Use one of the File Utilities (see **Chapter 6, File Utilities**).
- Go to the DOS shell (see below and **Chapter 7, Analysis**).
- Display additional curvetypes (see **Chapter 7, Analysis**).
- Optimize the data (see **Chapter 7, Analysis**).
- Perform calculations on the data (see **Chapter 7, Analysis**).
- Use the Snapshot function (see **Chapter 7, Analysis**).
- Recall other data files (see **Chapter 7, Analysis**).

Using DOS Commands

The **DOS Shell** function key on the Run System menu lets you temporarily suspend the 7 Series/UNIX Thermal Analysis software and go to the DOS prompt. When you select **DOS Shell**, the DOS prompt appears and you can enter DOS commands. To get back to the Thermal Analysis software, type **quit**, then press **Enter**.

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