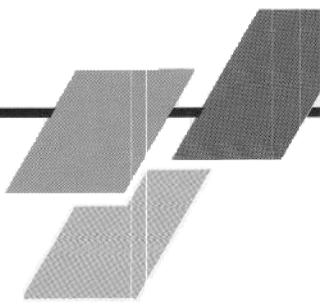


DMA Calibration Data Analysis Program

Version 4.0

For use with the
Du Pont Thermal Analyst 2000/2100

PN 996509.001 Rev. B
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Issued January 1988



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Using this Manual

- CHAPTER 1** Introduces the purpose and features of the DMA Calibration program.
- CHAPTER 2** Directs you on the methods used to prepare a backup copy of your DMA Calibration disk and install the program onto the Winchester disk.
- CHAPTER 3** Uses examples to take you step-by-step through the procedures used to load and start the program.
- CHAPTER 4** Describes the operations used to calibrate the 983 DMA.
- CHAPTER 5** Describes the operations used to calibrate the 982 DMA.
- CHAPTER 6** Provides you with the information needed to obtain written reports and plots of your results.
- CHAPTER 7** Discusses the equations, explains the program messages, and provides a function key outline.

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CHAPTER 1

Introducing the DMA Calibration Program

The DMA Calibration program can be used to calculate the following instrument calibration constants for 983 and 982 Dynamic Mechanical Analyzers (DMAs):

- Moment of inertia
- Spring constant
- Drive signal constant
- Parallel loss stiffness
- Series storage compliance
- Series loss compliance
- Phase zero (983 DMA only)
- Length correction (a sample-dependent constant)

As you obtain each constant, it is displayed on the screen to enable you to save the results in the module's (or Module Interface's) battery backed-up memory.

The DMA Calibration program has a summary screen that displays all calibration constants and allows you to change them from the keyboard. You can review these constants at any time on the Module Parameters screen under Instrument Control (see the *Du Pont Thermal Analyst 2000/2100 Operator's Manual*).

The length correction results and tabular reports can be printed on the dot matrix printer or optional digital plotter.

CHAPTER 2

Installing the Program

Creating a Working Copy

The first thing you should do when you receive your DMA Calibration Data Analysis program diskette is prepare a working (backup) copy and store the original in a safe place. Consult Chapter 2 of the *Du Pont Thermal Analyst 2000/2100 Operator's Manual* for instructions on creating working diskettes.

Installing Your Program on a Winchester

1. Load the TA Operating System, if it is not already loaded (see Chapter 2 of the *Du Pont Thermal Analyst 2000/2100 Operator's Manual*).
2. Press F11 DATA ANALYSIS. The Data Analysis screen will appear.
3. Press F8 (GoTo File Utilities). The File Utilities screen will appear.
4. Insert the DMA Calibration program disk into drive A.
5. Press F3 (Copy). The screen will prompt you to enter the source of file information.
6. Use the ARROW keys to select drive A as the source drive (the drive from which you will copy the program), then press ENTER.
7. Use the ARROW keys to select the program directory, then press ENTER. The screen will display the name of the program.

8. Make sure the cursor is over the name of the program, then press F3 (Select File). The screen will prompt you to enter the destination file information.
9. You are then prompted to enter the name you wish to assign to the file when it is copied to the Winchester disk. To use the same name as shown on the screen, simply press ENTER.

The disk drives will engage, and the program will be copied to the Winchester disk. After the file is copied, the File Utilities screen will appear.

CHAPTER 3

Getting Started

Loading the Program

When you are ready to use the DMA Calibration program, load it from disk storage to computer memory by following the steps below.

1. Turn on the DMA module. If you are calibrating a 982 DMA, also turn on its Module Interface.
2. Be sure the TA Operating System is active. The TA Operating System is activated by pressing the F1 (Start Du Pont TA) key immediately after turning on the computer or after the computer has been rebooted.
3. Check that the DMA is online (indicated in the Device Address table on the left side of the screen). If it is not, turn the module power on and press F5 (Auto Configure) to reconfigure the devices on the network.
4. Press F11 DATA ANALYSIS. The screen shown on the next page appears.

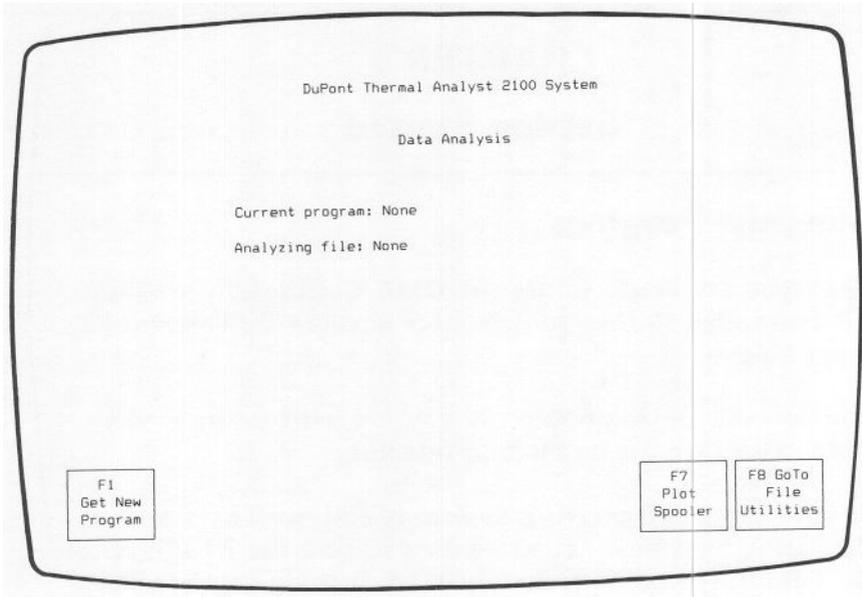


Figure 3.1
Data Analysis Screen

5. Press F1 (Get New Program). The screen will display:

Select drive: [A B C]

6. Use the ARROW keys to move the cursor to drive C, then press ENTER.

The screen will display a list of available programs as shown on the following page.

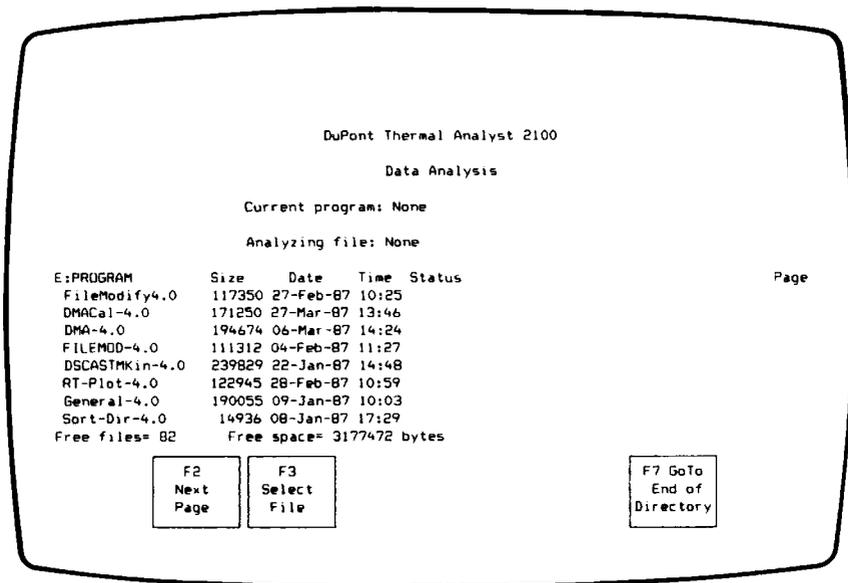


Figure 3.2
List of Programs Screen

7. Look for the DMACal program name in the list. You may need to press F2 (Next Page) to display the rest of the list.
8. Use the ARROW keys to move the cursor over the program name, then press F3 (Select File). The screen will display the program name and flash the word "Loading."

When the program is loaded, the "Ready" message will appear next to the program name.

9. Press F1 (Start Program) to begin the DMA Calibration program. The screen that appears depends on what type of DMA module is online (see Chapter 4 if you have a 983 DMA or Chapter 5 if you have a 982 DMA). The program will automatically decide which set of procedures to use based on which type of DMA is online.

The remainder of this chapter provides you with some general operating tips for your data analysis program.

Operating Tips

- When instructed in this manual to *enter* a response, type the response with the alphanumeric keys, then press ENTER. The cursor will automatically move to the next field in the screen. If you want to leave the response as it appears on the screen, simply press ENTER.
- The F8 (Accept This Form) key enters all information on a screen at once. Before pressing this key, check all user-input fields on the screen (always shown in green type) to ensure that they are correct or set as desired. To enter individual selections and proceed to the next prompt on a screen, use the ENTER key instead of F8 (Accept This Form).
- When you need to move backward through the program, press the ESCape key. In most cases, this will return you to the previous screen in the program.
- When you are prompted for input, you can determine the allowable range for each field by moving the cursor to the field and pressing F10 (HELP). A message will appear at the bottom of the screen in the following form:

Real number: x.xxx to x.xxx

When you enter your response, the HELP message will disappear.

- When instructed to move the cursor to a desired option, use the four gray ARROW keys to move the cursor up, down, left, and right. You can also use the SHIFT, ALT, and CTRL keys in combination with the ARROW keys to move the cursor more quickly. The relative speeds of these key combinations are:

ARROW (alone) = 1 pixel

SHIFT-ARROW = 5 pixels

ALT-ARROW = 10 pixels

CTRL-ARROW = 25 pixels

- To exit from the program at any point, press F11 DATA ANALYSIS. You can press F1 (Resume Program) to pick up the program where you left off or press F2 (Stop Program) to cancel the program entirely.
- This manual refers to the colors that appear on a color monitor. If you have a monochrome monitor, the colors will appear in varying shades of gray. See Table 3.1 to determine how each color appears on your screen.

Table 3.1
Appearance of Colors
on Monochrome and Color Monitors

Color Monitor	Monochrome Monitor
black blue red magenta green cyan yellow white	off dimmest ↓ dim ↓ bright ↓ brightest

NOTE

If you purchase a color monitor but do not install the color graphics software that comes with it, all colors will appear as white.

CHAPTER 4

Calibrating the 983 DMA

Overview

After you have started the DMA Calibration program, you will be requested to set up the DMA for each calibration through a series of screens. The order of the screens is controlled by the function keys at the bottom of each screen. In addition to summarizing these steps, this manual provides supplementary notes for further details, indented and marked with bullets (o).

CAUTION

Always use the locking pins when loading and unloading samples. Failure to do so can cause permanent damage to the pivots. Remove the locking pins before zeroing the arms.

NOTE

For best results, calibrate the DMA in the location where you intend to use it, and do not move it after it is calibrated.

There are several calibration procedures described in separate sections of this chapter. Each operation begins from the DMA Calibration Opening screen (see Figure 4.1 on page 12), which appears when you start the program and reappears upon completion of each procedure.

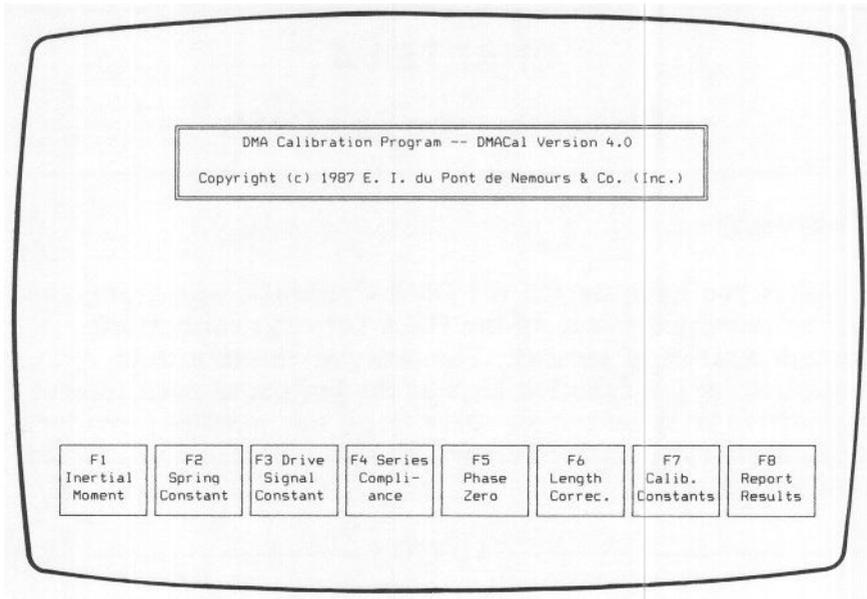


Figure 4.1
983 DMA Calibration Opening Screen

Most of the program operations are interdependent, using values calculated from the other calibrations; therefore, you must calibrate a new DMA in the specific order presented in this manual. If you perform the calibrations out of order, use Table 4.1 to determine which constants must be verified before you proceed (see page 68 for instructions on verifying a calibration).

Table 4.1
Interdependence of 983 Calibration Procedures

Procedure	Uses Calculations from:
F1 Inertial Moment	—
F2 Spring Constant	F1 Inertial Moment
F3 Drive Signal Constant	F1 Inertial Moment
F4 Series Compliance	F1 Inertial Moment F2 Spring Constant F3 Drive Signal Constant
F5 Phase Zero	F1 Inertial Moment F2 Spring Constant F3 Drive Signal Constant F4 Series Compliance
F6 Length Correction	F1 Inertial Moment F2 Spring Constant F3 Drive Signal Constant F4 Series Compliance

Starting the Calibration

Follow these steps before you begin calibrating the 983 DMA:

1. Turn on the DMA and allow it to warm up for 30 minutes.
2. Set up the DMA with a vertical clamping configuration (see the *983 DMA Operator's Manual*). Horizontal clamps cannot be used in the calibration procedures (except for length correction). Choose the vertical clamps you will be using most often in your experiments.
3. Make sure you know how to mount a sample properly (see the *983 DMA Operator's Manual*).
4. Locate the thin steel and thick steel standard samples. The thin steel standard is shipped with the DMA, and the thick steel standard is in the DMA accessory kit.

Measuring the Inertial Moment

F1
Inertial
Moment

The moment of inertia (J) is a measurement of how the instrument arms resist changes in motion. It is determined primarily by the mass of the arms, which should not change significantly during the lifetime of the instrument. Thus, this calibration needs to be performed only when the DMA module is first installed or after the drive assembly area has been serviced.

1. Press F1 (Inertial Moment). The following screen appears:

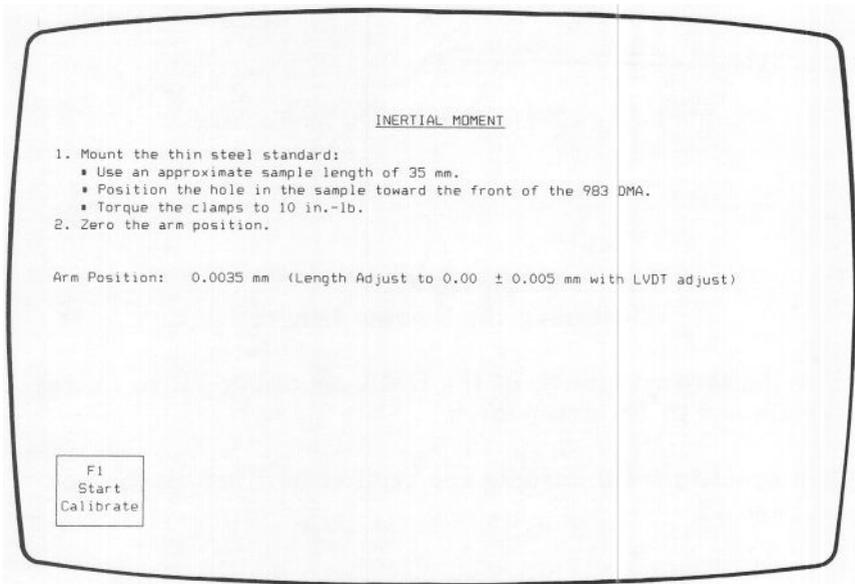


Figure 4.2
Moment of Inertia Screen

2. Follow the instructions on the screen.

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3).

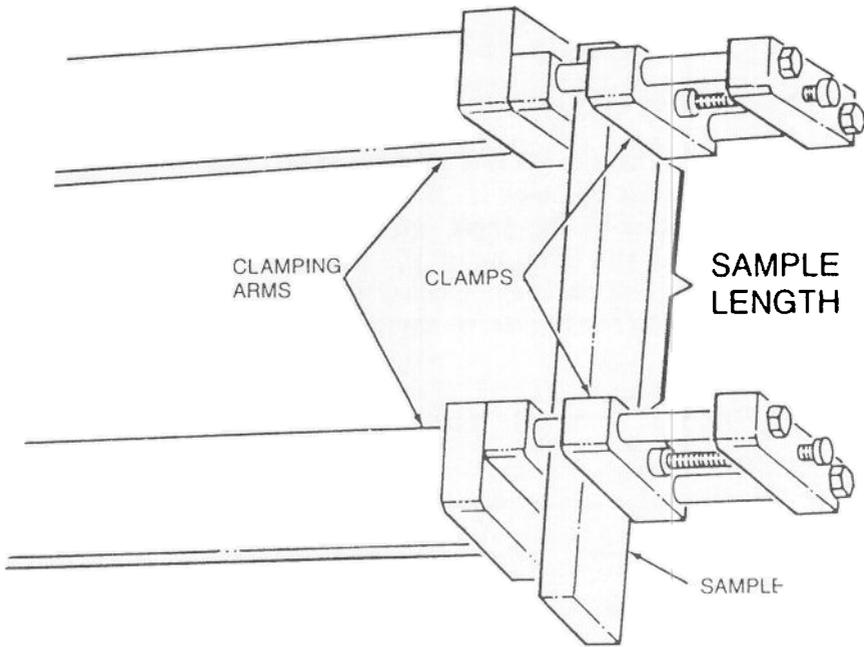


Figure 4.3
Measuring the Sample Length

- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 inch-pounds.
- The procedure for zeroing the arms is outlined in the box on page 17.

Zeroing the Arm Position

- a. Ensure that the sample is mounted correctly.
- b. Check that the SLIDE LOCK dial is tight (clockwise).
- c. Use the LVDT adjustment screw to zero the arm position to $\pm 0.005\text{mm}$.

3. Press F1 (Start Calibrate) when you have the arms zeroed.
4. Press F1 (Accept Value) when the frequency readout stabilizes between 15 and 22 Hz. If the frequency falls outside this range, press the ESCape key to go back to step 2, and adjust the sample length according to the guidelines in Table 4.2.

Table 4.2
Guidelines for Adjusting the Sample Length

Problem	Action
Frequency is too high.	Increase the sample length.
Frequency is too low.	Decrease the sample length.

After you press F1 (Accept Value), the screen shown on the next page appears.

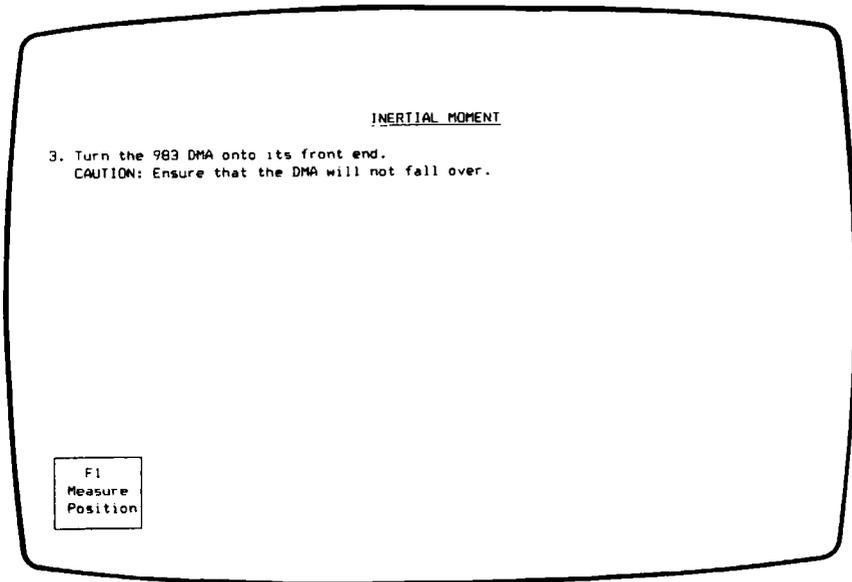


Figure 4.4
Moment of Inertia Screen

5. Follow the instructions on the screen.

WARNING

The 983 DMA weighs approximately 56 pounds. When turned on its front end, the DMA's center of balance is close to the front edge; be careful not to tip it too far forward.

- Hang a wire or paper clip (by itself) from the sample.

When you come to step 8 (page 19), you will need to hang a weight from the sample with this wire or paper clip so that the program can measure the change in arm displacement when a known weight is added.

6. Press F1 (Measure Position). The program measures and displays the arm position.

7. Press F1 (Accept Value) when the arm position readout stabilizes. (The arm position readout does not have to be zeroed, but it needs to be relatively stable.) The following screen appears:

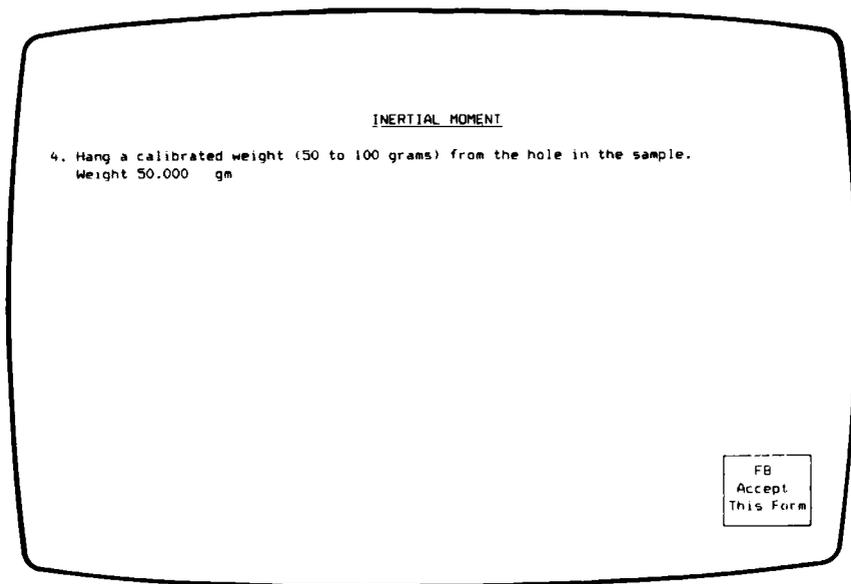


Figure 4.5
Moment of Inertia Screen

8. Follow the instructions on the screen.
 - Use the same paper clip or wire hung in step 5 to hang the weight from the sample.
 - Type in mass of the weight added, then press F8 (Accept This Form).
9. Press F1 (Measure Position). The program measures and displays the arm position.
10. Press F1 (Accept Value) when the arm position readout stabilizes. The program calculates and displays the inertial moment, which typically falls between 2.4 and 2.7 g m².

11. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

If the inertial moment is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, call your service representative.

12. Turn the DMA back to its normal position.

Calculating Flexibility

F2
Spring
Constant

This procedure calculates the instrument's spring constant, or parallel storage stiffness (K'). K' is a measure of the flexibility (springiness) of the pivots and is significant whenever a soft sample is under investigation, especially at low frequencies.

K' influences modulus values, particularly for samples with a resonant frequency below 10 Hz. Stiffer samples (resonant frequency above 40 Hz) are influenced more by the series compliance terms (see page 25).

Perform this calibration about once a month.

1. Press F2 (Spring Constant). The following screen appears:

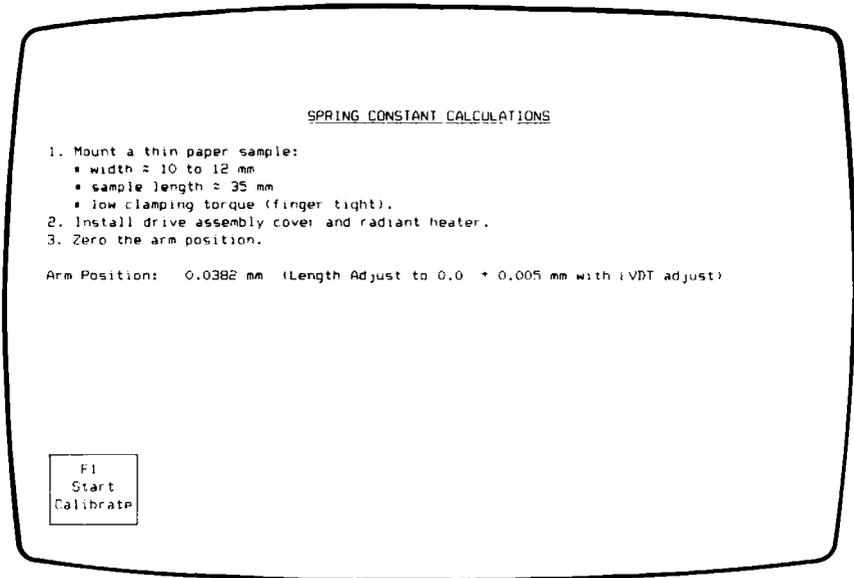


Figure 4.6
Spring Constant Screen

2. Follow the instructions on the screen.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3 on page 16).
 - Tighten the clamps manually; do not use the torque wrench.

NOTE

The drive assembly cover and radiant heater must be firmly installed to prevent stray air currents from affecting the measurements.

- The procedure for zeroing the arm position is outlined in the box on page 17.
3. Press F1 (Start Calibrate) when you have the arms zeroed.
 4. Press F1 (Accept Value) when the free arm frequency readout stabilizes between 1.8 and 2.0 Hz. The program calculates and displays the spring constant, which typically falls between 0.30 and 0.40 N m.
 5. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

If the spring constant is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, the instrument may have a faulty pivot; call your service representative.

Determining the Drive Signal Constant

F3 Drive
Signal
Constant

Using this function key allows you to calculate the drive signal constant (C') and the parallel loss stiffness constant (K_n''). The drive signal constant converts measured drive level to motor torque to determine how much energy is being applied to the system. The parallel loss stiffness is used in the modulus calculation to compensate for the mechanical friction (drag force) of the instrument. Both C' and K_n'' have a linear influence on damping calculations.

Perform this calibration about once a month.

1. Press F3 (Drive Signal Constant). The following screen appears:

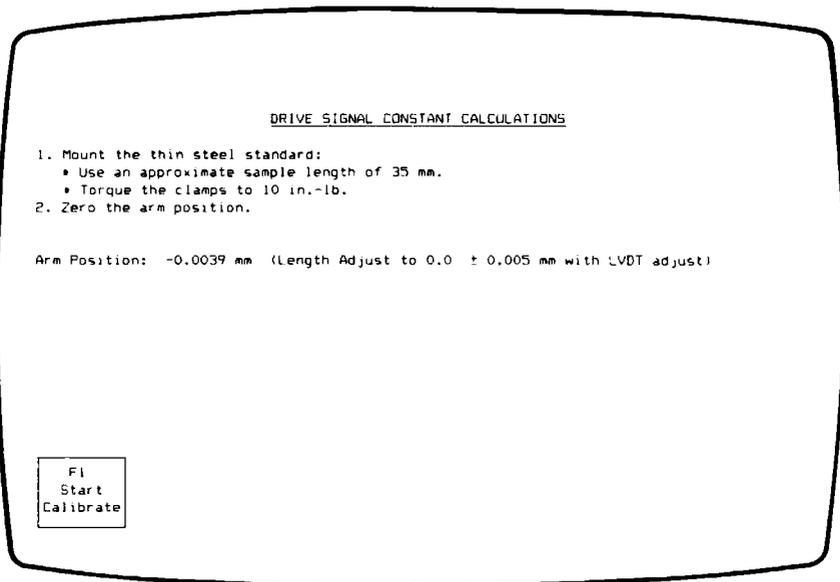


Figure 4.7
Drive Signal Constant Screen

2. Follow the instructions on the screen.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3 on page 16).
 - Use the torque wrench in the DMA accessory kit to torque the clamps to 10 inch-pounds.
 - The procedure for zeroing the arm position is outlined in the box on page 17.
3. Press F1 (Start Calibrate) when you have the arms zeroed.
4. Press F1 (Accept Value) when the frequency and drive signal readouts stabilize. The frequency should fall between 15 and 22 Hz. If the frequency falls outside this range, press the ESCape key to go back to step 2, and adjust the sample length according to the guidelines in Table 4.2 (page 17).

The program calculates and displays the drive signal and parallel loss stiffness constants. The drive signal typically falls between 0.015 and 0.026 mm/(mV sec²), and parallel loss stiffness should fall between 0.05 and 0.25 N m.

5. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

If one of the calibration constants is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, call your service representative.

Correcting for Stiff Samples

F4 Series
Compli-
ance

This operation requires a thick, stiff sample of known modulus to calculate the series storage compliance (J_C') and series loss compliance (J_{Cm}) of the instrument. The series compliance constants correct for the nonrigid responses of the instrument that occur with very stiff samples.

Perform this calibration about once a month.

1. Press F4 (Series Compliance). The following screen appears:

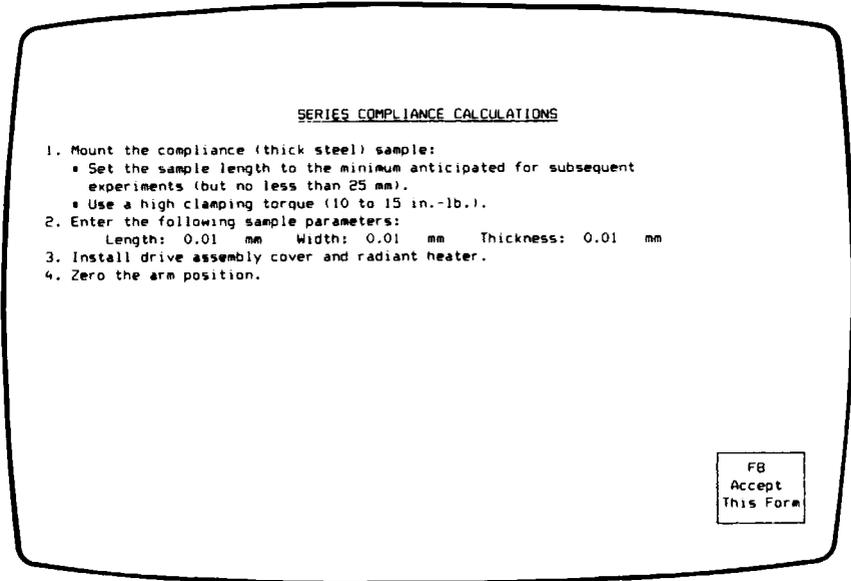


Figure 4.8
Series Compliance Screen

2. Follow the instructions on the screen.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3 on page 16).

- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 to 15 inch-pounds.
- Type in the sample dimensions, then press F8 (Accept This Form).

NOTE

Because the drive assembly cover and radiant heater affect the stiffness of the instrument, they must be installed firmly during this procedure.

- The procedure for zeroing the arm position is outlined in the box on page 17.

NOTE

Because the thick steel sample is very stiff, it is critical that the arm position be as close to zero as possible.

3. Press F1 (Start Calibrate) when you have the arms zeroed.
4. Wait for the frequency and drive signal to stabilize (approximately three minutes), then press F1 (Accept Value). The program calculates and displays the series storage compliance and series loss compliance. The storage compliance typically falls between 0.5 and 1.5 $\mu\text{m}/\text{N}$. Typical values for loss compliance range between 0.005 and 0.05 $\mu\text{m}/\text{N}$.
5. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

If one of the calibration constants is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, the clamps or slide lock may be loose, or the head assembly may not be firmly attached to the module base.

Correcting Instrument Phase Lag

F5 Phase Zero

In fixed frequency experiments, the instrument measures the phase lag between the drive signal and the arm position signal. This procedure corrects for the contribution of the instrument to sample phase angle measurements. A number of factors, including the instrument's electronic circuitry, its resonant frequency, and the ratio of drive signal to oscillation amplitude, contribute to phase angle errors.

The DMA Calibration program uses a thin steel sample, which has a known loss-to-storage modulus ratio ($\tan \delta = 10^{-5}$), to measure the instrument's contribution, then fits the measurements to a theoretical curve and displays the standard deviation of that fit. All phase correction data is stored internally in the module.

If you are using low-loss samples, perform this calibration at the beginning of each working day. Once a week is sufficient for high-loss samples.

1. Press F5 (Phase Zero). The screen shown on the following page appears.

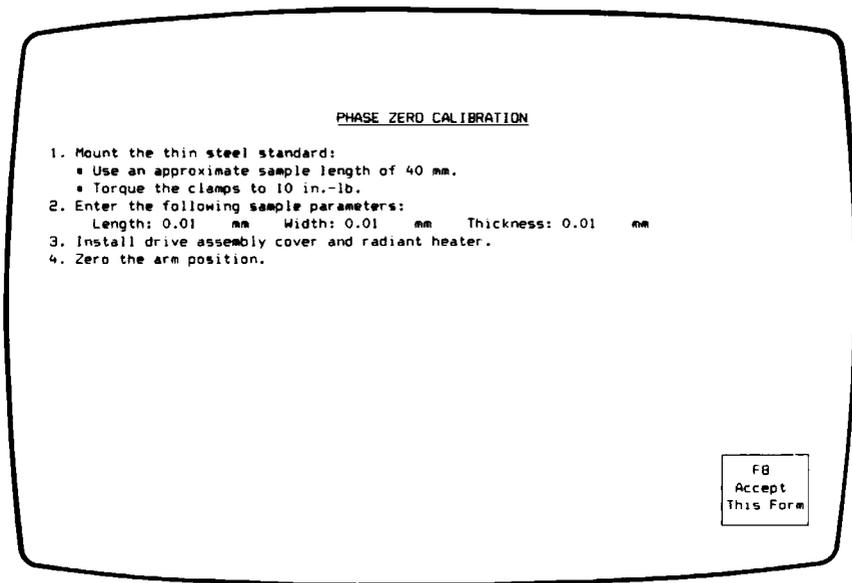


Figure 4.9
Phase Zero Step 1 Screen

2. Follow the instructions on the screen.

NOTE

The thin steel standard should be flat. If it appears bent, use a new one. (Extra standards are available from Du Pont.)

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3 on page 16).
- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 inch-pounds.
- Type in the sample dimensions, then press F8 (Accept This Form).

- The procedure for zeroing the arm position is outlined in the box on page 17.
3. Press F1 (Start Calibrate) when you have the arms zeroed. The program begins step 1 of the phase zero calibration, which takes approximately 20 minutes. If you wish to stop the procedure, press F1 (Cancel Phase Zero), and you will be returned to the screen in Figure 4.9.

NOTE

The arms do not move during the first four minutes of the phase zero calibration.

At the completion of Phase Zero step 1, the following screen appears:

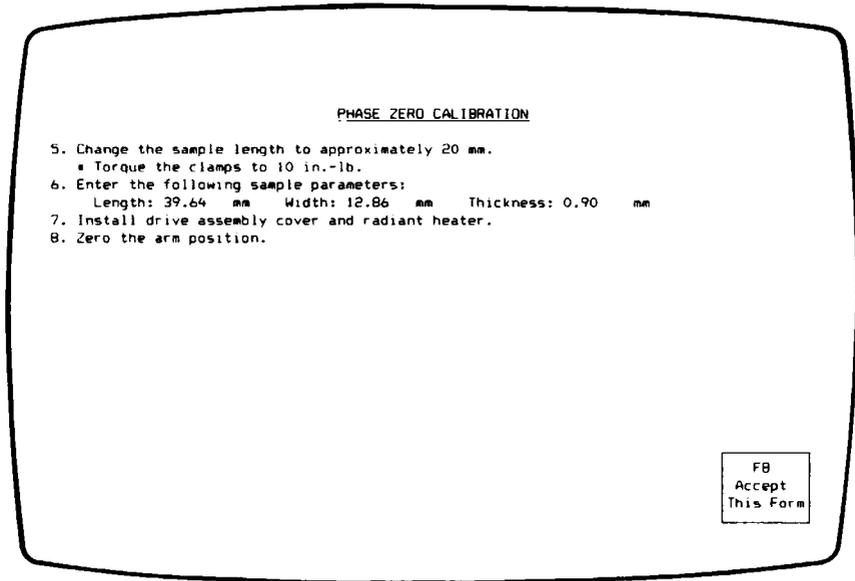


Figure 4.10
Phase Zero Step 2 Screen

4. Follow the instructions on the screen.
 - Insert the locking pins, loosen the clamp on the free arm, and loosen the slide lock. Then use the length adjust dial on the DMA to change the sample length.
 - Type in the new sample length, then press F8 (Accept This Form).
5. Press F1 (Start Calibrate) when you have the arms zeroed. The program begins step 2 of the phase zero procedure, which takes approximately fifteen minutes. If you wish to stop the procedure, press F1 (Cancel Phase Zero), and you will be returned to the Phase Zero Step 1 screen (see Figure 4.9).

At the completion of Phase Zero step 2, the program calculates and displays the standard deviation of the calibration, which should be less than 0.0006 radian.

6. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

If the standard deviation is too large, enter N and repeat the calibration, beginning with step 1. A large standard deviation usually means that the instrument was bumped during the calibration. If a large standard deviation persists, call your service representative.

Correcting for Sample Extension

F6
Length
Correc.

Because sample flexure actually extends beyond the clamp face into the center of the clamps, the sample length you measure must be corrected to reflect this “extension” of the real sample length. The length correction is a term that is added to the measured sample length to correct for this extension. Each sample type and clamping configuration has its own length correction factor.

Perform this calibration each time you use a different sample material, clamping configuration or when the length to thickness (L/T) ratio changes. After you complete the length correction calibration, you can plot your results if you wish (see “Plotting Length Correction Results”).

NOTE

This calibration is run in resonant mode. If your sample has a transition near room temperature, the results will be inaccurate.

CAUTION

Always remember to remove the locking pins before zeroing the arms and pressing F1 (Start Calibrate). If you attempt to start a calibration while the locking pins are still inserted, you will receive a motor control error message (see Chapter 7) and the data for the current measurement will be lost.

1. Press F6 (Length Correction). The screen shown on the following page appears.

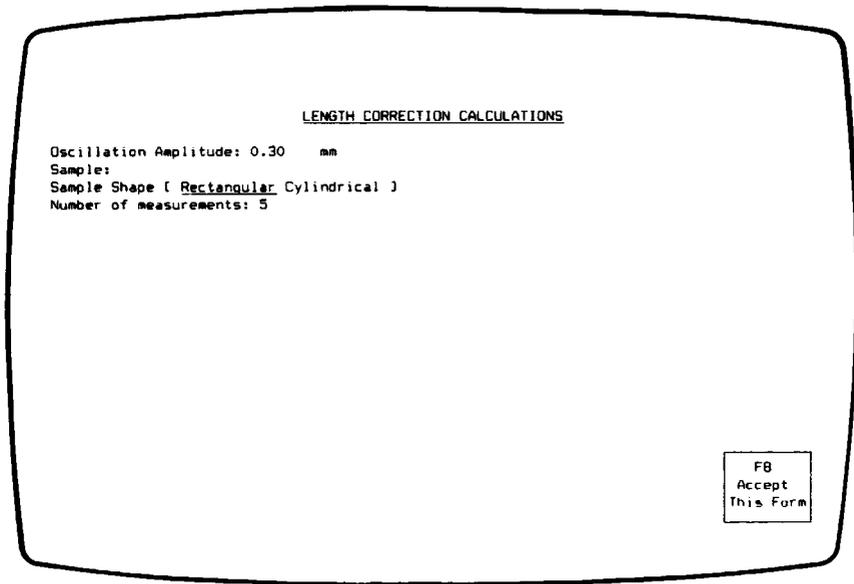


Figure 4.11
Length Correction Screen

2. Type or select the following:

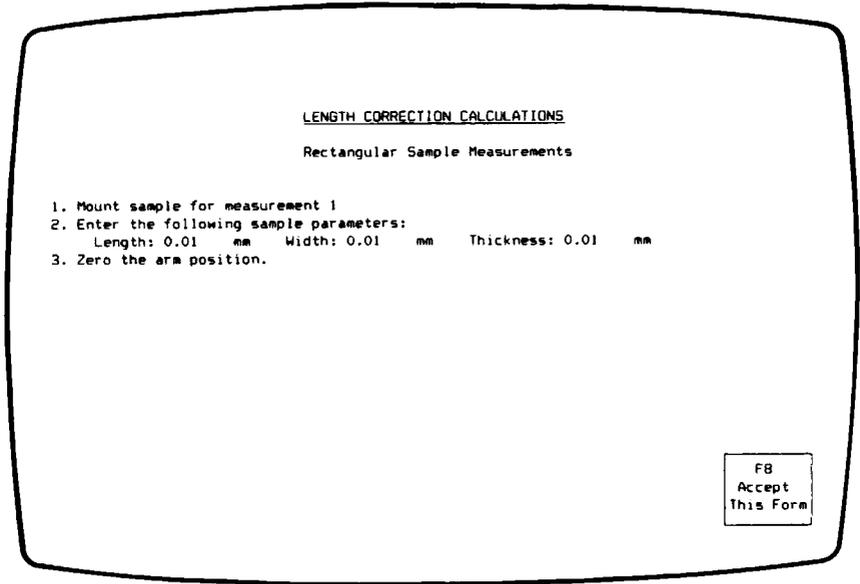
- The oscillation amplitude used most often in your experiments with this sample type.
- A description of the sample.
- The sample shape.
- The number of length measurements you wish to perform.

Then press F8 (Accept This Form).

NOTE

For best results, we recommend that you measure at least five different lengths.

The screen that appears depends on the sample shape:



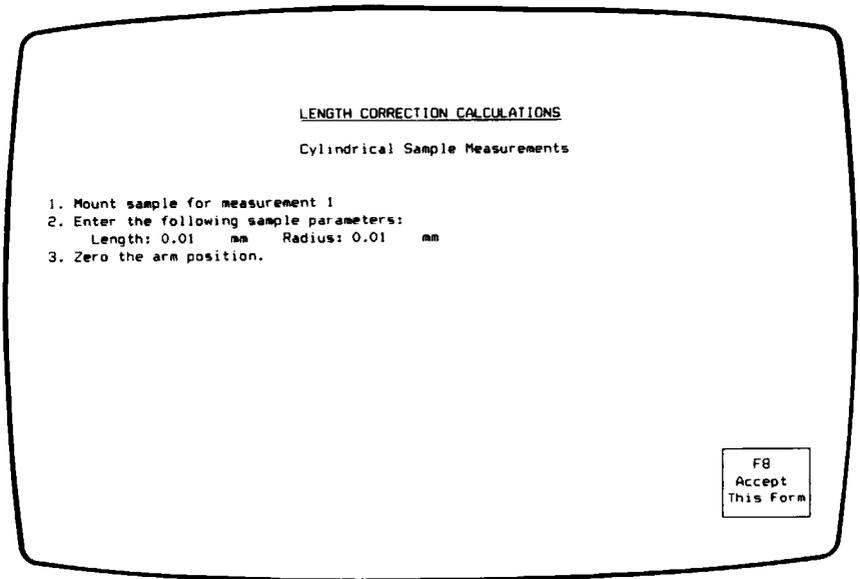
LENGTH CORRECTION CALCULATIONS

Rectangular Sample Measurements

1. Mount sample for measurement 1
2. Enter the following sample parameters:
Length: 0.01 mm Width: 0.01 mm Thickness: 0.01 mm
3. Zero the arm position.

FB
Accept
This Form

Figure 4.12
Rectangular Sample Measurements Screen



LENGTH CORRECTION CALCULATIONS

Cylindrical Sample Measurements

1. Mount sample for measurement 1
2. Enter the following sample parameters:
Length: 0.01 mm Radius: 0.01 mm
3. Zero the arm position.

FB
Accept
This Form

Figure 4.13
Cylindrical Sample Measurements Screen

3. Follow the instructions on the screen.
 - For the first length measurement, use the longest sample length you plan to measure. Your sample lengths should cover as wide a range as possible. Start with the maximum length, then work your way down to the minimum length in subsequent measurements.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 4.3 on page 16).
 - Use a consistent torque value for each measurement (10 inch-pounds for stiff samples, 3 to 5 inch-pounds for soft samples).
 - Type in the sample dimensions, then press F8 (Accept This Form).
 - The radiant heater is optional but recommended, especially if you are measuring a stiff sample.
 - The procedure for zeroing the arms is outlined in the box on page 17.
4. Press F1 (Start Calibrate) when you have the arms zeroed.
5. Press F1 (Accept Value) when the frequency and drive signal readouts stabilize. You are returned to the Sample Measurements screen.
6. Repeat steps 3 through 5 for each length measurement, working from the longest length to the shortest. Remember to insert the locking pins before loosening the clamps to change the sample length.

When you complete the final measurement, the program calculates the slope, corrected modulus, length correction, and standard deviation. The length correction value is normally positive.

7. Type Y and press ENTER to save your results in the 983 DMA's battery backed-up RAM.

NOTE

If you wish to see a plot of your length correction results, follow the instructions in the section “Plotting Length Correction Results” before unloading the DMA Calibration program. See page 76.

CHAPTER 5

Calibrating the 982 DMA

Overview

After you have started the DMA Calibration program, you will be requested to set up the DMA for each calibration through a series of screens. The order of the screens is controlled by the function keys at the bottom of each screen. In addition to summarizing these steps, this manual provides supplementary notes for further details, indented and marked with bullets (o).

CAUTION

Always use the locking pins when loading and unloading samples. Failure to do so can cause permanent damage to the pivots. Remove the locking pins before zeroing the arms.

NOTE

For best results, calibrate the DMA in the location where you intend to use it, and do not move it after it is calibrated.

There are several calibration procedures described in separate sections of this chapter. Each operation begins from the DMA Calibration Opening screen (see Figure 5.1 on page 38), which appears when you start the program and reappears upon the completion of each procedure.

CHAPTER 5

Calibrating the 982 DMA

Overview

After you have started the DMA Calibration program, you will be requested to set up the DMA for each calibration through a series of screens. The order of the screens is controlled by the function keys at the bottom of each screen. In addition to summarizing these steps, this manual provides supplementary notes for further details, indented and marked with bullets (o).

CAUTION

Always use the locking pins when loading and unloading samples. Failure to do so can cause permanent damage to the pivots. Remove the locking pins before zeroing the arms.

NOTE

For best results, calibrate the DMA in the location where you intend to use it, and do not move it after it is calibrated.

There are several calibration procedures described in separate sections of this chapter. Each operation begins from the DMA Calibration Opening screen (see Figure 5.1 on page 38), which appears when you start the program and reappears upon the completion of each procedure.

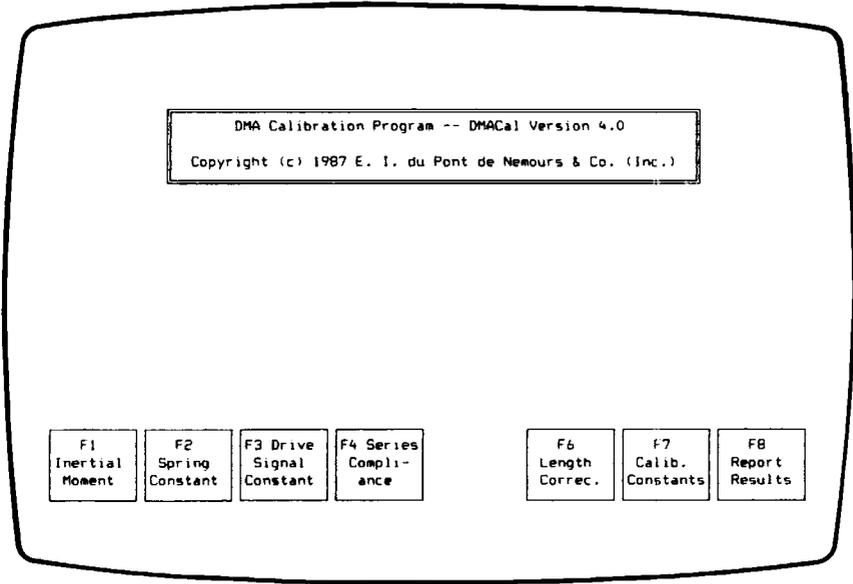


Figure 5.1
982 DMA Calibration Opening Screen

Most of the calibration procedures are interdependent, using values calculated from the other calibrations; therefore, you must calibrate a new DMA in the specific order presented in this manual. If you perform the calibrations out of order, use Table 5.1 to determine which constants must be verified before you proceed (see page 68 for instructions on verifying a calibration).

Table 5.1
Interdependence of 982 Calibration Procedures

Procedure	Uses Calculations from:
F1 Inertial Moment	—
F2 Spring Constant	F1 Inertial Moment
F3 Drive Signal Constant	F1 Inertial Moment
F4 Series Compliance	F1 Inertial Moment F2 Spring Constant F3 Drive Signal Constant
F6 Length Correction	F1 Inertial Moment F2 Spring Constant F3 Drive Signal Constant F4 Series Compliance

Starting the Calibration

Follow these steps before you begin calibrating the 982 DMA:

1. Turn on the DMA and its Module Interface and allow them to warm up for two hours.
2. Perform a null compensation on the 982 DMA (see the *982 DMA Operator's Manual*).
3. If the LVDT knob has been adjusted since the last calibration, adjust it to zero. Using the compliance (thick steel) sample in the DMA accessory kit, follow the procedure for electrical zeroing described in the *982 DMA Operator's Manual*.
4. Set up the DMA with a vertical clamping configuration (see the *982 DMA Operator's Manual*). Horizontal clamps cannot be used in the calibration procedures (except for length correction). Choose the vertical clamps you will be using most often in your experiments.
5. Make sure you know how to mount a sample properly (see the *982 DMA Operator's Manual*).
6. Locate the thin steel and thick steel standard samples. The thin steel standard is shipped with the DMA, and the thick steel standard is in the DMA accessory kit.
7. Use the F7 (Calibration Constants) key (see page 66) to check that the following constants are set to acceptable values:
 - Clamping distance
 - Poisson's ratio
 - Shear distortion

Whenever the module is reset with the F1 (Module Reset) key, all calibration constants in the Module Interface's battery backed-up memory are set to zero. If any of these constants are set to zero, enter an acceptable value from Table 5.2. The DMA Calibration program uses these constants in its calculations.

Table 5.2
Acceptable Values for 982 DMA Calibration Constants

Constant	Value
Clamping distance	8.0 mm
Poisson's ratio	0.44 typically 0.50 for rubbery materials 0.33 for glassy materials
Shear distortion	1.50 for rectangular samples 1.33 for cylindrical samples

Measuring the Inertial Moment

F1
Inertial
Moment

The moment of inertia (J) is a measurement of how the instrument arms resist changes in motion. It is determined primarily by the mass of the arms, which should not change significantly during the lifetime of the instrument. Thus, this calibration needs to be performed only when the DMA module is first installed or after the drive assembly has been serviced.

1. Press F1 (Inertial Moment). The following screen appears:

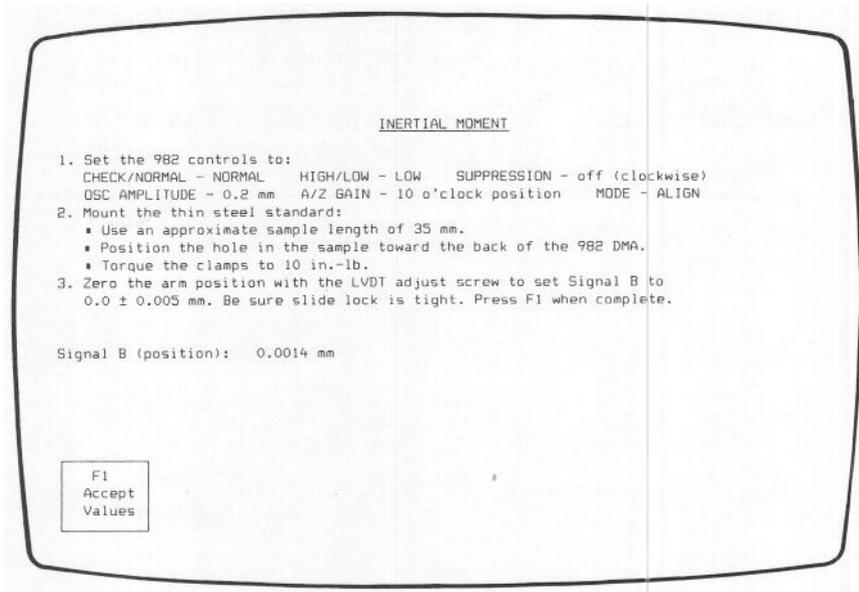


Figure 5.2
Moment of Inertia Screen

2. Follow the instructions on the screen.

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3).

Whenever the module is reset with the F1 (Module Reset) key, all calibration constants in the Module Interface's battery backed-up memory are set to zero. If any of these constants are set to zero, enter an acceptable value from Table 5.2. The DMA Calibration program uses these constants in its calculations.

Table 5.2
Acceptable Values for 982 DMA Calibration Constants

Constant	Value
Clamping distance	8.0 mm
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Measuring the Inertial Moment

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The moment of inertia (J) is a measurement of how the instrument arms resist changes in motion. It is determined primarily by the mass of the arms, which should not change significantly during the lifetime of the instrument. Thus, this calibration needs to be performed only when the DMA module is first installed or after the drive assembly has been serviced.

1. Press F1 (Inertial Moment). The following screen appears:

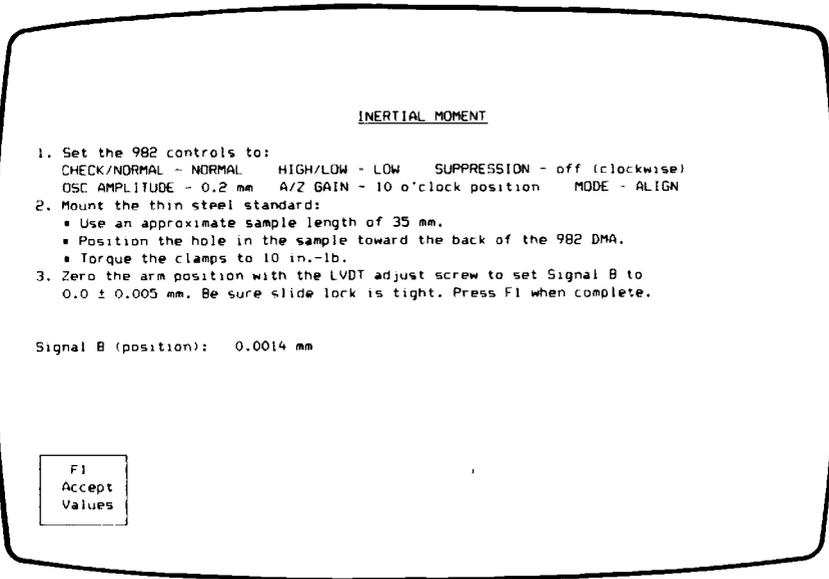


Figure 5.2
Moment of Inertia Screen

2. Follow the instructions on the screen.

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3).

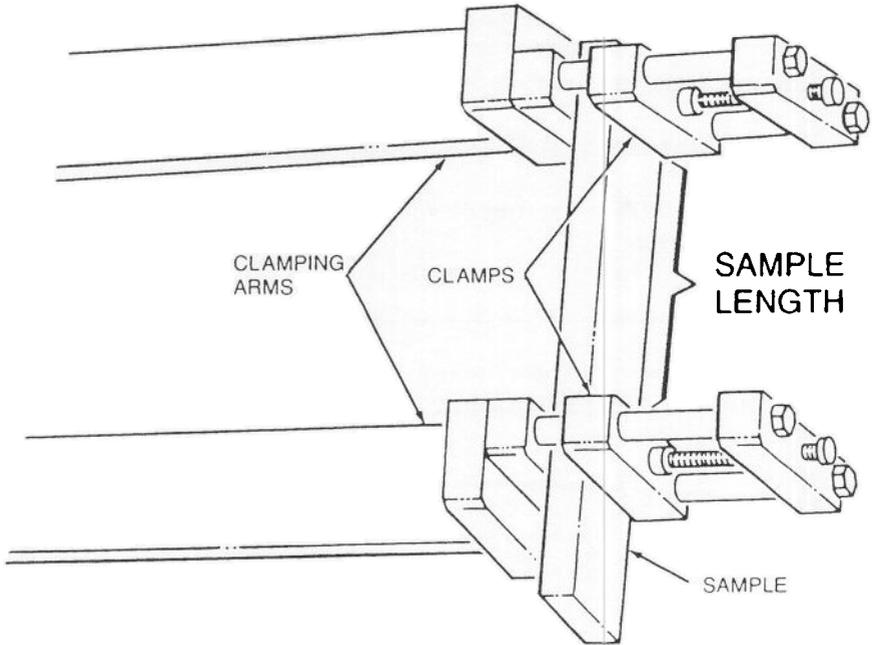


Figure 5.3
Measuring the Sample Length

- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 inch-pounds.
- The procedure for zeroing the arm position is outlined in the box on page 44.

Zeroing the Arm Position

- a. Ensure that the sample is mounted correctly.
- b. Check that the SLIDE LOCK dial is tight (clockwise).
- c. Use the LVDT adjustment screw to zero the arm position to $\pm 0.005\text{mm}$.

3. Press F1 (Accept Values) when you have signal B zeroed. The following screen appears:

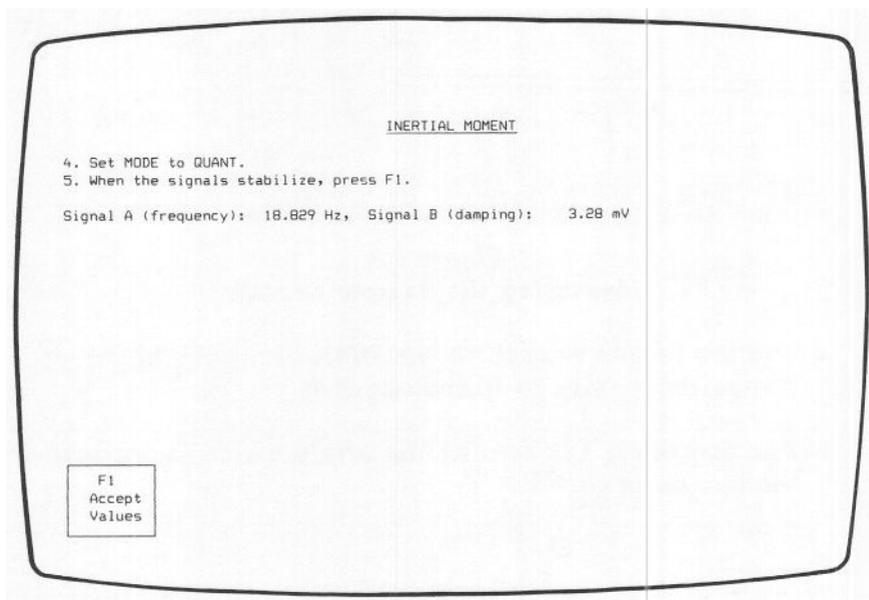


Figure 5.4
Moment of Inertia Screen

4. Follow the instructions on the screen.
5. Press F1 (Accept Values) when signals A and B stabilize. The following screen appears:

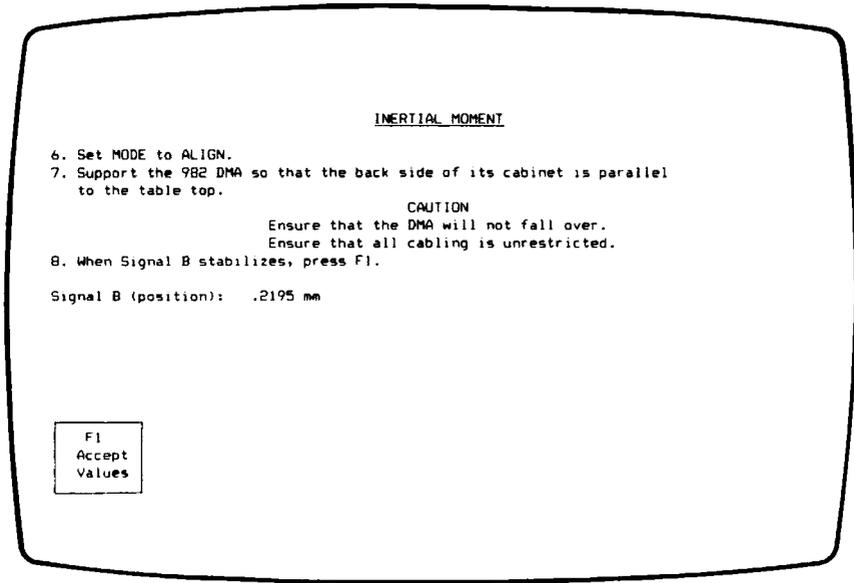


Figure 5.5
Moment of Inertia Screen

6. Follow the instructions on the screen.
 - Tip the DMA onto its back panel and raise it on four-inch supports (e.g., two-by-fours) to allow for clearance of the connectors and cables (see Figure 5.6).

—WARNING—

The 982 DMA module weighs approximately 40 pounds.

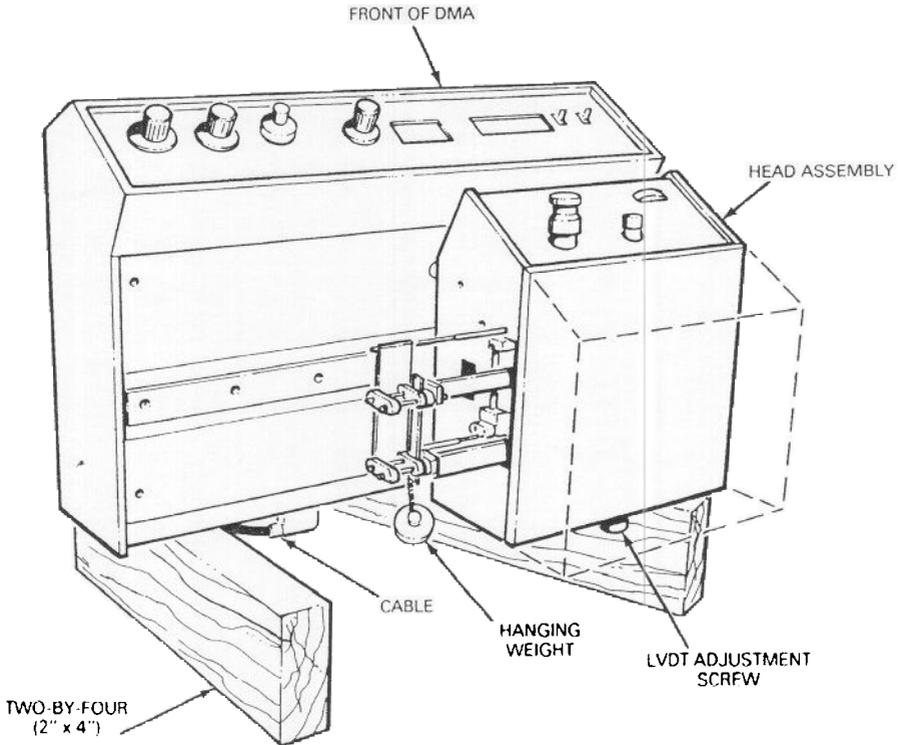


Figure 5.6
Placing the 982 DMA on its Back

- Hang a wire or paper clip (by itself) from the sample.

When you come to step 8 (page 47), you will need to hang a weight from the sample with this wire or paper clip so that the program can measure the change in arm displacement when a known weight is added.

7. Press F1 (Accept Values) when signal B stabilizes. The screen shown on the next page appears.

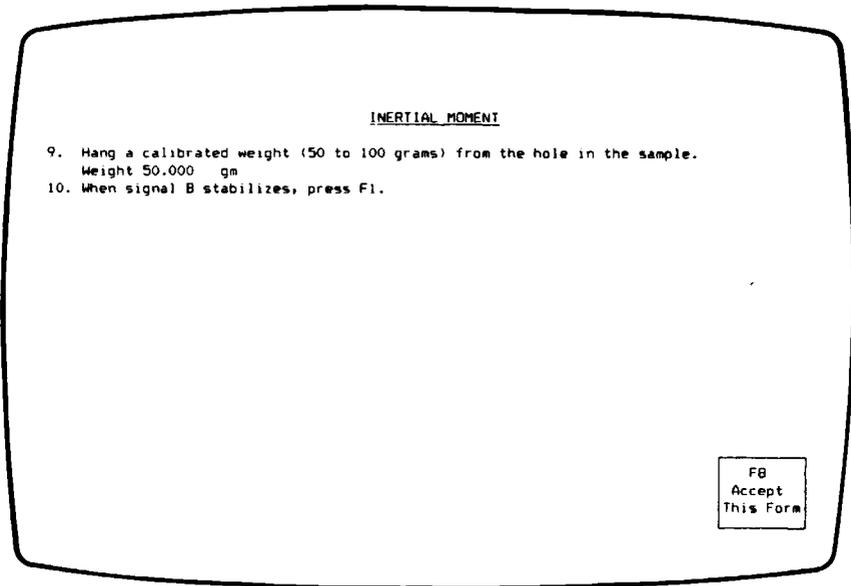


Figure 5.7
Moment of Inertia Screen

8. Follow the instructions on the screen.
 - Use the same paper clip or wire hung in step 6 to hang the weight from the sample.
 - Type in the mass of the weight added, then press F8 (Accept This Form).
9. Press F1 (Accept Values) when signal B stabilizes. The program calculates and displays the inertial moment, which typically falls between 2.4 and 2.7 g m².
10. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If the inertial moment is out of range, enter N and repeat this calibration, beginning at step 1. If you still get unacceptable results, call your service representative.
11. Turn the DMA back to its normal position.

F2
Spring
Constant

This procedure calculates the instrument's spring constant, or parallel storage stiffness (K'). K' is a measure of the flexibility (springiness) of the pivots and is significant whenever a soft sample is under investigation, especially at low frequencies.

K' influences modulus values, particularly for samples with a resonant frequency below 10 Hz. Stiffer samples (resonant frequency above 40 Hz) are influenced more by the series compliance terms (see page 58).

Perform this calibration about once a month.

1. Press F2 (Spring Constant). The following screen appears:

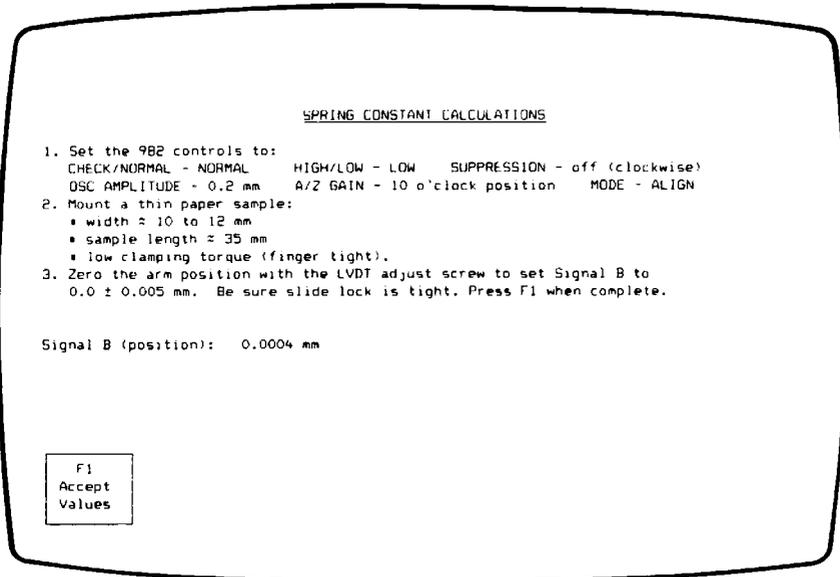


Figure 5.8
Spring Constant Screen

2. Follow the instructions on the screen.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3 on page 43).
 - Tighten the clamps manually; do not use the torque wrench.
 - The procedure for zeroing the arm position is outlined in the box on page 44.
3. Press F1 (Accept Values) when you have signal B zeroed. The following screen appears:

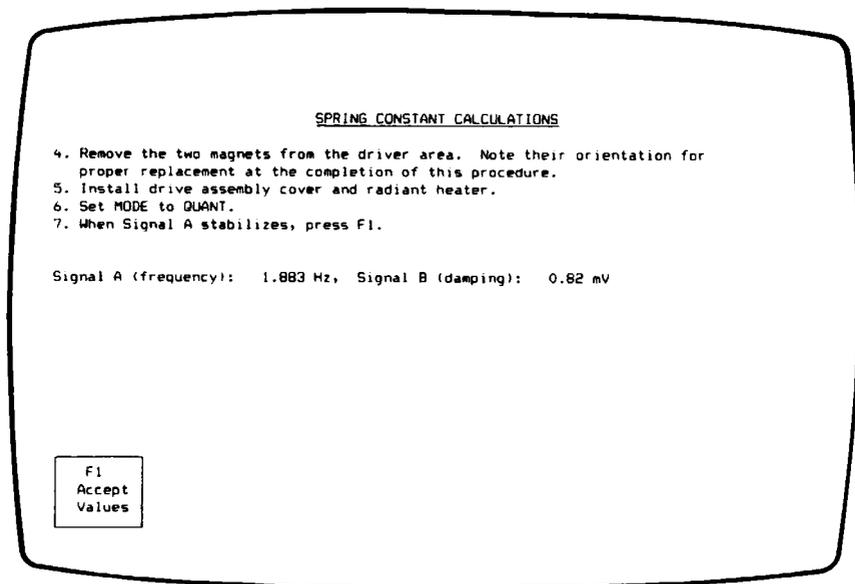


Figure 5.9
Spring Constant Screen

4. Follow the instructions on the screen.
 - Figure 5.10 on the next page shows the location of the driver magnets.

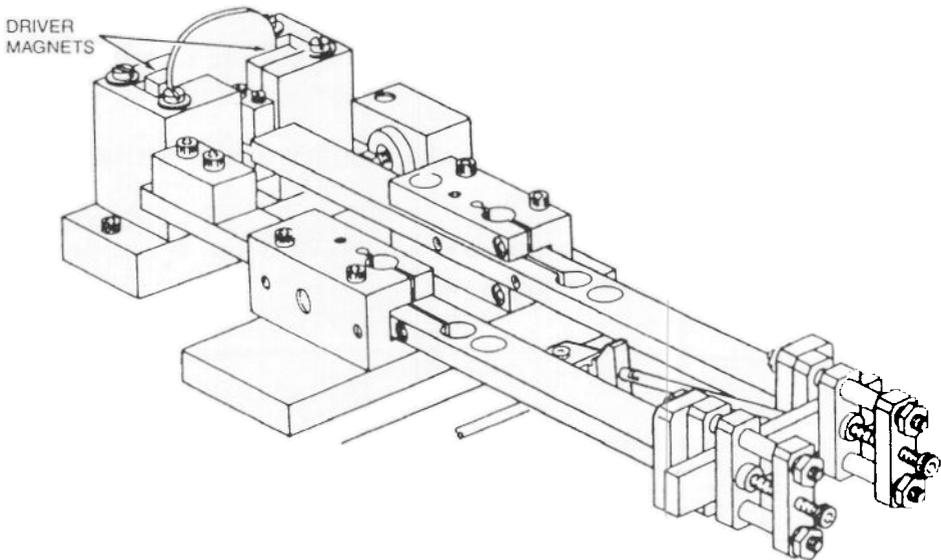


Figure 5.10
Location of the 982 Driver Magnets

NOTE

The drive assembly cover and radiant heater must be installed firmly to prevent stray air currents from affecting the measurements.

5. Press F1 (Accept Values) when signal A stabilizes between 1.8 and 2.0 Hz. You can help it stabilize by holding one of the arms between your thumb and forefinger to damp wide arm oscillations. The program calculates and displays the spring constant, which typically falls between 0.30 and 0.40 N m.
6. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If the spring constant is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, the instrument may have a faulty pivot; call your service representative.

7. Replace the driver magnets.

Determining the Drive Signal Constant

F3 Drive
Signal
Constant

Using this function key allows you to calculate the drive signal constant (C') and the parallel loss stiffness constant (K_n). The drive signal constant converts measured drive level to motor torque to determine how much energy is being applied to the system. The parallel loss stiffness is used in the modulus calculation to compensate for the mechanical friction (drag force) of the instrument. Both C' and K_n have a linear influence on damping calculations.

NOTE

The drive signal constant has been called the tan delta constant in previous literature. The name has been changed to make it consistent with 983 DMA documentation.

Perform this calibration about once a month.

1. Press F3 (Drive Signal Constant). The screen shown on the following page appears.

If the spring constant is out of range, enter N and repeat this calibration, beginning with step 1. If you still get unacceptable results, the instrument may have a faulty pivot; call your service representative.

7. **Replace the driver magnets.**

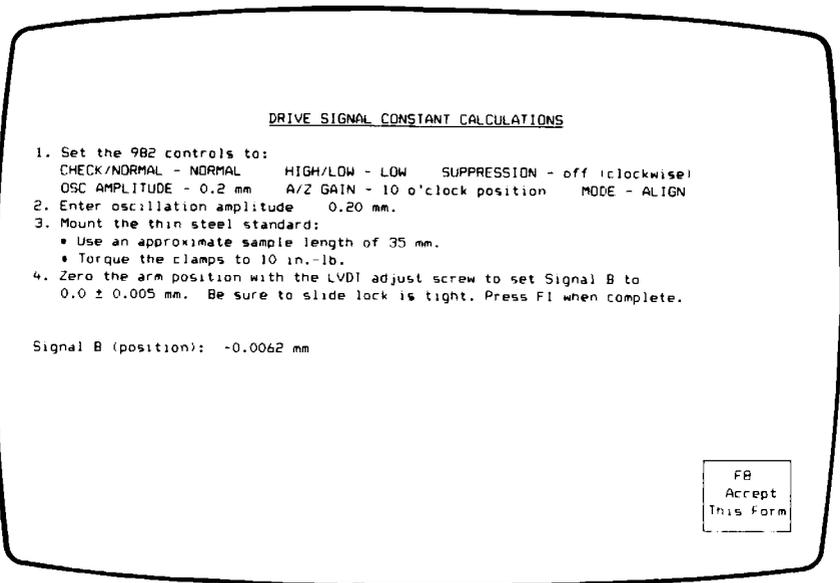


Figure 5.11
Drive Signal Constant Screen

2. Follow the instructions on the screen.
 - Type in the oscillation amplitude, then press F8 (Accept This Form).
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3 on page 43).
 - Use the torque wrench in the DMA accessory kit to torque the clamps to 10 inch-pounds.
 - The procedure for zeroing the arm position is outlined in the box on page 44.

3. Press F1 (Accept Values) when you have signal B zeroed. The following screen appears:

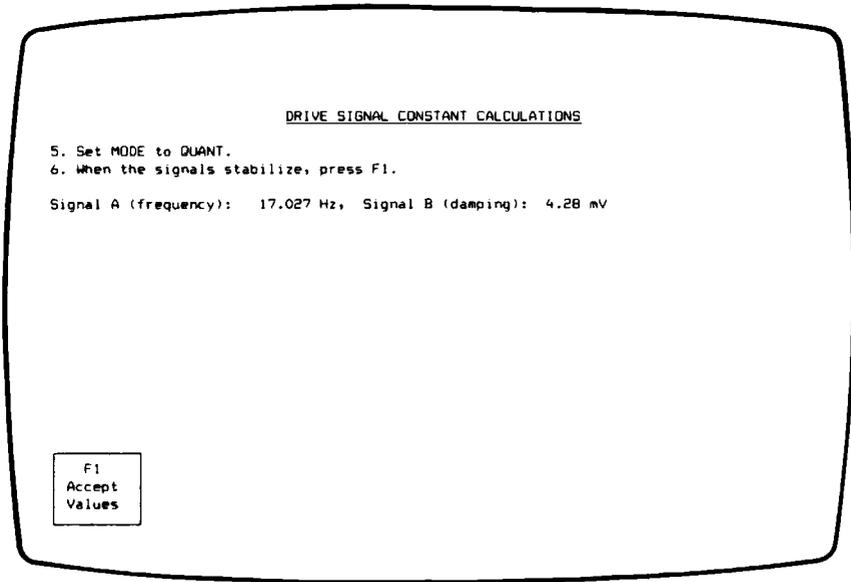


Figure 5.12
Drive Signal Constant Screen

4. Follow the instructions on the screen.
5. Press F1 (Accept Values) when signals A and B stabilize. The screen shown on the following page appears.

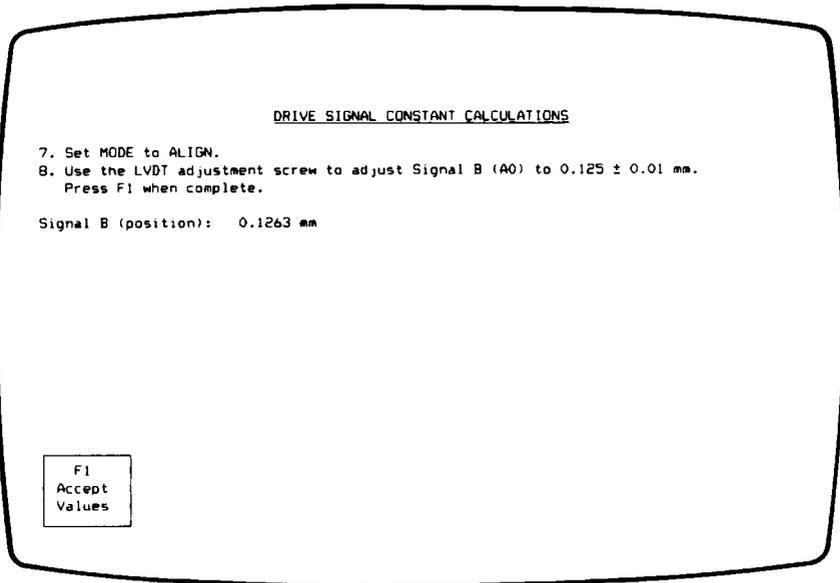


Figure 5.13
Drive Signal Constant Screen

6. Follow the instructions on the screen.
 - The LVDT adjustment knob is located on the back of the drive assembly.

7. Press F1 (Accept Values) when signal B is adjusted. The following screen appears:

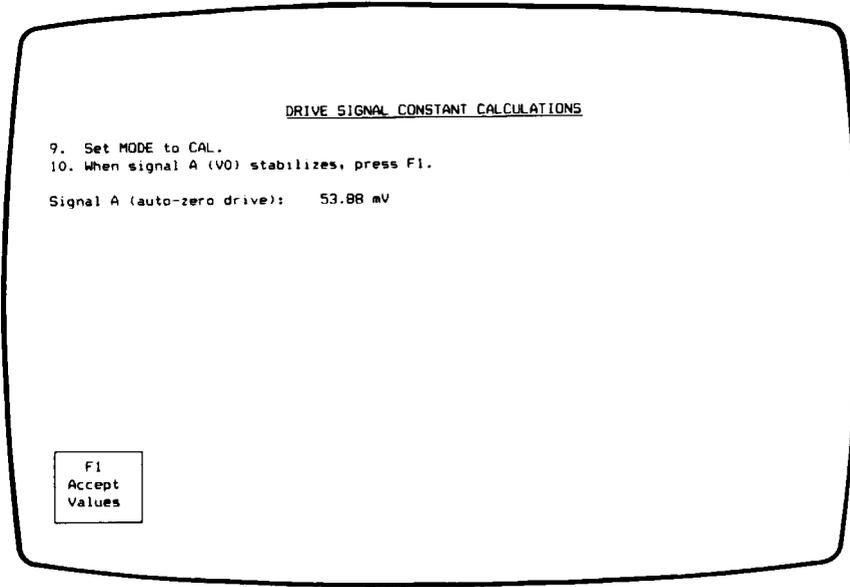


Figure 5.14
Drive Signal Constant Screen

8. Follow the instructions on the screen.

9. Press F1 (Accept Values) when signal A stabilizes. The following screen appears:

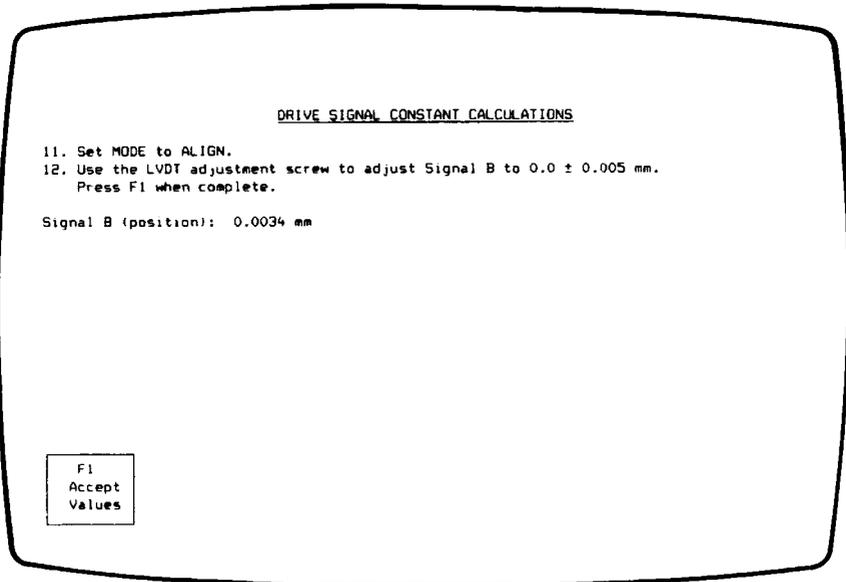


Figure 5.15
Drive Signal Constant Screen

10. Press F1 (Accept Values) when you have signal B zeroed. The program calculates and displays the drive signal and parallel loss stiffness constants. The drive signal constant typically falls between 0.02 and 0.04 mm/(mV sec²), and parallel loss stiffness should fall between 0.05 to 0.25 N m.
11. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If either of the constants is out of range, enter N and repeat the calibration, beginning with step 1. If you still get unacceptable results, call your service representative.

This operation requires a thick, stiff sample of known modulus to calculate the series storage compliance (J_C') and series loss compliance (J_{Cm}'') of the instrument. The series compliance constants correct for the nonrigid responses of the instrument that occur with very stiff samples.

Perform this calibration about once a month.

1. Press F4 (Series Compliance). The following screen appears:

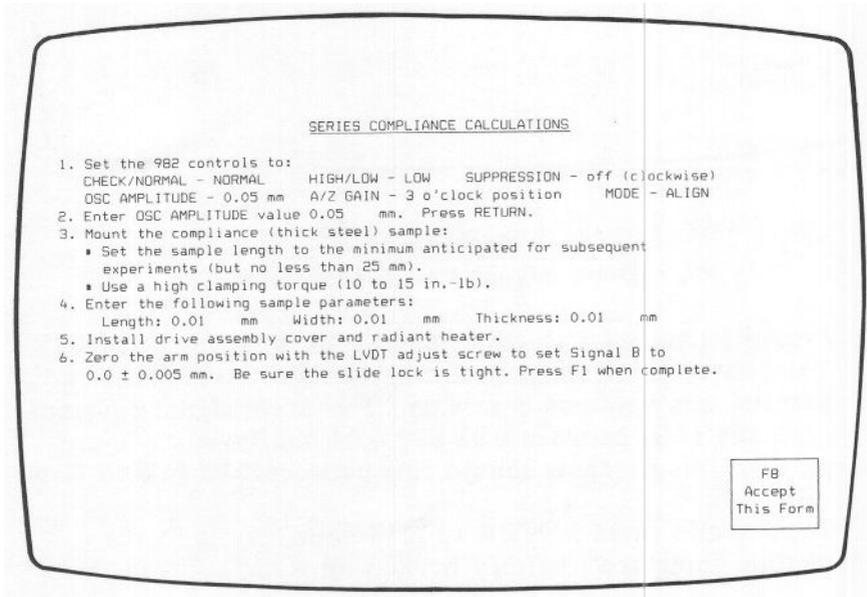


Figure 5.16
Series Compliance Screen

2. Follow the instructions on the screen.
 - If you use an oscillation amplitude other than the default (0.05 mm), be sure to type in your setting.

9. Press F1 (Accept Values) when signal A stabilizes. The following screen appears:

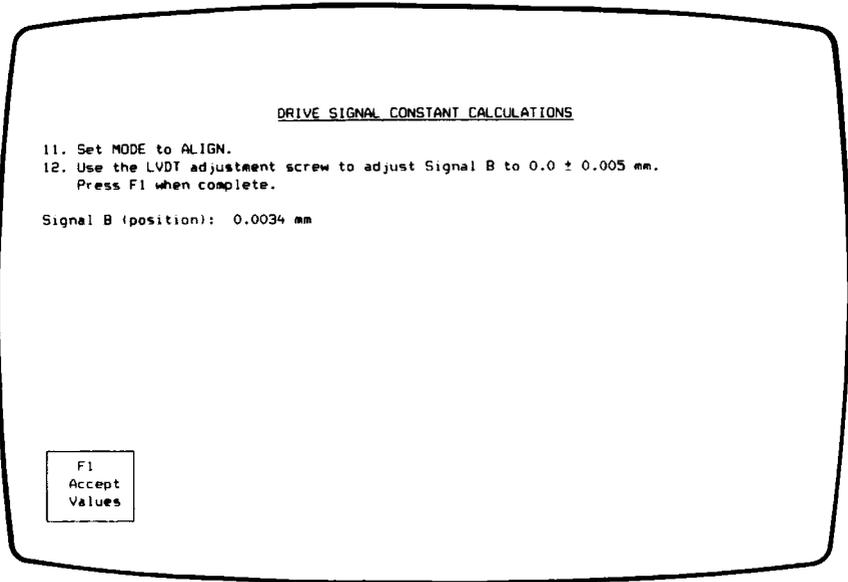


Figure 5.15
Drive Signal Constant Screen

10. Press F1 (Accept Values) when you have signal B zeroed. The program calculates and displays the drive signal and parallel loss stiffness constants. The drive signal constant typically falls between 0.02 and 0.04 mm/(mV sec²), and parallel loss stiffness should fall between 0.05 to 0.25 N m.
11. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If either of the constants is out of range, enter N and repeat the calibration, beginning with step 1. If you still get unacceptable results, call your service representative.

This operation requires a thick, stiff sample of known modulus to calculate the series storage compliance (J_C') and series loss compliance (J_{Cm}'') of the instrument. The series compliance constants correct for the nonrigid responses of the instrument that occur with very stiff samples.

Perform this calibration about once a month.

1. Press F4 (Series Compliance). The following screen appears:

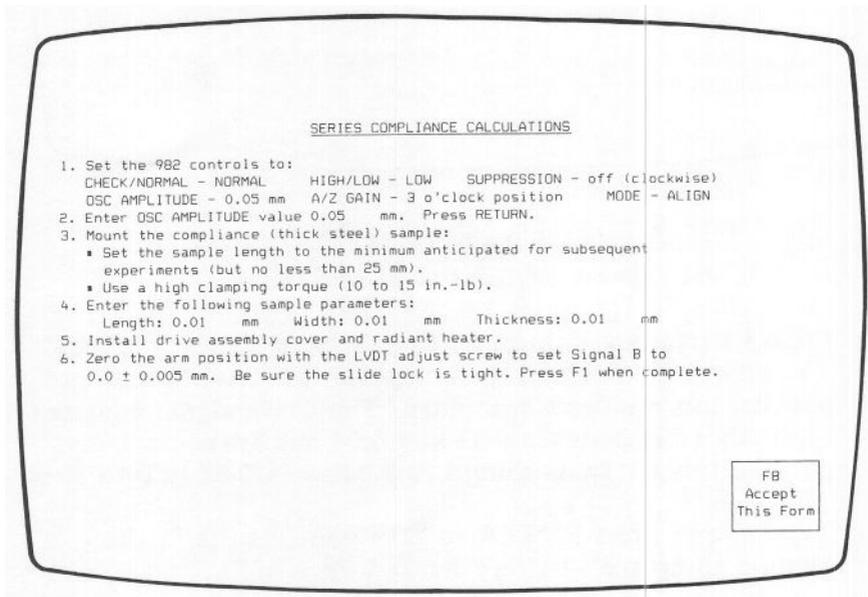


Figure 5.16
Series Compliance Screen

2. Follow the instructions on the screen.
 - If you use an oscillation amplitude other than the default (0.05 mm), be sure to type in your setting.

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3 on page 43).
- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 to 15 inch-pounds.
- Type in the sample dimensions, then press F8 (Accept This Form).

NOTE

Because the drive assembly cover and radiant heater affect the stiffness of the instrument, they must be installed firmly during this procedure.

- The procedure for zeroing the arm position is outlined in the box on page 44.

NOTE

Because the thick steel sample is very stiff, it is critical that the arm position be as close to zero as possible.

3. Press F1 (Accept Values) when you have signal B zeroed. The following screen appears:

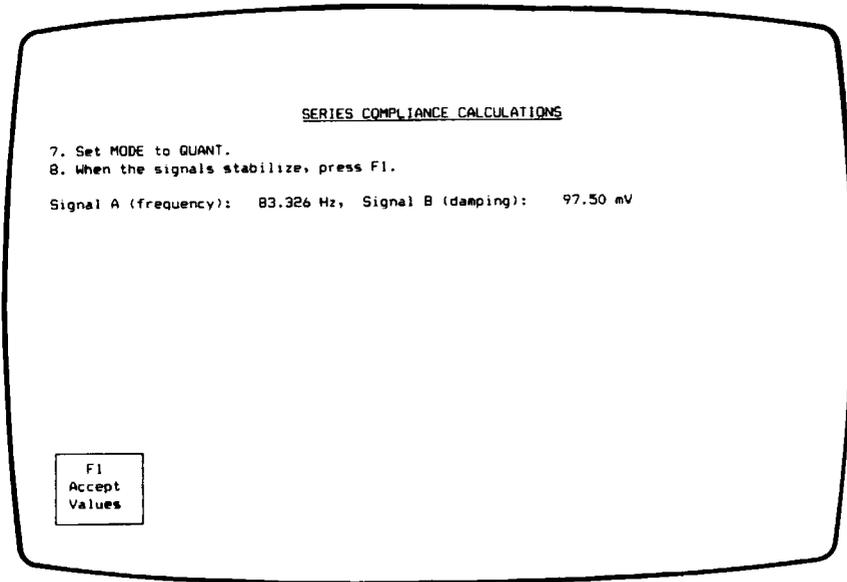


Figure 5.17
Series Compliance Screen

4. Follow the instructions on the screen.
5. Wait for signals A and B to stabilize (approximately 10 minutes), then press F1 (Accept Values). The program calculates and displays the series storage and series loss compliance. The series storage compliance typically falls between 0.5 and $2.0\mu\text{m}/\text{N}$. Typical values for series loss compliance range between 0.005 and $0.050\mu\text{m}/\text{N}$.
6. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If either of the constants is out of range, enter N and repeat the calibration, beginning with step 1. If you still get unacceptable results, the clamps or slide lock may be loose, or the head assembly may not be firmly attached to the module base.

- Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3 on page 43).
- Use the torque wrench in the DMA accessory kit to torque the clamps to 10 to 15 inch-pounds.
- Type in the sample dimensions, then press F8 (Accept This Form).

NOTE

Because the drive assembly cover and radiant heater affect the stiffness of the instrument, they must be installed firmly during this procedure.

- The procedure for zeroing the arm position is outlined in the box on page 44.

NOTE

Because the thick steel sample is very stiff, it is critical that the arm position be as close to zero as possible.

3. Press F1 (Accept Values) when you have signal B zeroed.
The following screen appears:

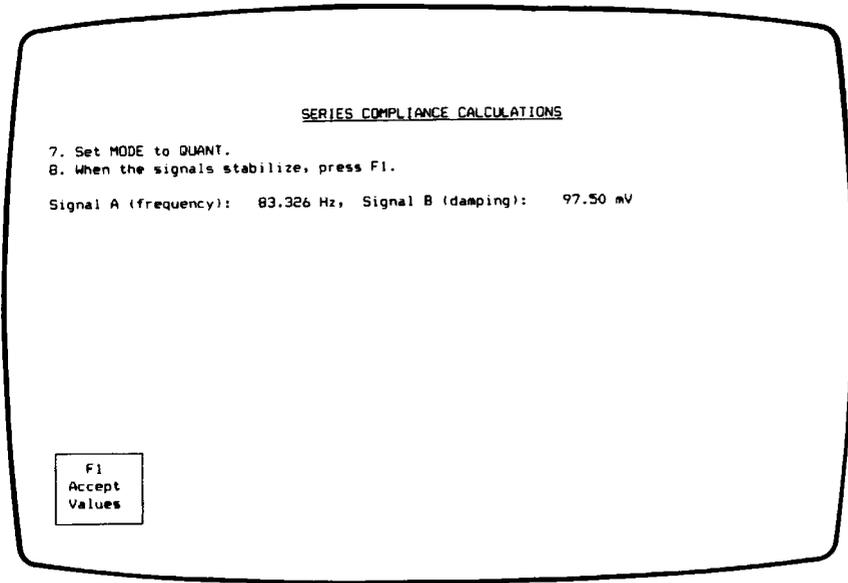


Figure 5.17
Series Compliance Screen

4. Follow the instructions on the screen.
5. Wait for signals A and B to stabilize (approximately 10 minutes), then press F1 (Accept Values). The program calculates and displays the series storage and series loss compliance. The series storage compliance typically falls between 0.5 and $2.0\mu\text{m}/\text{N}$. Typical values for series loss compliance range between 0.005 and $0.050\mu\text{m}/\text{N}$.
6. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

If either of the constants is out of range, enter N and repeat the calibration, beginning with step 1. If you still get unacceptable results, the clamps or slide lock may be loose, or the head assembly may not be firmly attached to the module base.

Correcting for Sample Extension

F6
Length
Correc.

Because sample flexure actually extends beyond the clamp face into the center of the clamps, the sample length you measure must be corrected to reflect this “extension” of the real sample length. The length correction is a term that is added to the measured sample length to correct for this extension. Each sample type and clamping configuration has its own length correction factor.

Perform this calibration each time you use a different sample material or clamping configuration. After you complete the length correction calibration, you can plot your results if you wish (see “Plotting Length Correction Results”).

1. Press F6 (Length Correction). The following screen appears:

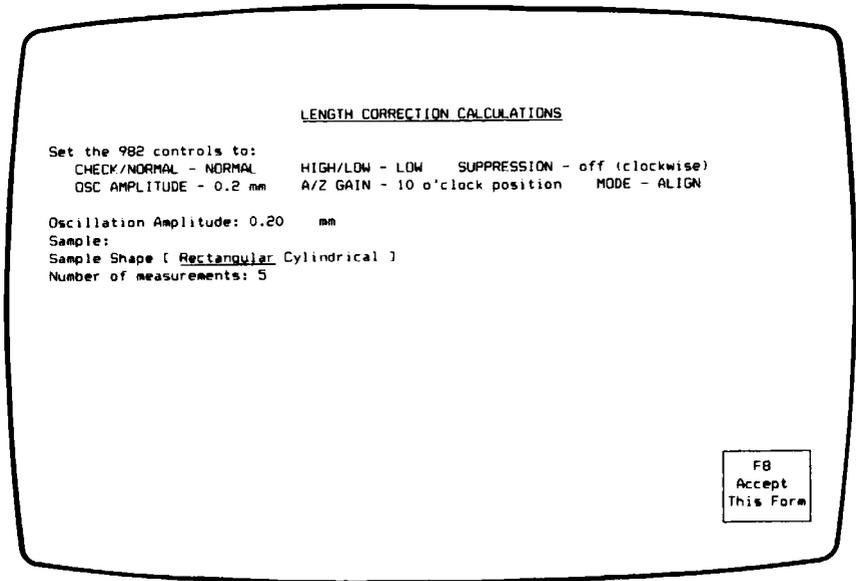


Figure 5.18
Length Correction Screen

2. Follow the instructions on the screen, then type or select the following:
- The oscillation amplitude you will use most often in your experiments with this sample type.
 - A description of the sample.
 - The sample shape.
 - The number of length measurements you wish to perform.
- Then press F8 (Accept This Form).

NOTE

For best results, we recommend that you measure at least five different lengths.

The screen that appears depends on the sample shape:

LENGTH CORRECTION CALCULATIONS
Rectangular Sample Measurements

1. Set MODE to ALIGN.
2. Mount sample for measurement 1
3. Enter the following sample parameters:
Length: 25.00 mm Width: 12.75 mm Thickness: 0.75 mm
4. Zero the arm position with the LVDT adjust screw to set Signal B to 0.0 ± 0.005 mm. Be sure the slide lock is tight. Press F1 when complete.

F8
Accept
This Form

Figure 5.19
Rectangular Sample Measurements Screen #1

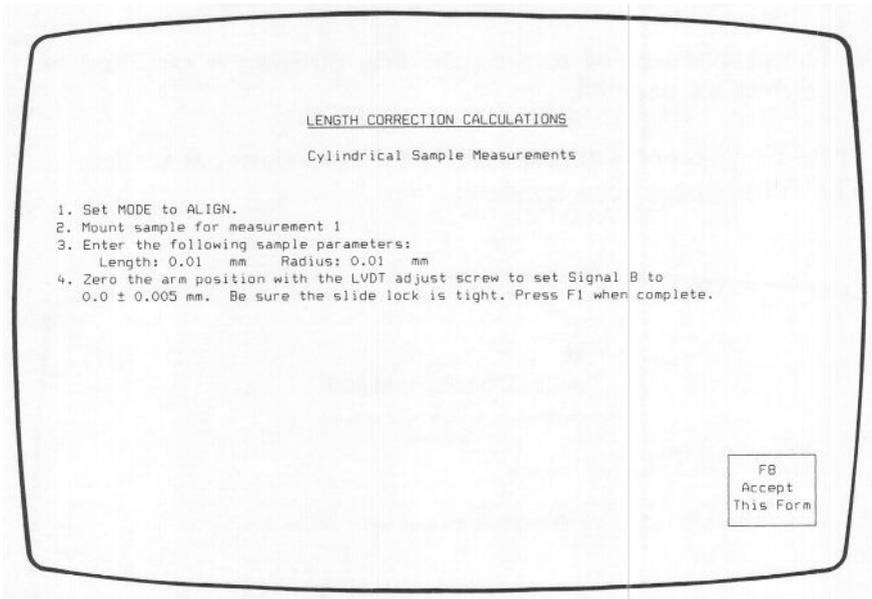


Figure 5.20
Cylindrical Sample Measurements Screen #1

3. Follow the instructions on the screen.
 - For the first length measurement, use the longest sample length you plan to measure. Your sample lengths should cover as wide a range as possible. Start with the maximum length, then work your way down to the minimum length in subsequent measurements.
 - Measure the sample length as the distance between the inner edges of the clamps (see Figure 5.3 on page 43).
 - Use a consistent torque value for each measurement (10 inch-pounds for stiff samples, 3 to 5 inch-pounds for soft samples).
 - Type in the sample dimensions, then press F8 (Accept This Form).

- The radiant heater is optional but recommended, especially if you are measuring a stiff sample.
 - The procedure for zeroing the arm position is outlined in the box on page 44.
4. Press F1 (Accept Values) when you have signal B zeroed. The following screen appears:

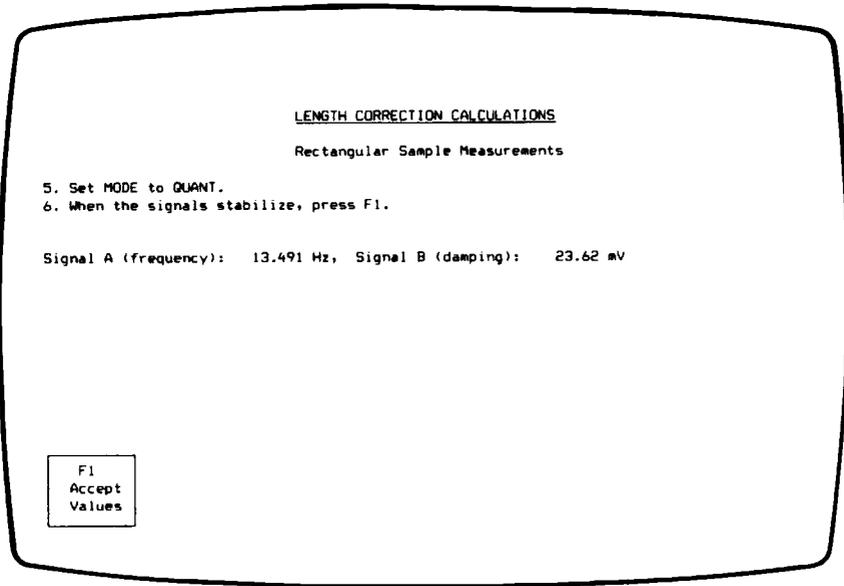


Figure 5.21
Sample Measurements Screen #2

5. Follow the instructions on the screen.
6. Press F1 (Accept Values) when signals A and B stabilize. You are returned to the first Sample Measurements screen.
7. Repeat steps 3 through 6 for each length measurement, working from the longest length to the shortest. Remember to insert the locking pins before loosening the clamps to change the sample length.

When you complete the final measurement, the program calculates the slope, corrected modulus, length correction, and standard deviation.

8. Type Y and press ENTER to save your results in the Module Interface's battery backed-up RAM.

NOTE

If you wish to see a plot of your length correction results, follow the instructions in the section "Plotting Length Correction Results" before unloading the DMA Calibration program. See page 76.

CHAPTER 6

Generating Reports and Plots

Viewing Your Results on the Screen

Displaying the Calibration Constants

F7
Calib.
Constants

The F7 (Calibration Constants) key on the DMA Calibration Opening screen enables you to view all of the calibration constants at once. When you press this key, the program reads the current values in the 983 module or 982 Module Interface and displays them on the screen:

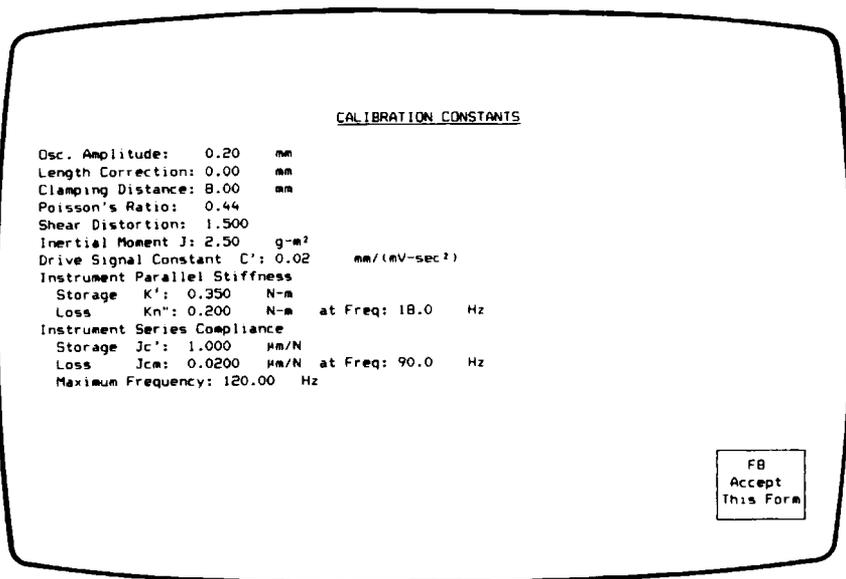


Figure 6.1
Default Calibration Constants Screen (983 DMA)

From this screen, you can change any of the values in the instrument's battery backed-up RAM by moving the cursor to the appropriate field, typing a new entry, and pressing F8 (Accept This Form).

Verifying Your Calibration

To verify the calibration constants, check that the values on the screen fall within or near the typical ranges shown in Table 6.1 or 6.2.

Table 6.1
Typical Calibration Values for the 983 DMA

Calibration Constant	Typical Values
Clamping Distance (B)	8.0 ± 0.2 mm
Inertial Moment (J)	2.4 to 2.7 g m ²
Drive Signal Constant (C')	0.015 to 0.026 mm/(mV sec ²)
Parallel Storage Stiffness (K')	0.30 to 0.40 N m
Parallel Loss Stiffness (K _n '')	0.05 to 0.25 N m
Series Storage Compliance (J _C '')	0.50 to 1.50 μm/N
Series Loss Compliance (J _{Cm} '')	0.005 to 0.050 μm/N
Maximum Frequency (f _∞)	110 to 140 Hz

Table 6.2
Typical Calibration Values for the 982 DMA

Calibration Constant	Typical Values
Clamping Distance (B)	8.0 ± 0.1 mm
Inertial Moment (J)	2.4 to 2.7 g m ²
Drive Signal Constant (C')	0.02 to 0.04 mm/(mV sec ²)
Parallel Storage Stiffness (K')	0.30 to 0.40 N m
Parallel Loss Stiffness (K _n '')	0.05 to 0.25 N m
Series Storage Compliance (J _C '')	0.50 to 2.0 μm/N
Series Loss Compliance (J _{Cm} '')	0.005 to 0.050 μm/N

Obtaining Reports

You can print plots and reports at any point in the program, but they will not show complete results until you have performed all of the calibration steps required.

Printing Results Reports

F8 Report Results

This function key on the DMA Calibration Opening screen enables you to print tabular reports of your calibration results, plot length correction results, and calculate the modulus values for a given sample. When you press F8 (Report Results), the following screen appears:

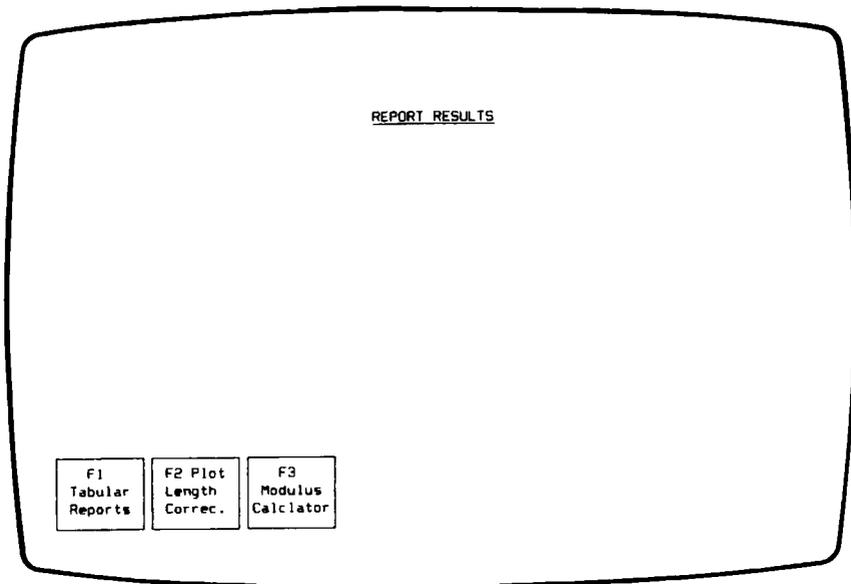


Figure 6.2
Report Results Screen

Printing Tabular Reports

F1 Tabular Reports

To obtain any one of three types of reports:

1. Press F1 (Tabular Reports). The screen prompts you to select the report type and the output device. The three report types are explained in Table 6.3.

Table 6.3
Report Types

Option	Result
Calculations	Prints the results of the instrument calibrations that have been performed for the current module (see Table 6.4). Constants that have not been changed since the program was loaded or the module type was changed are not reported.
Length Correction	Prints the results of the length correction calibration (see Table 6.5).
Instrument Parameters	Prints the calibration constants stored in the 983 (or 982 Module Interface) battery backed-up RAM (see Table 6.6).

2. Use the ARROW keys to select the report type and output device, pressing ENTER after each. The program prints the report, then returns to the DMA Calibration Opening screen.
3. Use the following three tables to interpret your printout.

Table 6.4
Calculations Report

Inertial Moment	
Added Weight	Mass of the hanging weight (M)
Arm Position (no weight)	Absolute arm position without hanging weight (P_0)
Arm Position (with weight)	Absolute arm position with weight (P_M)
Frequency	Resonant frequency of spring (thin) steel sample (f)
Inertial Moment	Instrument calibration constant J
Spring Constant	
Freq, free arm	Measured free arm frequency (f_0)
Parallel Storage Stiffness	Instrument calibration constant K'
Drive Signal Constant	
Osc Amplitude	Oscillation amplitude used during the calibration (a_n)
Frequency	Measured resonant frequency of the thin steel standard (f_n)
Drive Signal (Damping)	Measured amount of energy required to achieve resonance at the above oscillation amplitude (V_n)
Drive Transfer (983 only)	Drive voltage required for static displacement of the arm (V_0/A_0)
Arm Displacement (982 only)	Distance arm was displaced (A_0)

**Table 6.4
Calculations Report (continued)**

Drive Signal Constant (cont'd)	
Offset Drive (982 only)	Voltage required to return arm to zero position (V_0)
Drive Signal Constant	Instrument calibration constant C'
Parallel Loss Stiffness	Instrument calibration constant K_n'' at its measurement frequency
Series Compliance	
Osc Amplitude	Oscillation amplitude used during the calibration (a_m)
Frequency	Measured resonant frequency of the thick steel standard (f_m)
Drive Signal (Damping)	Measured amount of energy required to achieve resonance at the above oscillation amplitude (V_m)
Size	Sample dimensions (length x width x thickness)
ft	Theoretical resonant frequency for the compliance sample (f_t)
Maximum Frequency	Maximum instrument frequency obtained with an infinitely stiff sample (f_∞)
Series Storage Compliance	Instrument calibration constant J_C'
Series Loss Compliance	Instrument calibration constant J_{Cm}'' at its measurement frequency

Table 6.4
Calculations Report (continued)

Phase Zero (983 only)

Standard
deviation
of fit

The standard deviation showing how well the real data points from the phase zero calibration fit the theoretical equation

Table 6.6
Instrument Parameters Report

Oscillation amplitude	Current oscillation amplitude setting
Length correction	Correction factor added to the sample length to compensate for sample motion within clamps (ΔL)
Clamping distance	Distance from the arm center to the clamp face (D)
Poisson's ratio	The ratio of transverse contraction per unit dimension to the elongation per unit length when the sample is subjected to a tensile stress (σ)
Shear distortion	The distortion in a plane when the sample is under shear deformation (α)
Inertial moment	Instrument calibration constant J
Drive signal constant	Instrument calibration constant C'
Parallel storage stiffness	Instrument calibration constant K'
Parallel loss stiffness	Instrument calibration constant K_n'' and the frequency at which it was determined
Series storage compliance	Instrument calibration constant J_C'
Series loss compliance	Instrument calibration constant J_{Cm}'' and the frequency at which it was determined
Maximum frequency (983 only)	Maximum instrument frequency obtained with an infinitely stiff sample (f_∞)

Creating Plots

Plotting Length Correction Results

F2 Plot
Length
Correc.

Each time you perform the length correction calibration, you can plot the results with the F2 (Plot Length Correction) key. Once your results are plotted on the screen, they can be rescaled, customized, and hard copied using the procedures explained on the following pages.

NOTE

Make sure you plot the length correction results before unloading the DMA Calibration program or turning off the system. The length correction plot is not available if you have not performed a length correction calibration since the program was loaded.

1. Press F2 (Plot Length Correction) from the Report Results screen. The program draws an autoscaled plot of the ratio of the modulus to the corrected modulus ($E'/\text{corrected } E'$) versus X_i (reciprocal of the sample length) as shown in the figure on the following page.

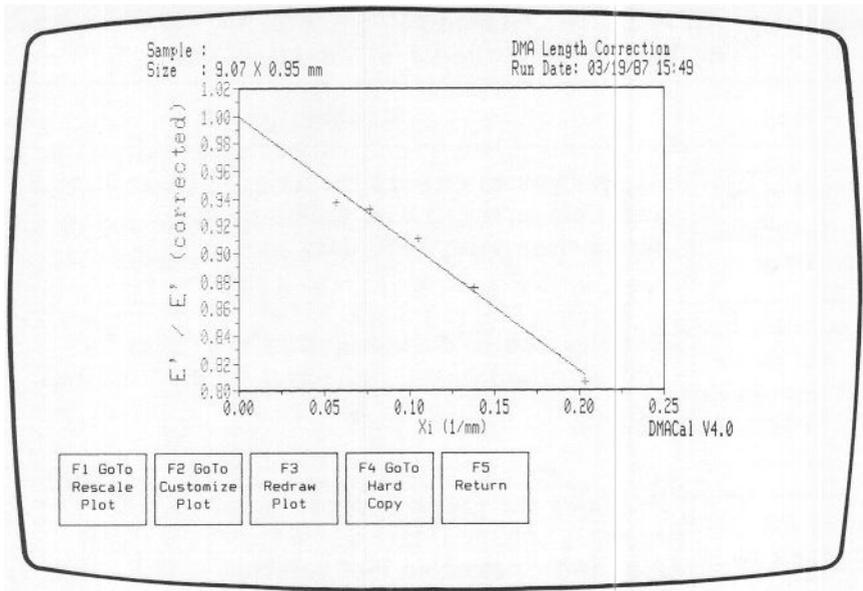


Figure 6.3
Length Correction Plot Screen

The function keys at the bottom of the Length Correction Plot screen are explained in the table on the following page. The keys marked with an asterisk are further detailed in the following sections.

Table 6.7
The Length Correction Plot Function Keys

Key	Explanation
* F1 GoTo Rescale Plot	Allows you to change the axis limits and label and tick intervals and set up plots for comparison with 1090 data.
* F2 GoTo Customize Plot	Enables you to custom-design your plot by adding annotations and selecting grid options.
F3 Redraw Plot	Redraws the graph according to the most recently selected options under the Rescale Plot and Customize Plot screens.
* F4 GoTo Hard Copy	Enables you to print your graph on the plotter or printer.
F5 Return	Returns you to the DMA Calibration Opening screen.
*This key is further detailed in a later section.	

Rescaling the Plot

When you press F1 (Go To Rescale Plot), the following screen will result:

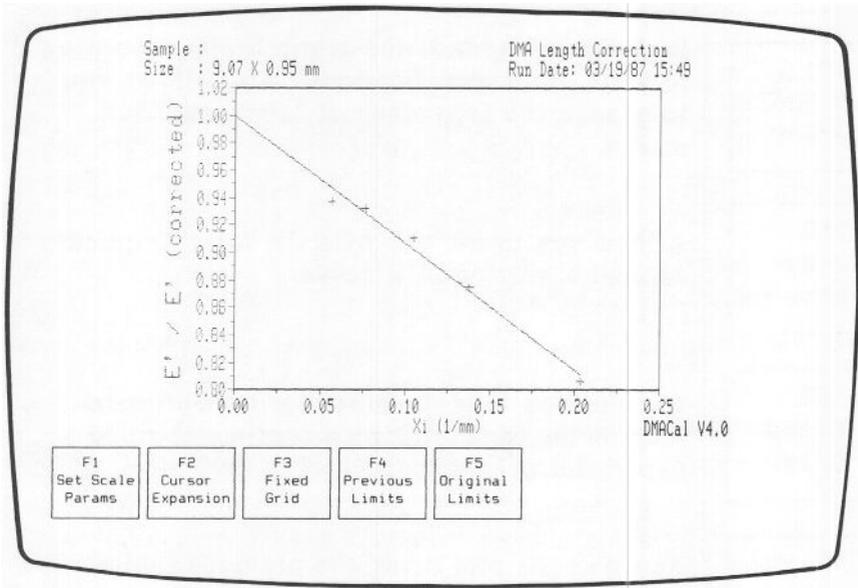


Figure 6.4
Rescale Plot Screen

At the bottom of the screen a set of function keys appears. These function keys allow you to perform the operations described in Table 6.8 on the next page.

Table 6.8
The Rescale Plot Function Keys

Key	Explanation
<div style="border: 1px solid black; padding: 5px; text-align: center;"> F1 Set Scale Params </div>	<p>Reduces or expands the graph limits according to your exact specifications. Also allows you to change the frequency of labels and tick marks.</p>
<div style="border: 1px solid black; padding: 5px; text-align: center;"> F2 Cursor Expansion </div>	<p>Allows you to use the ARROW keys to quickly expand a portion of a curve.</p>
<div style="border: 1px solid black; padding: 5px; text-align: center;"> F3 Fixed Grid </div>	<p>Enables you to scale your plot in centimeter increments for plotting on centimeter-ruled paper and/or comparison with 1090 plots.</p>
<div style="border: 1px solid black; padding: 5px; text-align: center;"> F4 Previous Limits </div>	<p>Redraws the plot using the previous limits.</p>
<div style="border: 1px solid black; padding: 5px; text-align: center;"> F5 Original Limits </div>	<p>Autoscales the plot to the minimum and maximum data values.</p>

The sections that follow will describe how to use each individual key.

Changing Axis Parameters

F1
Set Scale
Params

With the F1 (Set Scale Parameters) key, you can select exact start and stop limits for the x - and y -axes to reduce or expand the graph and change the label and tick intervals.

1. Press F1 (Set Scale Parameters) from the Rescale Plot screen. The following screen will appear:

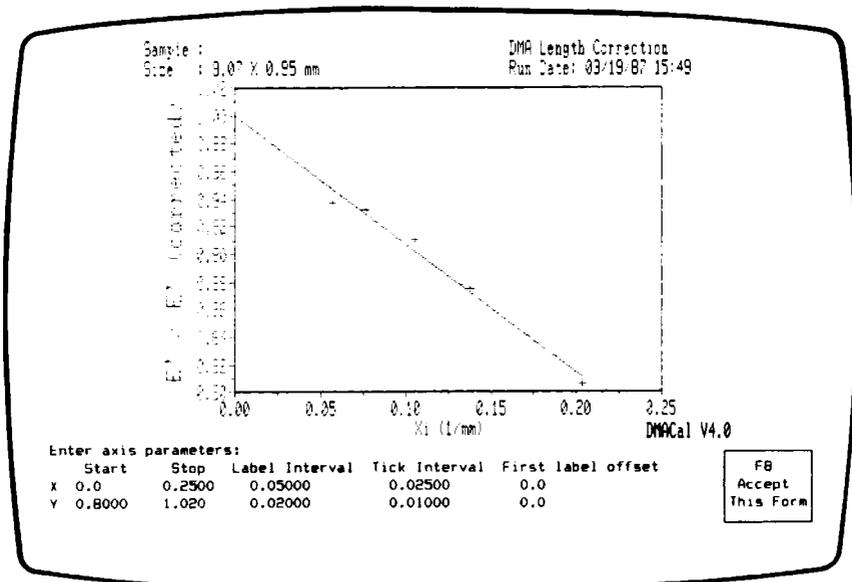


Figure 6.5
Scale Parameters Screen

2. Enter the new start limit for the x -axis. The start limit determines the signal value at which the x -axis begins.
3. Enter the new stop limit for the x -axis. The stop limit determines the signal value at which the x -axis ends.

4. Enter the interval you wish to appear between labeled points on the x -axis. This entry determines how often the x -axis is numbered. For example, if your x -axis is X_i , and you choose a label interval of 0.05, the x -axis is labeled as shown below:

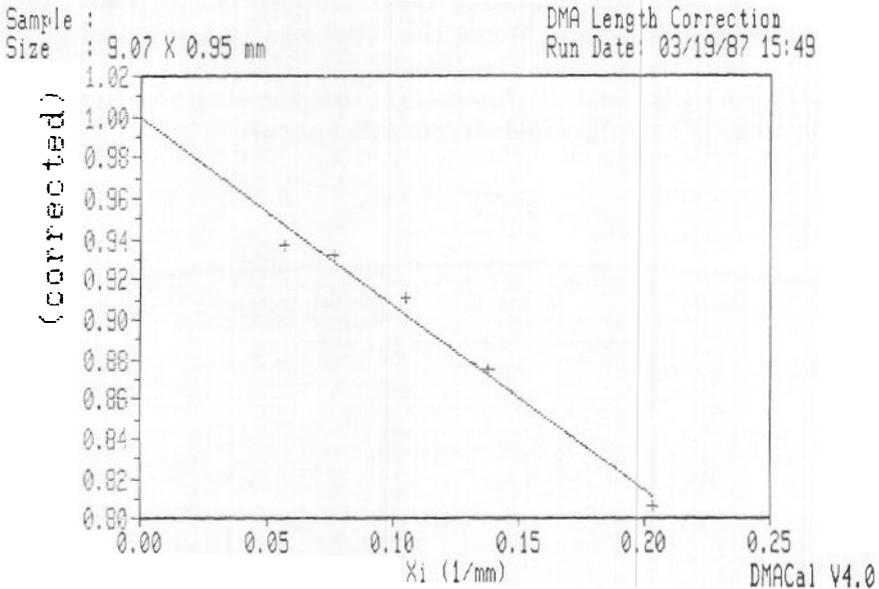


Figure 6.6
Label Interval of 0.05 on the X -axis

5. Enter the interval you wish to appear between tick marks for the x -axis. The tick interval determines how often the axis is marked to show increments in the signal. For example, if your x -axis represents X_i , and the tick interval is 0.025, the axis is ticked as shown on the next page.

Sample :
Size : 9.07 X 0.95 mm

DMA Length Correction
Run Date: 03/19/87 15:49

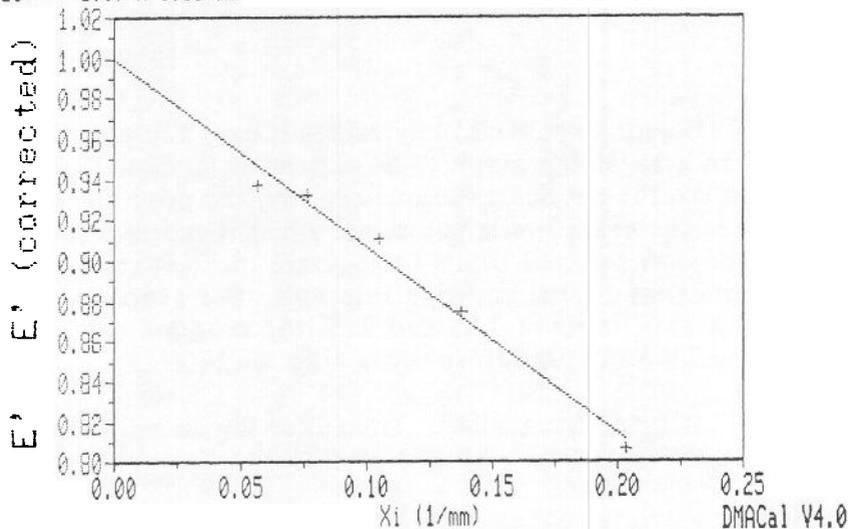


Figure 6.7
Tick Interval of 0.025 on the X-axis

6. Enter the offset to the place on the axis where you want the first label to appear.

The first label offset option allows you to start labeling the graph at any point you choose. For example, if the axis starts at 0.03, but you want the first label to appear at 0.05, you would enter 0.02 (the difference between the start limit and the actual point on the axis at which you want the first label to appear).

7. Repeat steps 2 through 6 for the y-axis. When you are satisfied with the scale parameters, press F8 (Accept This Form). The limits selected are shown in a red box, and the screen prompts:

Use these axis limits? Yes

8. Press F8 (Accept This Form) to redraw the plot with the new parameters. To answer "No," type N and ENTER to rescale the plot again.

Expanding a Portion of the Graph

F2
Cursor
Expansion

The F2 (Cursor Expansion) key makes it easy for you to mark off an area of the graph to be expanded to show further detail. Unlike the Set Scale Parameters key, the program does not use the exact limits you select when it redraws the plot; it uses your selected limits to generate the best start and stop limits and label and tick intervals. For example, if you select x-axis limits of 1.03 and 9.89, the program rounds them off to 1.0 and 10.0 before redrawing the plot.

1. Press F2 (Cursor Expansion). The following screen below appears:

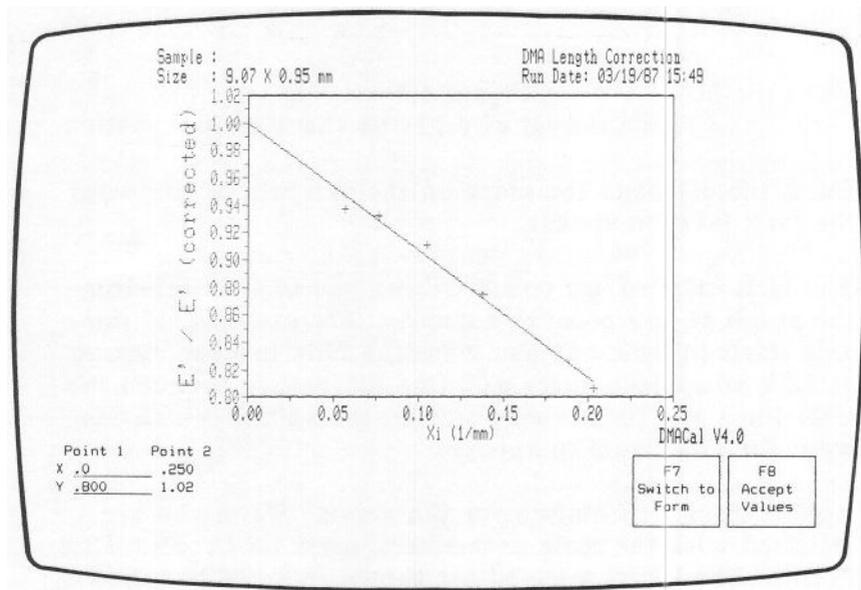


Figure 6.8
Cursor Expansion Screen

2. Enter the axis limits for points 1 and 2 using one of two modes:

- *Cursor mode.* This mode uses a red indicator line that can be moved with the ARROW keys.
- *Form mode.* In this mode, you enter numeric values to select the axis limits.

Both of these modes are explained as follows.

Using Cursor Mode

In cursor mode, you can select new axis limits by moving the axes with the ARROW keys. SHIFT-ARROW moves the cursor 5 pixels, ALT-ARROW moves the cursor 10 pixels, and CTRL-ARROW moves the cursor 25 pixels.

- Select point 1 (x - and y -axes) by using the ARROW keys. Move the vertical and horizontal indicators so that they cross at the desired point, then press ENTER. (You will notice that the numbers change according to the position of the indicator lines.)
- Repeat the procedure above for point 2.

NOTE

You can enter the values for points 1 and 2 in any order you wish. Use the F1 and F2 keys to switch between points 1 and 2.

The limits you selected are shown in a red box on the graph, and the screen prompts:

Use these limits? Yes

- Press ENTER or F8 (Accept This Form) to enter "Yes." The program will redraw the plot with the axis limits shown on the screen.

Enter N to reselect the plot limits.

Using Form Mode

In form mode, you can select new axis limits by entering numeric values directly from the keyboard.

- a. Press F7 (Switch to Form). The screen switches to form mode:

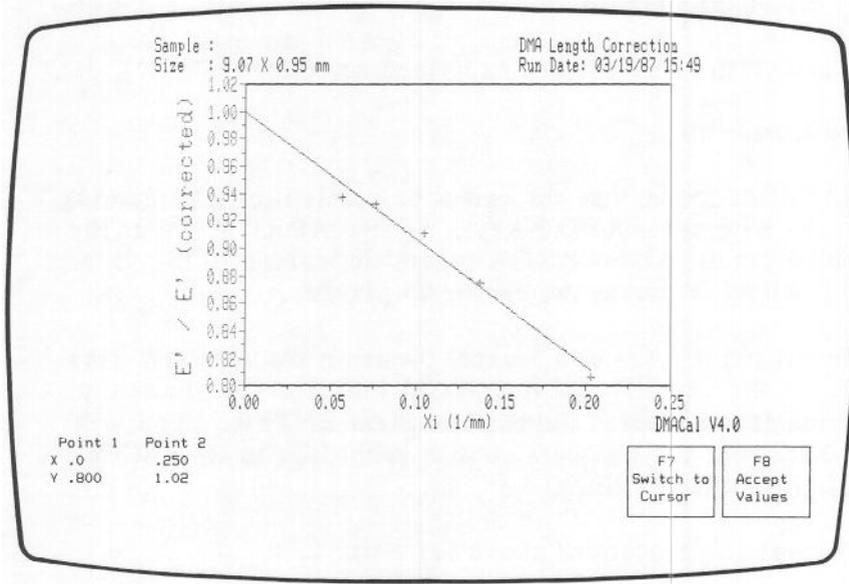


Figure 6.9
Form Mode Screen

Note that the F7 key changes to F7 (Switch to Cursor), which you can use to toggle back to cursor mode.

- b. Enter the number at which you want the x -axis to begin.
- c. Enter the number at which you want the x -axis to stop.
- d. Repeat steps b and c for the y -axis. The limits you selected are shown in a red box, and the screen prompts:

Use these limits? Yes

- e. Press F8 (Accept This Form) to redraw the graph with the axis limits shown, or enter N to reselect the limits.

Comparing Your Plots with 1090 Data

F3 Fixed Grid

The F3 (Fixed Grid) key enables you to set up the axis scaling in centimeter intervals on the plotter. The centimeter scaling allows you to overlay your plots on Du Pont 1090 Thermal Analyzer plots for comparison.

With the Fixed Grid key, you can specify:

- The number of units contained in each centimeter of a sheet of centimeter-ruled graph paper.
- The frequency with which the centimeter-ruled graph paper is labeled.

NOTE

You may need to change the plotter reference points (P1 and P2) on your plotter to obtain plots in precise centimeter increments (see your Hewlett-Packard plotter manual).

1. Press F3 (Fixed Grid) from the Rescale Plot screen.

The screen shown on the following page appears.

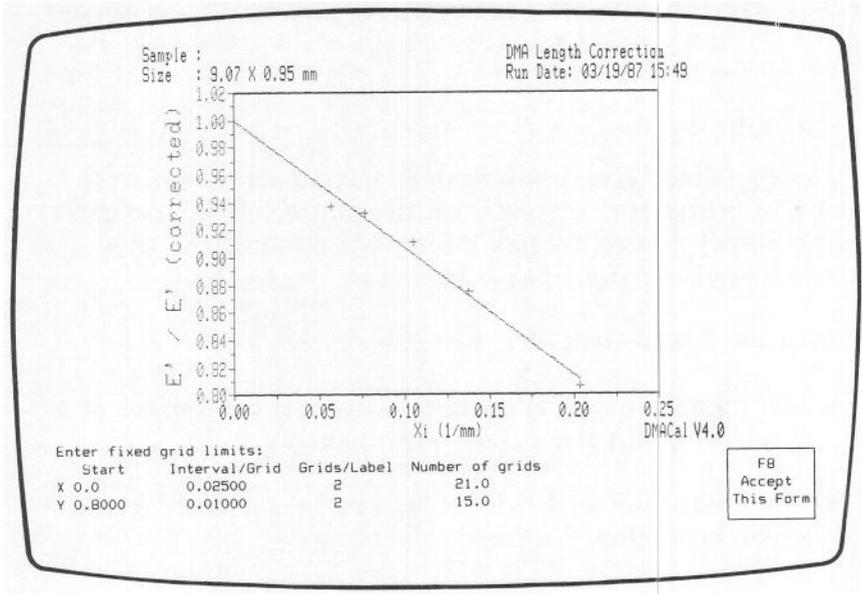


Figure 6.10
Fixed Grid Screen

2. Enter the start limit for the x-axis.
3. Enter the desired number of intervals per grid (number of temperature or time units plotted per centimeter).
4. Enter the number of grids (centimeters) you want to appear between each label on the x-axis. For example, if you enter 2, the x-axis will be labeled at every second centimeter.

The number of grids indicates the number of centimeter grids that will appear on the axis. This number cannot be changed.

5. Repeat steps 2 through 4 for the y -axis. The program uses your selections to determine the stop limits for the x - and y -axes. The resulting limits are shown in a red box, and the screen prompts:

Use these limits? Yes

6. Press F8 (Accept This Form) to redraw the plot with the axis limits shown, or enter N to reset the axis limits.

Redrawing the Previous Plot

F4
Previous
Limits

The F4 (Previous Limits) key redraws the plot as it appeared before the *most recent* scale changes. Only the scaling options selected from the Rescale Plot screen are affected by this key; any changes made in annotation remain in the most recent settings.

If you press the Previous Limits key repeatedly, the screen toggles between the two most recent screens (see Figure 6.11); it does not continue to move backward until the original plot is obtained.

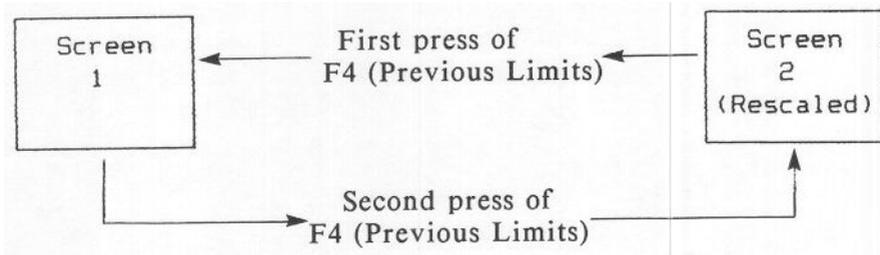
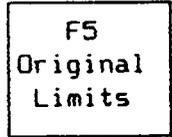


Figure 6.11
Action of the Previous Limits Key

Redrawing the Original Plot



The F5 (Original Limits) key autoscales the curves to the minimum and maximum data values, determining the best limits and tick intervals for that range. Only the scaling options selected from the Rescale Plot screen are affected by this key.

Customizing Your Plot

You can add annotations and grids to the Length Correction plot. This is accomplished with a set of function keys on the Customize Plot screen, accessed by the F2 (GoTo Customize Plot) from the Length Correction Plot screen. The following screen appears:

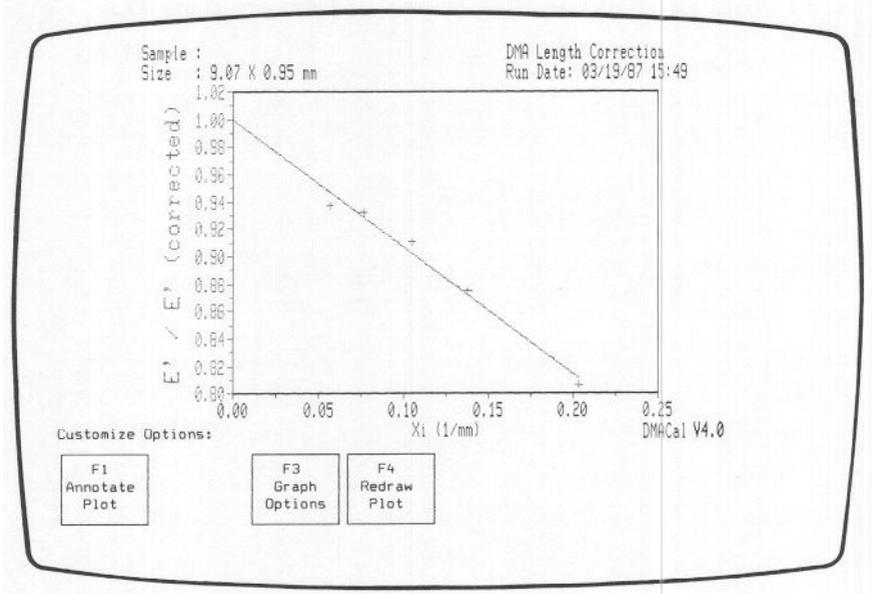


Figure 6.12
Customize Plot Screen

The Customize Plot function keys are detailed on the following pages.

Creating Annotations

F1
Annotate
Plot

The F1 (Annotate Plot) key is used to place comments using alphanumeric characters anywhere on the plot. The description given here covers all annotation options available to you. However, you can skip steps labeled "OPTIONAL" and vary the order of the steps if you wish.

1. Press F1 (Annotate Plot) from the Customize Plot screen. The following screen appears:

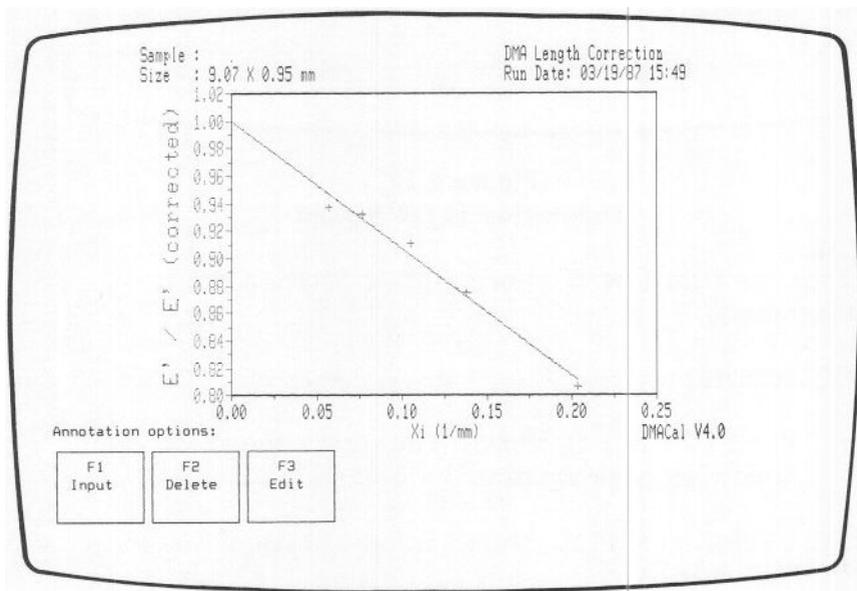


Figure 6.13
Annotation Options Screen

2. Press F1 (Input) from the Annotation Options screen. The Annotation Input screen appears.

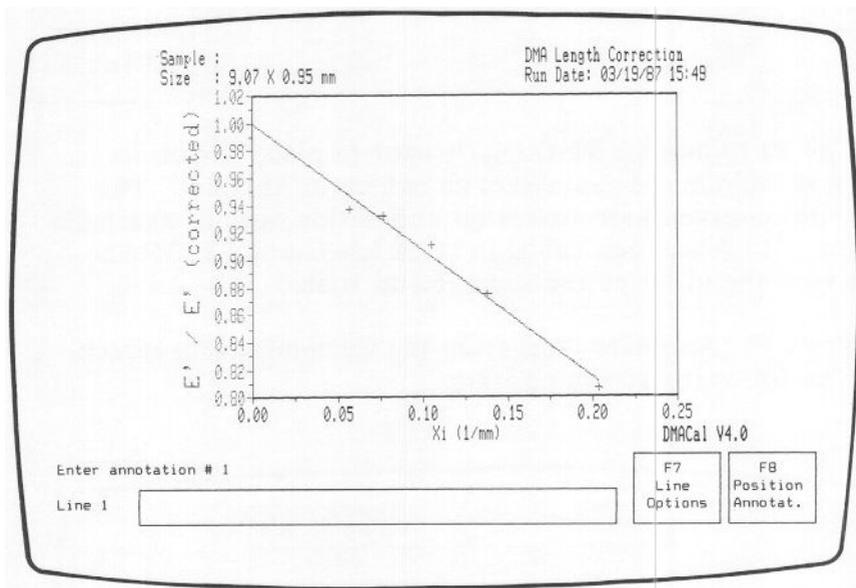


Figure 6.14
Annotation Input Screen

3. Type the first line of your graph annotation (up to 50 characters).
4. OPTIONAL:
 - a. Press F7 (Line Options). The screen shown on the following page appears.

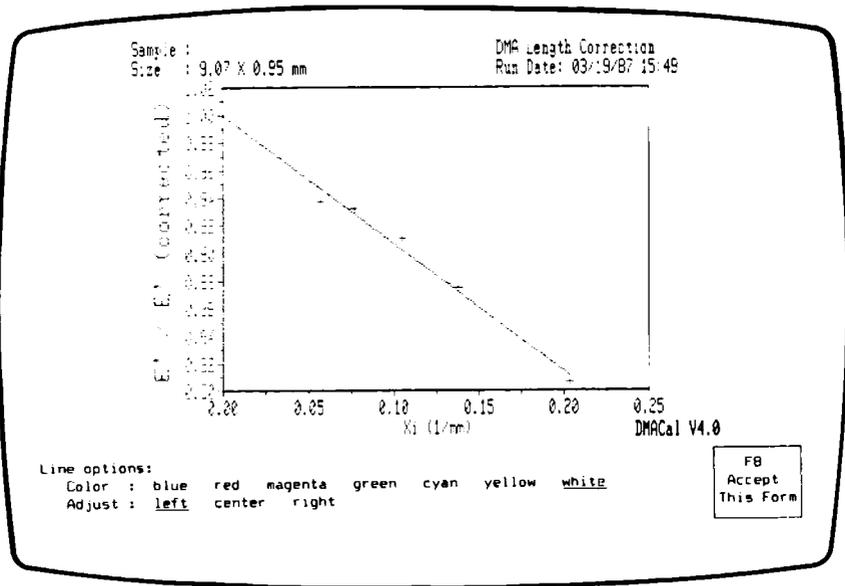


Figure 6.15
Line Options Screen

- b. Use the ARROW keys to select the color in which you want the annotation line to appear, then press ENTER. The color you choose becomes the default color for subsequent lines in the annotation; previously entered lines are not affected.
- c. Use the ARROW keys to select the line adjust option, then press ENTER (see Table 6.9 for explanation). The line adjust option you choose becomes the default setting for subsequent lines in the annotation; previously entered lines are not affected.

After you select the adjust option, the Annotation Input screen reappears.

Table 6.9
Line Adjust Options

Option	Result	
Left	Lines are left justified: <div data-bbox="323 256 902 360" style="border: 1px solid black; padding: 5px;"> Slope of line equals -1. </div>	
Center	Lines are centered: <div data-bbox="325 416 904 520" style="border: 1px solid black; padding: 5px;"> Slope of line equals -1. </div>	
Right	Lines are right justified: <div data-bbox="325 587 904 691" style="border: 1px solid black; padding: 5px;"> Slope of line equals -1. </div>	

5. Press ENTER, then repeat step 3 if you want to add another line to the annotation. If you want to change the line options for each line, repeat step 4. After you type your last line, press F8 (Position Annotation). The screen on the following page appears.

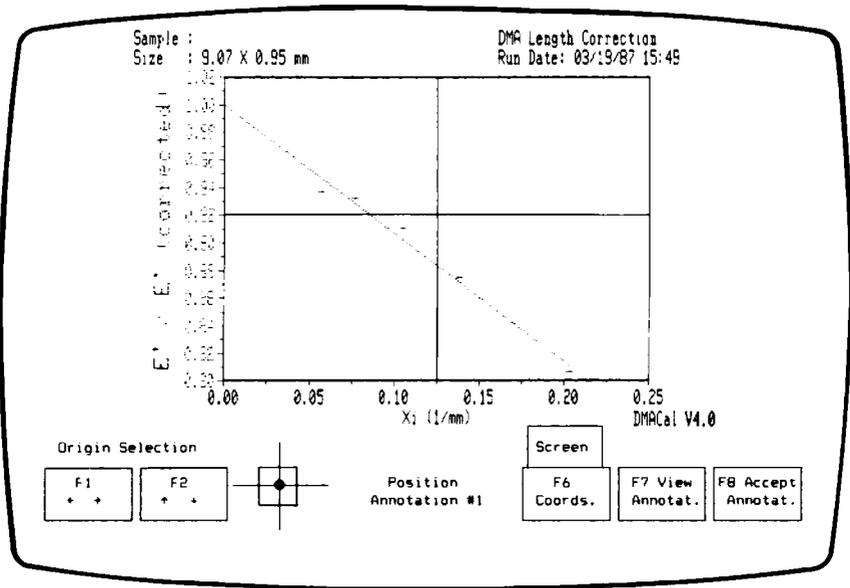
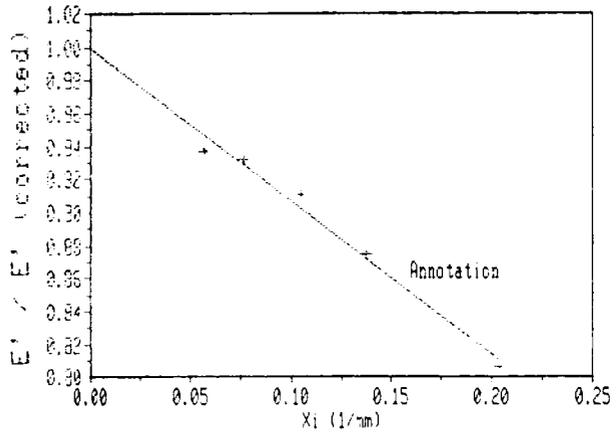


Figure 6.16
Annotation Position Screen

6. Press F6 to choose screen or curve coordinates for annotation positioning. The coordinates you choose affect how the annotation is located when the plot is rescaled (see Figure 6.17).

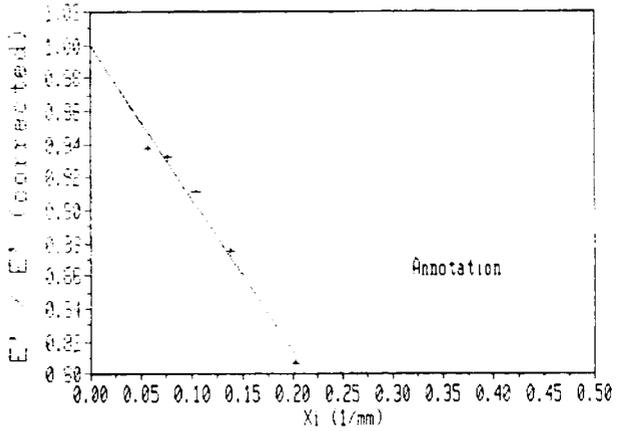
Before Rescaling



After Rescaling

Screen Coordinates

The annotation is located according to its position on the screen, regardless of how many times you later rescale the plot.



Curve Coordinates

The annotation is located according to its position on the axes.

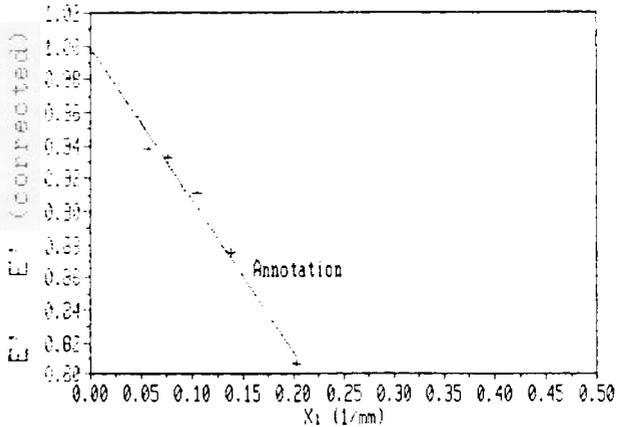


Figure 6.17
Screen versus Curve Coordinates

7. Press F1 and F2 repeatedly to move the white position indicator box (in the diagram to the right of the keys). The position of this box determines where the annotation will originate in relation to the red cross-hairs on the graph. For example, if you move the box to the lower right corner of the cross-hairs (see Figure 6.18), the origin of the annotation box will be its upper left corner. The origin you choose is the area of the box that will appear where you place the cross-hairs in step 8.

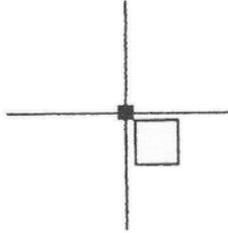


Figure 6.18
Annotation Position Indicator Box in Lower Right Corner

8. Use the ARROW keys to move the cross-hairs to where you want the annotation to appear, then press F8 (Accept Annotation).

NOTE

To move the cross-hairs quickly, hold down the SHIFT, ALT, or CTRL key, then press the ARROW key. The relative speeds are:

ARROW (alone) = 1
SHIFT-ARROW = 5

ALT-ARROW = 12
CTRL-ARROW = 25

— NOTE —

If you are not certain where you want to place the annotation, press F7 (View Annotation) instead of F8 (Accept Annotation). The annotation will appear on the screen temporarily. To reposition the annotation, move the cross-hairs again, and the annotation will disappear. When you are satisfied with the placement of the annotation, press F8 (Accept Annotation).

After you press F8 (Accept Annotation), the Annotation Options screen reappears. You can now:

- Input another annotation by pressing F1 (Input). The annotation number increases by one.

— NOTE —

The maximum number of annotations per graph is 10, and the maximum number of lines for all annotations is 50.

- Delete an annotation by pressing F2 (Delete) (see page 101).
- Edit an existing annotation by pressing F3 (Edit) (see page 102).
- Return to the Customize Plot screen by pressing ESCape.
- Return to the Length Correction Plot screen by pressing ESCape twice.

Deleting Annotations

F2
Delete

To remove any annotations from the screen, follow these steps:

1. Press F2 (Delete) from the Annotation Options screen. If your graph contains only one annotation, the program deletes the annotation and prompts:

Okay?

Skip to step 3.

If your graph contains more than one annotation, the screen prompts:

Select annotation #: 1 (1--x)

(x is the highest annotation number). Go on to step 2.

2. Type the number of the annotation you wish to delete, then press ENTER. The program deletes the annotation you selected, then prompts:

Okay?

3. Press ENTER to complete the deletion or enter N to restore the deleted annotation.

NOTE

Any existing annotations following the one you delete (i.e., with a higher number) are renumbered. For example, if you have three annotations and you delete annotation #2, annotation #3 then becomes annotation #2.

Editing Annotations

F3 Edit

You can change the annotations already placed on the graph using the following procedures:

1. Press F3 (Edit) from the Annotation Options screen. If your graph contains only one annotation, the annotation temporarily disappears, and the first line appears on the bottom left of the screen. Skip to step 3.

If your graph contains more than one annotation, the screen prompts:

Select annotation #: 1 (1--x)

(x is the highest annotation number). Go on to step 2.

2. Type the number of the annotation you wish to edit, then press ENTER. The annotation you selected temporarily disappears, and the first line appears on the bottom left of the screen.
3. From this point, you can change the text of the annotation, change the color, or relocate the annotation by following steps 3 through 8 of "Creating Annotations" (pages 94 to 99).

Selecting a Grid Type

F3
Graph
Options

You can use the F3 (Graph Options) key to enhance your plot with grids originating from the tick marks on each axis.

1. Press F2 (Graph Options) from the Customize Plot screen. The following screen appears:

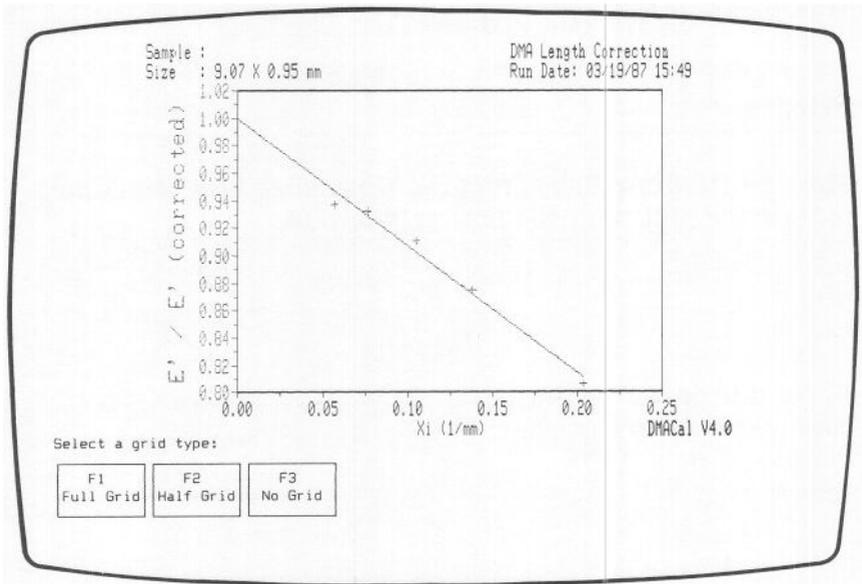


Figure 6.19
Graph Options Screen

2. Press one of the displayed function keys to select the grid type you wish to use. The three grid options are explained in Table 6.10.

Table 6.10
Curve Grid Options

Key	Explanation
F1 Full Grid	All ticks on the axes are used in creating the grid.
F2 Half Grid	Only the labeled ticks on the axes are used in creating the grid.
F3 No Grid	No grid is drawn.

3. Press F4 (Redraw Plot) from the Customize Plot screen to redraw the plot with the new grid option.



Printing Plots

F4 GoTo Hard Copy

To obtain a copy of the plot currently on the screen:

1. Press F4 (GoTo Hard Copy) from the Length Correction Plot screen. The screen prompts you to select the output device:

Select a device for the hard copy.

F1 Plotter	F2 Printer
---------------	---------------

2. Check that the plotter or printer is online, then press F1 (Plotter) or F2 (Printer). As the plot is printed, the F1 key changes to:

F1 Abort

You can cancel the hard copy command by pressing F1. When the F1 (Abort) key disappears, you can go on to another operation.

If your system is equipped with the plot spooler, which comes with the Simultaneous Run/Analyze option, the F1 (Abort) key will disappear more quickly; the system is now free for other operations while a plot is being made. No extra steps are necessary to activate the plot spooler; it is activated automatically each time you start a plot with the F4 (GoTo Hard Copy) key. After you start a plot, you can go on to another operation as soon as the F1 (Abort) key disappears from the screen.

The F7 (Plot Spooler) key on the Data Analysis screen enables you to control the spooler once a plot has been started. Press this key, and you will see the Plot Spooler Control screen, shown below:

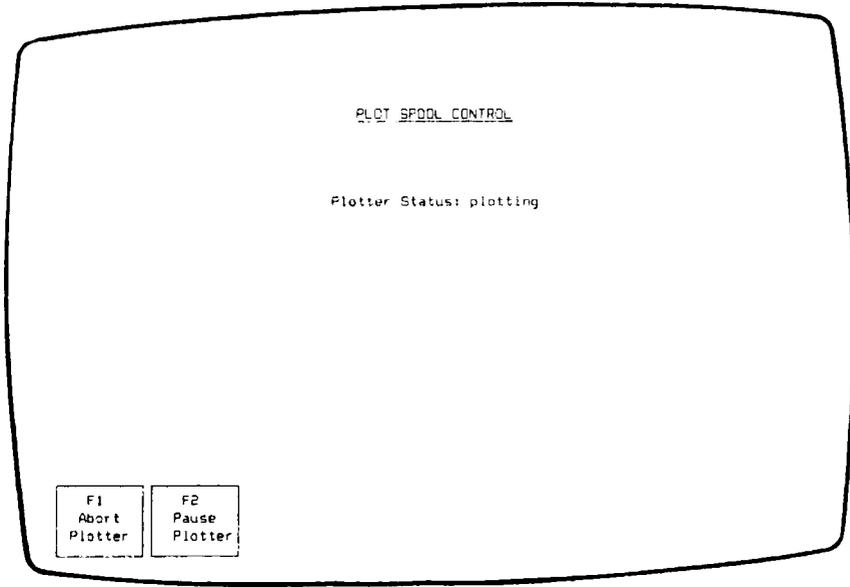


Figure 6.20
Plot Spooler Control Screen

The function keys on the Plot Spooler Control screen are explained in Table 6.11.

Table 6.11
Plot Spooler Control Screen Function Keys

Key	Explanation
<p align="center">F1 Abort Plotter</p>	<p>Cancels the plot in progress. If you wish to restart the plotter after pressing this key, you must return to the program and repeat the hard copy request.</p>
<p align="center">F2 Pause Plotter</p>	<p>Temporarily pauses the plotter. You can then resume right where you left off with the F2 (Resume Plotter) key or start the plot over from the beginning with the F3 (Restart Plotter) key.</p>
<p align="center">F2 Resume Plotter</p>	<p><i>Appears after you press F2 (Pause Plotter).</i> Causes the plotter to resume plotting where it left off when you pressed F2 (Pause Plotter).</p>
<p align="center">F3 Restart Plotter</p>	<p><i>Appears after you press F1 (Abort Plotter).</i> Restarts the plot that was interrupted, from the beginning.</p>

If you press F7 (Plot Spooler) when the plotter is not being used by a data analysis program, you will see the following message instead of the Plot Spooler Control screen:

Plotter Status: inactive

—NOTE—

Attempts to plot data while the plot spooler is paused will cause the following message to appear:

Plotter not available.

If you see this message, you must press the F7 (Plot Spooler) key and F2 (Resume Plotter) or F3 (Restart Plotter) before using the plotter.

NOTE

The DMA Calibration program uses the calibration constants of the currently or most recently connected DMA to perform the modulus calculations. Make sure the calibration constants are satisfactory (see page 67) before you use the modulus calculator. If no values are available from a DMA module, the program uses the default values shown on the Calibration Constants screen on page 67.

1. Press F3 (Modulus Calculator) from the Report Results screen. The screen prompts you to select the sample shape.
2. Use the ARROW keys to select the sample shape, then press F8 (Accept This Form). The screen that appears depends on the sample shape you select:

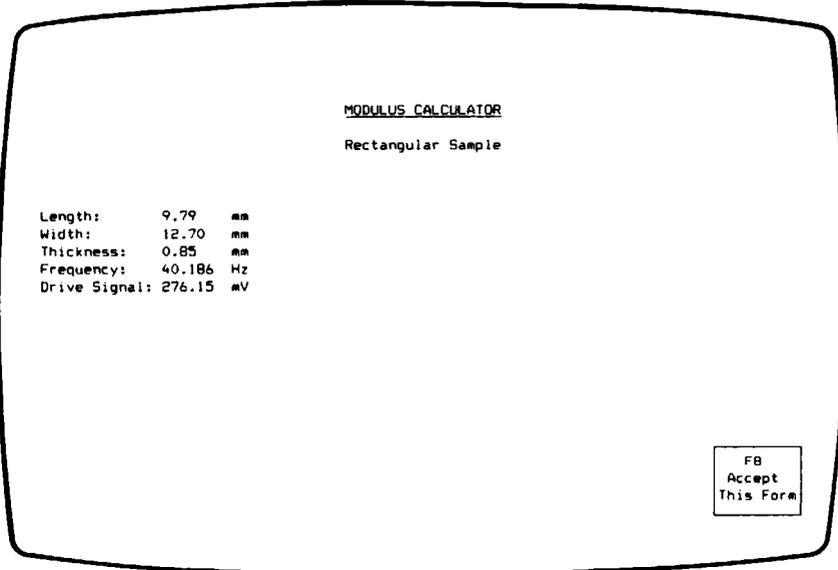


Figure 6.21
Rectangular Sample Parameters Screen

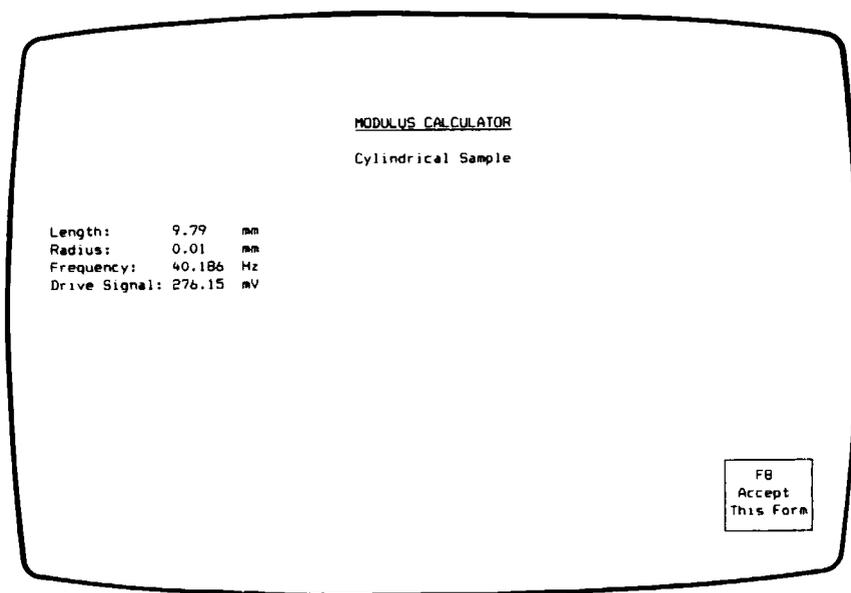


Figure 6.22
Cylindrical Sample Parameters Screen

3. Type in the requested sample parameters, pressing ENTER after each entry. The program calculates and displays the shear storage, shear loss, flexural storage, and flexural loss moduli and tan delta.

CHAPTER 7

Reference Information

Discussion of Equations

The measurements and equations involved in each DMA calibration are described below.

Moment of Inertia

The resonant frequency of the DMA is based on the moment of inertia of the arms and the stiffness (elastic restoring force) of the system. Thus, once the stiffness and resonant frequency are known, the moment of inertia can be derived easily from the following simplified relationship:

$$\begin{array}{c} \text{Moment} \\ \text{of} \\ \text{Inertia} \end{array} = \frac{\text{Sample Stiffness}}{\text{Resonant Frequency Squared}}$$

In the first step of the moment of inertia calibration, the resonant frequency of the system with the thin steel sample is measured (15 to 20 Hz). In the second step, the instrument is placed on its side so that a known force (50 to 100 gram weight) can be applied to the system. The resulting deflection of the arms is then measured to give the stiffness of the system. The single arm moment of inertia is then calculated as follows:

$$J = \frac{M G R^2}{8 \pi^2 f^2 \Delta P}$$

where:

M = mass of hanging weight (g)

G = gravitational acceleration = 9.80 m/sec²

R = distance from center of flexure pivot to clamp surface
(0.12785 m)

ΔP = change in measured arm position due to mass (M)

= $P_m - P_0$ for 983 DMA

= $P_0 - P_m$ for 982 DMA

f = resonant frequency

P_m = arm position with mass

P_0 = arm position without mass

Spring Constant

When no sample is mounted, the instrument will oscillate at a resonant frequency determined by the moment of inertia and the spring constant of the flexure pivots. Thus, if we know the resonant frequency of the instrument with no sample and the moment of inertia, we can easily calculate the spring constant. However, the moment of inertia and spring constant of each arm can vary slightly, and when no sample is mounted, only the driven arm can be measured. Thus, we use a paper sample, which contributes virtually nothing to the measurements, to tie the two arms together and allow a composite measurement of the spring constant of the two pivots.

After the resonant frequency of the system is measured, the spring constant is calculated as follows:

$$K' = 4\pi^2 f_0^2 J$$

where:

f_0 = frequency obtained with no sample (Hz)

J = moment of inertia (kg m^2)

Drive Signal Constant (983 DMA)

In the first step of the drive signal constant calibration, the resonant frequency of the system with the thin steel sample is measured to obtain the total stiffness of the system. As the motor starts, the instrument performs a static displacement of the arms and measures the drive voltage required to deflect the arms by a known amount. This yields a ratio of arm displacement (mm of deflection) to drive (mV):

A_0 (arm displacement)

V_0 (drive signal)

This ratio, in combination with the measured resonant frequency (f_n), is used to calculate C' :

$$C' = \frac{A_0 f_n^2}{V_0}$$

In the second step, we assume that the steel sample has a negligible loss ($\tan \delta = 10^{-5}$) compared to the instrument contribution. The drive signal needed to maintain resonance with the steel sample is measured and then used in the calculation for K_n'' :

$$K_n'' = \frac{4\pi^2 C' V_n}{a_n} J$$

where:

C' = drive signal constant (mm/(mV sec²))

V_n = drive signal required to maintain oscillation amplitude a_n (mV)

a_n = measured oscillation amplitude (mm)

J = moment of inertia (kg m²)

Drive Signal Constant (982 DMA)

The equation for C' for the 982 DMA differs slightly from that for the 983:

$$C' = \frac{A_0 f_n^2 \kappa}{V_0}$$

where:

A_0 = arm displacement (mm)

f_n = measured resonant frequency (Hz)

κ = instrument electronics factors (0.06444)

V_0 = drive signal required to move the arms the distance A_0
(mV)

The formula for K_n is identical to the 983 equation.

Series Compliance

In principle, measuring instrument compliance terms requires an infinitely stiff, no-loss sample, which of course does not exist. Instead, the series compliance calibration uses a very stiff steel sample of known modulus and dimensions and measures its resonant frequency and drive signal. The theoretical resonant frequency of the sample (f_t) is then calculated and used with the observed frequency to calculate the instrument contributions and obtain J_C' :

$$J_C' = \frac{B^2}{8\pi^2 J (f^2 - f_0^2)}$$

where:

- B = distance between pivot centers (mm)
- J = moment of inertia (kg m^2)
- f = maximum instrument frequency (Hz)

$$f_{\infty}^2 = \frac{(f_t^2 - f_0^2)(f_m^2 - f_0^2)}{f_t^2 - f_m^2} + f_0^2$$

- f_0 = frequency obtained with no sample (Hz)
- f_t = theoretical resonant frequency for compliance sample (Hz)

$$= \frac{W T^3 B^2 E}{8\pi^2 L^3 J} + f_0^2$$

- W = sample width (mm)
- T = sample thickness (mm)
- E = flexural modulus of the compliance sample (200 GPa)
- L = sample length (mm)
- f_m = measured resonant frequency (Hz)

The observed drive signal is used to calculate the instrument loss (J_{Cm}):

$$J_{Cm} = J_{C'} \frac{\left[C' \left(\frac{V_m}{a_m} \right) - \left(\frac{V_n}{a_n} \right) \left(\frac{f_m}{f_n} \right) \right]}{(f_m^2 - f_0^2)}$$

where:

C' = drive signal constant (mm/(mV sec²))

V_m = drive signal for compliance sample (mV)

V_n = drive signal measured during drive signal constant calibration (mV)

f_m = measured resonant frequency for compliance sample (Hz)

f_n = frequency at which K_n was measured (Hz)

a_m = oscillation amplitude for compliance measurement (mm)

a_n = oscillation amplitude for drive signal constant measurement (mm)

f_0 = frequency obtained with no sample (Hz)

Phase Zero (983 DMA only)

In the phase zero calibration, the 983 DMA first performs an internal measurement to calibrate the internal phase lag in the electronics. Next, the instrument starts moving the arms and measures the phase angle of the thin steel sample ($\tan \delta = 10^{-5}$) at two different levels of drive-to-oscillation-amplitude ratio by using two different sample lengths. It measures each phase over a range of frequencies, then fits the measurements to a theoretical curve and calculates the standard deviation of that fit. All phase correction data is stored internally in the module.

Length Correction

The length correction procedure measures the modulus of a sample at a variety of sample lengths, then extrapolates the modulus values to an infinitely long sample. The length correction is negligible with an infinitely long sample, so the extrapolated modulus is used to back-calculate what length correction is needed to obtain the same modulus for a real sample length.

Sample storage modulus is first calculated using the measured sample dimensions and instrument constants with the length correction constant (ΔL) assumed to be zero. The resulting E' values are then plotted versus ξ . For rectangular samples, ξ is computed as follows:

$$\xi = \frac{1}{L} \frac{2(1 + \sigma) T^2 \alpha + 3L^2}{2(1 + \sigma) T^2 \alpha + L^2}$$

For cylindrical samples:

$$\xi = \frac{1}{L} \frac{6(1 + \sigma) r^2 \alpha + 3L^2}{6(1 + \sigma) r^2 \alpha + L^2}$$

where:

σ = Poisson's ratio

T = thickness of a rectangular sample (mm)

L = measured sample length (mm)

r = radius of a cylindrical sample (mm)

α = shear distortion factor.

The program then fits a line through the points. The zero intercept of the line (where $\xi = \text{zero}$) is the corrected modulus (E' (corrected)), which represents the modulus of an infinitely long sample. The slope of the line is $-E'$ (corrected) ΔL . To obtain corrected shear modulus, divide E' (corrected) by $2(1 + \sigma)$. The length correction value is the same for shear and flexural modulus and is normally positive.

Discussion

Typical values for the length correction are between 0.1 and 4.0 mm. Negative values may indicate over-compression in the clamps or a problem with the length correction data. Examination of the length correction plot is recommended to detect possible bad points or curvature.

It is reasonable to use the same length correction when working with similar samples and using the same torque value. Torque values of 10 inch-pounds should be used for hard

samples (below glass transition). When running elastomeric (soft) sample experiments, use the constant tension springs on the clamps to maintain a reliable clamping pressure.

Often it is desirable to estimate the DMA length correction for certain samples when running quantitative DMA measurements. The length correction (ΔL) depends primarily on two factors:

- Sample stiffness
- Sample thickness

As the sample stiffness and thickness increase, the length correction also increases.

Figure 7.1 (pages 118 to 119) shows estimates of the length correction for different types of samples. You can use these graphs to:

- Estimate ΔL when time does not permit you to carry out an actual length correction measurement.
- Confirm that the length correction you obtained during a measurement is reasonable.

To use the figure, select the graph number corresponding to your sample material type in Table 7.1 (page 120). Then read the estimated length correction for your particular sample thickness from the graph. An example estimate based on Figure 7.1 is given on page 120.

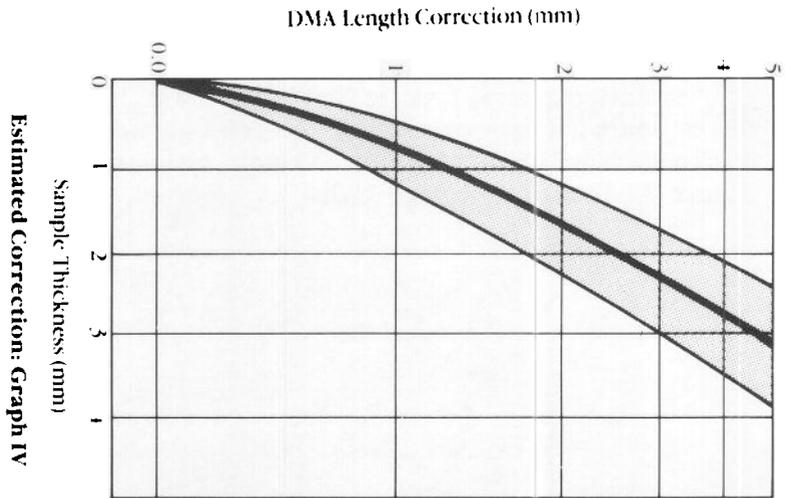
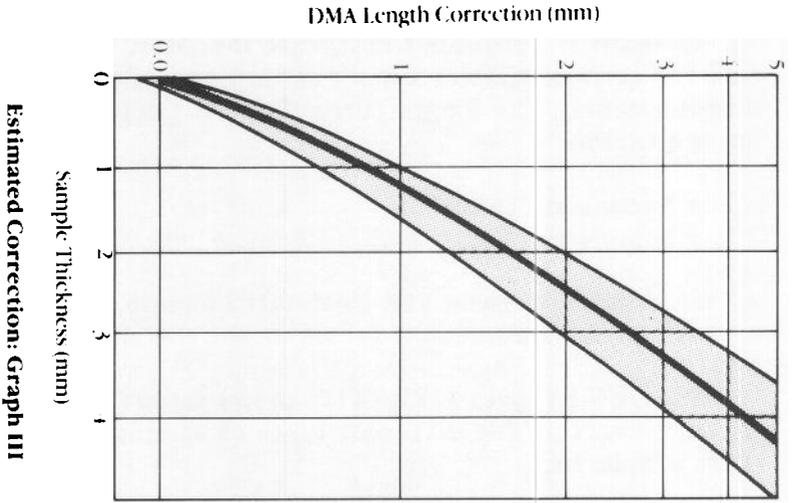
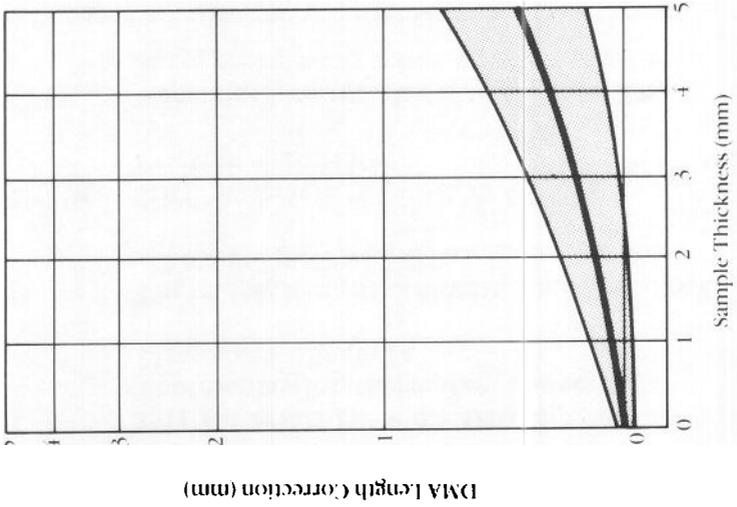
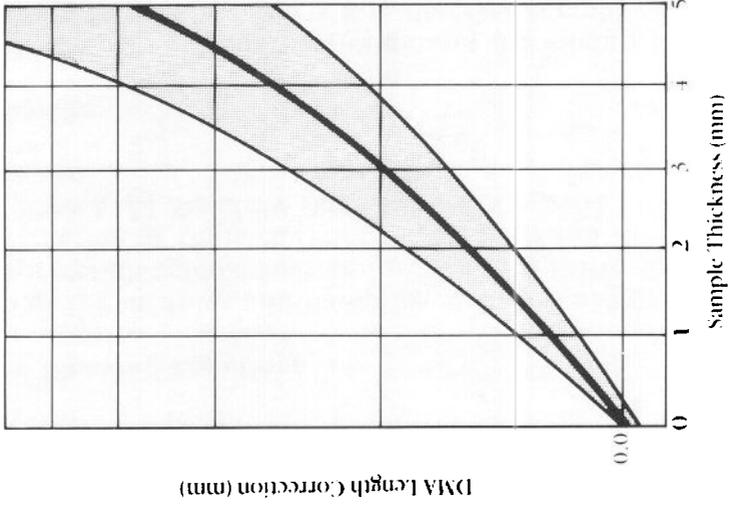


Figure 7.1
Length Correction Estimates



Estimated Correction: Graph I



Estimated Correction: Graph II

Table 7.1
Length Correction Sample Types

Classification	Type of Sample
Graph I	Soft materials ($E' < 0.5$ GPa) Thermoplastics/thermosets above T_g Elastomers above T_g
Graph II	Thermoplastics, thermosets, and elastomers below T_g ($E' = 2$ GPa)
Graph III	Stiff materials ($E' = 10-20$ GPa) Friction materials
Graph IV	Very rigid materials ($E' = 50-200$ GPa) Reinforced composites with matrix below T_g

Length Correction Estimation Example

A 3-mm thick sample of polycarbonate at 25°C is a thermoplastic below its glass transition and has a storage modulus (E') of 2.3 GPa. Its classification is therefore type II (see Table 7.1). According to Graph II, a 3-mm thick sample of this type has an estimated ΔL of 1.0 mm.

Modulus and Damping Equations

The following modulus and damping equations are used in the length correction calculations and by the modulus calculator. The derivation of these equations is explained in the Appendix to the *DMA Standard Data Analysis Program Operator's Manual*.

Primary Equations

The following set of equations calculates the moduli by combining the results of equations that describe the physical instrument with parameters derived from viscoelastic beam-bending theory. These equations were derived to account for the contribution of instrument calibration constants to sample modulus. The instrument correction is subtracted from the

raw modulus (storage or loss), and the result is multiplied by the sample geometry to yield the corrected modulus.

The calculations are complicated by the fact that the instrument correction terms β and γ are dependent on G' and G'' . They are solved by an iterative process that stops when the change in modulus is less than one percent.

(1) Shear storage modulus (GPa):

$$G' = (2Jk^2 - 2K') \frac{\beta^2 + \gamma^2}{\beta} \frac{L'}{B^2A} - \frac{\gamma}{\beta} G''$$

(2) Shear loss modulus (GPa):

$$G'' = [2J(wd) - 2K''] \frac{\beta^2 + \gamma^2}{\beta} \frac{L'}{B^2A} + \frac{\gamma}{\beta} G'$$

(3) Flexural storage modulus:

$$E' = 2(1 + \sigma) G'$$

(4) Flexural loss modulus:

$$E'' = 2(1 + \sigma) G''$$

(5) Tan delta:

$$G''/G' \text{ or } E''/E'$$

where:

Combined Quantities

$$\beta = \alpha + \frac{L'^2A}{24(1 + \sigma)I} + \frac{A}{L'} (J_C'G' + J_C''G'')$$

$$\gamma = (A/L') (J_C'G'' - J_C''G')$$

$$k^2 = \frac{M_0 \cos \delta}{2J\theta_0} + \omega^2$$

$$(\text{wd}) = \frac{M_0}{2J\theta_0} \sin \delta$$

$$\omega = 2\pi f$$

$$M_0 = (2J/R) 4\pi^2 C'V \quad (\text{N m})$$

$$\theta_0 = a/R$$

$$L' = \text{effective sample length (mm), measured } L + L$$

$$B = \text{distance between pivot centers (mm)}$$

$$= \text{sample length} + 2(D)$$

$$A = \text{cross-sectional area of sample (mm}^2\text{):}$$

$$T \times W \text{ for rectangular samples}$$

$$\pi r^2 \text{ for cylindrical samples}$$

$$I = \text{geometric inertial moment of sample cross section (mm}^4\text{):}$$

$$T^3W/12 \text{ for rectangular samples}$$

$$\pi r^4/4 \text{ for cylindrical samples}$$

Measured Signals

$$V = \text{drive signal (mV)}$$

$$f = \text{frequency (Hz)}$$

$$\delta = \text{phase angle (radians)}$$

$$= \text{measured signal for fixed frequency mode}$$

$$= \pi/2 \text{ for resonance mode}$$

Experimental Parameters

$$T = \text{thickness of rectangular sample (mm)}$$

$$W = \text{width of rectangular sample (mm)}$$

$$L = \text{length of sample (mm)}$$

$$r = \text{radius of cylindrical sample (mm)}$$

$$a = \text{oscillation amplitude (mm)}$$

Sample Constants

- ΔL = length correction constant (mm)
 σ = Poisson's ratio
 α = shear distortion factor, which has the following suggested values:
1.50 for jaw-clamped rectangular samples
1.33 for jaw-clamped cylindrical samples
1.00 for plate-clamped samples ($L/T < 1$)

Instrument Constants

- D = clamping distance (mm)
 R = distance from flexure pivot center to point at which a is measured in instrument calibration (127.85 mm)
 J = single arm inertial moment (kg m^2)
 C' = drive signal constant ($\text{mm}/(\text{mV sec}^2)$)
 K' = instrument parallel storage stiffness (N m)
 K'' = instrument parallel loss stiffness (N m)
 $= K_n'' f/f_n$
 f_n = frequency at which K_n'' was measured (Hz)
 $J_{C'}$ = instrument series storage compliance ($\mu\text{m}/\text{N}$)
 $J_{C''}$ = instrument series loss compliance ($\mu\text{m}/\text{N}$)

$$= J_{Cm}'' \frac{k^2/2\pi}{f_m}$$

- f_m = frequency at which J_{Cm}'' was measured (Hz)
 f_∞ = maximum frequency obtained with an infinitely stiff sample (Hz)

Error Messages

Definitions and solutions for the error messages specific to the DMA Calibration program are listed alphabetically below. Other messages you may see while operating the program are explained in the *Du Pont Thermal Analyst 2000/2100 Operator's Manual*.

Annotation number is out of range.

Problem: You selected an annotation number that does not exist.

Solution: Reenter the annotation number, making sure it is not higher than the total number of annotations on the graph. Remember that the annotations are renumbered and the highest possible number decreases by one each time you delete an annotation.

Calculations did not converge. Hit ENTER to continue, ESC to quit.

Problem: The variation in modulus did not converge to less than one percent within 100 iterations.

Solution: Press the ENTER key to continue the modulus calculation for another 100 iterations. If you still receive this message, press the ESCape key to stop the modulus calculation. The program returns to a point where data inputs (e.g., sample size, frequency) can be verified and changed to allow the modulus calculation to be retried.

Drive signal is not stable to within +/- 0.2%.

Problem: The sample is in resonance, but the drive signal could not be stabilized within 0.2% of its absolute value. This is often caused by surrounding bench-top or building vibrations.

Solution: Place the DMA on a vibration-free surface. Alternatively, you can accept the frequency and

drive signal values (if the drive signal appears stable) with somewhat reduced accuracy in the resulting calibration constant.

Module is not online.

Problem: The DMA Calibration program cannot be run because the DMA is not online.

Solution: Turn the module or Module Interface on, then use the F5 (Auto Configure) key on the System Configuration screen to reconfigure the devices on the network.

Motor control error.

Problem: The 983 DMA motor is not functioning properly. This error message is always accompanied by a more specific module error in the screen status line.

Solution: Look up the module error message in the box on page 126 for specific instructions.

No annotation exists.

Problem: You attempted to edit or delete an annotation, but none exists on the graph.

Solution: No action required.

No calculations to report.

Problem: None of the calibration constants has been changed since the DMA Calibration program was loaded or the current module was selected, so the Calculations report is not available.

Solution: Select the Instrument Parameters report if you wish to view the current instrument calibration constants. The Calculations report will become available only after you have completed at least one of the calibration procedures.

Motor Control Errors	
Err 83	Arm zero offset too large. Check arms (locking pins) and sample.
Problem:	The DMA was unable to bring the arm position to zero when it tried to start the motor.
Solution:	Check that the locking pins have been removed, that the arms rotate freely, and that the sample is mounted correctly. Bring the arm position closer to zero with the LENGTH ADJUST dial.
Err 84	Cannot start oscillation. Check arm and sample alignment.
Problem:	The instrument could not maintain sample oscillation.
Solution:	Check that the locking pins have been removed, that the arms rotate freely, that the sample is mounted correctly, and that the clamps are properly tightened.
Err 90	Phase calibration error. Measured phase exceeds specification.
Problem:	The measured phase angle exceeded the instrument's specifications. The calibration procedure will continue, but the results may be inaccurate.
Solution:	Call your Du Pont service representative.

No length correction values to plot.

Problem: The length correction procedure has not been run since the DMA Calibration program was loaded or the current module was selected, so the Length Correction plot is not available.

Solution: You must perform the length correction calibration before you can obtain a plot of the results.

No length correction values to report.

Problem: The length correction procedure has not been run since the DMA Calibration program was loaded or the current module was selected, so the Length Correction report is not available.

Solution: You must perform the length correction calibration before you can obtain a tabular report of the results.

No longer in calibration mode.

Problem: You interrupted the DMA Calibration program by pressing F1 (Start) or F2 (Stand By) on the Instrument Control screen or F1 (Motor Drive On/Off) on the Signal Control screen.

Solution: Press one of the DMA Calibration Opening screen function keys to restart your calibration.

No more lines.

Problem: (1) You have reached the maximum of 50 annotation lines per graph, or (2) you pressed ENTER while editing an annotation that does not contain any more lines.

Solution: Delete some lines in the current annotations if you want to add more lines.

No room for more annotation.

Problem: You have created the maximum number of annotations per graph (10).

Solution: Delete one of the current annotations if you want to add a new one.

Not connected to a DMA.

Problem: The currently connected module is not a 982 or 983 DMA.

Solution: Check that the 983 DMA module or 982 DMA Module Interface is properly configured, then press F5 (Auto Configure) on the System Configuration screen to reconfigure the devices on the network. If you have a multimodule system, check that the desired module has been selected via the Instrument Control screen.

Plotter disk space is full.

Problem: For the plot spooler to free up the system while a plot is being made, it must write the plot information in a temporary file on the Winchester disk; however, the Winchester disk is full, so the plot has been aborted.

Solution: Delete a file or files from the Winchester disk to make room for the plotter file by using the F5 (Delete Files) key under File Utilities. Repeat your request for a hard copy.

Plotter is not available.

Problem: (1) The plotter is being used, (2) the plotter is not turned on, (3) you do not have a plotter hooked up to your system, or (4) the plot spooler is paused.

Solution: Try the following:

- (1) Wait for the plotter to complete the current task.
- (2) Make sure the plotter's power switch is on.
- (3) Check for loose cable connections.
- (4) If the optional plot spooler is installed, check

that the spooler has not been intentionally put on "Pause." To check, press F11 DATA ANALYSIS and then F7 (Plot Spooler) for a display of current spooler status.

Then repeat your hard copy request.

Printer is not available.

Problem: (1) The printer is being used, (2) the printer is not turned on, (3) the printer is not online, or (4) you do not have a printer hooked up to your system.

Solution: Try the following:

- (1) Wait for the printer to complete the current task.
- (2) Make sure the printer's power switch is on.
- (3) Check for loose cable connections.

Then repeat your hard copy request.

Program not licensed for use on this system.

Problem: The DMA Calibration program can be run only on the Du Pont 2000/2100 system for which it was purchased.

Solution: Use a copy of the program that is licensed for your system. The label on the original program diskette shows the serial number of the system with which it is compatible. To determine the serial number of your system, look on the System Configuration screen by pressing F12 INSTRUMENT CONTROL, then F8 (GoTo Configure System).

Function Key Outline

When you have learned how the DMA Calibration program works, you may find the function key outline on the next page useful for stepping quickly through the program. Only the F8 (Report Results) key is outlined, because it is the only key that has several submenus of keys. Note that frequently occurring but self-explanatory keys, such as F8 (Accept This Form), are not shown.

The outline begins at the DMA Calibration Opening screen, which you can reach by following the steps in the box below.

Loading a Data Analysis Program

1. Press F11 DATA ANALYSIS.
2. Load the program:
 - a. Press F1 (Get New Program).
 - b. Select the drive.
 - c. Move the cursor to the desired program, then press F3 (Select File).
3. Press F1 (Start Program).

DMA Calibration Function Key Outline

Opening Screen Function Keys:

F1 Inertial Moment	F2 Spring Constant	F3 Drive Signal Constant	F4 Series Compli- ance	F5 Phase Zero	F6 Length Correc.	F7 Calib. Constants	F8 Report Results
---------------------------------	---------------------------------	---------------------------------------	-------------------------------------	----------------------------	--------------------------------	----------------------------------	--------------------------------

└ F8 Report Results:

F1 Tabular Reports	F2 Plot Length Correc.	F3 Modulus Calclator
---------------------------------	-------------------------------------	-----------------------------------

- └ F1 - Send tabular report to printer or plotter.
- └ F2 - Plot length correction calibration results.

F1 GoTo Rescale Plot	F2 GoTo Customize Plot	F3 Redraw Plot	F4 GoTo Hard Copy	F5 Return
-----------------------------------	-------------------------------------	-----------------------------	--------------------------------	---------------------

└ F1 GoTo Rescale Plot:

F1 Set Scale Params	F2 Cursor Expansion	F3 Fixed Grid	F4 Previous Limits	F5 Original Limits
----------------------------------	----------------------------------	----------------------------	---------------------------------	---------------------------------

- └ F1 - Set axis scaling parameters: start, stop, and tick intervals.
- └ F2 - Use cursor to enter axis limits.
- └ F3 - Set axis scaling with fixed grid.
- └ F4 - Set axes to previous limits.
- └ F5 - Set axes to original limits from Length Correction Plot screen.

└ F2 GoTo Customize Plot:

F1 Annotate Plot	F3 Graph Options	F4 Redraw Plot
-------------------------------	-------------------------------	-----------------------------

- └ F1 - Add text annotation to plot.
- └ F3 - Select a grid option.
- └ F4 - Redraw plot with most recently selected parameters.

DMA Calibration Function Key Outline (Continued)

F3 Redraw Plot: Redraw plot with most recently selected parameters.

F4 GoTo Hard Copy: Send a copy of the plot to -

F1 Plotter

F2 Printer

F5 Return: Return to DMA Calibration Opening screen.

F3 - Calculate modulus for user-input parameters.

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